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AP-42 Section Number: 9.10.1.2

Reference Number: 1

Title: Beet-Sugar Technology, Third Edition

McGinnis, R. A.

Beet Sugar Development Foundation

1982

BEET-SUGAR TECHNOLOGY

Third Edition

EDITED BY

R. A. McGinnis

PUBLISHED BY

BEET SUGAR DEVELOPMENT FOUNDATION

P. O. Box 1546, Fort Collins, Colo. 80522

FIRST EDITION 1951
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ASSIGNED TO THE BEET SUGAR
DEVELOPMENT FOUNDATION, 1968
SECOND EDITION 1971
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THIRD EDITION
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BEET SUGAR DEVELOPMENT FOUNDATION

PRINTED IN THE UNITED STATES OF AMERICA

STUDIO OF PRINTCRAFT, INC.
FORT COLLINS, COLORADO 80524

Dedication

Respectfully dedicated to
JAMES H. FISCHER,
Secretary-Manager of the Beet Sugar
Development Foundation
for three decades
whose intelligent and devoted
services to the Beet-Sugar
Industry, are an
inspiration to its members.

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FOREWORD

The first edition of Beet-Sugar Technology, published in 1951, was an outgrowth of a series of articles on the manufacture of beet sugar which appeared in *Spreckels Sugar Company's house* organ, "Honey Dew News." According to Carl J. Moroney, then president of the Company, "It was planned at the very outset that the material in the series of articles referred to, when completed would be published in permanent book form for the benefit, not only of our Company's own employees, but of all workers in and students of the sugar industry generally."

The planning of the book did not adequately provide for its unexpected popularity. Very soon after its publication the supply of the first edition was exhausted. Even though several very fine books had at this time become available on beet sugar manufacturing technology, none was in the English language.

The North American Industry pondered the options of either reprinting the first edition or publish a second edition to fill this void. It was the officers and directors of the Beet Sugar Development Foundation who decided that a second edition was the most plausible approach and that such a venture should have technical and financial support from the industry as-a-whole.

It was good fortune to have such a decision coming concurrent with the retirement of the first edition editor from his post with Spreckels Sugar Company. All rights were acquired and Dr. R. A. McGinnis agreed to proceed with a new and considerably expanded publication which would carry a 1971 date.

Within a decade, a rapidly dwindling supply became apparent and a decision to either reprint the "new" second edition or publish a third edition was to be made. A study revealed that nearly 25 percent of the second edition required correction, revision or updating. Again the officers and directors of the Beet Sugar Development Foundation elected to underwrite and publish a new edition with Dr. McGinnis serving as editor and a majority of the contributors repeating as authors and revisors.

It is our sincere wish that this edition again will serve in strengthening the technical base for the art of beet sugar manufacture. Again, we hope that it will serve as a tribute to the many technologists, living and dead, who by their intelligence and labors have established for posterity the impressive structure of technology described herein. We gratefully acknowledge with deep appreciation the many hours of composing, writing and proofing which have been donated by all who have contributed to this book. It has been a labor of love and pride. May it all serve the industry well.

THE OFFICERS AND DIRECTORS
Beet Sugar Development Foundation

PREFACE

As with the previous editions, this book is addressed to three classes of readers: the beet-sugar factory technologists, for instructional and reference purposes, the sugarbeet growers and others connected with or interested in the industry for general information, and professional engineers, chemists and technical college students, as a reference manual.

In scope, all phases of beet-sugar manufacture are included, with the exception of organization, financing, and marketing.

The differing requirements of the three reader groups call for both elementary and advanced treatments. To meet these requirements, elementary discussions have been given of most subjects, with additional advanced material on subjects and unit operations peculiar to the beet-sugar industry. In other words, unit operations which are given advanced treatment in other modern books are here given less space.

All sections of the book have been brought up to date as required. In addition, the chapter on "Crystallization" has been greatly expanded.

The sections on "The Barium Saccharate Process" and "Recovery of Amino Acids" have been deleted as no longer of interest to the North American Industry. Two of the Appendices have also been removed, as of questionable value.

New revising authors of this edition include Glen W. Yeager, R.E. Wyse, R.M. Sequeira, W. Akeson, J.M. Buchert, Donald L. Stewart, J.H. Leavell, C.T. Chua, M. Bolinder, and S.S. Martin.

The revisors of this edition have been drawn from the following sources (employed, or formerly employed by): The Amalgamated Sugar Company (2), American Crystal Sugar Company (2), Calgon Corporation (1), Canadian Sugar Factories (1), The Great Western Sugar Company (3), Holly Sugar Corporation (5), Johns-Manville (1), Michigan Sugar Company (2), Minn-Dak Farmers Cooperative (2), Amstar Corporation, Spreckels Sugar Division (14), Tandam Associates (1), U.S.D.A. Crops Research Laboratories (2), and Western States Machine Company (1).

Major credits, after the authors, must be given to the reviewers of the individual sections, whose constructive comments helped form the final test. Three men, W.W. Blankenbach, the late L.T. Jensen and P.H. Lott reviewed virtually every section. Reviews of a large number of the individual sections were made by the late Orville Barber, W.W. Barr, Burnall Brown, F.F. Coons, F.G. Eis, W.D. Foley, R.S. Gaddie, E.M. Hartmann, G.W. Miles, Jr., L.W. Norman, the late Guy Rorabaugh, H.G. Rounds, D.J. Stewart, A.L. Stock, E.F. Trojan, and M.A. Woods.

Certain individuals gave particularly valuable help with specific sections: "Sucrose Formation," W. Akeson, R.C. Johnson, Kent Nielsen, P.B. Smith, and R.R. Wood; "Chemistry," A. Carruthers (England), T. Chadwick, M. Cleary, R.E. Hallbeck, J.R. Johnson, R.B. Lew, the late R.M. McCready, Daniel Muller, Whitney Newton, II, the late G. Rorabaugh, K.W.R. Schoenrock, and J.P. Thomas; "Juice Purification." S. Heiner, C.H. Iverson, A.P. Nielsen, and H.G. Rounds; "Filtration," S.L. Force; "Evaporators," F.F. Coons, C.A. Summers; "Dried Pulp," C.H. Iverson; "Electrical Power," S. Heiner; "Process Control," K. Corrado; "Instruments," J.E.A. Rich; "Technical Accounting," H.O. Ebell, the late H.F. Haney, and J.E. Hedde.

Sincere thanks are due to individuals who furnished materials for the various sections, in the forms of data, drawings, graphs, photos, or other information: "Agriculture," A.C. Parker, L. Henry; "Sucrose Formation," A.A. Armer, R.T. Johnson, G.G. Nordstrom; "Chemistry," G.W. Maag, R.M. Sequeira; "Tare Laboratory," W.W. Barr, S.E. Bichsel, C.O. Caprin, J. Cancelarich, P. Cherniavsky, R.A. Fogg, M.G. Frakes, H.F. Haney, J.E. Hedde, J.R. Johnson, L. Langlois, M.C. Mertes, J.M. Silver, and Claes Weibull (Sweden); "Beet Storage," S. Anderson, J. Angus, S. Bass, P. Brimhall, R.A. Fogg, J.W. Hall, R.C. Johnson, G. Nichol, J. Nieder, D.L. Oldemeyer, F.B. Russell, E.L. Swift, J.C. Tanner, and R.E. Wyse; "Diffusion," F.M. Sabine (deceased) and C. Steele; "Juice Purification," O. Barber, S.E. Bichsel, H. Brüniche-Olsen (Denmark), P.J. Hatch, J.A. Hupfer, and S. Stachenko; "Lime and Carbon Dioxide," D.J. Annis, V.J. Azbe, P.L. Reiber, and W.H. Weisz; "Molasses," M.G. Auth; "Electrical Power," R.J. Horrath; "Process Control," J.P. Schratz; "Instruments," J. Roberts and J.E.A. Rich; "Technical Accounting," W.W. Barr and J. Landyshev.

Many of the special drawings for the illustrations were made by Dennis Fischer. The authors of the chapters on Chemistry and Crystallization made most of their own drawings, and the special foldout sheet was drawn by the editor.

As before, it was a pleasure to have experienced the full cooperation of J.H. Fischer and his staff at Fort Collins. Jim Fischer's talents were used in every phase of the book's production, from miscellaneous art work through all the details of a large-scale printing project. His years of experience in the publication of other books and of the Society's Journal were very valuable.

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Chapter I

AGRICULTURE*

General and Distribution of Sugarbeets. The sugarbeet crop has been developed only during the past 110 years in the five continents of the world. By 1979 the share of the world sugar production from sugarbeets had increased to 42% of the total. Sugarbeets are grown on an estimated 20 million acres in 41 countries of the world. It is an essential factor in the agriculture and industry of many of these countries, not only for the protection of a supply of home-grown sugar, but also as an important feed for livestock.

Growing of beets is important in most European countries for the protein supplied in the beet foliage, as well as the carbohydrate in sugarbeet pulp. Russia is the largest producer of beet sugar, with more than twelve million acres reported. Poland produces 1 375 000 and France 1 370 000 acres. Acreage planted by other major producing nations includes West Germany 1 085 000, England 538 000 and Italy 595 000. In the United States the acreage planted to sugarbeets in 1979 was 1.16 million. The crop is grown in 17 of the 50 states, and grows in the winter months below sea level in the Imperial Valley of California and in the summer in Colorado at an altitude above 5 000 ft.

As with most American crops, records show phenomenal increases in the per-acre sugarbeet production. The average root yield per acre 50 years ago (U.S. 1915-1924) was 9.75 tons, whereas the last 10-year period has reached 19.92 tons for the United States (1). Figure 1-1 shows the increase in yield during the 60-year period 1915 to 1976.

Figure 1-1. A 60-year record of yields in U.S. beet-producing areas, 1915-1976.

Botany and Growth Character of Sugarbeets. The sugarbeet is a member of the Goosefoot *(Chenopodiaceae)* family, together with red beets, spinach, pig weed, lambsquarter, and Russian thistle. The species *Beta vulgaris* includes sugarbeets, table beets, Swiss chard, and mangel-wurzels.

The sugarbeet is a deep-rooted plant often extending its roots to a depth of six to eight feet in mellow soil. Calculations made by Wadleigh (20) indicate that a single beet plant extending its roots seven feet in alluvial soil would have at least 10 000 square feet of root surface (see Figure 1-2).

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*Original by P.B. Smith, revised by Glen W. Yeager

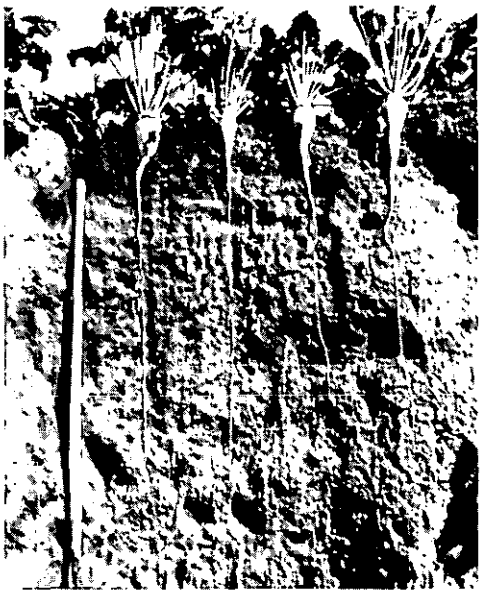

Figure 1-2. In friable soil the fine, hairlike roots of the sugarbeet plant may extend, as in this photo, to a depth of six feet.

The sugarbeet plant grows most successfully in northern latitudes. However, it is a plant which adapts itself to many climatic conditions (14), based on suitable soil acidity. In the United States, it grows in the hot climates of Arizona and the Imperial Valley of California, as well as in the cold climates of North Dakota and Minnesota and in the Canadian provinces of Quebec, Manitoba, and Alberta.

In controlled greenhouse studies at the California Institute of Technology (16,17) involving variations in nutrition, hours of light, light intensity and changes in day and night temperatures, all these factors affected the development of foliage, sugar percentage, and root weight. It was noted that, in a general way, a day-time maximum average of 70 °F and a night minimum average of 50 °F gave optimum production of sugar. A total of 56 different climates were included in these studies. Some strains of beets better adapt to hotter climates, some to cooler climates, and some do fairly well in a wide range of conditions of day-length, and temperature variation.

Purchase of Beets. The sugarbeet crop is produced by the grower under a participating purchase contract. Its arrangement is unique in American agriculture, in that the grower shares directly in the proceeds from the sale of sugar, calculated back to each ton of beets.

The exact terms vary with the contracts prevailing in the different areas of the United States. All are similar on two points when establishing the price paid for the beets: first, the percent sugar content of the beet roots, and second the net selling price for the sugar in the market, less freight and certain sales charges. In determining the sugar content, either the average sugar content from individual grower's beets as delivered, or the average sugar content of all growers' beets as sliced in the entire campaign, may be used. In the latter case, grower's samples also may be taken, and the proportion of each individual grower's sugar analysis to the average content will determine his payment.

Clauses common to most purchase contracts cover manner of harvest, price of seed, and distribution of payments as quantities of sugar are sold. Certain provisions relating to the growing of the crop are included.

In recent years, some sugarbeet producing areas have also measured beet purity and other nonsucrose chemical constituents as a means of determining the extractable sugar per ton of beets. These data become a part of the equation in determining the basis of payment for sugarbeets.

Sugar Control Legislation. It is interesting to note that in 1789 the second law passed in George Washington's first term of office was a tariff that included sugar.

In May 1934, the Jones-Costigan Act was passed by Congress which controlled sugar production and consumption. The Sugar Act served the United States sugar industry very well for 40 years. All federal legislative control ceased on December 31, 1974 when Congress failed to extend the Sugar Act.

Recent Developments. The first grower-owned beet-sugar cooperative in the United States was organized in 1973 when sugarbeet producers in the Red River Valley of Minnesota and North Dakota acquired ownership of the American Crystal Sugar Company. Three other cooperatives were formed in 1974, two of which are located in the Red River Valley of the north and the third in central Minnesota. One of the three cooperatives organized in 1974 merged with the grower-owned American Crystal Sugar Company in 1975.

In late 1974, returns for sugar reached an all-time high due to a superficial sugar shortage worldwide. These high price levels continued into 1975, followed by rapidly declining values resulting in the entire industry being subjected to severe economic pressures.

In 1975, 1.516 million acres of sugarbeets were harvested in the United States which yielded 29 270 000 tons of beets. Sugarbeet acreage declined to the point where 1 120 000 acres were harvested in 1979 which produced 21 937 000 tons of beets.

Sugar production from sugarbeets in the United States in 1979 was approximately 2.6 million tons compared to 4.0 million tons in 1975. Much of this decline can be attributed to a reduction in sugarbeet area as the result of factory closings.

In 1974, there were 53 operating beet sugar factories in the United States. These plants were located in 18 states, from Michigan and Ohio to the Pacific Coast. Since that time, eleven sugarbeet processing plants have closed including all of the factories (four) owned and operated by one company. Sugarbeets were grown in 17 states in 1979.

For several years, California was the leading sugarbeet producing state, planting 30 percent of the United States acreage. The Red River Valley of the north is now the leading producing area.

Seed Production and Development. Since 1936 the American beet industry has developed a home-grown seed production program which essentially supplies its needs. Seed is grown mainly in Oregon and Utah. It is produced by "the overwintering method" (9). Since the sugarbeet is a biennial, seed can be produced from a fall planting of seed in a climate which produces growth, but will be cold enough to produce vernalization, yet not severe enough to kill the root. Seed planted in August and September will send up seed stalks in March and produce seed for harvest in the late summer of the following year.

Commonly, sugarbeets produce a multiple-germ seed ball which, when planted, results in several seedlings emerging from one spot in the soil. This plagued sugarbeet production until two refugee Russian scientists, Drs. V. F. and Helen Savitsky, while living in the United States, discovered and developed plants producing single germ seeds. This monogerm gene has been transferred to the principal commercial varieties now grown in the United States. The United States Department of Agriculture and commercial company research staffs have joined in the task of developing varieties to suit variable conditions of environment and disease. Strains developed by Federal researchers are distributed through the Beet Sugar Development Foundation to each of the company breeding stations in the United States. The objective of this effort is to build better varieties with higher yield potential and better quality as raw material for the factories.

The varieties offered farmers are monogerm and may be of two types: polyploid and diploid hybrid. The latter is the most popular among plant breeders of this country. Following the pattern of corn plant breeders, these probably are two-, three-, or four-way hybrids. Male sterility is employed in the monogerm parent with the final cross generally using a multigerm pollen parent. This permits the mechanical separation of the multigerm fraction, allowing the beet grower to plant seed that is 98% monogerm. The unprocessed monogerm seed is flat in general conformation and is not subject to good precision drilling.

These single germ seeds, with the corky material having five points, makes for uncertain seed fill in drills. The problem has been worked on by numerous investigators. Bainer, in 1940, worked on mechanical separation of multigerm seed in segments (2). Some of these devices were tried on single-germ seed with varying degrees of success.

It was not until the rice huller was employed in Colorado in 1955 by Walters and Smith (21) that a satisfactory means was found to round off the edges and permit careful sizing of the corky seed units. A combination of diameter and thickness grading, together with gravity separation, has made it possible to provide different size groups for issue to beet growers.

Another plan employed by some sugar companies to overcome the irregular shape of the seed, is to coat it and produce a rounded pill or pellet. In the past 35 years several investigators have explored the potential of various materials for seed coating, including carbon dust, pumice, clay, bentonite, and silica. Fungicides and powdered fertilizer also have been included in the coatings. The pellet is built up in size and customarily has three sizes, expressed by the screen grades of 9/64 to 10/64" known as mini pellets, 9/64 to 11½/64" regular size, and 12/64 to 13/64" extra large. The resulting round shape has enabled drills to drop seed more accurately at uniformly spaced intervals.

Research continues for coating additives to hasten germination, produce a stronger seedling and more effectively protect it from diseases. Another possibility being explored is to double-coat the seed so that it will germinate only when suitable temperatures prevail for continuous seedling growth. This might be quite helpful in areas where spring frosts sometimes cause replanting.

Preparation of the Seed-bed and Planting. The outcome of the beet crop is largely determined by the preparation of the seed-bed. The system of land management, crop sequence, depth of plowing, time of plowing, fertilizer practice, and herbicide usage prior to the time of beet emergence are all important factors which produce a proper seed-bed environment for the seeds and seedlings. In areas where

winter freezing helps mellow soils, it is an advantage to plow in the fall. Fall plowing generally produces 5-20% increases in tonnage with fine-textured soils. It can be recommended on all except sandy or sandy-loam soils.

Beet yields have been shown to be higher on soils plowed 10 to 12 in. deep when compared with 8 in. or less. It is a good rule, however, in any one year, not to turn up on the surface more than 1 in. of new soil as it tends to crust and interfere with seedling emergence.

The time of planting depends on weather, the length of the growing season, and the harvest date. In regions where the growing period is limited, it is advantageous to plant as early as possible to extend the growing season.

With certain soils, particularly following a crop of potatoes or other deep-tilled crop, it is advantageous to merely surface-work the soil in preparation for beets. This relatively new system is known as minimum tillage and reduces weed trouble. Experiments conducted in Ohio and Nebraska have shown that it is possible to plant beets in the same rows that grew either soy-beans or sorghum the previous year. In this practice herbicides are applied, post-emergence, and in some areas a side dressing with nitrogen fertilizer is practiced ahead of thinning. The stalks left from the previous year's crop are pushed into the middle between the rows with a wide duckfoot when planting. This generally produces an excellent germinating condition for the beet seed.

The trend in many sections of the United States is to space out the seed three or four inches apart when planting, as seen in Figure 1-3. Even wider spacing is used under favorable conditions, thus eliminating the thinning operation.

Figure 1-3. An excellent distribution of monogerm seed, planted to a stand, no thinning required. This weed-free field has been carefully cultivated and is growing vigorously.

All of the United States beet crop is now drilled with processed monogerm seed. The size of the seed is fitted carefully to the cell size, and planters are generally operated at speeds below three miles per hour. A typical twelve-row drill, also equipped for incorporation of a pre-emergence application of herbicide, is shown in Figure 1-4. Many farmers, realizing that speed of drilling is very important to the uniformity of stand, now use 8-, 12-, 16- and even 24-row planters. This enables a greater acreage to be seeded in a day when soil conditions are optimum. Depth of seeding varies from 0.5 to 1.5 in. depending on type of soil, possibility of rain or irrigation for germination, and temperature of soil.

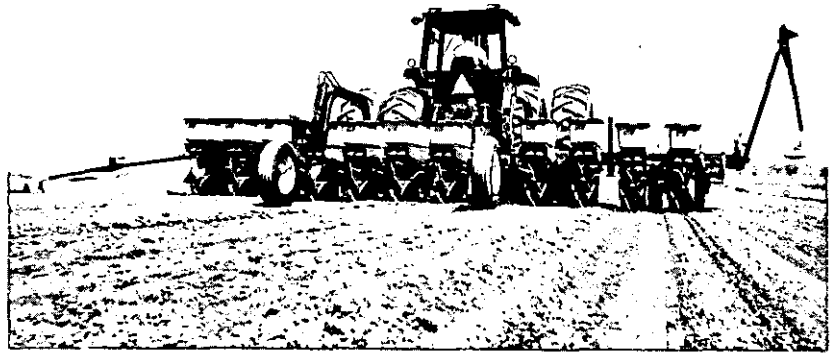

Figure 1-4. A typical 12-row drill, equipped with incorporators for applying liquid selective herbicide in bands on each side and under the beet seed to control weeds.

Fertilizer Fundamentals and Crop Rotation. Four nutrient factors influence growth: water, carbon dioxide, organic matter, and minerals. Roots grow only in moist soil. On the other hand, roots do not develop properly in water-saturated soils because of the exclusion of oxygen. Water is necessary for the process of photosynthesis (see Chapter II).

The activity of the fine beet-roots is related to temperature as well as moisture (5). Dry and cold soil retard root activity and reduce the ability of the roots to absorb phosphorus and other nutrients.

Soil organic matter is very important for the production of maximum yield and is lower in sandy soils than in clays. Well-rotted animal manure is probably the best source for addition to the soil, but in practical farming systems animal manure is too limited in supply. If organic matter is to be supplied in amounts for maximum production, it must come from crop roots and residues. This source can best be provided by proper rotation with legumes, and from additions of mineral fertilizers.

In order to obtain the maximum benefits from mineral nutrients added, the soil acidity must not be overlooked. A pH above 6.5 is advantageous for assimilation of some mineral nutrients, but also is necessary for a lime-loving crop such as sugarbeets.

A proper soil environment for this crop is one where there is ample aeration between soil particles, without an obstructing hardpan that prevents water percolation. A well-flocculated soil permits a deep penetration of the beet's tap root and a maximum use of the whole soil structure to a depth of several feet.

Minor element deficiencies can usually be observed in the appearance of the growing plants. A shortage of potassium, for example, will be displayed by a yellowing of the leaf blade and a reduction in petiole length. These conditions reduce photosynthesis, resulting in a decrease of sugar production.

For optimum growth the sugarbeet requires potassium more than any of the other elements. Most acid soil areas require fertilization with potassium for maximum results.

When limited in supply, phosphorus reduces early growth, final maturity, and yield (7). When phosphorus is very limited, a "Black Heart" disease develops, and the root is easily subjected to rot. The foliage between the leaf veins turns brown or

black in color, and the leaf curls inward. Because phosphorus moves very little in the soil, it is helpful to apply an adequate amount of this element to the preceding legume crop which is included in many crop rotations (8).

In addition to primary nutrients, the secondary elements sulfur, calcium, and magnesium are very essential to proper growth of a healthy sugarbeet plant. The minor or micro elements, if lacking or in oversupply, can greatly affect the crop, even though there is a good supply of primary and secondary elements present. These minor elements include: boron, often short in Michigan soils; copper, important in photosynthesis; iron, essential to chlorophyll formation and proper storage of sugar; molybdenum; manganese, beginning to become short in some humid areas; sodium, which appears beneficial to sucrose formation; and zinc, which can be very toxic in oversupply. Ulrich observes that (18,19) petiole analysis is helpful for guidance.

Beet Quality Improvement. The quality of sugarbeets, i.e., beet purity and sugar content, started to decline in the mid-1950's. Studies were initiated in the Imperial Valley of California in 1957 to determine the reasons why beet quality in this normally high quality area started a downward trend. A more intensive and extensive approach to this problem began in 1970 in the Imperial Valley and the Hereford, Texas, district. This program was soon expanded to include Northern California and the Rocky Mountain district.

It has long been known that nitrogen has a very profound effect on both yield and sugar. Nitrogen is a growth element and is essential for maximum yield. However, if excessive amounts of nitrogen are still available at harvest time, sugar content is depressed. Having the "right" amount of nitrogen available to the beet crop for maximum yield and sugar content and for the most efficient sugar production is the goal of the beet quality improvement program.

The complete program involves (1) soil (2) petiole and (3) brei testing for nitrate nitrogen.

The brei nitrate test measures the amount of nitrate nitrogen in every sample of beets taken for sugar analysis. This test, therefore, is a measure of the amount of nitrate nitrogen in beets at harvest time. It does not necessarily reflect the amount of nitrate nitrogen available to the crop at any other time during the season. Since this test is conducted on every sugar sample (and each sample is from an approximate 20 ton load), the average nitrate nitrogen value for each contract is usually based on an evaluation of a sample of beets taken from less than an acre. The actual measurement of nitrate nitrogen is made on exactly the same filtrate sample on which sugar is determined. The brei nitrate nitrogen test has proved to be a very valuable guide for managing the growing of succeeding crops.

The secret to high sugar and high yield is to provide ample nitrogen during most of the growing season, but make it unavailable 4 to 6 weeks before harvest. With growth slowed due to lack of nitrogen at this time, other forces such as cool temperatures (below 50 °F) and lack of moisture can further enhance the accumulation of sugar.

Why do excessive amounts of nitrogen in beets at harvest time appear? There can be several reasons, but the single most obvious reason is simply too much nitrogen in the soil — for whatever reason. If there were large quantities of nitrogen remaining in the soil from previous fertilization and then fertilizer were applied, it would result in excessive amounts of available nitrogen.

Consequently, to find out just how much nitrogen was remaining in the soil, many fields have been sampled to a depth of 4 feet and analyzed for nitrate nitrogen. Needless to say, there has been a tremendous variation in the amount of nitrogen found — from virtually none to over 1 000 pounds per acre of nitrate nitrogen per four feet of depth!

It has been reported that 10 pounds of nitrogen are needed per ton of beets; therefore, only 250 pounds are needed for a 25-ton per acre crop. For 306 grower contracts sampled over the years 1972 through 1976 in central California, it was found that the greatest amount of extractable sugar per acre was obtained with only 8.7 pounds of nitrate nitrogen (available from soil and fertilizer) per ton of beets.

The petiole test has been found to be a very simple means of finding out the nitrate nitrogen status of beets during the growing season. In this procedure, the middle portion of the most newly matured leaves is taken from many different beets in a field. They are then analyzed for nitrate nitrogen.

All these tests — brei, soil and petiole nitrate are valuable aids in seeking the goal of high yield, high sugar, efficient sugar production and maximum monetary return.

Chemical and Mechanical Control of Weeds. The best methods of weed control combine both chemical and mechanical practices with a proper crop sequence. This last factor is very important and was brought out by a four-year study at Colorado State University (3). The number and identity of weed seeds after certain crops is shown in Table 1-1.

Table 1-1 Weed Seedling Numbers per 400 g of Soil.

Beets Following	Kochia	Pigweed	Grasses	Lambs-quarter	Total Weeds
Barley	42.9	31.6	14.5	18.2	109.4
Corn	66.6	43.6	48.2	6.6	165.7
Beans	15.7	7.1	11.4	8.6	44.2

Likewise a comparison was made of the differences in weed populations with and without chemical weed control. In Table 1-2 are data showing that there were about 50% fewer weeds in the crop treated with herbicides as compared with mechanical weeding.

The trends today are toward making fewer trips across the fields in production of the sugarbeet crop. This reduces the compaction of the soil by heavy tractors and equipment. It is possible to raise better crops of sugarbeets by use of pre- and post-emergence herbicide treatments to replace some of the machine cultivations and hand hoeings formerly thought necessary.

Table 1-2 Weed Seedling Numbers per 400 g of Soil.

Weed Control	Kochia	Pigweed	Grasses	Lambs-quarter	Total Weeds
Chemical	32.8	19.3	13.8	7.9	74.3
Nonchemical	50.7	35.6	35.6	14.4	138.7

The system employed to fit the chemical and mechanical tools together, will vary from area to area, as well as from field to field (13). Working out the spring-time techniques to fit the conditions depends on a number of factors, including: 1) the type of soil; 2) the emergence pattern of the beet seedlings; 3) the kind of annual weeds; 4) the herbicide application incorporated in the soil at planting time; 5) the use of a post-emergence chemical; 6) the use of lay-by herbicide; 7) machine thinning; and 8) the kind of machinery available for mechanical practices. Added to these are variability of weather, crust, and need of irrigation to germinate beet seed and activate the chemicals. Further complications come from disease and insects.

The simple objective is to eliminate labor and reduce costs of production. In areas where sufficient rainfall occurs for seed germination, the recommended procedure is to plow, preferably in the fall; follow the minimum tillage concept by going once over in the spring with a field cultivator, then plant, followed by a liquid herbicide mixture of TCA and Pyramin, or TCA, Pyramin plus Nortron, applied back of the press-wheel in a 7 to 10 inch band; after the beets have emerged, cultivate as close to the row as possible. Post emergence weed control is accomplished by the timely application of Betanal or Betanex. It is desirable to have a population of 25 000 to 30 000 beets per acre.

The above system must be modified when applied to irrigated areas. The most successful method is the use of a lister to form ridges in the fall, after plowing, at center distances equal to the row width of the planter to be used. A bed planter is used to level off the ridges which makes an ideal seedbed. Three techniques are used to control weeds with herbicides, namely: pre-emergence, post-emergence and lay-by. The most effective chemicals for pre-emergence weed control include Nortron, Ro-Neet and Avadex. These herbicides are applied either by power incorporation, ridge cover or by broadcasting. Post-emergence chemicals include Betanal, Betanex and Pyramin W and are applied with a band sprayer on both sides of the beet rows and directed into the row covering a band of 8 to 10 inches. The lay-by applications are made by incorporating Treflan or Eptam in the irrigation water or with a broadcast sprayer. Figure 1-5 shows excellent weed control accomplished by good agronomic practices coupled with timely application of recommended herbicides.

Figure 1-5. Outstanding weed control in a field with double row beds, 14 inches between the rows on the bed and 26 inches across the furrow.

Chemical Control of Insects and Diseases. The subject of insects and diseases is vast and important. For identification of insects and diseases, reference is made to Asa C. Maxson's book on the subject (10). During the last 30 years much has been learned about control of insects and diseases affecting the production of sugarbeets. Both chemical and biological controls are being investigated. Recommendations for controls are suitable only for a short period of time, due to the rapid rate at which new chemicals are being formulated. The most recently approved chemicals are customarily listed in handbooks such as the USDA No. AH452 (12).

There are about 15 insects considered of importance in one or more areas of the United States, including the soil insects: cutworms, wire worms, white grubs, root lice, flea beetles, root maggots and garden centipedes. The foliar insects include: webworms, leaf miners, yellow bear caterpillars, alfalfa loopers, aphids, spider mites, army worms, alkali beetles, lygus, blister beetles, grasshoppers and leafhoppers.

Some of the insects listed carry virus diseases. Curly top is carried by the beet leafhopper, *Circulifer tenellus* Baker. Beet savoy is transmitted by the *Piesma cinerea* (Say) plant bug. Various mosaics and both European and western yellows are transmitted by at least two species of aphids, the most common being the green peach aphid.

Diseases caused by fungal bacteria can be classified into root, foliar and seed-borne types. New fungicides and treatment processes are being tested constantly, and great progress has been made in recent years. The newer seed treatments and treaters are providing greater protection against seedling diseases.

The most common seedling disease is "blackroot." Several organisms and fungi cause blackroot, such as: *Rhizoctonia, Phoma, Pythium, Fusarium* and *Aphanomyces.* Mature roots may be attacked by various types of fungi also, but the most common is *Rhizoctonia solani. Erwinia, Fusarium, Phoma, Verticillium* and *Sclerotium* should be included as root rots of economic importance in some areas. Development of resistant strains is bringing about great increases in production. Research by the plant breeder has been rewarded by new varieties with increased resistance to *Cercospora beticola,* curly top, European and western yellows and Powdery Mildew. One can be optimistic about the eventual solution offered by genetic selections for resistance to the diseases. Figure 1-6 illustrates a healthy sugarbeet crop resulting from good crop rotation, planting disease-resistant seed and adherence to sound management practices.

Resistant varieties coupled with the use of fungicides and insecticides are needed to control some diseases. Crop rotation is important, as it reduces the incidence of diseases.

The Environmental Protection Agency has had a profound influence on sugarbeet production. The agency has banned the production and application of many chemicals which gave good control of various pests and diseases. Such pesticides and fungicides were alleged to be carcinogenic.

Cercospora is controlled in humid sections by spraying at 10- to 14-day intervals as a preventive measure with Duter or Kocide. Powdery Mildew is controlled by the application of sulfur.

Nematodes, of which some 4 600 species have been identified, are roundworm pests that attack both plants and animals. There are three known to be of economic importance to sugarbeets. The most common is a cyst form called

Heterodera schachtii, which can be found in every sugarbeet producing area in the United States. It is most common in California, Idaho, Oregon, Colorado and Nebraska. There are two forms of root knot nematodes, *Meloidogyne* spp and *Heterodera marioni* cornu, known to be damaging to beets.

Heterodera schachtii has so many host plants that a strict rotation of nonhost crops many times is not successful because of host weeds that may not be controlled. Steele (15) found that 23 out of 49 families studied were hosts to this species. Of 283 genera and 535 species investigated, 218 species within 39 genera were hosts. For more than three decades geneticists have been seeking immune strains of sugarbeets. There are a number of so-called wild species of beta that are resistant to attack. Intensive genetic selection work has been conducted by Dr. Helen Savitsky and others of the United States Department of Agriculture, Science and Education Administration, at Salinas, California, in splitting chromosome genes for nematode immunity when combined with beta vulgaris species.

Figure 1-6. A disease-free field that has received timely and precise management practices.

At the present time the best control of all of the species of nematodes affecting sugarbeets is through soil fumigation. The two most common chemicals are D-D, made by the Shell Oil Co., and Telone, made by the Dow Chemical Co. These are chiseled into the soil at rates of 20 to 30 gallons per acre, to at least a 7-inch depth and placed 12 inches apart across the beet rows at least one week in advance of planting. Recent evidence indicates that materially reduced rates of application can be made directly under the beet row, 12 inches or deeper, in the fall of the year, with a ridge left over the chisel area. This is smoothed off and beets planted in the sterilized zone the following spring.

The Environmental Protection Agency announced in November 1979 that it intends to suspend registrations of pesticide products containing Dibromochloropropane (DBCP) (22). Shortly thereafter, several chemical companies announced they would voluntarily cancel registrations of certain products which contain DBCP (23). This list includes Telone, a reliable soil fumigant used to control nematodes that attack the sugarbeet crop.

Beet Thinning Methods. Space planting precisely graded monogerm seed, including pelleted seed, has become a common practice in beet growing areas

throughout the United States. Michigan is the leading state where over 95% of the acreage is space planted and not thinned.

Precision and timing of each operation are the key factors in the success of "planting to a stand." The application of an approved herbicide at the time of planting is recommended.

Many growers carefully space plant the seed at four-inch intervals resulting in a stand that lends itself to successful thinning by the use of an American-designed electronic thinning machine as shown in Figure 1-7.

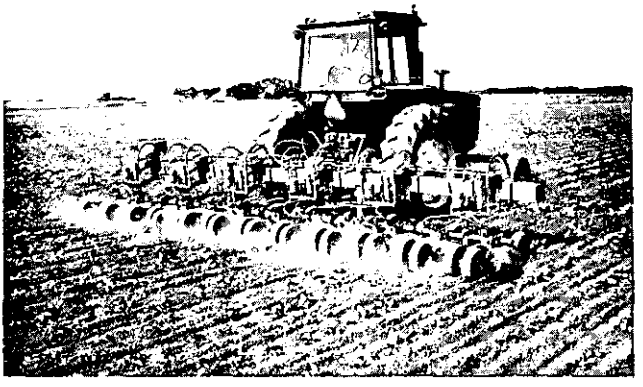

Figure 1-7. Six-row electronic thinner leaving a good stand in a field that was space planted. Note the weed-free field.

Either space planting to a final stand or this procedure combined with the use of an electronic thinning machine, coupled with the use of recommended herbicides, has significantly reduced or eliminated the need for field labor.

Sugarbeet Irrigation Practice. The irrigation practices for sugarbeets vary with the producing area, but are generally dependent on temperature, type of top soil and subsoil, amount of slope, amount of rainfall, availability of irrigation water, and length of growing season. The actual water requirements vary considerably, based on the transpiration rate and the growth rate of the crop.

Generally the sugarbeets benefit from moderate or light irrigation as soil air is needed for optimum growth. Frequent, light irrigations rather than heavy, infrequent waterings are most effective. Deficient soil moisture puts a stress on the plant and wilting takes place, decreasing the crop growth. In the early season beets do not require as much water as in the latter portion. Over-irrigation leaches out the nitrogen, and the crop may have a yellow appearance and experience a decrease in sugar content and yield. "In well-developed root systems the resistance to water movement in the soil does not appear to be significant when the water potential is above −1.0 Bar," says Gardner (4). "This movement is roughly proportional to the difference between the water potential in the plant and that in the soil."

In common practice irrigators endeavor to keep the top 18 in. of soil moist, but not overly wet (6). Too much water keeps the subsoil soggy and is often the cause of sprangled roots in the same manner as a hardpan layer. Well-aerated soil develops better root systems and results in higher yields. It is important to drain excess soil water in order to prevent damage to the crop.

The maintenance of a favorable water balance in the crop is dependent on adequate absorption of moisture from the soil. The harder a beet plant has to work to get moisture from the soil, the slower is its growth. The transpiration rate is not of concern, as the plant restores the needed moisture from the soil.

The most common method of irrigation is with furrows between the rows of beets. Some farmers follow a practice of irrigating every other row. Rows irrigated are alternated from irrigation to irrigation, so that all rows are watered throughout the season. If this were not done, large amounts of nitrogen would accumulate between the dry rows. This program conserves water and prevents over-irrigation, particularly on flat land. Depending on the shape of the fields, the length of run generally should not be more than 800 ft. This prevents water at the top of the field from penetrating to a depth below root absorption. Through use of syphons or gated pipe the amount of water turned into each furrow can be regulated so there is a uniform distribution over the field without undue waste of water. Figure 1-8 depicts the even distribution of water that results from the use of syphon tubes.

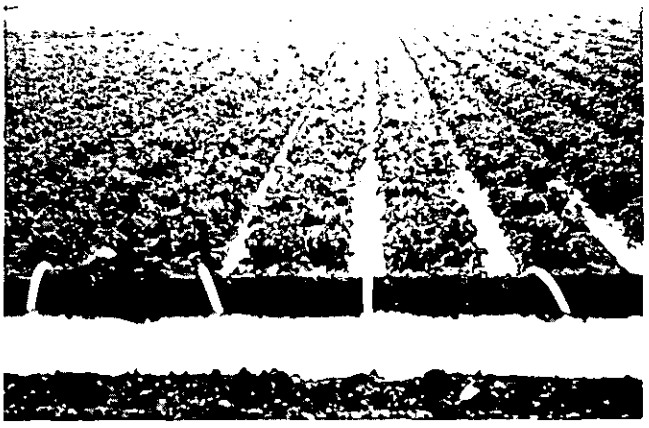

Figure 1-8. The use of siphon tubes to distribute water evenly to a good field of double row beets planted on beds.

A relatively new practice is being used with sprinklers in pump irrigation areas, without any expenditure for leveling the field. A pump is placed in the center of each quarter-section of land to be watered. The nozzles are placed on a wheel frame device that rotates in a circle by use of water pressure or electric motors. Depending on the amount of head at the well, the water is distributed in amounts sufficient to soak 12-18 in. per irrigation. Sandy soils absorb better than clays but require more frequent applications. Each of these systems will cover approximately 100 to 140 acres depending on percolation rate. There are a number of different types on the market.

Machine Harvest of Roots. In the period following World War II the sugarbeet industry in the United States started developing machines to harvest the crop. The first windrow field loaders originated in 1939, and the first root harvesters appeared during the last two years of World War II. About 7% of the sugarbeet crop was machine-harvested in some fashion in 1945. The development of machines was very rapid, and by 1949 half of the sugarbeet acreage in the United

States was mechanically harvested. Virtually all harvesting of beets has been mechanized since 1953.

Harvesters are of two types: those that top the beet root in the ground and those that dig the root first and top it out of the ground. A possible further division is into machines that load directly into trucks as the roots are lifted, and machines that carry the beets in a tank or cart and then unload into a truck.

Many sizes of beet harvesters are available to farmers depending on the size of the acreage involved. Single-row machines are popular as fitting the needs of small "family-sized" operations, where the grower does all the harvesting by himself. There are two-, three-, four-, and even six-row harvesters to fit the requirements of almost any size operation. Certain machines fit bed planting where the rows might be 14 to 16 in. apart on the bed. Other harvesters can be adjusted to handle rows varying from 20 to 30 in. apart. The most popular row spacing, however, is 22 in. However, 30 in. rows are standard in California and Texas.

There probably is not one harvester universally popular with all farmers. The trend is toward multiple row harvesting as the acreage handled by each operator becomes greater. Four- and six-row units are most prevalent at the present time. Figure 1-9 shows a recently improved machine handling six rows at a time.

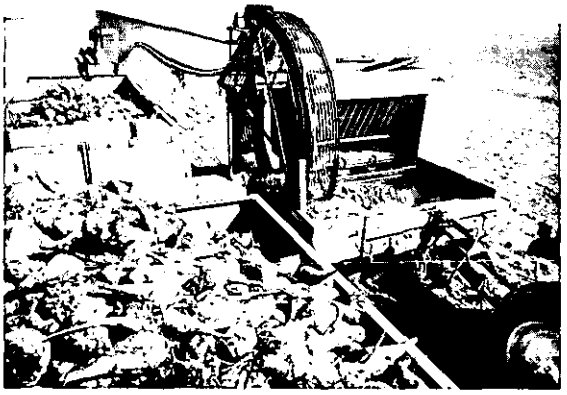

Figure 1-9. A high capacity John Deere beet harvester called "the wheel" which elevates the beets to the cross conveyor for delivery into a truck.

Climatic and soil conditions change harvesting methods and designs. For example, in Southern California where extremely dry, hard soil conditions exist during April, May, and June, the roots are generally lifted first and then topped. On the other hand, in Northern California where heavy rainfall often interrupts the harvest in the fall, the common procedure is to cease harvest until the wet cycle is passed. In the northern growing areas of the United States, where soil freezing occurs, the harvest proceeds even under severe soil conditions in order to complete the harvest before loss of the crop. Present machines have been successful under most situations.

During the past 20 years engineers have been adding refinements to the beet harvester making it more efficient, dependable and increasing its capacity to deliver cleaner beets to a truck. In good conditions harvesters are capable of delivering 40 acres daily. Large, custom-type machines in good yields have recorded up to 1 000 tons in a single day. A six-row machine today will replace 60 to 90 workers in the

hand operations of 35 years ago. Improvements are needed to reduce damage to beets in the screening operation and still maintain the capability of separating dirt and trash.

Methods of Handling Beet Tops. It is contended that the by-products from the average production of beets will provide as much net livestock feed as can be grown on the same acreage devoted to grain or alfalfa. This would include the thermal units of energy in beet tops, pulp and molasses. The largest value, of course, is in the beet tops which on the average, according to E. J. Maynard (11) produce in excess of 200 lbs of dry matter per ton of beets.

Normally the green weight of the tops is about equal to the weight of the roots as harvested. As tops contain varying amounts of moisture depending on how green they are when fed, it is customary to consider them in terms of "tops per ton of beets" rather than on actual weight of tops fed. There are at least three methods of handling beet tops:

1. The flail-type machine Figure 1-10 beats or cuts off the foliage at the top of the crown, followed by a scalper which uniformly removes a portion of the crown material. This chops up the tops and scatters them on the ground to be a source of organic material and fertilizer. The mineral value is considered to be worth $50 to $60 per acre if promptly incorporated into the soil.

2. The multiple-row harvester with topper and digger on the same tractor scatters the green material over the field with some opportunity for field pasturing by livestock. A modification of this method is to follow with a side-delivery hay-rake to windrow the tops. Generally these are left in the field to air dry and are hauled in dry.

3. A separate tractor can be used from the one employed in digging the roots. Normally this is a three- or six-row delivery of the tops to a three- or six-row windrow. The beet tops are customarily left in the windrow until they dry to a moisture content of about 75%. They can also be ensiled fresh and green, but there is some liquid lost unless a blotting material is placed on the bottom of the silo to absorb this high protein liquid.

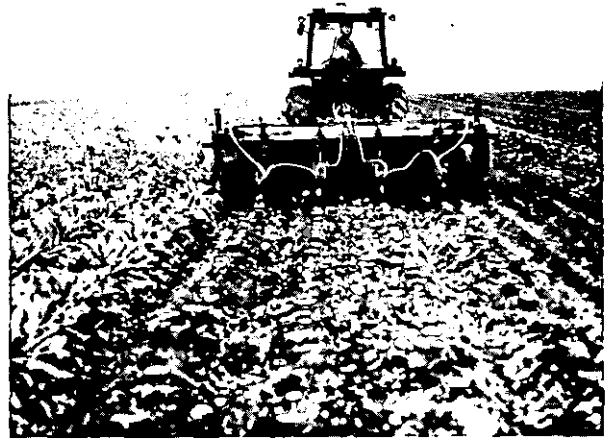

Figure 1-10. A flail-type machine removes remaining petioles and leaves after the major portion of crowns, petioles and leaves have been removed and windrowed.

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Chapter II

THE SUGARBEET AND CHEMISTRY*

The Sugarbeet and Sucrose Formation

Introduction

Development of the sugarbeet was initiated in Europe some 200 years ago by genetic selection for high sucrose content within forage beets. Forage types possessing white roots, a conical shape, and a below-ground growth habit were observed to be highest in sucrose content. The early selections contained an estimated 8 to 10 percent sucrose, but through years of selection, contents of 18 to 20 percent are now common. The modern sugarbeet is currently the only major crop that was not known in prehistoric times. Therefore, the modern sugarbeet, grown worldwide for its high sucrose content, is truly a product of modern plant breeding.

The Sugarbeet Plant

Growth Habit

Taxonomically the sugarbeet is classified as *Beta vulgaris,* as are red garden beets, chard, and mangel-wurzel. The genus *Beta* is part of the Chenopodiaceae family which contains many drought-resistant, salt-tolerant species. *Beta vulgaris* and other closely related *Beta* species originated in the Mediterranean area.

The sugarbeet is a biennial plant and as such requires 2 years to complete its life cycle. During the first year, only the vegetative portion of the plant develops. Following overwintering, or a 10 to 12 week period of below 6 °C, reproductive growth is induced (vernalization), and seed production begins. If no cold induction occurs, the sugarbeet plant will remain vegetative indefinitely.

The sugarbeet is unique because it stores large amounts of sucrose as opposed to starch, the more common storage carbohydrate. Roots containing 20 percent sucrose on a fresh weight basis are approximately 80 percent sucrose on a dry weight basis. About 90 percent of the soluble solids in a sugarbeet root are sucrose. Therefore, the sugarbeet root is an example of a remarkable adaptation of an organ to store high concentrations of sucrose.

The Shoot

The shoot consists of the petioles and leaf blades. Although the crown is anatomically a foreshortened stem, it is commonly considered as a part of the root (Figure 2-1). The shoot forms a rosette of 30 to 70 leaves, the number depending upon genetic and environmental factors. The leaf blade is an extremely important part of the sugarbeet plant because sucrose is synthesized in this organ. Mature, fully expanded leaf blade size increases to a maximum early in the growing season and then declines, presumably as the demand of the root for photosynthate exceeds that of new leaves.

The blade contains a profuse network of vascular tissue and minor veins. (The vascular system of a plant consists of xylem and phloem tissue for the transport of water and nutrients within the plant. Xylem is nonliving tissue primarily involved in the transport of water and minerals from the root to the leaves. Phloem is living tissue responsible for the transport of organic substances). The minor veins are closely

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associated with the photosynthetic apparatus and are of particular importance in the exchange of materials to and from the leaf tissue. Minor veins form such a profuse network that each 33 micron length of vein services approximately 20 mesophyll cells (9, 16).

Gaseous exchange between the leaf and the surrounding atmosphere occurs through apertures called stomates. Simple stomates are found in the epidermal cells on both the upper and lower surfaces of the leaf. Stomates regulate the uptake of carbon dioxide and release of water vapor by opening and closing in response to environmental and physiological conditions.

The primary function of the petiole is to display the leaf in a manner which will maximize light interception. The proportion of the total above-ground biomass that is petiole is very responsive to environmental influences such as stand density, nitrogen fertilization and light intensity. The petiole consists of large vascular bundles through which sucrose is translocated from the leaf to the root, and where water and nutrients are translocated from the root to the leaves.

Figure 2-1. The Sugarbeet Plant.

The Root

The sugarbeet taproot of economic importance consists of a crown, hypocotyl and true root. The crown is stem tissue containing leaf buds and supports the rosette of leaves. The vascular system of the petiole becomes highly branched and convoluted in the crown and then becomes organized into concentric rings at the transition zone between hypocotyl and crown. The size of the crown relative to that of the total root depends greatly on variety and nitrogen fertilization (5, 38).

The hypocotyl region is the transition zone between the crown and the true root. This region is the thickest part of the taproot and contains no grooves or lateral roots. In the mangel, the hypocotyl comprises the major bulk of the root. No differences in chemical composition are apparent between the hypocotyl and the true root.

The surface of the taproot is creamy white in color and relatively smooth except for two parallel grooves on opposite sides containing the lateral roots. In cross section, the dominant feature of the taproot is the alternating concentric rings of vascular and parenchymal tissue. The vascular rings are laid down early in the ontogeny of the root by the production of supernumerary cambia (1). The supernumerary cambia produce vascular bundles and parenchymal cells by rapid cell division. The increase in root size during the growing season is the result of continued cell division in the cambial region of the vascular rings and cell enlargement. The root may contain 8 to 13 vascular rings at harvest time, but the 5 to 6 rings which represent 70 to 80 percent of the root bulk at harvest are already formed by the time the root is 1 cm in diameter (Doney, personal communication, 1978, 1, 27).

The root dry matter (about 25% fresh weight) is made up of insoluble cell wall material (17%), sucrose (73%) and soluble compounds (10%). The insoluble cell wall material, known as "marc," is made up of approximately equal portions of cellulose, hemicellulose and pectic substances (26). The cellulose and hemicellulose give support to the cell while the pectic substances act as cementing agents holding the cells together (Figure 2-2).

Figure 2-2. Electron micrograph of a portion of a sugarbeet root cell. Note very large vacuole (V) and thin cytoplasm (C), nucleus (N), middle lamella (ML), mitochondrian (M).

The chemical composition of the root varies considerably among the various parts. The crown is normally 1 to 3 percent lower in sucrose than the rest of the root and contains a higher proportion of nonsucrose constituents. The crown area is approximately 125% higher in sodium and amino acids and 60% higher in potassium and reducing sugars than the remainder of the taproot (3, 4, 13, 38). However, these differences depend greatly on variety and nitrogen fertilization (5, 38).

The parenchymal cells, where most of the sucrose in the root is stored, are thin walled and highly vacuolated (Figure 2-2). The cytoplasm, which contains the organelles and enzymes involved in cellular metabolism, is extremely thin and tightly appressed to the cell wall. The vaculole, the site of sucrose storage, makes up the remaining volume of the cell (95%). Therefore, these cells are capable of storing large amounts of sucrose.

The sucrose content is highest in the cells near the vascular bundles (6, 24). For this reason, roots with the greatest number of rings tend to be highest in sucrose percentage (29). However, this has not proven to be a feasible criterion for screening breeding lines.

In present sugarbeet varieties, an inverse relationship exists between sucrose content and root yield. Attempts to increase sucrose content by genetic means often result in reduced yield and vice versa. The same inverse relationship holds true for the effect of environmental conditions on sucrose content and yield. Those agronomic practices or climatic conditions that promote root yield result in lower sucrose content. This relationship may be explained by cell size (8). Roots that have a small average cell size tend to have higher sucrose content while roots that have a large average cell size tend to be high yielding and low in sucrose content (27). Agronomic practices, such as high nitrogen fertilization or widely spaced stands, promote large roots and large cells and a reduced sucrose content. Genetic selections for high sucrose content tend to be those with small cell size. The relationship between cell size and sucrose content may be a factor controlling photosynthate partitioning in sugarbeets and deserves further study.

Sucrose Synthesis

Sucrose is synthesized in the cytoplasm of mesophyll leaf cells from chemical precursors and energy formed in the chloroplast during photosynthesis. In the sugarbeet, carbon dioxide is fixed to ribulose 1,5 biphosphate by the enzyme ribulose 1,5 biphosphate carboxylase (Figure 2-3). The resulting unstable intermediate breaks down immediately to form two molecules of 3-phosphoglyceric acid. The 3-phosphoglyceric acid then reacts with ATP via phosphoglyceric kinase to form the more chemically reactive and, therefore, more easily reduced 1,3 diphosphoglyceric acid. The 1,3 diphosphoglyceric acid formed is subsequently reduced to glyceraldehyde 3-phosphate (GAP) by glyceraldehyde 3-phosphate dehydrogenase. GAP is in equilibrium with its isomer, dihydroxyacetone phosphate (DHAP). These two three-carbon compounds are readily transported across the chloroplast membrane or they may undergo an aldol condensation reaction catalyzed by an aldolase to form fructose 1,6 diphosphate. Eventually fructose and glucose monophosphates are formed by the action of phosphatase and isomerase enzymes. Glucose or fructose monophosphate formation may occur in the cytoplasm or in the chloroplast.

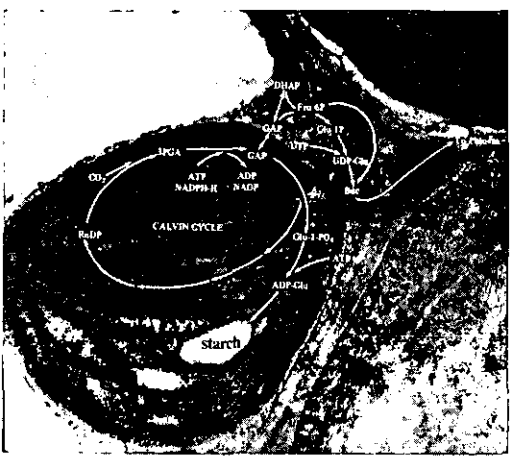

Figure 2-3. Path of carbon dioxide fixation and sucrose synthesis during photosynthesis. Pathway of sucrose synthesis and movement have been superimposed over actual structures where the events occur, i.e., chloroplast, cytoplasm and cell wall.

Sucrose is formed in the beet leaf in two steps. Sucrose phosphate is first formed by the enzyme sucrose phosphate synthetase (UDP glucose: fructose-6-phosphate transglucosylase). The reaction catalyzed by this enzyme is as follows:

Fructose-6-phosphate + UDP-glucose ⟶ sucrose-phosphate + UDP. Free sucrose is then formed by the action of a phosphatase on the primary product, sucrose phosphate. This reaction is highly irreversible. Sucrose is the first product of photosynthesis which does not contain a high-energy phosphate bond. Free glucose and fructose are subsequently formed from sucrose by the action of an acid invertase.

Sucrose Transport

Sucrose synthesized in the mesophyll cells of the leaf is either utilized for growth and maintenance of the source cell or transported to other parts of the plant. The sucrose to be transported out of the leaf moves from the cytoplasm into the intracellular spaces (apoplast) of the leaf mesophyll cell where it moves by diffusion to the companion cells of the phloem (15, 18). The loading of the companion cells of the minor veins is against a concentration gradient, and is, therefore, an energy-requiring (endergonic) process (14, 33). The membrane transport of sucrose into the companion cells of the phloem sieve elements occurs with the cotransport of a proton (H^+). An H^+ gradient between the free space and the companion cell cytoplasm provides the electrochemical gradient which drives the sucrose uptake mechanism (19). The mechanism is specific for sucrose and occurs without hydrolysis of the sucrose molecule (17, 21). This phloem loading step is critical because it establishes a concentration gradient between the leaf and the root, thus driving the translocation mechanism.

The transport of assimilates (sucrose) is from source tissue to sink regions. The source tissue is actively photosynthesizing leaves. The sink regions are any actively growing tissues which utilize sucrose. Young leaves act as sinks until they sucrose is driven by a proton gradient from the vacuole to the cytoplasm and a stage, there is a net export of sucrose to other sink regions. Therefore, in rapidly growing leaves, the products of photosynthesis are utilized locally and sucrose export is nil (10, 22). Leaves of intermediate age transport sucrose both to the root for storage and to the younger, developing leaves to support the metabolic activities required for growth. Mature leaves are the major suppliers of sucrose for transport to the root. During early stages, when top growth predominates, most of the sucrose is used to maintain top growth. After approximately 40-50 days, however, the root becomes the dominant sink for available photosynthate (11, 32). At this stage, the root receives or has the strongest attraction for the sucrose synthesized in the leaves.

The rate of sucrose transport from the leaf to the root may occur at rates up to 135 cm/hr (28). The rate and quantity of sucrose transported depend on many factors including plant age, time of day, light intensity (15), nitrogen nutrition (31) and temperature.

Sucrose Storage

The sucrose arriving in the root is partitioned to sucrose, nonsucrose dry matter and as a substrate for respiration. Respiration is required to provide energy for growth and maintenance for the cell and utilizes approximately 20% of the translocated sucrose. About 16% of the sucrose is utilized in producing cell wall and soluble metabolites. The remainder, about 64%, is stored in the vacuole.

Upon arrival at the root, sucrose is unloaded from the phloem by an as yet unknown mechanism. Evidence exists suggesting that phloem unloading occurs into the cytoplasm of the immediately adjacent parenchymal cells. The sucrose then moves symplasticly (in the cytoplasm) away from the vascular region. Although the sucrose is unloaded into the cytoplasm, the plasmalemma membrane possesses a rapid exchange capability for sucrose. As a result of this exchange reaction, the concentrations of sucrose in the cytoplasm and in the cell wall rapidly equilibrate, making it difficult to elucidate the actual transfer mechanism of sucrose away from the vascular region. However, as the sucrose moves away from the vascular region, it is rapidly absorbed and stored in the vacuoles of parenchymal cells in the vascular rays and interring area. The process concentrating sucrose in the vacuole to levels of 0.7 to 0.8 molar occurs at the tonoplast membrane bounding the vacuole. There is now good evidence that the sucrose moves across the tonoplast in conjunction with the transport of potassium, i.e., potassium-sucrose cotransport (30). The uptake of sucrose is driven by a proton gradient from the vacuole to the cytoplasm and a potassium gradient from the cytoplasm to the vacuole. These electrochemical gradients provide the energy necessary to drive the sucrose uptake mechanism. This mechanism is capable of concentrating sucrose in the vacuole by 100 fold over levels in the cytoplasm. The entire process of phloem loading translocation and uptake into the storage vacuole of root storage cells occurs without sucrose hydrolysis (20, 35).

Sucrose Metabolism

The mechanism by which sugarbeet plants regulate sucrose utilization for growth, cell maintenance, and storage in root tissue is not well understood; however, sucrose synthetase may play a major role (25). Sucrose synthetase catalyzes the following reaction:

$$\text{UDP-glucose} + \text{fructose} \rightleftharpoons \text{sucrose} + \text{UDP}$$

The reaction is easily reversible ($K_{eq} = 1.3$) and operates primarily in the direction of sucrose degradation *in vivo*. Therefore, the enzyme produces the sugar nucleotide, UDP-glucose, which is a precursor for many oligo- and polysaccharides. Sucrose synthetase activity is not found in tissue containing chlorophyll.

Sucrose can also be broken down by invertase. Two forms of invertase exist in sugarbeet, one active at pH 7.0, the other at pH 5.0 (34). Both types exist in all parts of the plant. Acid invertase catalyzes the breakdown of sucrose to glucose and fructose and also catalyzes the transfer of fructose to acceptors such as sucrose, thus forming the kestose sugars. However, the role of acid invertase (both in the leaf and in the root) and its importance to sucrose metabolism, remains a mystery. Roots of young seedlings and all sugarbeet leaves contain high levels of acid invertase. Acid invertase activity in mature beet roots is very low but its activity would appear to be sufficient to supply the required hydrolytic activity to support respiration during storage. The decline in acid invertase activity and the increase in sucrose synthetase during the first 40 days of growth have been proposed to be related to sucrose storage capacity (23, 25), but this relationship is still obscure. Levels of neutral invertase activity in the root are significant but its importance is unknown.

Respiration

The energy required for growth and maintenance of the sugarbeet plant is derived from the controlled enzymatic oxidation of sugars in a process called respiration. Respiration occurs in small, sub-cellular organelles called mitochon-

dria. Respiration occurs in all parts of the beet, both during the day and night, and is of particular importance in storage where it accounts for the major loss of sucrose in the absence of mold growth (36). The rate of respiration reflects differences in variety, temperature, the concentration of oxygen and carbon dioxide in the surrounding atmosphere, injury, and fungal infection.

In respiration, the hexose sugars formed by inversion of sucrose are oxidized and cleaved to form two pyruvic acid molecules via the glycolytic pathway in the cytoplasm. The pyruvic acid enters the mitochondria where it is converted to acetyl co-enzyme A with the release of carbon dioxide by the pyruvate dehydrogenase enzyme system. Acetyl Co-A then enters the tricarboxylic acid cycle (TCA cycle) where it is oxidized to CO_2. The acid intermediates of the TCA cycle also serve as carbon skeletons in the synthesis of amino acids and other more complex compounds.

Concurrent with the oxidation reactions of the glycolytic and TCA cycles is the production of the reduced form of nicotinamide adenine dinucleotide (NADH) which is subsequently reoxidized to NAD and water via the mitochondrial electron transport chain. The energy released in the oxidation of NADH by the electron transport system is conserved as adenosine triphosphate (ATP) by oxidative phosphorylation. Therefore, the energy released during the oxidation of carbohydrates is trapped in the high-energy phosphate bonds of ATP. This energy in ATP is used to drive endergonic reactions which synthesize the chemical compounds and drive the metabolic processes required for growth such as ion uptake, sucrose accumulation, and protein, fat, and polysaccharide synthesis.

The proportion of energy trapped in high-energy phosphate bonds varies and is a function of mitochondrial respiration efficiency. Sugarbeet root mitochondria are more efficient (trap more of the released energy in ATP) when they are growing rapidly than in the fall or under storage conditions. Respiratory efficiency has been shown to be related to growth rates and to hybrid vigor (7).

Patterns of Sucrose Accumulations

In most sugarbeet growing areas of the U.S., the concentration of sucrose in the root and the size of the root increase in essentially a linear fashion throughout the growing season (2, 12). Therefore, the longer the growing season, the higher the yield and the higher the sucrose content. However, late in the growing season, changes occur in the nonsucrose soluble solids which result in improved processing quality. The insoluble cell wall (pulp) fraction increases and at the same time becomes less soluble at high extraction temperatures (70 °-80 °C). The decreased solubility is a result of the conversion of pectins from soluble to more insoluble forms (37). Amino acid levels decline as nitrogen becomes deficient and free amino acids are converted to proteins. As a result of these chemical conversions, the efficiency of lime defecation increases, resulting in higher thin juice purities (39).

Because of its indeterminate vegetative growth habit, sucrose content and quality are very sensitive to changes in the environment. The environmental factors affecting qualtiy and sucrose concentration at harvest time are not well understood. One important and well-documented factor is nitrogen fertilization; however, climate, genetics, agronomic practices and length of the growing season are all interrelated and extremely important in their effects on harvest quality.

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Chemistry of the Beet and Processing Materials*

The composition of sugarbeets varies widely, depending on the genetic strain, agronomic factors of soil and fertilization, weather conditions during growth, incidence of plant disease, degree of maturity, and the treatment between harvesting and slicing. The percentage of sugar in mature beets ranges from 10 to 22%. The purity, the percentage of sucrose in the soluble dry matter, varies between 76 and 89%. Median values for North American factories for the 1979 campaigns were 15.5% sugar and approximately 85.0% purity.

As of all living tissues, the major component of the sugarbeet is water, amounting to about three-quarters of its weight. The beet is comprised of a liquid or juice phase, and an insoluble or solid phase, in a weight ratio of about 19:1.

Composition of Juice Phase

Water is again the largest individual component, and the juice contains approximately 25% of solids by weight, which are either in true or colloidal solution. Of these dissolved solids, by far the greatest portion is sucrose, with small amounts of other organic compounds and still smaller quantities of inorganics. The water-soluble nonsucrose organic constituents can, in turn, be separated into about equal quantities of nitrogenous and of nitrogen-free compounds.

Considering the nitrogen-free substances, there are included here other carbohydrates in small amounts, mostly glucose and fructose, raffinose and kestoses. There are likewise traces of arabinose and galactose. The organic acids are represented by various mono-, di-, and tricarboxylic acids, both unsubstituted and hydroxy acids. The juice also contains small quantities of pectic material with saponins, lecithin, and traces of numerous other organic compounds, such as the vegetable fats, which, in this case, are esters of glycerol and oleic, palmitic and erucic acids.

Of the nitrogenous compounds, protein fragments make up the largest fraction. These may be present in process juices as either colloidal or, under certain conditions, partially soluble suspensions. The nitrogenous organics, which cannot easily be coagulated or precipitated, are generally classed as "interfering" or "harmful" nitrogen. They make processing difficult. The internal quaternary ammonium salt, betaine, is the most abundant nitrogenous component, followed by the amides — mostly glutamine and asparagine — amino acids, purines and pyrimidines, and ammonia and nitrates.

The inorganic constituents, totaling about 0.5% of the whole beet, include as major constituents the cations of potassium, calcium, magnesium, and sodium, and the anions phosphate, chloride, and sulfate, each series arranged in order of abundance.

Composition of the Solid Phase

The insoluble portion of the beet, the *marc,* amounts to from 5 to 8% of the beet weight, nearly one-half of which is bound water. This can be freed from the true marc by drying with heat.

Of the true marc, one-half is insoluble pectic material, the composition of which is quite variable. The other half is made of about equal quantities of

*by R.A. McGinnis. Revised by R.M. Sequeira. Sections of the 1st ed., Chapter VII, by Prof. and Mrs. J. Dedek are included. These sections are enclosed in quotation marks.

cellulose, and of other associated structural materials such as lignin, and the hemicelluloses araban and galactan. In the marc there are also proteins, saponins, and trace quantities of miscellaneous organic and inorganic substances.

The chemical components of the beet will be considered in more detail under the general headings: nitrogen-free compounds, nitrogenous compounds, and inorganic substances.

NITROGEN-FREE ORGANIC COMPOUNDS

Carbohydrates

Carbohydrates were often considered to be hydrates of carbon, having the general formula, $C_n(H_2O)_m$. However, the term carbohydrates has been expanded to include substances which do not conform exactly to this formula, and a better definition would be: polyhydroxy aldehydes or polyhydroxy ketones or substances which yield such compounds on hydrolysis. Carbohydrates may be classified as monosaccharides, disaccharides, or polysaccharides, referring to the number of separate monosaccharide units contained in the molecule. Monosaccharides are simple carbohydrates which cannot be hydrolyzed.

Monosaccharides

The pentose (5 carbon) L-arabinose, a component of the hemicellulose of the beet marc, and the hexoses, D-galactose (a constituent of the trisaccharide, raffinose, occurring in the beet), D-glucose and D-fructose are important in the beet-sugar manufacturing process. The latter two monosaccharides are products of sucrose hydrolysis (commonly called inversion).

All of the monosaccharides mentioned above will reduce Fehling's solution, and are termed *reducing sugars*, although because of the small amounts of arabinose normally present in the process materials, the term usually refers, in practice, only to fructose and glucose.

The *invert sugars*, glucose and fructose, so called because the observed optical rotation of sucrose solutions is changed by its hydrolysis or *inversion* to a negative value, are quite reactive under conditions of high pH and high temperature. They may be destroyed so rapidly that they may accumulate in factory materials only in traces, although actually formed in considerably larger quantities.

Specific Rotation. The optical rotatory power of a sugar is given by the expression

$[\alpha]_D^t = \frac{100\,\alpha}{lc}$ where $[\alpha]_D^t$ is the specific rotation at a temperature the optical path length, l, of the sugar column is in decimeters, and the concentration, c, is in g/ml (62). The D indicates the rotation is measured at the sodium D line, although any other wavelength may be used. Recent work by the U.S. Bureau of Standards is seeking to establish the wavelength of the helium-neon laser as a standard wavelength for sugar polarization.

Inversion. The inversion or hydrolysis of sucrose, and the change of the specific rotation of the solution proceeds as follows:

$$C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6 \quad (1)$$

$$[\alpha]_D^{20}\ +66.5^\circ \qquad +52.7^\circ \quad -92.4^\circ \quad (2)$$

$$-19.8^\circ$$

Many of the chemical reactions of the reducing sugars may be grouped into those involving a) the carbonyl group, and b) the hydroxyl (OH) group.

a) Carbonyl Reactions

1) Additional reactions of glucosides: condensation reactions forming osazones with phenylhydrazine, oximes with hydroxylamine, and addition reactions with HCN to form cyanohydrins.

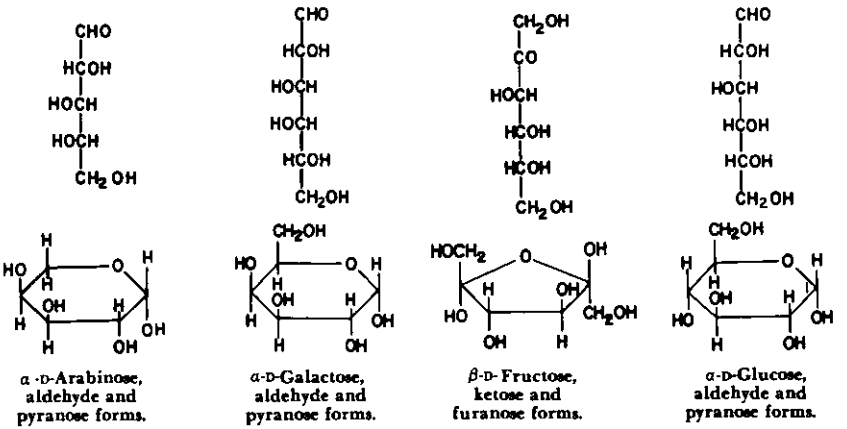
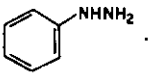
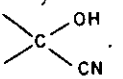


Figure 2-6. Monosaccharides, open and closed chain configurations.

Glucoside. A glycoside involving glucose. A glycoside is a sugar derivative in which carbon 1 of the internal hemiacetal is bonded to another group. This bond is referred to as the glycosidic bond.

Osazone. A compound formed by treating aldoses or ketoses with phenylhydrazine  . Osazones have characteristic melting points and other physical constants, which are useful in identifying sugars.

Oxime. A compound formed by reaction of aldehydes or ketones with ylamine, NH_2OH, and characterized by the group $=N-OH$ attached directly to a carbon atom.

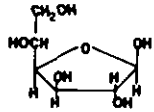
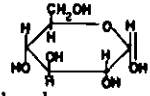
Cyanohydrin. A compound formed by the reaction of aldehydes and ketones with HCN, and containing the group  .

2) Oxidation to mono- and dibasic hydroxyacids.

3) Reduction to alcohols

4) In warm, alkaline solutions, caramelization, and formation of acidic products.

Carbohydrate Nomenclature. A *furanose* is a sugar which contains a 5-membered ring of four carbons and one oxygen, as shown in the furan form

of glucose,  or glucofuranose. A *pyranose* is a sugar which contains a 6-membered ring of five carbons and one oxygen as shown by the pyran form of glucose,  or glucopyranose.

Geometric Isomerism. Referring to the glucopyranose structure, the carbon designated as no. 1 is asymmetric, and thus two stereoisomers exist, α if the OH group is located below the plane of carbon atoms as represented by the Haworth formulas, and β, if above.

The furan or pyran forms are made by linkage of the aldehyde oxygen attached to carbon 1, with carbon 4 or 5. The aldehyde form of glucose, or ketone form of fructose are in equilibrium with the ring structures in solution. The crystalline sugars are generally in the α or β form, and *mutarotation* is the change of optical rotation occurring as a result of the equilibrium of the various forms, after dissolution of the crystalline sugars.

Optical Isomerism. The aldehyde form of glucose illustrated in Figure 2-6, is the D-glucose isomer, and its specific rotation is positive. If the positions of the H and OH on each of the carbons 2, 3, 4, and 5 are transposed, it becomes L-glucose, and the specific rotation is reversed. However, D and L do not refer to directions of rotation; D-fructose has a negative specific rotation. They refer ultimately to the configurations of D- and L-glyceraldehyde as described by the fundamental studies of Emil Fischer. Another basic system for the specification of configurations of all substances exhibiting configurational isomerism is useful to circumvent ambiguities resulting from the overlapping of individual systems of nomenclature. The symbols R and S are used (11).

In acid solutions, under dehydrating conditions, hexoses can lose three molecules of water, forming hydroxymethylfurfural, which then degrades into formic and levulinic acids. In addition, other reactions may occur, yielding disaccharides, and saccharides of higher molecular weight (Figure 2-7).

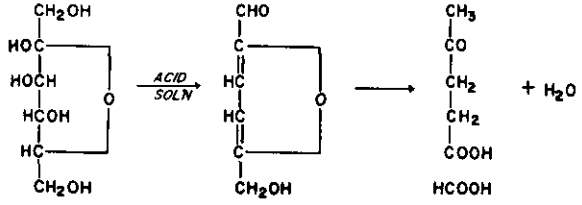


Figure 2-7. Typical degradation of a reducing sugar. Conversion of fructose to hydroxymethylfurfural and then to levulinic and formic acids.

In hot, alkaline solutions, invert sugars rapidly are converted into acids and colored decomposition products. The destruction of glucose is preceded by its isomerization to fructose and mannose. From a practical viewpoint, even traces of invert sugars will decompose and cause significant increases in the colors of process solutions. One molecule of glucose or fructose usually yields two molecules of acid; thus the inversion of one molecule of sucrose yields four molecules of acid, and thus can form four equivalents of lime salts.

The colors of the alkaline decomposition products are greater when formed at lower alkalinity. From an operating standpoint, the most desirable method of destruction of invert sugar in the diffusion juice is by liming the juice at a relatively low temperature, and then heating it to 80 °C or above. Figure 2-8 summarizes the results.

Bichsel (5a) has developed a kinetic method for the study of steady state concentrations in alkaline process liquids, which permits estimation of the destruction of glucose and fructose to be made with some precision.

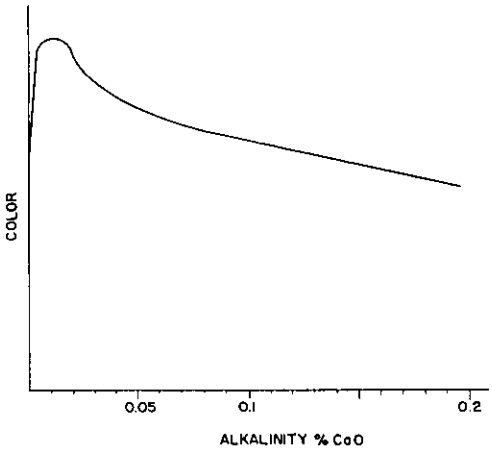


Figure 2-8. Color produced by destruction of invert sugars. The influence of alkalinity. *(Vasatko-Kasjanov, 1938).*

Tables 2-1 and 2-2, compiled by R. M. Silin from his own studies (79), summarize his data.

Table 2-1 Acidic Products of the Alkaline Reactions of Invert Sugars (79)

Acid	Formula	Comments
glucinic	$C_{12}H_{22}O_{11}{\cdot}H_2O$ (?)	A reducing material, brownish.
apoglucinic	$C_9H_{10}O_5$ (?)	Formed by alkaline degradation of glucinic acid. Blood-red salts.
saccharummic	$C_{14}H_{18}O_{11}$ (?)	Brown, reducing, decomposes on standing to form humic substances.
molassic	$C_6H_6O_3$	Dark brown, soluble in alcohols.
humic	$C_{60}H_{52}O_{24}(COOH)_4$	Dark, formed in soil from vegetable decomposition.
saccharinic	$C_6H_{12}O_6$	Soluble. Can form a lactone, saccharin.
lactic	$C_3H_6O_3$	

b) **Hydroxyl Reactions**

1) Formation of esters, or reaction products of acids and alcohols, including acetyl ($CH_3-\overset{O}{\overset{\|}{C}}-$) derivatives:

2) O-methyl derivatives, CH_3-O-), or ethers:

3) saccharates with metals.

c) **Microbial or Biological Reactions.** The reducing sugars are metabolized by microorganisms and yeasts yielding, among other substances, various alcohols, lactic acid, butyric acid, and dextran.

Table 2-2 Solubility of Some Acids and Salts Formed from Reducing Sugars in Alkaline Solution* (79)

(s = soluble, p = precipitated by)

	Free Acid	Basic Lead Acetate + Ammonia	Basic Lead Acetate	Lead Acetate	Ba $(OH)_2$ or Ca $(OH)_2$
glucinic acid	s	p	p	s	s
apoglucinic acid	s	p	p	p	s
saccharummic acid	s	p	p	p	p
molassic acid	p	p	p	p	s
humic acid	p	p	p	p	p
saccharinic acid	s	p	s	s	s
lactic acid	s	s	s	s	s

*All sodium and potassium salts are soluble

Table 2-3 Water Solubility of Monosaccharides in Percent Concentration by Weight

	Temperature, °C								Ref.
	0	10	20	25	30	55	60	90	
D-glucose	35.0	41.6	47.7	—	54.6	—	74.7	84.6	(77)
D-fructose	—	—	78.9	—	81.5	88.1	—	—	(77)
D-galactose	10.3	—	—	68.3	—	—	—	—	(47)
D-arabinose	—	16.9	—	—	—	—	—	—	(47)

Table 2-4 (77) Physical Properties of Monosaccharides

	Specific Rotation, $[\alpha]_D^{20}$	Molecular Weight	Melting Point °C	Crystalline Form	Density g/ml
D-glucose	+52.7	180.16	146	rhombic needles	$1.544^{\frac{25}{4}}$
D-fructose	—92.4	180.16	102-104	needles	$1.598^{\frac{20}{4}}$
D-galactose	+80.2	180.16	167	hex. tablets	
DL-arabinose	+104.5	150.13	160	rhombic prisms	

Disaccharides

Sucrose. The only disaccharide of importance in beet materials is sucrose (saccharose, cane sugar, beet sugar, α-D-glucopyranosyl-β-D-fructofuranoside). See Figure 2-9.

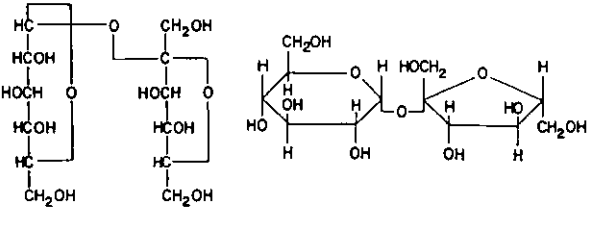


Figure 2-9. Fischer-projection and Haworth-type structural diagrams of sucrose.

a) *Physical Properties (48).*
Molecular weight: 342.303
Thermodynamic properties:
normal entropy = 86.1 cal/°K per mole (66)
heat of formation = 530.8 kcal/mole
enthalpy of combustion = −1351.3 kcal/mole (43)
heat of combustion = 1351.3 kcal/mole

Data on crystal form: (4,84a,92a) Monoclinic, with monoclinic, hemimorphic (sphenoidal) crystals: ratio of axes. a:b:c = 1.2595:1:0.8782, angle β = 103°30'. Crystals have a prismatic habit, strongly influenced by impurities.
Crystals are triboluminescent (*emit light on fracture*).
Specific heat: 99.3 cal/mole at 20°C.
Electric conductivity not measurable.
Dipole moment 3.1 × 10^{-18} dynes$^{1/2}$ cm^3 (58)
Dielectric constant: between 3.5 and 3.85 (64)
Specific magnetic susceptibility: −0.57 (37)
Density: 1.5879 g/ml $\frac{15°}{4}$

Linear thermal-expansion coefficient: 0.0028–0.0050%/°C, depending on axis (75)
Melting point: decomposes at 185-186°C (56)
On slowly melting sucrose, an amorphous state is produced, which slowly crystallizes on standing.

b) *Aqueous Solutions.*
Physical properties of sucrose solutions are described in Chapter XII.
The specific rotation of sucrose is $[\alpha]_D^{20}$ + 66.53 (5).

The acid dissociation constants are: pk_1, 12.7 at 25°, 12.3 at 40°, and with some uncertainty, 11.6 at 80°, pk_2, 13.1 at 25°

Francotte *et al.* have reported the following values at 25°C (37a): pK_1 = 12.02, pK_2 = 12.56, pK_3 = 12.01

c) **Reactions in Aqueous Solutions**[1]. "Chemically, sucrose is rather unstable, being easily hydrolyzed, especially in acid solutions, to glucose and fructose. These are even more reactive sugars, which decompose into acids and colored products. It is important to recognize that in the presence of heat and high alkalinity sucrose can form oxalic acid, resulting in the deposition of calcium oxalate scale in the evaporators and vacuum pans (88). The great number of hydrophilic OH groups it contains make it easily soluble in water, even though its molecular weight is high. These hydroxyl groups can be esterified. The hydroxyl groups are further able to dissociate ionically, so that sucrose will behave as a polybasic acid in alkaline solutions (57), and can form alcoholates, or saccharates."

d) **Reactions in Acid Solution.** Under the catalytic action of hydrogen ions, and to a smaller extent of hydroxyl ions, sucrose hydrolyzes, forming glucose and fructose. In so doing, the dry substance in solution increases by the weight of the water molecule, or 5% (see Figure 2-10).

The inversion is a bimolecular reaction between sucrose and water. However, in dilute solution, the water concentration is effectively constant, making it a unimolecular reaction. Further, since in factory practice only small amounts of sucrose are allowed to be destroyed, the sucrose concentration, also, may be considered constant, reducing the reaction to the zero order, where the rate is proportional to the catalyst.

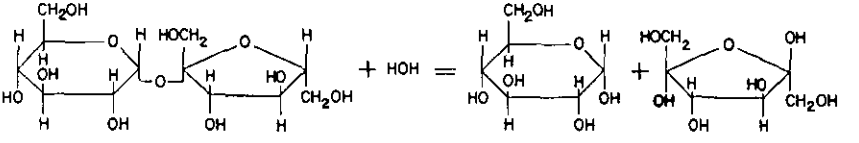


Figure 2-10. Inversion of sucrose to form glucose and fructose.

[1]See refs. 55a, 58a, 59a

In this reaction I is the quantity of invert formed, and S_0 is the original sucrose concentration, t is the time in minutes, and K is the rate constant:

$$I = KS_0 t \quad (3)$$

The rate constant is proportional to the hydrogen ion concentration,

$$K = K_0 [H^+]. \quad (4)$$

K_0 is a constant, independent of the hydrogen ion concentration, but which increases rapidly with temperature. If S_0 and I are expressed in any consistent units, values of the rate constant, K_0, for different temperatures are, 50°, 0.1145 M^{-1}; 60°C, 0.3806 M^{-1}; 70°C, 1.182 M^{-1}; 80°C, 3.303 M^{-1}; 90°C, 8.922 M^{-1}; 100°C, 26.797 M^{-1}.

e) **Reactions in Alkaline Solution.** Sucrose is resistant to reducing agents, showing only a slight effect with Fehling's solution.

In hot, alkaline solutions, decompositions occur. The primary product is lactic acid. It has been noticed that when such decomposition occurs in the evaporators, about three equivalents of acid are formed per mole of sucrose destroyed. Hirschmüller states that as a rule of thumb, if one assumes that the sucrose loss in a limed solution of pH 12 (20°C) held for one hour at the boiling point is 0.5%, approximate estimates may be made of sucrose losses under other conditions. This is done by estimating that one unit *increase* in pH or 10°C increase in temperature involves a three times increase in sugar loss, and in the case of small losses, these are directly proportional to the time.

The hot, alkaline decomposition of sugar causes a browning effect, with formation of largely undefined coloring substances, probably not similar to those mentioned in the alkaline degradation of invert.

The alkali and alkaline-earth metals form saccharates, in which a metal ion replaces an H ion in one of the OH groups, and in this reaction sucrose behaves as an acid. Calcium and barium saccharates are discussed in detail in Chapter XVIII.

Sucrose is not easily oxidized, except by strong oxidizing agents such as permanganate, peroxide, or nitric acid. Likewise reduction requires strong methods, such as the use of hydrogen in the presence of metallic catalysts.

f) **Hydrolysis by Enzymes.** Sucrose may be hydrolyzed by the action of various enzymes, and a common example is with the use of the enzyme, invertase, to invert sugars. The bacterium *Leuconostoc mesenteroides,* frequently encountered in the sugar factory as "frog-spawn," secretes an enzyme, α-D-glucopyranosyl transferase, which converts sucrose to fructose and dextran. Dextran has molecular weights up to many millions (52). *Bacillus subtilis* can correspondingly bring about the formation of the high-molecular-weight polysaccharide, levan.

Dextran and levan are frequently formed in appreciable amounts in beets in storage under unfavorable conditions, and can constitute a real problem. These substances can partially seal filter cloths, and have seriously impeded rotary vacuum filtration of first carbonation sludge in a number of instances. Both dextran and levan appreciably affect the polarizations of the process juices. Their presence in appreciable quantities can cause error in the measurement of the sugar entering the factory.

Trisaccharides

The only trisaccharide found in significant quantities in all sugarbeets is raffinose. Under certain circumstances kestoses are found, and identification has been made of the trisaccharide, planteose.

Raffinose. Raffinose (melitose, mellitriose, gossypose) is a trisaccharide (see Figure 2-11) which occurs in sugarbeets to the extent of 0.3-1.2% or more of the sugar present (10,65a). The amount in beets increases with prolonged, cool storage, and since it is as chemically resistant as sucrose, it accumulates in the molasses. Since it forms insoluble calcium saccharates, it is precipitated along with the sucrose in the Steffen process, greatly increasing the raffinose content of Steffen molasses. Referred to the anhydrous form, in solution it is nearly twice as dextro-rotory as sucrose, and its presence affects the polarization for sucrose determination, which may lead to sugars polarizing over 100 °S.

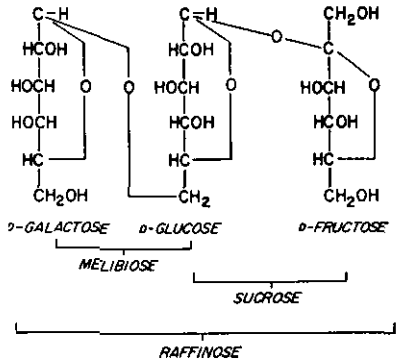


Figure 2-11. Raffinose.

Raffinose interferes with the crystallization of sucrose, primarily by adsorption on specific crystal faces. This effect can be significant in processing in certain beet-growing areas. The effect assumes technical importance when the raffinose concentration in solution exceeds 0.5% on sucrose (50).

Raffinose influences the crystal habit of sucrose, a result of the reduction of normal growth rates along certain crystallographic axes (49). At levels ranging from zero to 0.5% raffinose on sucrose, no significant deformation occurs. A distinctive cubic appearance is evident at levels of 0.5% to 1.0% or more. Peculiar flat crystals are induced as the raffinose level approaches 2.0% on sucrose. At even higher concentrations the crystals become thin, narrow plates, sometimes almost needle-like.

a) **Physical Properties**

Crystal form: needles or prisms. Normally crystallizes as the pentahydrate, $C_{18}H_{32}O_{16}{\cdot}5H_2O$

Molecular weight: anhydride, 504.45
pentahydrate, 594.45

Melting point: anhydride, 118-119 °C
pentahydrate, 80 °C.

Boiling point: pentahydrate, decomposes at 130 °C

Solubility: pentahydrate: water, 14 g/100 ml at 20 °C, freely soluble, hot.
ethanol, slightly soluble
methanol, insoluble

Specific rotation: $[\alpha]_D^{20}$ anhydride, +123.1 (77)
pentahydrate, +105.2 (66b)

Because its presence increases the polarizations of factory materials, it is practically important to be able to estimate its concentration, analytically. While paper chromatographic methods are currently in widest use (13,17,55,97), thin-layer chromatography can be used to advantage (89), and an enzymatic-photometric procedure holds promise (61).

b) **Chemical Reactions.** Raffinose does not reduce Fehling's solution.

In very dilute acid solution, or with invertase, a partial hydrolysis occurs in which the fructofuranose ring splits off, leaving melibiose, a disaccharide composed of D-galactose and D-glucose, and the freed D-fructose. Still more rigorous acid treatment splits the melibiose into its components. On the other hand, under the catalysis of the enzyme α-galactosidase, the raffinose splits into sucrose and D-galactose.

Minor Trisaccharides

Kestoses. Kestoses are fructosyl sucroses, formed from one molecule of sucrose and one of fructofuranose. Three configurations of this trisaccharide have been found in beets. These are designated 6-kestose, 1-kestose, and neo-kestose (8) (Figure 2-12).

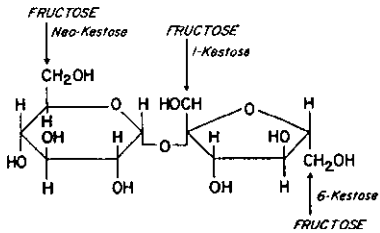


Figure 2-12. Kestoses. Sucrose molecule, showing points of attachment of fructose to form the three known kestoses.

Recently Binkley, *et. al.* (7) has found a nonfermentable trisaccharide, planteose, in small amounts in beet-sugar materials [α-D-galactopyranosyl-(1—6)-β-D-fructofuranosyl-(2—1)α-D-glucopyranoside].

Also a tetrasaccharide, stachyose, in which raffinose is attached to another molecule of galactose, was isolated in small amounts from beet molasses by Schneider, *et. al.* (69) [α-D-galactopyranosyl-(1→6)-α-D-galactophranosyl-(1→6)-α-D-glucopyranosyl-(1→2)-β-D-fructofuranoside].

Miscellaneous Carbohydrates

a) **Inositols.** The inositols are are hexahydroxycyclohexanes. *Myo*-inositol has been found in beets in the amount of 0.4% on sugar (see Figure 2-13). Brown and Serro in 1952 (74) first isolated and identified 1-0-(α-D galactosyl)-myo-inositol, which they named "galactinol." These workers obtained galactinol from Steffen molasses. Later, Dutton, using a modified procedure, isolated galactinol from

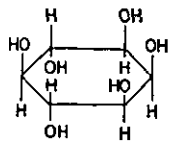


Figure 2-13. *myo*-inositol.

straight-run molasses (34). He found the galactinol content to be somewhat less than 0.2%. This substance reacts weakly to certain aldose tests, does not react to ketose tests, but does react with ammoniacal silver nitrate. Neither *myo*-inositol or galactinol are affected by carbonation, but they are precipitated in the Steffen process and *thus accumulate in Steffen molasses. Galactinol is optically active*, the $[a]_D^{20} = +135.6°$, and the polarization drops to a low value after hydrolysis in the presence of melibiase. It normally exists as the dihydrate, $C_{12}H_{22}O_{11} \cdot 2\ H_2O$, m.p. 220-222 °C, solubility in water at 20 °C, 99.8 g/100 g water.

Myo-inositol has a m.p. 225-227 °C, no optical activity, and water solubility of 14 g/100 ml of solution at 25 °C.

b) **Hemicelluloses.** Hemicelluloses are carbohydrate polymers. Chemically they have no relation to cellulose, and the name has arisen because they are frequently found in association with cellulose.

Araban and galactan are polysaccharides occurring in conjunction with pectic substances as constituents of the cell walls. Araban makes up about 25-30% of the pectic substances. Galactan is present in relatively small quantities. Both are dissolved from the cell walls by dilute alkaline solutions, and on heating with dilute acids, break up into their chief building blocks, L-arabinose and D-galactose.

Araban is water soluble (80), optically active $[\alpha]_D^{20} = -129°$, and has a molecular weight of 10 000. The basic structure is thought to be L-arabofuranose, connected with an α-glycoside linkage (Figure 2-14).

Sugarbeet galactan has neither been isolated nor has its structure been elucidated.

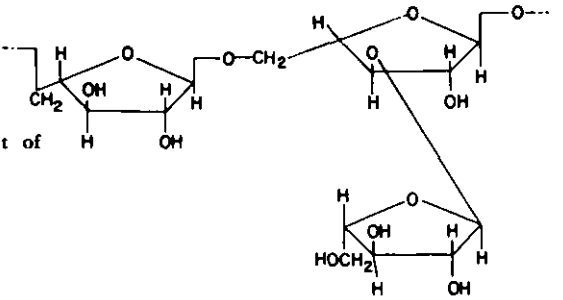


Figure 2-14. Repeating unit of araban.

Araban and galactan, liberated from the pectin complex present in the beet, pass unaltered through the normal purification process to the molasses. Quantities of araban ranging from 100 to 500 mg per kg have been found in various molasses (19), the higher levels being associated with the operation of diffusers using makeup rich in ammonia. Araban and galactan are removed from thin juice by activated carbon.

Polysaccharides of High Molecular Weight

Cellulose

Cellulose, $(C_6H_{10}O_5)_n$ makes up the major portion of vegetable fiber and tissues, and consists of building blocks of glucose, connected in very long and very stable molecules with β-glycosidic connections (Figure 2-15). Chemically, cellulose is relatively inert, and is quite insoluble in water and dilute acids or bases, but is soluble in Schweitzer's reagent [Cu $(NH_3)_4$] $(OH)_2$. Nevertheless, it can be digested

by ruminants with the aid of intestinal bacteria, for whom it has a real food value. It has been found to make up about 26-27% of the beet marc (39).

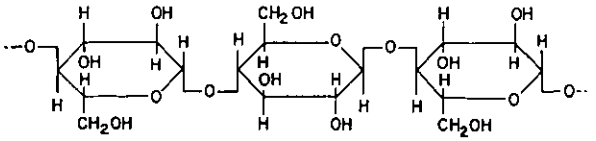


Figure 2-15. Repeating unit of cellulose.

Lignin

Lignin $(C_{10}H_{13}O_3)_n$, is probably arranged structurally in units of $-(C_6H_4)(OCH_3)C_3H_6O-$ joined with cellulose to contribute strength to the cell walls. It is thought to be related to coniferyl alcohol $(CH_3O)(OH)C_6H_3-CH:CHCH_2OH$. It is insoluble in water or dilute acid or alkaline solution, and remains with the pulp, where it is apparently without food value.

Pectic Material

The cell walls are largely made up of pectic substances, the central lamella being almost 100% pectic substance. The total content in beets varies from 2.5 to 10% on ds, being higher in immature beets. It is the largest component of the beet dry substance, after sucrose. Dissolved pectin is also found in the cell sap, as well as in immature beets (0.008-0.015%) (51). The pectin content decreases markedly in long term, warm beet storage (1). Since there have been a number of changes in terminology, current standardization is as proposed by Henglein (42).

Pectic material is a collective designation. *Protopectin* is the portion insoluble in water, which forms *pectin* on hydrolysis. Pectin in turn is polygalacturonic acid, partially or wholly esterified. A *pectinate* is a salt of pectin. *Pectic acid* is unesterified polygalacturonic acid, and a *pectate* is a salt of pectic acid.

As already mentioned, the hemicelluloses araban and galactan are usually found associated with pectic materials, as are cellulose and lignin, all of which may be connected with true pectic substance by secondary valences or esterifications.

The building unit of pectin is D-galacturonic acid (63,71) (Figure 2-16).

Pectin contains long polygalacturonic acid chains, with α -glycosidic linkages, which contain about 7% by weight of methylated carboxyl groups $(-CO-O-CH_3)$ and 6% by weight of α-acetyl groups $(CH_3CO)-$. Beet pectin contains more acetyl groups than other vegetable pectins, and thus has a lower jelling power. The

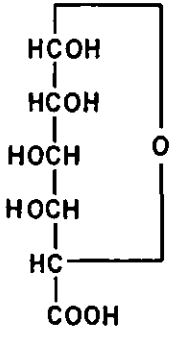


Figure 2-16. D-galacturonic acid.

molecular weight of beet pectin, (about 20,000) is much lower than those of apple or citrus pectins, which range between 150 000 and 200 000.

Beet pectin is optically active, $[\alpha]_D = +130°$, and after de-esterification this rises to $+230°$ (81). Because pectins are not easily defecated with lead acetate, the error of apparent purities is increased, and sucrose losses may be simulated. Free pectin is quite soluble in 20 parts of water, but its calcium salts are insoluble.

Beet pectin will hydrolyze in acid, or much more easily in alkaline solutions, with the loss of the methyl ester groups as methyl alcohol, and the loss of the acetyl groups as acetic acid. Finally the pectin chain is hydrolyzed by acid to give units of lower molecular weight, which are not precipitable by lime. Pectic acid is quite stable to alkalies.

In Figure 2-17 is a diagram which summarizes the pectic relationships (81).

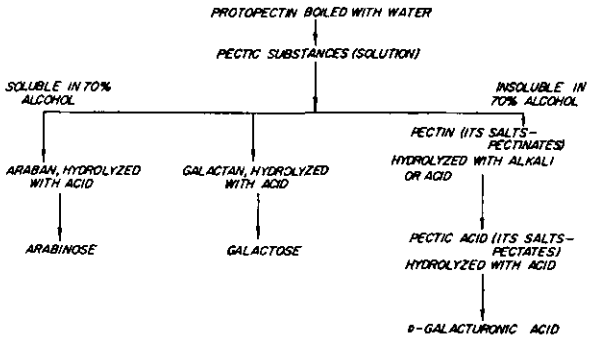


Figure 2-17. Relationships of pectic materials. (Silin).

In the process, care must be taken to avoid acid hydrolysis of pectic substances which results in solution of pectin and pectinates and their products, pectic acid and pectates. The hydrolysis of the methyl ester groups is much more rapid at elevated temperatures. The rate of hydrolysis is at a minimum at 5.2 pH (84), but increases as the pH is either lowered or raised. At 7 pH the rate is 8× that at 5 pH.

Dedek (29) found that colloidal micelles of pectin probably behave as cation exchangers. This property may tend to increase the natural alkalinity. He notes that the "larger-molecule pectins, if present, will form, on liming, voluminous colloidal, strongly hydrated precipitates, even highly viscous jelly-like solutions, settling and filtering with great difficulty. The lower-molecular weight decomposition products, either produced in the beets or during the liming of the juices, seem to influence the crystallization of sucrose, and to form turbidities which are difficult to remove by filtration." There are indications that the precipitation of proteins in cossette scalding may be enhanced by the presence of pectins (30).

Activity of certain enzymes has been noted in breaking down pectic structures.

Organic Acids

Many organic, nitrogen-free acids occur in the sugar beet in small quantities, and more are produced in the factory materials as the products of decomposition of larger organic structures, under the influence of enzymes, microorganisms, or liming.

Table 2-5 lists the acids commonly identified together with some of the characteristics important in processing.

TABLE 2-5 ORGANIC ACIDS FOUND IN BEET MATERIALS meq/kg

ACID NAME	Chemical Structure	Class	Source	Typical (96) Amounts in [2](a) Beets [3](b) Molasses	Place of Elimination in Process	Ca-Salt Water Solubility	Acid Dissociation Constants, 25 °C	Optical Activity $[\alpha]_D^{20}$
FORMIC ACID	HCOOH	Monobasic	Decompositions Beets	(a) 0.02 (b) 5	not elim.	16.2 (0°) 18.4 (100°) g/100ml	1.76×10^{-4}	
ACETIC ACID	CH_3 COOH	Monobasic	Decompositions Beets.	(a) <0.01 (b) 13.5 (with butyric)	not elim.	37.4 (0°) 29.7 (100°) g/100 ml	1.75×10^{-5}	
BUTYRIC ACID n-Butyric acid ethylacetic acid butanoic acid	CH_3 CH_2 CH_2 COOH	Monobasic	Decompositions Beets	(a) <0.01 (b) 2.0 (with acetic)	not elim.	22.0 (0°) 17.8 (100°) g/100 ml	1.48×10^{-5}	
OXALIC ACID ethanedioic acid	COOH COOH	Dibasic	Decompositions Beets	(a) 0.08 [4] (b) 1.5	partly in defecation, evaporators (see text)	0.00067 (15°) 0.0014 (95°) g/100 ml	6.5×10^{-2} 6.1×10^{-5}	none
MALONIC ACID propanedioic acid methanedicarboxylic acid	COOH CH_2 COOH	Dibasic	Beets			0.44 (0°) 0.72 (100°) g/100 ml	1.64×10^{-3} 2.1×10^{-6}	none
SUCCINIC ACID butanedioic acid amber acid ethylenesuccinic acid	COOH CH_2 CH_2 COOH	Dibasic	Beets			0.193 (10°) 0.89 (80°) g/100 ml	6.6×10^{-5} 2.8×10^{-6}	none

TABLE 2-5 continued

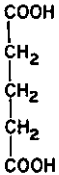
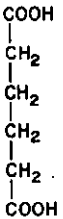
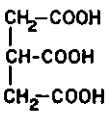
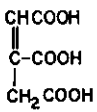
GLUTARIC ACID pentanedioic acid 1-3-propanedicarboxylic acid		Dibasic	Beets		not elim.	Sl. Sol.	4.60×10^{-5} 6.0×10^{-6}	none
ADIPIC ACID hexanedioic acid 1, 4-butanedicarboxylic acid		Dibasic	Beets		not elim.	Sl. Sol.	3.90×10^{-5} 5.29×10^{-6}	none
TRICARBALLYLIC ACID 1, 2, 3 propanetricarboxylic acid		Tribasic	Beets			Sl. Sol. 0.29%	3.25×10^{-4} 2.65×10^{-5} 1.48×10^{-6}	none
ACONITIC ACID 1, 2, 3 propenetricarboxylic acid equisetic acid citridic acid achilleic acid		Tribasic	Beets Decompositions		not elim.	Sol.	1.58×10^{-3} 3.5×10^{-5}	none
GLYCOLIC ACID hydroxyacetic acid hydroxyethanoic acid		Hydroxy	Beets	(a) 0.01 (b) 4	not elim.	Sol.	1.48×10^{-4}	none

TABLE 2-5 continued

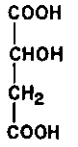
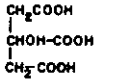
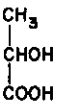
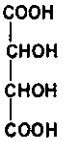
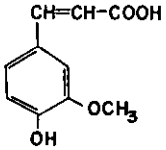
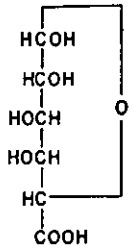
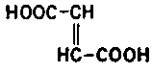
ACID NAME	Chemical Structure	Class	Source	Typical (%) Amounts in [a] Beets [b] Molasses	Place of Elimination in Process	Ca-Salt Water Solubility	Acid Dissociation Constants	Optical Activity $[\alpha]_D^{20}$
L-MALIC ACID L-hydroxysuccinic acid apple acid		Hydroxy	Beets	(a) 0.08 (b) 3	not elim.	0.812 (0°) 1.224 (37.5°) g/100 ml	4×10^{-4} 9×10^{-6}	—3.07° (salts from —9.84° to —7.13°)
CITRIC ACID 2-hydroxy-1, 2, 3-propanetricarboxylic acid β-hydroxytricarballylic acid		Hydroxy	Beets	(a) 0.52 (b) 5	partly in defecation, evaporators	0.25 (30°) 0.210 (95°) g/100 ml	18° 8.2×10^{-4} 1.77×10^{-5} 3.9×10^{-6}	none
DL-LACTIC ACID lactic acid racemic lactic acid α-hydroxypropionic acid 2-hydroxypropanoic acid		Hydroxy	Beets	(a) 0.01 (b) 20.9	not elim.	3.1 (0°) 7.9 (30°) g/100 ml	1.38×10^{-4}	none
L-TARTARIC ACID ordinary tartaric acid L-2, 3-dihydroxybutanedioic acid		Hydroxy	Beets		defecation	0.0032 (0°) 0.0078 (37.5°) g/100 ml	1.04×10^{-3} 6.9×10^{-5}	+11.98°
GLYOXYLIC ACID formylformic acid glyoxalic acid oxoethanoic acid		Aldehyde	Beets		not elim.		4.6×10^{-4}	none

TABLE 2-5 continued

FERULIC ACID 4-hydroxy-3-methoxycinnamic acid 3-methoxy-4-hydroxycinnamic acid 3-(4-hydroxy-3-methoxyphenyl) propenoic acid		Beets				
D-GALACTURONIC ACID		Beets	(a) 0.06	defecation	insol.	muta-rotation +98.0° → +53.0°
FUMARIC ACID		Beets	(a) 0.01		2.11 (30°) g/100 ml	

(1) in pressed juice
(2) g/100 g total solids
(3) meq/100 g 80% ds molasses

In general, organic acids may be regarded as intermediate or end products of changes in carbohydrate structures. In the plant, they serve a useful purpose as buffers to maintain favorable pH environments. While in the sugarbeet itself the inorganic acids predominate, in diffusion juice the organic acids make up the greater portion. See Table 2-6.

Table 2-6 Relative Distribution of Organic and Inorganic Acids in Beets and Diffusion Juice (96)

	Percent of Total Acids	
	Beets	Diffusion Juice
Inorganic		
Phosphoric	40.0	25.3
Hydrochloric	13.3	13.7
Sulfuric	5.8	5.3
	59.0	44.1
Organic		
Citric	20.0	18.4
Oxalic	6.7	10.7
Malic	5.0	4.7
Acetic		6.8
Lactic		8.8
Succinic		
Glycolic		
Galacturonic	7.8	6.6
Pyroglutamic		
Formic		
Fumaric		
Butyric		
unidentified	1.5	0.6
	41.0	55.9

Acids with Insoluble Lime Salts

For practical purposes, acids may be classified either as having relatively insoluble or relatively soluble calcium salts. Prominent examples of the insoluble class are oxalic, tartaric, and citric acids, together with the inorganic sulfuric and phosphoric acids. "These acids are, in the form of potassium and sodium salts, very important as a source of the so-called 'natural alkalinity,' formed in accordance with the reaction: $An'' + CaO + H_2O = CaAn + 2OH^-$ where An'' is the anion of such an acid, and CaAn is its insoluble lime salt. The NaOH or KOH so formed is the natural alkalinity, and plays a very important role in the precipitation of Ca from soluble lime salts in the second carbonation."

There are a number of unusual features. The solubility of calcium oxalate increases with increasing lime content, and decreases with increasing sugar content (67), and, indeed, the solubility of all calcium salts increases somewhat in alkaline sugar solutions, probably due to complex formations. Spengler and Brendel (87) actually found that calcium oxalate goes into solution in main liming, but it is precipitated again in carbonation. Oxalate, it has been pointed out, can be formed as a result of a Cannizaro disproportionation reaction (between two molecules of certain aldehydes in alkali, so that one is reduced to the corresponding alcohol, while the other is oxidized to the acid) of glyoxylic acid. It has also been suggested that it may result from the decomposition of so-called oxalogenic substances such as oxamide, allantoin, and sucrose. More recent critical studies indicate quite clearly that calcium oxalate deposition as scale in evaporators is not due to any of the above reactions but can be accounted for in terms of the solubility product of calcium oxalate. This solubility product, in the presence of 30% sucrose, is half what it is in

solutions containing 10% sucrose. (12). That oxalate formation from invert degradation is negligible in comparison with the oxalate which is not removed in juice purification by liming and gassing with carbon dioxide was strikingly demonstrated in experiments involving the use of radioactive fructose (20).

Citric acid usually precipitates as the tricalcium citrate. Its solubility also decreases with increasing sucrose concentration, and it can form very highly supersaturated solutions, from which crystals can slowly deposit in sugar-end equipment, and even appear as flakes in the sugar itself. Citric acid, when heated, can undergo dehydration, eliminating a molecule of water to form aconitic acid (Figure 2-18).

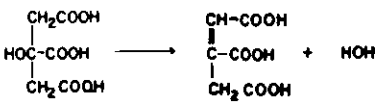


Figure 2-18. Formation of aconitic acid from citric acid.

Acids with Soluble Lime Salts

"Acids which give easily soluble lime salts are, from the point of view of juice purification, of lesser importance. They increase the ionic strength of juices, thus influencing the dissociation constants of such substances as carbonic acid. If present as free acids, they give rise to the formation of lime salts. Their pK, if low, is also responsible for less efficient deliming in second carbonation. Oxyacids are able to form complex calcium salts, and they thus also increase the lime content of carbonated juices. In alkaline, limed, and carbonated juices, they act as buffers, affecting the pH-alkalinity relationship in the juices. They are, through their cations (24,25), strongly melassigenic."

Basic lead acetate precipitates all the organic acids with the exception of glycolic, lactic, and malic. The optical activity of malic acid could be a factor contributing to cause errors in sucrose polarizations of leaded solutions.

Saponins

Saponin, or sugarbeet glycoside, appears to be located primarily just under the skin of the sugarbeet root, where it may protect the beet against invasions of microorganisms, since it is quite toxic to lower forms of life, including fish, although harmless to man.

Chemically, saponins are characterized by aqueous solutions of very low surface tension, and are responsible for the high foaming power of diffusion juice. Saponins are insoluble in acids, and are precipitated by heavy metals.

Saponins consists of a pentacyclic triterpene, called the sapogenin, tied with a glucosidic linkage to an acidic carbohydrate, such as glucuronic acid. It was first conclusively shown by Ehrlich and Rehorst (35) and van der Haar (41) that beet saponin contains a sapogenin of oleanolic acid linked to a molecule of glucuronic acid. Eis and co-workers (36) first showed the sugarbeet saponin to be primarily responsible for floc formation in carbonated beverages made from beet sugars.

With the aid of thin-layer chromatography, van Duuren (92) found that there are probably at least six different sapogenins present, all belonging to the β-amyrin group. In addition to oleanolic acid, he was tentatively able to identify quillaic acid.

Eis found three species present: the free aglycone, oleanolic acid; the glycoside, composed of oleanolic acid and glucuronic acid; and a glycoside-salt complex, cleaving into glucuronic and oleanolic acids on hydrolysis (Figure 2-20).

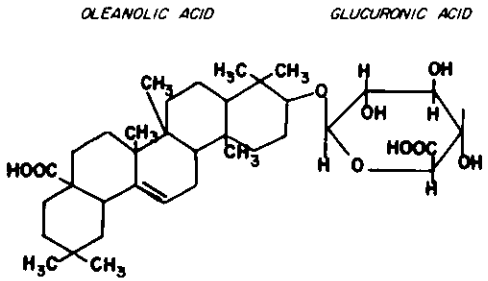


Figure 2-20. Sugarbeet saponin (36).

Carbonated Beverage Floc

Floc formation in carbonated beverages is complicated, since saponin serves as a "trigger" substance, and traces of other acid-insoluble substances in solution, such as defoaming oils, will join with the saponin to add to the bulk of the visible flocs. In the absence of saponin, however, no floc appears on acidification, but only a general light turbidity.

Normal lime-carbon dioxide purification, when carefully and optimally conducted, will eliminate over 97% of the saponin which is present at from 5000 to 10 000 ppm on ds extracted in the diffusion juice. The small amount passing through the process is, however, selectively adsorbed in the sugar crystal faces, and, in general, more than 2 ppm in the crystalline sugar will cause unpleasant appearing flocs when a dilute water solution of the sugar is acidified.

The amount of floc in the sugar can be affected in many ways. If the diffusion is conducted at a lower pH, at least theoretically, less floc is extracted. The use of predefecation increases floc elimination. Vigorous defecation, with large amounts of lime, high first carbonation alkalinities and lime addition to second carbonation increase the saponin precipitation. Once past the purification stage, effort can only be made to keep the saponin in solution, or to remove it by adsorbing agents. Since the saponin is more soluble at high pH values, attempts have been made to keep the second carbonation endpoint relatively high (0.028% CaO alkalinity) and to keep the massecuite pH above 8.6 (60,68). This was done successfully by the steady addition of caustic soda to the white pan massecuite while boiling, although at the sacrifice of color increase and purity lowering (38). It was accomplished without detriment by "careful operation" an appreciable part of a campaign (54). This has also been accomplished by careful control of second carbonation and sulfitation, essentially preventing a pH lowering across the evaporators.

One manufacturing company alkalizes the white massecuite in several of its factories by steadily adding caustic soda to the third or fourth evaporator effect. It is also common practice to add soda ash to the remelt-sugar melters for the same purpose.

Removal by adsorbing agents is more successful, and this is accomplished in all of the five factories of one company by passing the thick juice through beds of regenerable, granular carbon (Pittsburgh CAL or Nuchar), with the added benefit of additional color removal (2). Most anion exchangers will nearly completely remove saponin from treated process liquids.

Lipids

The beet fats are mostly located in the protoplasm portion of the cell walls. Estimates of the lipid contents of beets vary from 0.01% (65) to 0.03% (82) on beets.

The approximate composition of the lipids (45,65,82), assuming the 0.03% value, and 17.9% soluble ds in the beets, is: oleic acid, $CH_3(CH_2)_7CH{:}CH(CH_2)_7COOH$, 0.01% on beets; erucic or brassidic acid, $CH_3(CH)_{14}COOH$, 0.003% on beets; lecithin, phytosterols, 0.01% on beets.

The acids are probably present in the form of glycerides.

Phytosterols are unsaponifiable plant sterols characterized by the presence of a saturated phenanthrene ring (see Figure 2-21), with an additional 5-member ring fused at the 1,2 position.

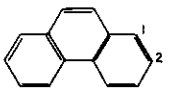
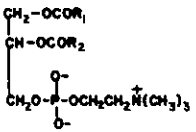


Figure 2-21. Phenanthrene ring.

Lecithin is a generic name for a 3-carbon chain structure, as shown in Figure 2-22, where R_1 and R_2 are fatty acid residues. Since lecithin contains a substituted ammonia group (quarternary nitrogen), it is to some extent a vegetable base, and is saponified by alkalies, yielding salts of fatty acids, phosphoric acid, and choline (82).

The fatty acids and portions of the decomposition products of lecithin are eliminated in the juice purification process. The phytosterols, in very small amounts, may pass through to the molasses.

Figure 2-22. Lecithin.



NITROGENOUS COMPOUNDS

Since the beet contains 1% or more of nitrogen-containing compounds, and since they mostly become parts of the diffusion juice, their importance from the technical viewpoint is very great. While the percentage of nitrogen varies widely with types of fertilization, beet strains, and growing conditions, Table 2-7 gives a typical distribution of nitrogenous compounds in an average European beet.

Table 2-7 Nitrogenous Materials in Beets (83)

	% on Beets	% of Total
Amino acids and others	0.042	21.0
Amides	0.015	7.5
Ammonia	0.005	2.5
Nitrates	0.002	1.0
Proteins and peptides	0.115	57.5
Betaine and choline	0.020	10.0
Purines	0.001	0.5
Total	0.200	100.0

The nitrogenous materials which are not removed in standard juice purification have been called "interfering" or "harmful" nitrogen. The substances which are removed are: 1) proteins, coagulated by lime; and 2) ammonia, expelled from the alkaline solutions.

Some amino acids are adsorbed by the first carbonation precipitate, but betaine and choline, nitrates, the purines, and most of the amino acids are molasses formers.

Amino Acids

With the one exception of γ-aminobutyric acid which is found only in the cell sap, all the amino acids are constituents of both the proteins of the cell structure, and of the cell sap. The amino acids commonly found in the sugarbeet, with some of the characteristics important for the factory processing, are listed in Table 2-8.

An amino acid is an organic acid in which one or more of the hydrogen atoms of the carbon chain has been replaced by an NH_2 group. Thus amino acids are amphoteric: i.e., they can act either as acids or as bases. In alkaline solution, the NH_2 group adds a molecule of water and becomes the basic NH_3OH group, and this can form salts with an acid (Figure 2-23). The carboxyl group can react with bases in the normal manner.

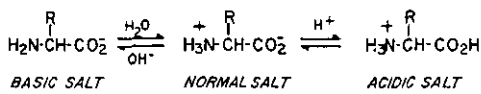


Figure 2-23. Amphoteric characteristics of amino acids. Basic salt form, as in the sodium salt. Normal salt form as in the free amino acid. Acidic salt form, as in the amino acid hydrochloride

Most of the amino acids are water soluble, and all are soluble in alkaline solution. None are precipitated by lime, but some small amounts may be adsorbed and removed by the first carbonation precipitate.

Amino acids are frequently classified in three groups: the predominantly acidic amino acids, which comprise lysine, arginine, and histidine; and the neutral amino acids.

The equilibria of the $[OH^-]$ groups with the amino group and the $[H^+]$ with the carboxyl groups are, of course, affected by the pH of the solution. At a certain pH, the electrical charges within the amino acid molecule are at a state of equality, or are "isoelectric." At this point the molecule will not move in an electrical field. Since amino acids are hydrophilic substances, they are normally protected from coagulation by an adsorbed sheathing of water. At the isoelectric point this protection is lost, and the acids tend to flocculate. These isoelectric points are, for the acidic acids, about 3 pH, for the basic acids from 8-11 pH, and for the neutral acids about 6 pH.

"Amino acids react with Ca^{++} to form complex cations $(CaAn^+)$ (23,40) thus increasing the total amount of lime in the juices. Carbonic acid is readily bound by them in the form of carbamates (76) which are changed into carbonates relatively slowly. Amino acids act as very efficient buffers, and lower the pH of second carbonation juices, which means also a lowering of the $[CO_3^=]$ (9), and therefore a less efficient deliming. Dark colored products may be formed, through reaction of the amino groups with the CO of the reducing sugars according to Maillard's, or the browning reaction (59). They are melassigenic, either by themselves or through their cations." All are optically active excepting glycine and γ-aminobutyric acid, and the magnitudes of activity are strongly dependent on the pH of the solution, being capable of reversing the polarization with pH change. Therefore apparent purity determinations may provide erroneous information because of changes in optical activity, which will affect the estimation of sucrose losses.

TABLE 2-8 AMINO ACIDS FOUND IN BEET MATERIALS

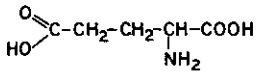
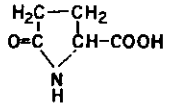
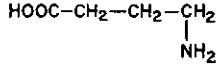
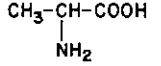
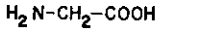
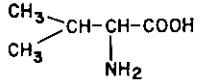
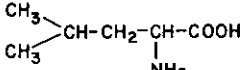
Name	Chemical Structure	Typical Amounts in Beet Molasses and Diffusion Juice	Solubility in Water	Dissociation Constants	Rf	Optical Activity $[\alpha]_D^{(t)}$	Other Common Forms
L-GLUTAMIC ACID glutaminic acid 2-aminopentanedioic acid α-aminoglutaric acid 1-aminopropane-1,3,-dicarboxylic acid		diff. juice [a] 137 mg/l molasses [b] 4.17%	25°, 8.64 g/l 100°, 140 g/l	pK_{a_1} = 4.07 pK_{a_2} = 9.47 pK_b = 11.90	acid, 0.40 anion, 0.35	22.4° +31.2° (c = 1.00 in 6N HCl)	D, DL
L-PYROGLUTAMIC ACID 2-pyrrolidone-5-carboxylic acid L-pyroglutamic acid 5-oxo-2-pyrrolidinecarboxylic acid glutimic acid; glutiminic acid α-amino glutaric acid lactam glutamic acid lactam			Soluble			20° —11.9° (c = 2 in wtr)	none
γ-AMINO BUTYRIC ACID γ-amino-*n*-butyric acid 4-amino-butannic acid piperidic acid		molasses 0.30%	freely soluble	25° $K_a = 3.7 \times 10^{-11}$ $K_b = 1.7 \times 10^{-10}$			none
L-ALANINE L-α-alanine L-α-aminopropionic acid L-2-aminopropanoic acid		diff. juice 38 mg/l molasses 0.28%	25°, 166.5 g/l 100°, 373.0 g/l	pK_a = 9.37 pK_b = 11.6	0.59	15° +14.7°	DL
GLYCINE aminoacetic acid aminoethanoic acid glycocoll		molasses 0.30%	25°, 25 g/l 100°, 67.2 g/l	pK_a = 9.78 pK_b = 11.65	0.42		
VALINE α-aminoisovaleric acid 2-amino-3-methylbutanoic acid		diff. juice 29 mg/l molasses 0.30%	25°, 88.5 g/l 65°, 102.4 g/l	25° pK_a = 9.7 pK_b = 11.7	0.75	23° +22.9° (c = 0.8 in 20% HCl)	L, DL
L-LEUCINE α-aminoisocaproic acid 2-amino-4-methyl-pentanoic acid		diff. juice 33 mg/l molasses 0.55% (incl. isoleucine)	25°, 24.26 g/l 100°, 56.38 g/l	25° pK_a = 9.7 pK_b = 11.7	0.79	26° +15.1° (6N HCl, 58 mols. HCl per mol. leucine)	DL

TABLE 2-8 continued

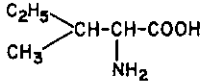
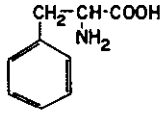
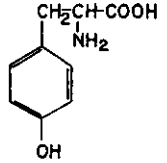
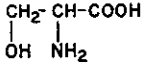
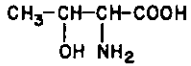
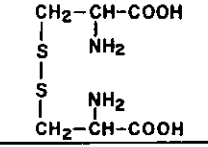
Name	Chemical Structure	Typical Amounts in Beet Molasses and Diffusion Juice	Solubility in Water	Dissociation Constants	Rf	Optical Activity $[\alpha]_D^{[t]}$	Other Common Forms
L-ISOLEUCINE α-amino-β-methylvaleric acid 2-amino-3-methylpentanoic acid		diff. juice 42 mg/l molasses vide supra	25°, 41.2 g/l 100°, 82.6 g/l	25° pK_a = 9.7 pK_b = 11.7		20° +11.29° (c = 8) (in water)	DL, *L-allo-*
L-PHENYLALANINE β-phenylalanine α-aminohydrocinnamic acid α-amino-β-phenylpropionic acid		molasses 0.09%	25°, 29.6 g/l 100°, 99.0 g/l	25° pK_a = 9.24 pK_b = 11.42	0.82 (H_2O-butanol)	20° —35.1° (c = 1.94) (in water)	D, DL
L-TYROSINE β-(p-hydroxyphenyl) alanine α-amino-p-hydroxy hydrocinnamic acid		molasses 0.21%	25°, 0.45 g/l 100°, 5.65 g/l	25° pK_a = 9.11 pK_{a_2} = 10.07 pK_b = 11.80	0.62	20° −8.64° (c = 4 in 63% HCl)	D, DL
L-SERINE β-hydroxyalanine 2-amino-3-hydroxypropanoic acid 1-amino-2-hydroxypropionic acid α-amino-β-hydroxypropionic acid		molasses 0.03%	25° ca. 250 g/l	25° pK_a = 9.15 pK_b = 11.79	0.43	20° —6.83° (1.5 g in 15 g aq. soln.)	DL
L-THREONINE α-amino-β-hydroxybutyric acid 2-amino-3-hydroxybutanoic acid		diff. juice 26 mg/l	freely sol.		0.51	26° —28.3° (c = 1.1)	DL
L-CYSTINE 3-3'-dithiobis(2-aminopropanoic acid) dicystein; β,β'-dithiodialanine α-diamino-β-dithiolactic acid β,β'-diamino-β,β'-dicarboxydiethyldisulfide bis (β-amino-β-carboxyethyl) disulfide		molasses 0.05%	25°, 0.112 g/l 100°, 1.142 g/l	pK_a = 8.00 pK_a = 10.25 pK_{b_1} = 11.95 pK_{b_2} = 12.96	0.16	20° −223.4° (c = 1.00 in N HCl)	D, DL, *meso-*

TABLE 2-8 continued

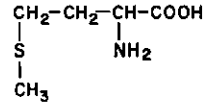
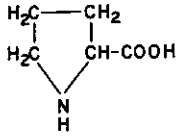
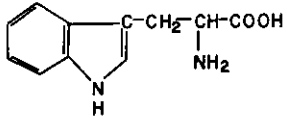
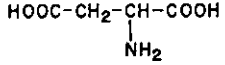
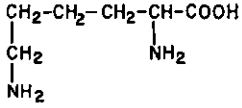
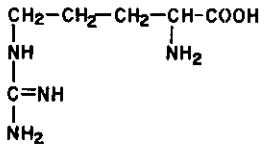
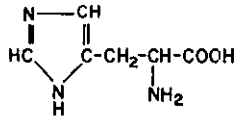
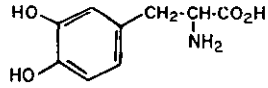
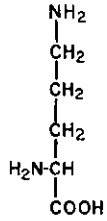
Name	Chemical Structure	Typical Amounts in Beet Molasses and Diffusion Juice	Solubility in Water	Dissociation Constants	Rf	Optical Activity $[\alpha]_D^{(t)}$	Other Common Forms
DL-METHIONINE α-amino-γ-methylmercaptobutyric acid 2-amino-4-methylthiobutanoic acid γ-methylthio-α-aminobutyric acid			25°, 33.81 g/l 100°, 176.0 g/l	25° pK_a = 9.21 pK_b = 11.72	0.77		L, D
L-PROLINE 2-pyrrolidinecarboxylic acid		molasses 0.11%	25°, 100 g wtr dissolves 162 g	pK_a = 10.60 pK_b = 12.0	0.85	23.4° −85.0° (c = 1)	DL
L-TRYPTOPHAN *1*-α-amino-3-indolepropionic acid 2-amino-3-indolylpropanoic acid *1*-β-3-indolylalanine *1* (−) tryptophan			25°, 11.4 g/l 100°, 49.9 g/l	pK_a = 9.39 pK_b = 11.62		23° −31.5° (c = 1)	none
L-ASPARTIC ACID aminosuccinic acid asparagic acid asparaginic acid		diff. juice 100 mg/l molasses 0.37%	20°, 1 g diss. in 222.2 ml wtr. 100°, in 18.6 ml wtr.	25° pK_{a_1} = 3.86 pK_{a_2} = 9.82 pK_b = 11.93	acid, 0.32 anion, 0.25	18° +4.7° (c = 1.3)	
L-LYSINE α-ε-diaminocaproic acid 2,6-diaminohexanoic acid		molasses 0.03%	freely sol.	pK_a = 10.53 pK_{b_1} = 5.05 pK_{b_2} = 11.82	acid, 0.71 cation, 0.53	20° +14.6° (c = 6.5)	DL

TABLE 2-8 continued

Name	Chemical Structure	Typical Amounts in Beet Molasses and Diffusion Juice	Solubility in Water	Dissociation Constants	Rf	Optical Activity $[\alpha]_D^{(t)}$	Other Common Forms
L-ARGININE l-amino-4-guanidovaleric acid		molasses 0.03%	21° satd. soln. contains 15%, w/w	pK_a = 12.48 pK_b = 4.96	acid, 0.76 cation, 0.60	20° +12.5° (c = 3.5)	
L-HISTIDINE α-amino-4 (or 5)-imidazole propionic acid glyoxaline-5-alanine		molasses 0.03%	25°, 41.9 g/l	pK_a = 9.18 pK_b = 7.90		20° −39.74° (c = 1.13)	DL
L-DOPA β-(3,4-dihydroxyphenyl)-L-alanine β-(3)-4-dihydroxyphenyl)-α-alanine 2-amino-3 (3,4-dihydroxyphenyl) propanoic acid		(c) present in leaves and roots	25° 165 mg/100 ml			20° −14.28°	DL, D
L-ORNITHINE α-δ-diaminovaleric acid 2,5-diaminopentanoic acid		(c) present in thin juice, leaves and roots	freely soluble in water	at 25° K_a = 1.74 × 10^{-11} K_{b_1} = 4.46 × 10^{-6} K_{b_2} = 8.70 × 10^{-13}	0.67 for the cation 0.47	25° +11.5° (c = 6.5)	DL

(a) see ref. (90) (b) see ref. (70) (c) see ref. (66a)

Most amino acids are fermentable by yeasts, and this takes place with the loss of one C atom as CO_2, the assimilation by the yeast of the nitrogen in the form of ammonia, and the yield of the appropriate alcohol. When beet juices are purposely fermented to produce ethyl alcohol, the chief undesirable constituents are isoamyl and isobutyl alcohols, which result from the fermentation of leucine and valine.

Of the individual amino acids, glutamic and aspartic are present in the largest quantities. The mother substance of glutamic acid in the beets, glutamine, gives rise to glutamic acid and pyroglutamic (pyrrolidone-carboxylic) acid when passing through the process. Glutamic acid is of interest because it is the only amino acid which tends to split off water. Glutamine will form pyroglutamic acid directly with the loss of ammonia, and with additional hydration will form glutamic acid. Glutamic acid is in equilibrium with pyroglutamic acid with appropriate loss or gain of water (Figure 2-24). In final molasses, most is in the form of pyroglutamic acid, and this, by treatment with strong acid or base, can be returned to the glutamic acid form.

Glutamic acid is of economic importance because of the value of its monosodium salt as a flavor enhancer.

The very important amino acid, Dopa (3,4 dihydroxyphenylalanine) has been found (66a) mainly in beet leaves, usually when there are some *Cercospora* leaf spot lesions, and the presence of Dopa may be related to leaf-spot resistance.

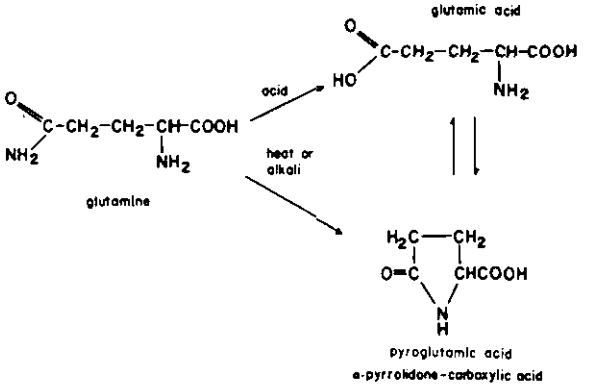


Figure 2-24. Relations between glutamine, glutamic acid, and pyroglutamic acid.

Amides, Ammonium Salts, and Nitrates

Amides

An amide is an organic acid in which the OH group of the carboxyl is replaced by an NH_2 group. In the sugarbeet, these are chiefly glutamine (L-glutamine, 2-aminoglutarimic acid, glutamic acid 5-amide) and asparagine (α-aminosuccinamic acid, altheine, asparamide, agedoite) the amides of glutamic and aspartic acids, and smaller amounts of oxamic acid, the amide of oxalic acid (see Figures 2-24 and 2-25). The amide-nitrogen in beets is about 0.02%, and is increased by a lack of water during the beet-growing period (33) and by high-nitrogen fertilizers. Oxamic acid has been present in beets at about 0.004%. Amides are effec-

tively neutral, as the NH_2 group loses much of its basic nature when located adjacent to the carboxyl group. Nevertheless, salts can be formed with strong acids. The amides are water soluble, and are not precipitated with lime. They are also not precipitated by basic lead acetate, and since they are optically active: glutamine $[\alpha]_D^{23} = +6.1°$; asparagine $[\alpha]_D^{20} = -5.42°$ and the activity can change or reverse with the pH, they constitute additional sources of error in the determination of sucrose by polarization.

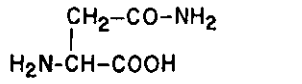


Figure 2-25. Structures of asparagine and oxamic acid.

Under factory processing conditions, they decompose under liming and especially at high tempertures to form soluble lime salts and evolve ammonia.

$$2\,R\text{-}CONH_2 + Ca^{++} + 2OH^- = 2R\text{-}COO^- + Ca^{++} + 2\,NH_3 \qquad (5)$$

In a vigorous defecation, this reaction can be essentially completed, but in most factories the reactions continue through the balance of the process, including pan boiling, with steady evolution of ammonia and lowering pH values, which may result in sucrose inversion and corrosion. "However, the loss of alkalinity may prove advantageous, by protecting evaporators from calcium carbonate scale formation (28), and in facilitating the boiling and crystallizing of low purity materials." The hydrolysis of oxamic acid yields, of course, scale-forming oxalic acid.

Ammonium Salts and Nitrates

There are only very small contributions of ammonium salts from sugarbeets. However, ammonia is very important in the process, being formed, as described, in large quantities from the hydrolysis of amides and the decomposition of glutamine, and it is "chiefly responsible for the loss of alkalinity in the factory. The NH_4^+ ion is an acid ($NH_4^+ = NH_3 + H^+$, pK = 9.5) and therefore ammonium salts may invert even alkaline, warm sucrose solutions, and cause corrosion of evaporators (27)."

Nitrates are normally present in very small quantities in beet materials. Their chief importance is in their reduction by certain thermophilic bacteria to nitrites, and the reaction of these nitrites with bisulfites following the sulfitation of thin juices, to form the dipotassium salt of imidodisulfonic acid, $HN(SO_3K)_2$, which can precipitate with and raise the ash of the product sugar (18,72). However, under certain conditions, significant concentrations of nitrates can be found in molasses.

Proteins

Proteins are made of amino acid units joined together by the peptide linkage (–CO–NH–), and this is sometimes called the *Biuret group,* since it gives a violet color in the Biuret test, when treated with NaOH and $CuSO_4$ (Figure 2-26).
Only a small portion of the proteinaceous material is extracted, either in solution or suspension, into the diffusion juice. The relative amounts of the different amino

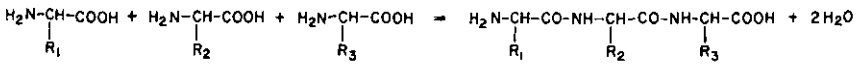


Figure 2-26. Linkage of amino acids to form proteins.

acids in the beet structure are not the same as found in the cell sap or in the diffusion juices.

"The colloidal solutions formed by proteins are, in the native state, rather stable. Their denaturization; that is, the loss of their regular three-dimensional structure, may be caused by extreme conditions of heat, pH, and mechanical treatment. Solutions of such proteins are relatively unstable, and can precipitate."

"Proteins are polyelectrolytes, having a large number of acidic and basic groups of very different pK. Their state of ionization depends on the pH of the solution. They have, like amino acids, a certain region of pH where the total of their positive and negative charges is zero, and in this isoelectric state they precipitate, if previously denatured." There are more acidic amino acids in the protein structures than there are basic. Thus, beet protein must be regarded as acidic, and consequently "for sugar juice proteins, this isoelectric region is in the neighborhood of pH 3.2 (93). At higher pH the ionization of their basic groups is diminished ($-NH_3^+ = -NH_2 + H^+$), and they are predominantly negatively charged and able to form salts, or proteinates with metallic cations. Polyvalent cations, even bivalent ones, especially of high molecular weight, are then able to precipitate proteins. Apparently they form undissociated salts, thus diminishing the number of charged, hydrophilic groups, which help the proteins to stay in solution. If, however, by further decreasing the pH, the new, weakly acidic groups become ionized and therefore impart new electrical charges to the protein molecule, either precipitation will not occur at all, or precipitates already formed will be repeptized and dissolve.

"The colloidal micelles of proteins of high molecular weight behave as ion exchangers. They are easily attacked on heating, and under extreme conditions of pH lead to products more highly peptized, and therefore in more stable solution. Eventually, they cannot be precipitated at all (Figure 2-27).

"It has been shown (31,32) that two different methods for the determination of protein nitrogen have given results which are in agreement in the analysis of diffusion juice samples, but are increasingly different when the samples are taken through the various stages of processing, up to the molasses, at which point the difference is the greatest. Splitting the polypeptide chain, through acidic, basic, or enzymatic action, liberates new acidic and basic groups, with the formation of NH_3," and these groups proceed through to the molasses.

"Proteins of the diffusion juice, which have been precipitated by lime, augment the amount of colloidal, slimy precipitate, which settles very slowly, and is rather difficult to filter. Proteins are able to depress the activity of the calcium ion, and to bind carbonic acid in the form of carbamates, thus increasing the apparent

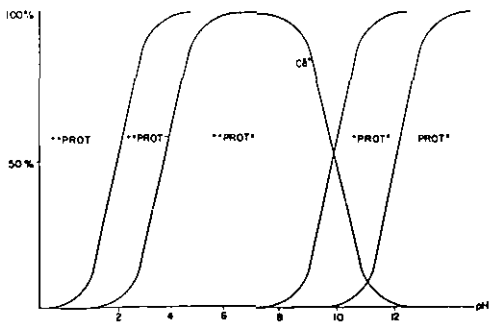


Figure 2-27. Ionization of proteins (*ovalbumin, Ettisch-Schulz*).

solubility product of calcium carbonate and other sparingly soluble lime salts (22), and even facilitating the formation of supersaturated, but relatively stable solutions of these salts."

It has been shown (94), that as beets deteriorate, the amount of protein that can be precipitated by lime decreases: i.e., protein destruction occurs in such beets before processing.

"Proteins are strongly surface active, give rise to foams, and may affect the velocity of crystallization of sucrose by adsorption on the crystal faces. On the other hand, they may assist in the agglomeration of calcium carbonate particles in the carbonation sludges, and by means of ion exchange (29) even take part in the formation of natural alkalinity, and thus in the deliming of second corbonation juices."

Nitrogenous Bases, Purines, and Pyrimidines

Nitrogenous Bases

The nitrogenous bases all contain the trimethylammonium group, $-\tilde{N}(CH_3)_3$, which gives them their basic characteristics. "They act as buffers and may simulate natural alkalinity in second carbonation, depressing the ionization of carbonic acid, and hence enhancing the deliming of juices. Again, they are able to bind carbonic acid as carbamates. When volatile, like trimethylamine, $N(CH_3)_3$, they may cause loss of alkalinity (26). They are probably melassigenic." The three most prominent nitrogenous bases are betaine, choline, and allantoin.

Betaine, which is the largest component of the "harmful" nitrogen, is present in beets as about 0.1%. The metabolic function of betaine in the plant is unknown (98) (Figure 2-28). It is noteworthy that betaine is present in much larger amounts in the members of the beet family, the *chenopodiacae*, than in any other plants. There is also more betaine in beets low in sugar (85). It is remarkable in its resistance to destruction under extreme chemical conditions, and it passes unchanged through the process as a melassigenic agent. While betaine is assimilated in the digestive tracts of ruminants, it is mostly excreted, unaltered. Unfortunately betaine has defied all efforts to find any large-scale economic use. It exists as an internal quaternary ammonium salt.

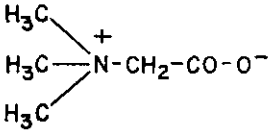


Figure 2-28. Betaine.

Choline, a quaternary ammonium ion related to betaine, exists in the beet only as part of the cell-wall material, lecithin, mentioned under "lipids." However, lecithin decomposes somewhat in diffusion and extensively in defecation to free choline, and fatty acids. Choline itself decomposes to a small degree in defecation to give trimethylamine and $CH_2OH\text{-}CH_2OH$, but the greater part goes through to the molasses. The conversion of allantoin to allantoic acid (Figure 2-29) occurring both during beet juice purification and in the evaporation of clarified juices, was first described in 1960 (16). Comparison of analytical data obtained on molasses produced over different campaigns seems to agree with the view (15) that the allantoin content of beets may increase during periods of prolonged drought.

Again, allantoin can be derived from urea (Figure 2-30), and this is important since on liming, this reaction is reversed and some urea and glyoxylic acid are pro-

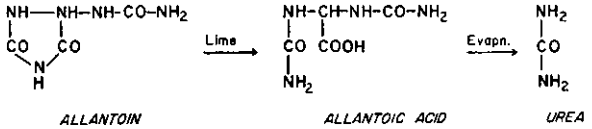


Figure 2-29. Conversion of allantoin to allantoic acid and to urea, in course of factory processing *(Carruthers et al.)* (16).

duced. The glyoxylic acid in itself proceeds through internal oxidation-reduction to form oxalic and glycolic acids, while the urea is degraded to CO_2 and NH_3. Again, most of the allantoin goes to the molasses unchanged. Allantoin is found in sugarbeets in the amount of about 0.015%.

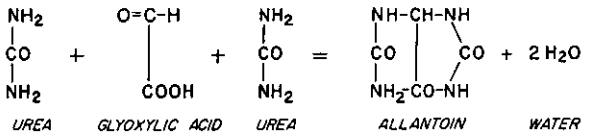


Figure 2-30. Formation of allantoin from urea and glyoxylic acid.

Purines

The purine bases are components of nucleic acids and complex proteins of the cell nuclei. They are present in very small amounts, together about 1-2% of the nitrogenous compounds. They are all sparingly soluble, and weakly alkaline in solution. The amino purines found are adenine, guanine, xanthine, and hypoxanthine.

Pyrimidines

Grouped with the pyrimidines, uracil, and cytosine, are the ribosides, guanosine, uridine, and cytidine (the D-ribosides of guanine, uracil, and cytosine), and the desoxyriboside thymidine. All are vital constituents of plant tissue.

As far as is known, none of the above are removed in juice purification, and pass through to the molasses.

Stark and co-workers (91) first isolated several of these large compounds, and Table 2-9 is a summary of almost all findings in the literature, including those of Janacek (53) and Vavruch (95).

Tables 2-9. Purines, Pyrimidines, and Nucleosides in Beet Materials (mg/l)

	European Beets		California Beets	
		(Vavruch)	(Stark, *et al*)	
Compound	(Janacek)	Pressed Juice	Pressed Juice	Molasses
Adenine	28	1	40	450
Guanine	80	3	25	300
Hypoxanthine	52	4	35	30
Xanthine	50	5	?	
7-Methylxanthine	28	6	?	
Uracil		probably	60	21
Cytosine			5	
Guanosine		2	?	
Uridine			135	630
Thymidine			20	45
Cytidine			10	

Analytical data differing somewhat from the findings of Stark, *et al.*, for the distribution of purines, pyrimidines, and nucleosides have also been reported (15).

Table 2-10 lists important chemical and physical data of the nitrogenous bases, purines, and pyrimidines.

Coloring Materials

The colored substances which develop during the manufacturing process are of three general types: melanins, a general term for blackish pigments formed in vegetable tissues; melanoidins, or the products of the Maillard reaction, especially prominent in evaporator juices; and caramels.

Melanins

The reactions producing dark colors in the diffusion juice are very complex (44). Primarily enzymatic oxidation of tyrosine by the enzyme, tyrosinase, may be responsible for the major portion of the color. The first stage of the oxidation is said to be the introduction of an OH into the No. 4 position of the ring structure to form "Dopa" 3,4-dihydroxyphenylalanine. Further oxidation leads to O-quinoid intermediates, and finally to indole structures (Figure 2-31).

It is worthy of note that these colored compounds are not dissolved, but are present in suspension, and may be filtered from the clear, yellowish juice. The enzyme acts best at pH values between 6 and 8, and it is inactivated when heated. The coloring matter is mostly formed in the interval between the slicing of the beet and the heating of the juice in the diffuser. All of this coloring matter is removed in juice purification. The color formation can be repressed by the presence of sulfur dioxide.

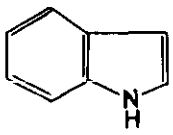


Figure 2-31. Indole ring.

Melanoidins

This is a general term for coloring matter formed by the Maillard reaction, between reducing sugars and amino compounds. This type of reaction proceeds throughout the sugar end of the factory, in the evaporators and in the crystallizing procedures. The reactions have been extensively studied and are, again, most complex (14). Excellent reviews of this subject were given by Danehy and Pigman (21), and Hodge (46). The browning reaction can be effectively inhibited by the presence of a small concentration of sulfur dioxide.

Caramels

These coloring materials are formed from the destructive heating of sucrose solutions, and in a properly conducted factory they do not occur to an appreciable extent. The chemical nature of the coloring substances formed is largely unknown.

TABLE 2-10 NITROGENOUS BASES, PURINES AND PYRIMIDINES

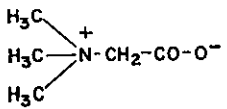
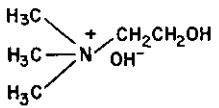
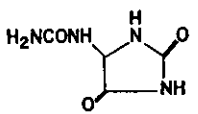
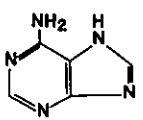
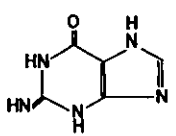
Name	Chemical Structure	Class	Solubility in Water	Optical Activity Solution pH Dissociation Constants
BETAINE trimethylglycine; lycine; oxyneurine; trimethylglycocoll (carboxymethyl)-trimethyl-ammonium hydroxide anhydride		Nitrogenous Base	ca. 25°, 160 g/100 ml	Sat. Soln, 8.0 pH
CHOLINE β-(hydroxyethyl) trimethylammonium hydroxide (trimethyl-2-hydroxyethyl ammonium hydroxide sincaline; bilineurine; amanitine; gossypine vidine; bursine; luridine; fagine		Nitrogenous Base	Sol.	
ALLANTOIN 5-ureidohydantoin glyoxyldiüreide cordianine		Nitrogenous Base	ca. 25°, 0.53 g/100 ml Sol. in hot water	hot water, 5.5 pH
ADENINE 6-aminopurine vitamin B_4		Amino purine	ca. 25°, 1 g diss. in 2000 ml 100°, 1 g diss. in 40 ml	neutral
GUANINE 2-aminohypoxanthine 6-oxy-2-amino purine		Amino purine	ins. cold Sol., acids and alks.	40° $K_b = 6.09 \times 10^{-4}$ $K_a = 1.19 \times 10^{-10}$

TABLE 2-10 continued

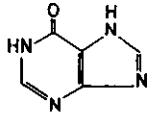
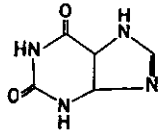
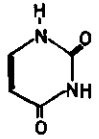
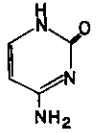
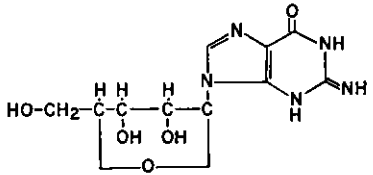
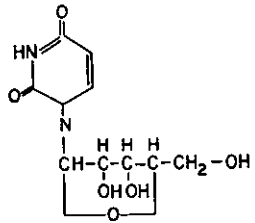
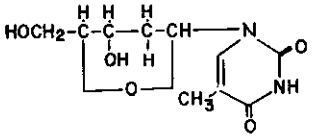
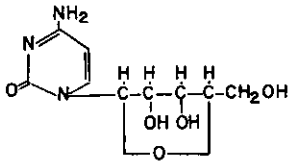
Name	Chemical Structure	Class	Solubility in Water	Optical Activity Solution pH Dissociation Constants
HYPOXANTHINE 6 (1H)-purinone sarcine sarkin 6-oxypurine		Amino purine	ca. 25° 1 g diss. in 1400 g 100°, 1 g diss. in 70 g Sol. in acids, alks.	25° pK_b = 8.7
XANTHINE 2,6 (1,3)-purinedione 2,6-dioxopurine		Amino purine	16°, 1 g diss. in 14.5 l 100°, 1 g diss. in 1.4 l freely sol., acids, alks.	40°, $K_b = 6.09 \times 10^{-14}$ $K_a = 1.19 \times 10^{-10}$
URACIL 2,4 (1,3)-pyrimidinedione 2,4-dioxopyrimidine		Pyrimidine	25°, 0.36 g in 100 g	pK = 9.45
CYTOSINE 4-amino-2 (1)-pyrimidone 4-amino-2-oxo-1,2-dihydropyrimidine 2-oxy-6-aminopyrimidine		Pyrimidine	1 g diss. in 130 ml	$pK_1 = 4.60$ $pK_2 = 12.16$

TABLE 2-10 continued

GUANOSINE guanine riboside vernine		Riboside	18°, 1 g in 1320 ml 100°, 1 g in 33 ml Sol. in acids, bases	$[\alpha]_D^{20} = -60.5°$ (p = 5 in 0.1N NaOH)
URIDINE Uracil riboside		Riboside	Sol.	$[\alpha]_D^{20} = +4°$ (c = 2)
THYMIDINE thymine-2-desoxyriboside		Riboside	Sol.	$[\alpha]_D^{25} = +30.6°$ (c = 1.029)
CYTIDINE cytosine riboside 1-β-D-ribofuranosylcytosine		Riboside	freely sol.	$[\alpha]_D^{25} = +31°$ (c = 0.7)

INORGANIC COMPOUNDS

The disposition of the mineral matter in beets is unknown, but surely most of it is combined with organic compounds.

Table 2-11 lists ash analyses from Silin (78).

Table 2-11 Ash Analysis of Sugar Beets (Russian).

Substance	% of beet	% of total ash
K_2O	0.25	42
Na_2O	0.25	8
CaO	0.08	13
MgO	0.08	13
P_2O_5	0.09	15
SO_3	0.02	3
Al_2O_3, Fe_2O_3	0.01	2
SiO_2	0.01	2
Cl	0.01	2
	0.60	100

(Note: the metals are not present in the beets as oxides. They were converted to this form in the analysis).

Various spectrographic analyses of beet brei ash have shown traces of barium, lead, boron, iron, cobalt, copper, magnesium, molybdenum, nickel, ruthenium, silver, selenium, silicon, strontium, thallium, and zinc. The physiological significance of many of these elements has not been established.

Boron is known to have connection with certain disease conditions, and magnesium deficiency has been associated with a yellowing of the beet leaves. The ash content of beet-factory process juices is, relative to cane processing, very small, and in general there is not nearly the concern over ash, other than residues of the lime reagent added in processing.

Sodium and potassium cations represent two of the nonsucrose components present in appreciable quantity, which are not removable in normal processing and lead to increased molasses production and reduced sucrose extraction. Most formulas which predict sucrose extraction are based on the ash component, and perhaps the amino nitrogen and invert contents. It is common practice in Europe and more recently in at least one U.S. beet-growing area to determine these impurities in the tare laboratory. The grower's payment is then based on the calculated extractable sucrose.

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Chapter III

THE TARE LABORATORY

Payment for Beets*

In North America sugarbeets are grown by farmers and sold to the processors. Payment is made by three general methods, differing extensively in details. 1) In most of the United States, the grower is essentially paid for the sugar in the beets he delivers. 2) At the time of writing, growers in areas including parts of Northern Utah, Central and Eastern Idaho, the Red River Valley in Minnesota, and a small part of Ohio are paid on the net weight of beets they deliver, which are assigned the average sugar content of the cossettes sliced. 3) In Canada the grower shares in the sugar manufacturers' profits, in proportion to the net weight of clean beets he delivers, without consideration of the sugar content of the beets.

The *net weight* of beets delivered by the grower must be determined for the application of all three methods. By this is meant the weight of beets, free of dirt and trash, with the crowns trimmed in a specified manner. The crown-trimming requirement arises from the much lower sugar content of the crowns.

Net Weight

Beets are delivered by the grower to a company receiving station, which may be at a factory, or at some distance. In either case, after receipt, the beets may then be piled for long-term storage. When the receiving station is not at the factory, eventually the beets are reloaded into railcars, transport trucks, or, in one case, barges, for conveying to the factory.

At the receiving station the grower's loaded truck is weighed, and the beets are discharged into a hopper. The empty truck is then positioned under the elevated screens over which the beets are passed, and receives the dirt and trash separated. The truck is then weighed a second time with the dirt and trash, and the difference between the two weights is the first net weight. In some areas the screened dirt and trash are not returned to the grower's truck, but are separately weighed by a weighing hopper or belt. In such cases the *first net weight* is obtained by subtracting the weight of the separated *tare material* from the gross difference in the weights of the loaded and empty truck.

Sampling the Beets

The aim of sampling is to ensure that each sample taken should accurately represent the load in tare material and sugar content. This can only be approximated, since beets from the same field and even from the same row vary greatly in physical characteristics and sugar percentage, as shown by the data in Tables 3-1 and 3-2.

The probability that a single sample of beets weighing perhaps 30 lbs will accurately represent a load of from 7 to 20 tons of beets is very small. The most that can be expected is that the average of a large number of loads will yield statistically satisfactory results.

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*by R. E. Hallbeck

Table 3-1 Variation in Size and Sugar Content of 50 Beets Growing Consecutively in a Row. U. S. 33. Woodland, California, 1948

S = percent sugar W = clean weight of beet, grams

Location 1				Location 2			
S	W	S	W	S	W	S	W
11.8	401	13.5	745	10.1	232	9.0	574
13.1	284	11.9	711	12.4	997	14.0	639
9.2	1052	16.7	705	10.8	263	13.2	2315
8.7	142	9.6	590	11.3	452	15.0	313
12.9	724	15.1	639	6.3	388	13.8	417
13.7	854	14.6	257	15.7	1218	15.1	709
9.6	129	10.4	688	14.3	472	14.9	360
13.7	1816	13.4	158	15.0	140	12.6	1043
8.5	298	14.6	724	12.5	520	14.1	444
15.7	830	10.5	356	11.8	718	11.4	461
14.1	662	8.6	417	11.6	1427	9.4	465
11.9	369	15.2	537	12.2	296	12.4	823
16.7	377	11.1	591	7.5	860	15.0	757
7.4	151	14.5	307	13.4	862	9.5	626
10.0	671	12.1	717	14.7	346	12.9	1047
4.4	331	14.9	543	14.2	1392	13.4	896
13.2	1376	15.0	745	14.0	272	10.6	301
13.8	381	12.1	212	15.1	872	6.5	173
9.1	492	12.6	653	6.5	1285	11.0	165
11.9	276	13.0	382	8.7	666	11.9	1160
12.8	694	14.1	624	11.0	1587	11.8	270
15.3	837	14.4	449	13.0	650	12.6	1420
12.6	169	13.1	320	9.2	430	9.5	326
16.1	1438	13.3	194	7.0	253	12.2	1382
17.2	746	15.0	660	13.2	700	8.2	576

Table 3-2 Variation in the Average Sugar Content of 48 Beet Samples Selected from the Same Load. Each Sample Contained 7 Beets and were Selected each to Contain 1 Large Beet, 4 Medium Beets and 2 Small Beets

S = percent sugar W = weight of clean beets, lbs.

S	W	S	W	S	W	S	W
15.3	29.0	15.3	22.1	14.8	30.6	15.6	23.7
14.4	26.9	16.1	21.8	15.7	29.5	15.4	23.4
17.3	25.0	15.7	21.7	14.7	25.5	16.0	22.5
15.3	24.7	12.8	21.5	14.2	25.3	14.3	21.6
14.1	24.6	16.0	21.5	14.5	24.7	15.0	21.6
15.7	23.9	13.0	21.1	15.1	24.6	16.4	21.5
15.6	24.0	16.2	20.0	13.7	24.2	14.8	21.1
15.5	24.4	13.2	20.0	13.5	24.2	15.2	21.3
15.3	23.2	15.2	19.7	12.1	23.9	15.8	21.0
15.2	23.5	14.7	19.7	14.5	24.3	14.5	20.6
15.2	22.8	15.4	19.3	15.5	23.3	16.5	20.7
15.8	22.5	17.1	18.3	12.4	23.3	15.0	20.4

Good Sampling Practices

1) All errors, particularly biased ones, should be eliminated as far as possible. Random errors, which are hard to remove, are of secondary importance since they will average out in a large number of samples.

2) As much dirt and trash (weeds and beet leaves) as possible should be removed and accounted for before the sample is taken. This requires good field-topping and cleaning, as well as efficient removal of tare material at the beet receiv-

ing station. If the amount of dirt and trash remaining in the beets is small, a relatively large sampling error will have a correspondingly smaller effect.

3) The sampling equipment should be physically located so that a representative sample of beets and tare material can be taken.

4) The timing of the sampling during the discharge of the beets should be specified. Preferably it should be carried out at the half-way point. If the load to be sampled is a two-part unit, such as a truck and trailer, and only one sample is to be taken, sampling should alternate from the front to the rear portion, from load to load.

5) The samples should be as large as is economically and physically practicable, and should be uniformly sized.

6) As many loads as possible should be sampled.

7) The specified sampling procedure should be rigorously followed, with sufficient supervision and inspection to ensure it.

Samplers

The usual sampler consists of a pan set in a cradle, mounted on an arm so that it can be passed through a stream of falling beets. The pan must pass quickly through the beet stream, remaining just long enough to be filled, and not long enough to be overfilled. In the case of overfill, dirt from the beets which bounce off will remain in the pan, and give a falsely high tare content.

Since beets tend to bounce off the apron at the end of the dirt-removing screens, and the dense dirt tends to flow down close to the apron, under the beets, it is important that the sampling pan pass through the whole stream. If this is not the case, the amount of dirt in the sample will be too low. Once caught in the pan, no beet should be discarded, since this tends to increase the ratio of dirt to beets.

A newer type of sampler uses a pan which is first passed inverted through the beet stream. It is then restored to its upright position and returned through the beet stream, collecting the sample. By inverting the filled pan over a funnel, the sample is discharged into a sample bag.

In areas where individual sugar testing is not done, the beet samples are weighed at the receiving station. The loose dirt and trash are first removed, followed by removal of the dirt adhering to the beets by brushing or scraping, and finally the beets are decrowned in accordance with the specifications of that particular area. The cleaned *tared* sample is again weighed and the correction applied to the gross weight, or to the first net weight, to give the *second net weight* of the load. This is the weight of *beets purchased* used in calculating the grower's payment.

Analysis of Sugar Content*

It is outside the scope of this book to detail methods of analysis. Nevertheless, the procedure of estimating sucrose content of 15 to 80 lb beet samples, in numbers up to 6000 samples a day in a single tare laboratory, represents a unique analytical procedure, which is believed to be without parallel in other industries. The Sachs-le Docte procedure (5) is universally used for this purpose, although the Pellet analysis is sometimes made for check purposes. Since the taring procedures are also done in sugar-determining laboratories, they are included in the following discussion.

*by R. A. McGinnis

Preparatory Steps and Sachs-le Docte Procedure

1. Sample preparation. 1.1. Determination of sample gross weight. 1.2. Removal of loose dirt and trash. 1.3. Trimming leaves and crowns. 1.4. Beet cleaning. Alternatives: 1.4.1. Brushing or scraping the beets. 1.4.2.1. Washing the beets. 1.4.2.2. Drying the washed beets. 1.5. Weighing the cleaned beet sample. 1.6.1. Securing the beet brei. 1.6.2. Mixing the brei. 1.6.3. Sampling the brei.

2. The Sachs-le Docte analysis. 2.1. Weigh out a normal weight or 26 g of brei into a cup, and add a volume of lead solution, sufficient to give a total liquid-phase volume, which includes the liquid in the brei and that added, of 200 ml. The volume of solution to be added varies with the volume of the beet marc, and is set by agreement in different areas at values between 177 and 179.1 ml. At least the following alternatives are used: 2.1.1. To the weighed sample of brei, add the indicated volume of water plus a measure of dry lead acetate powder; 2.1.2. Put approximately 26 g of brei on the pan of a proportional balance, and mix with the correctly proportioned quantity of lead solution delivered by the balance; or 2.1.3. Put a quantity of brei on the pan of a proportional balance, add a proportional volume of water disbursed by the balance, mix, and finally add a constant volume of lead solution shortly before completion of mixing. The sum of the volumes of water metered out by the balance, and of the lead solution, is the correctly proportioned liquid volume for the weight of brei.

2.2. Digestion. Alternatives: 2.2.1. Cold digestion (6). At room temperature the mix is blended or macerated with a high-speed rotor for from one to several minutes (9,11).

2.2.2. Hot digestion (7). The mix is held at 80 °C in closed metal capsules for about 30 minutes, and then is cooled for a similar length of time to 20 °C or standard temperature.

2.3. Filtration. 2.3.1. The mix is poured into a filter paper or porous filter capsule, and filtered. Options: 2.3.1.1. A filter aid may be added before filtration. 2.3.1.2. The first few ml of filtrate, if turbid, may be returned to the filter.

2.4. Polarization. 2.4.1. The clear filtrate is polarized in a sacharimeter. 2.4.2. The percent sugar is read and recorded. Options: 2.4.2.1. The filtrate may be stirred before polarization. 2.4.2.2. A few drops of glacial acetic acid may be stirred into the filtrate.

3. Calculations. 3.1. Using the primary data of the gross weight of the beets in the load, and the first net weight, the gross weight of the sample and its clean weight, the *second net weight* of the load is calculated (usually not in the tare laboratory), and multiplied by the percent sugar in the clean beet sample to give the amount of sugar in the clean load of beets. 3.2. The calculated data are recorded with the load and contract number, or with other means of designating the grower and delivery load concerned.

Alternative Aluminum Clarification Procedure*

To eliminate the risks associated with handling and disposing of toxic lead compounds, many tare laboratories now substitute aluminum salts for lead salts as extract clarificants. Analytical results are identical (11). Aluminum does not adequately clarify factory juices or other highly-colored extracts; for these lead or other special-purpose clarificants still are used.

4. Sample preparation is unchanged from section 1. above.

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*By S.S. Martin

5. Aluminum clarification. 5.1. Weigh out 26.0 g brei and add aluminum clarifying solution to give a total liquid phase volume of 200.0 ml. The clarifying solution should contain 0.2-0.5 g aluminum per liter and may be prepared from aluminum chloride or aluminum sulfate. A proportioning balance usually is employed as described in 2.1.2. 5.2 Blend the mixture at room temperature for one-half to several minutes. (Hot digestion is avoided with aluminum clarification because of the risk of sucrose inversion in the acidic solution.) 5.3. Filter the mixture through a moderately retentive filter paper, with or without filter aid.

6. Polarization. 6.1. Determine polarization to the filtrate. A short-pathlength automatic polarimeter is particularly suitable, although longer pathlengths and manual instruments may be used.

Tare Laboratory Equipment

Sample Scales. Steps 1.1 and 1.5 require sensitive scales, which both indicate and record the weights of the sample on a ticket or tape.

Beet Toppers. In many laboratories the top trimming is done by hand with knives. Other laboratories use motor-driven toppers, which top the beets with either a flat or a conically-shaped cut.

Beet Washers. In many laboratories the dirt is removed from the beets by hand, by brushing or scraping. In others the beets are washed in mechanical washers. The Great Western batch washer (Figure 3-1) is widely used. This unit washes and discharges one sample at a time. Automated and some nonautomated laboratories use continuous washers, which wash a number of samples at a time, keeping them separate. The Ogden and the Carver-Ogden (see Figure 3-2) are examples of this type.

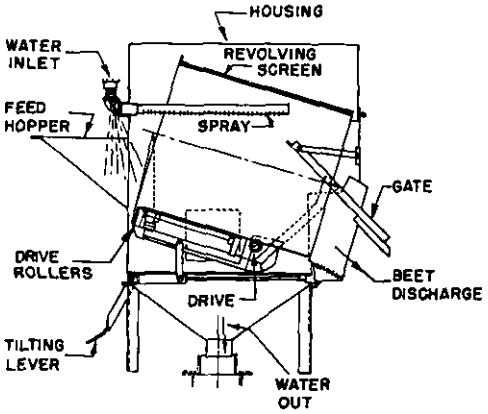

Figure 3-1. The Great Western Beet Washer.

Beet Driers. Washed beets are normally dried in tunnel driers, through which the washed samples pass continuously in open-mesh baskets. Drying is done with very large volumes of gas-heated air. If the beets are over-dried, the weights will be low, and the determined sugar content correspondingly high. If the beets are not dried sufficiently, the reverse occurs. In proper drying, the beets emerging from the drier will be dry to the touch, but cool.

Obtaining the Brei. The beet brei, or "sawdust," is made through the use of rasps or saws. Very finely divided brei is made by the Keil-Dolle (3) or Ninegar rasps (4). The use of multiple circular gang saws permits more rapid throughput, as with the Bensel (1), the Spreckels (12) or the Weibull saw.

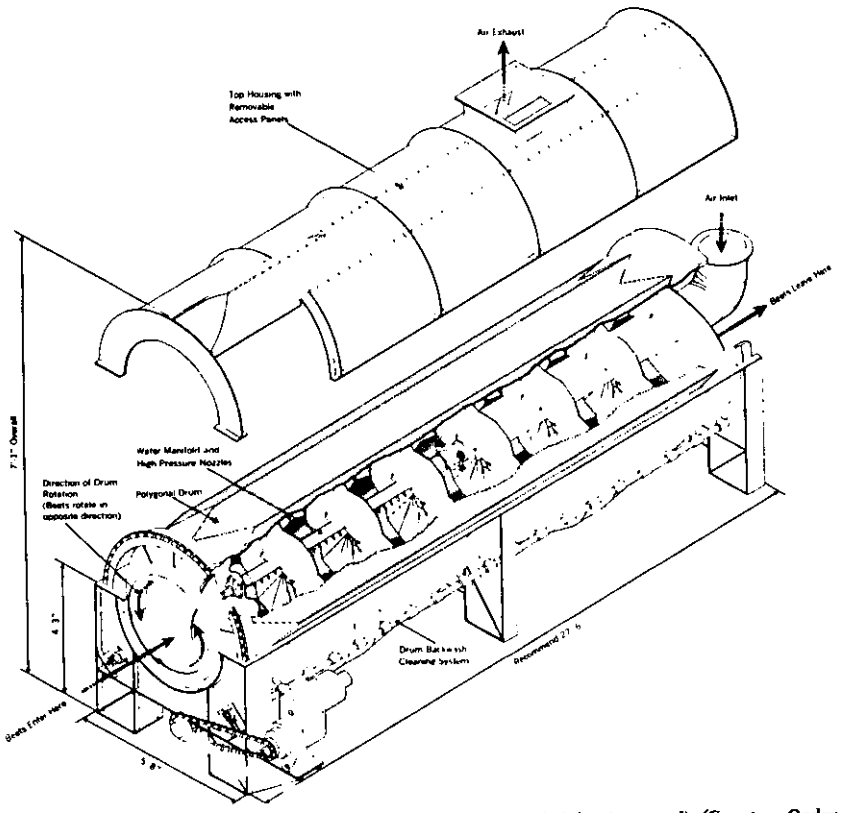

Figure 3-2. Carver-Ogden continuous beet sample washer and drier (patented). *(Courtesy Ogden Iron Works, Ogden, Utah)*

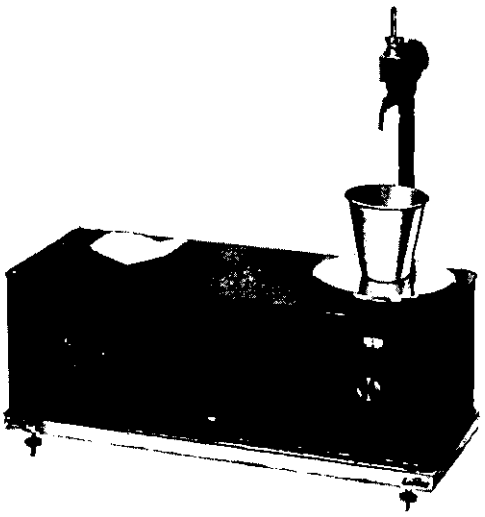

Figure 3-3. Reyers automatic dispenser balance. *(Manufactured by Reyers and Zoom, N.V., Holland. Courtesy, LaPine Scientific Company, Chicago)*

Mixing the Brei. Step 1.6.1 must be properly carried out, as otherwise the precision and accuracy of analysis are seriously damaged. Failure to adequately mix the brei is probably the one most important cause of low precision in modern tare laboratories, when either the rasp or saws are used.

Taking the Brei Sample. In most laboratories this is done by hand, by weighing out a 26 g portion on a balance. There is at least one mechanical device which takes approximately the correct weight of brei by suction with a piston device.

Adding the Lead Solution. The lead clarifying solution is generally made up using three parts of lead subacetate to one part of yellow litharge of lead. The solution concentration is measured with a Brix spindle, and is frequently used at about 6 °Brix. This solution will hereafter be referred to as the "lead solution." In many laboratories the addition is done by adding to exactly 26 g of pulp, the specified volume of solution with an automatic pipette. Proportioning balances have come into considerable use during the last decade (Figure 3-3).

Typical of these balances is the Reyers balance. The operator places 26 g ± 2 g onto the left-hand pan, and the balance automatically dispenses the correct volume (through its weight) of the lead solution into the beaker on the right-hand pan. For accurate results, the lead solution must be kept exactly at the right concentration and temperature, and the head in the gravity tank feeding the balance must be kept constant. The precision and accuracy of the Reyers balance is ample for its purpose. Another good proportional balance is manufactured by Nils Weibull AB.

Digestion. In the hot procedure, the digestion is carried out by standing the metal capsules containing the samples in hot baths, and similarly in cooling. In the cold analysis it is customary to mix the brei-lead solution mixture with high speed blenders or mixers for a short period. In automated laboratories, this is readily automated by the use of traveling belts or turntables equipped with mixers.

Filtration. This normally manual procedure is likewise readily automated.

Polarization. The manually operated and balanced saccharimeter is most widely used, and is always used for ultimate reference for accuracy, if not for precision.

The Koontz Printing Polariscope (10) is in use in almost all Great Western

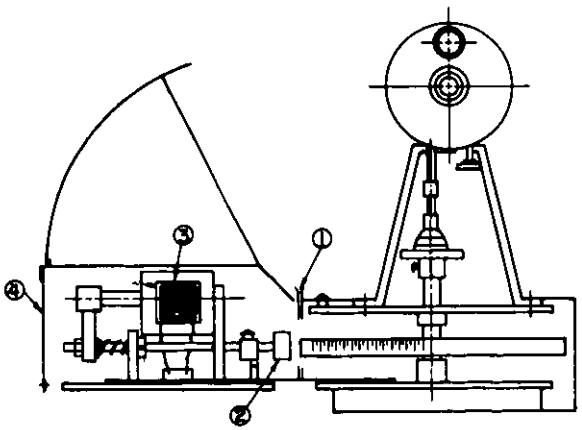

Figure 3-4. The Koontz printing polariscope. 1) slot for ticket, 2) plunger, 3) solenoid, 4) housing. ***(Courtesy, The Great Western Sugar Company)***

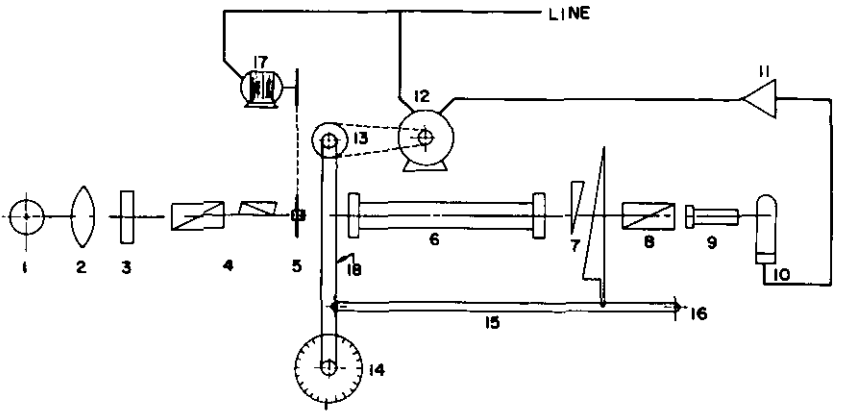

Figure 3-5. Spreckels-Bernhardt automatic saccharimeter. This instrument combines a Bausch and Lomb visual saccharimeter with additional components for automatic balancing of the bipartite photometric field, and print-out of the percent sugar. Light from lamp 1 passes through lens 2, filter 3, Lippich polarizer 4, scanner 5, sample tube 6, quartz compensator 7, analyzer 8, and telescope 9 to phototube 10. Scanner 5 is a semi-circular aperture which revolves at line frequency, driven by synchronous motor 17. When the photometric field is unbalanced, the intensity of the light reaching the phototube fluctuates at line frequency. The electrical output from the phototube, after amplification by 11, is applied to the phase-sensitive motor 12. The speed and direction of rotation of this motor is a function of the degree and direction of unbalance of the photometric field. Pulley 13 is driven by the motor and coupled through cable 18 to printer disc 14 and lever 15. The lever is supported at pivot 16, and coupled to quartz compensator 7. Movement of the printer mechanism ceases when the photometric field is balanced. Manual actuation of the printer mechanism imprints the °S of the sample on a tare ticket. *(Courtesy, Spreckels Sugar Company)*

factories. This device saves the time used in reading the vernier, which is considerable (Figure 3-4).

The first fully automatic balancing and printing unit was the Spreckels-Bernhardt (2,2a), which has been in use in all Spreckels Sugar Company tare laboratories since 1947 (Figure 3-6), as well as in the Holly laboratories at Hereford, Texas, and Worland, Wyoming.

DFS System

In the state of Michigan, an entirely different method of determining sugar in beet samples is used, the DFS system (Dexter, Frakes, Snyder). This is used for the factories of the Michigan and Monitor Sugar Companies. The principles and details are complex, and reference is made to the publication (8).

In brief, beet samples to be analyzed for sugar are not weighed, but only brushed free of adhering dirt. Care is taken not to remove any leaf tissue adhering to the crown. The whole sample, complete with crown, as delivered, is passed through the brei saws. The brei is squeezed in a cloth, obtaining 44 ml of juice, which is placed in a tightly covered plastic container, and immediately frozen.

Each day's samples are delivered the next morning, frozen, to the central sugar-testing laboratory, and placed in frozen storage. One truck delivers the samples from seven tare laboratories.

Samples from the freezer are thawed in a water bath to 20 °C. Dry lead subacetate is introduced, vigorously stirred in, and the solution filtered. The filtrate is polarized in a Bendix unit, and the percent sugar is printed on the sample ticket, which was originally placed on the sample in the tare laboratory. Details of the computation for use as basis for the grower's payment are in the reference. During the

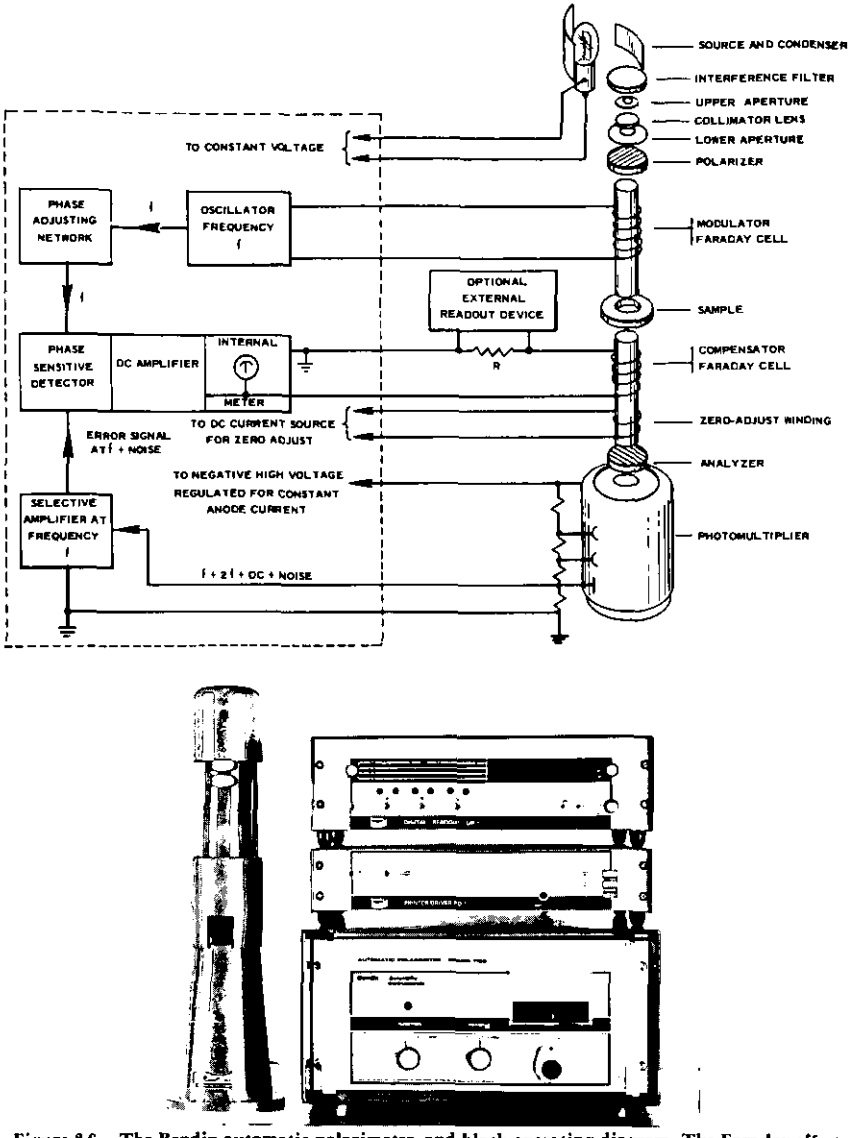

Figure 3-6. The Bendix automatic polarimeter, and block operating diagram. The Faraday effect is used to modulate the polarized light, and to determine the sample's optical rotation. If sample is optically inactive, no light reaches the photo multiplier, as the polarizer and analyzer are "crossed." In operation, an a-c current from oscillator at frequency f (380Hz) flows through Faraday modulator coil, causing alternate deflection of plane of polarization in clock- and counterclock-wise directions. This results in flow of a-c of too small f (760Hz) from the photo multiplier to selective amplifier. An optically active sample in the light path shifts the plane of polarization from the polarizer, and the output current from the photo multiplier is distorted by the a-c, f, resulting from the presence of the sample. The selective amplifier passes only a signal of f to the phase-sensitive detector, which emits a d-c signal proportional to the optical rotation of the sample. The d-c amplifier feeds the d-c to the Faraday compensator. An internal meter or external readout device indicates the size of the compensator current, which is a measure of the optical rotation of the sample. *(Courtesy, Bendix Scientific Instruments)*

most recent campaign, 72 400 samples were analyzed in five weeks by five workers, working 46 hours a week.

Automated Tare Laboratories

There are four types of automated tare laboratories in use in the United States: the Weibull, Kenway, Modified Toledo, and Venema. The first two will be described briefly and the third in some detail.

Weibull Analytical Laboratory

Two tare laboratories, at the Garland, Utah, and Moses Lake, Washington, formerly used factories of the Utah-Idaho Sugar Company, use sugar-testing equipment furnished by the firm of Nils Weibull AB, Malmö, Sweden.

As with all of the tare laboratories described in this section, the tare samples in bags are run from the receiving dock on overhead carriers, like meat carcasses, with the tops of the sample bags down. The suspended bags of beets are weighed, the beets are dropped from the bag into a hopper, and the empty bag is again weighed. The empty weight is automatically subtracted from the full weight.

The sample is run into a Carver-Ogden beet washer and drier, after which the beets are top-tared. The sample is then passed through saws, which pass through slots in the beet-containing basket, to produce the brei. The beet slabs are conveyed to the factory. The brei is caught on a belt, and mixed on the belt by a rubber-vaned wheel. The mixed brei drops into a mixing bowl set on scales, so that the flow of brei stops at about 80 g. Water is automatically added here, at the ratio of three parts water to one part of brei, less 10 g. The bowl is placed manually on a turntable unit, equipped with eight separate disintegrators, with rotors turning at 13,000 rpm. After the blending, the bowl is removed and 10 ml of concentrated lead solution is added. The mixing bowls are automatically washed and dried at this table. The bowl is then taken by the operator to the filtration apparatus, where filtration is done by suction, through porous disposable capsules. The filtrates are then automatically run through a Bendix polarimeter, and the readout is automatically printed for the accounting system by an adding machine.

The accounting system uses automatically-operated adding machines located at the gross and net weight scales, and at the polarimeters. Figure 3-7 shows a portion of the equipment formerly at the Moses Lake laboratory.

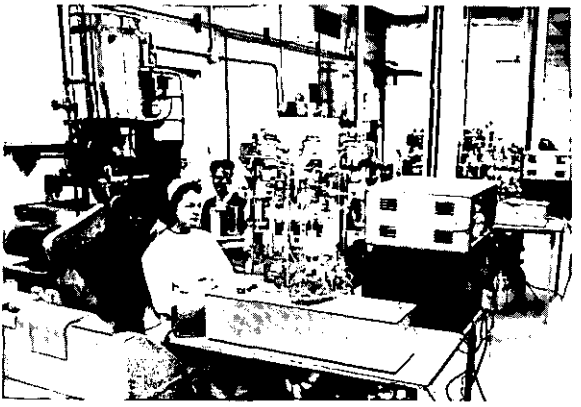

Figure 3-7. Weibull tare laboratory analytical equipment formerly at Utah-Idaho factory, Moses Lake, Washington. Operator in foreground is at one of two filtration-polarization stations. ***(Courtesy, Nils Weibull AB, Malmo, Sweden)***

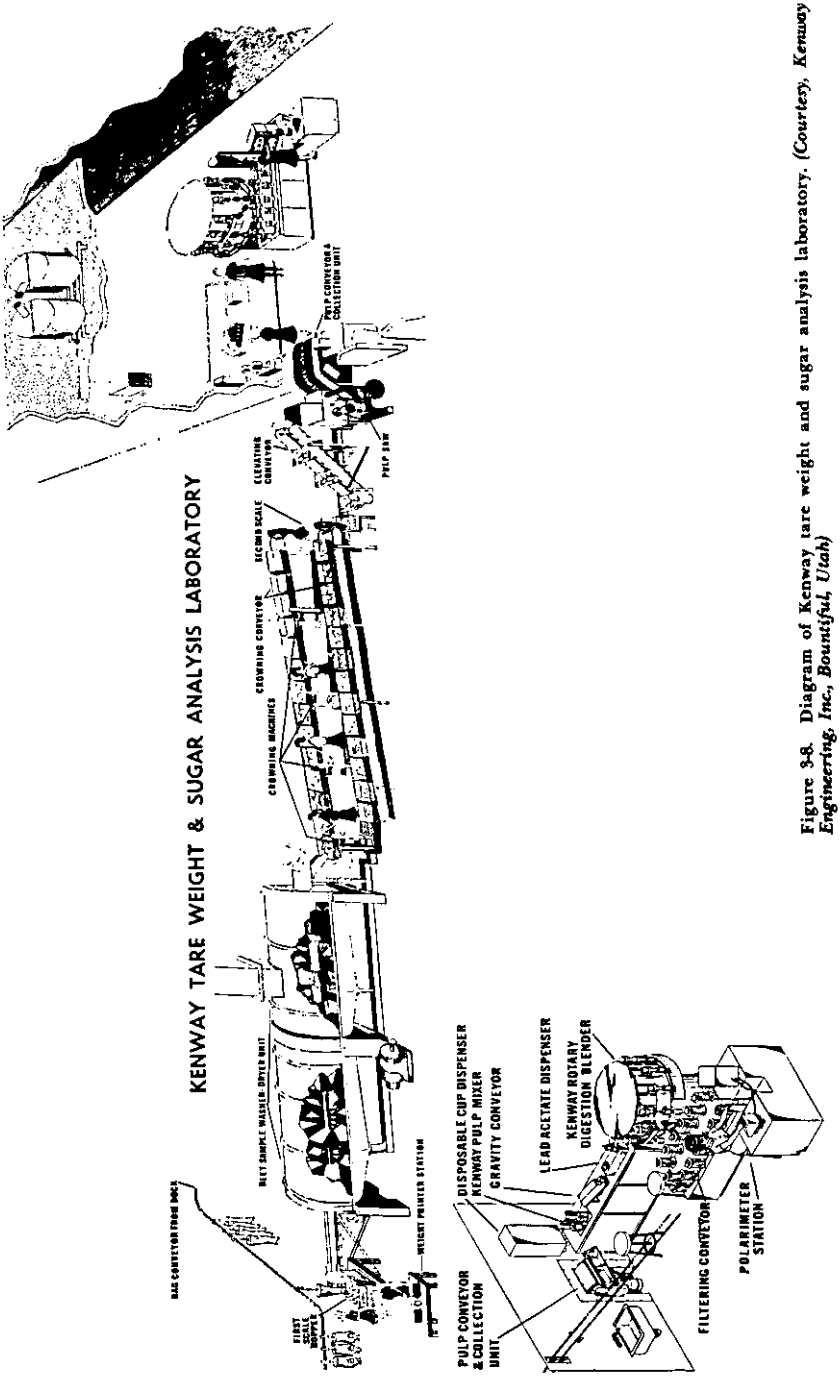

Figure 3-8. Diagram of Kenway tare weight and sugar analysis laboratory. *(Courtesy, Kenway Engineering, Inc., Bountiful, Utah)*

Kenway Automated Laboratory

Laboratories designed and constructed by Kenway Engineering, Inc., are located at the Chaska[1], Minnesota, plant of American Crystal Sugar Company, and at the Goodland, Kansas, factory of The Great Western Sugar Company (Figure 3-8). Individual installations are made to fit regional requirements.

Kenway laboratories include as equipment: 1) Overhead conveyors to move the samples in bags to the tare room. 2) Automatic printing and dumping gross and net weight scales. 3) Carver-Ogden beet washers and driers. 4) When beets are trimmed, conveyors of tandem pairs of pans, are moved past operator-topping stations. At the Chaska laboratory, the conveyor has been changed to the single-pan wide, one row type. 5) Beet saws yielding very fine brei. 6) Automatic brei-collection belt. 7) Brei mixers in disposable cups. 8) Reyers proportioning balances. 9) Rotary digestion blenders operating at 13 000 rpm, adjustable time controls, and automatic washing and drying of the blending cups. 10) Automatic pouring of the blended mixtures into filter cups which have had prior automatic addition of filter aid. 11) Bendix automatic polarimeters, with automatic recording of the sugar percentage on the sample ticket which has been automatically conveyed in sequence from the taring end by way of a ticket conveyor.

In present installations, the following automatic operations are used: 1) Gross weighing, recording, and dumping into a washer feed conveyor. 2) Sequencing of sample cards for net weight printing and conveying to the polarimeter station for automatic sugar percent imprint. 3) Sample sequencing through the whole system, by compartmentable equipment and a sequencing limit switch logic control system that times each function at 15-second intervals. 4) Washing and drying. 5) Transport past topping stations, if used. 6) Discharge of dirt and trash to waste flume. 7) Loading, weighing, and printing in net weight scales. 8) Conveying clean beets to the factory if sugar is not to be determined. 9) Introduction into the beet saws. 10) Collection of brei. 11) Preliminary brei mixing. 12) Proportioning of lead solution to the sample of brei. 13) Blending for controlled time, dumping blended sample into a filter, and washing and drying the blending cups. 14) Addition of filter aid to filter cup. 15) Percent sugar determination and recording on sequenced sample card.

The Kenway equipment is simple, and most of its various units are of proven soundness.

Amalgamated Automated Laboratory*

Amalgamated has two tare laboratories which determine tare and sugar content of all beets delivered to the Company. The laboratories differ in design and to some extent in operating principles. One laboratory is located at the Nyssa, Oregon,factory. The second and newer laboratory is at the Mini-Cassia factory.

The Nyssa laboratory, is described in detail in the 2nd Edition of Beet Sugar Technology on pages 87-90. The Mini-Cassia laboratory, constructed in 1974 entirely different in design, is discussed in the following.

Operating Objectives and Description

The Mini-Cassia laboratory is composed of two lines — one for tare only, the other for sugar content. The Nyssa laboratory also is composed of two lines — both of which determine tare and sugar content. Since the sugar content, determined on a

[1]No longer operating.

*by J. R. Johnson

tared sample, is significantly higher than the sugar content determined on a sample of clean roots but with top tare still on, it is obvious that sugar content determined on beets with the top tare intact more nearly represents the sugar entering the factory.

A study of the tare laboratories in the United States and in some European laboratories indicated that no one design completely fit our requirements. Consequently, our engineers selected equipment from various suppliers. This equipment was interfaced with computers and electronic/mechanical automation concepts,resulting in a high-performance laboratory unit.

Sample bags are of a different color, one color for sugar and the other for tare. Sample designations alternate on loads delivered daily by each grower. The result is a daily average for tare and sugar for each grower, which in turn are weighted according to tons delivered during the harvest. Grower identification is noted on a special card which is inserted into a Monroe CR-2 card reader as the sample enters the sugar or tare line at the laboratory.

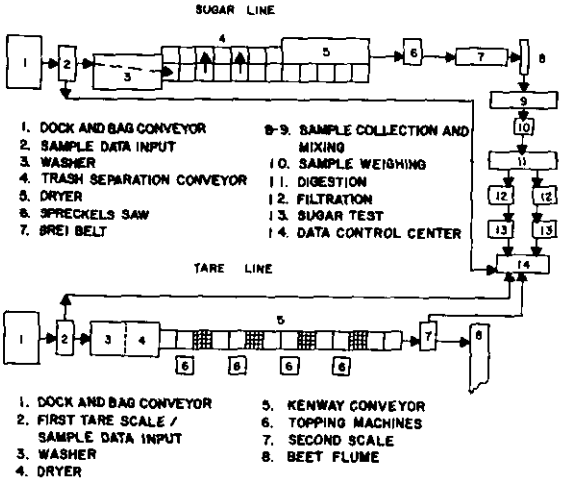

Figure 3-9. Block diagram of flows in tare and sugar sides of the Amalgamated tare laboratory, Mini-Casia Factory, Rupert, Idaho. (Courtesy of The Amalgamated Sugar Co.)

Beet scales are the load-cell type. Beet washers are Ogden Iron Works standard tare laboratory type.

The tare line conveyor is a Kenway Engineering type, and the sugar line conveyors are of Toledo Scale Company design. The brei saw is a Spreckels 8-blade unit. Brei is conveyed from the saws on a rubber belt to the collector and transferred to the mixing cups. This equipment is of modified Toledo design. Approximately 80.0 grams of brei is manually transferred to a Weibull cup on an automatic-zero-set Mettler balance. The cup is then placed in the water metering stand where water is added proportional to the weight of brei. The final dilution ratio is 177 ml water per 26.0 grams brei. The water added is set at 10.0 ml less the total requirement. This 10.0 ml volume is added as 55 Brix lead acetate on the Weibull digestion turntable.

After digestion the sample is manually poured into a U&I Incorporated, designed filter station. From here the filtrate is automatically sent to the Bendix

automatic polarimeter station where the sugar content is measured. The filtrate then flows to the Orion 701 nitrate ion equipment prior to being discharged to the waste solution system.

Each step in either the tare or sugar line is interfaced with Texas Instrument 960-B computers and the Texas Instrument Silent 700 Electronic Data terminals. A back-up data system feeds information to a punch tape which records sample identification, serial number, gross and light weight, sugar content and nitrate ion in each sample. The computer system records all information on Texas Instruments tape after the sample has passed the final step. As an example — critical data such as sample identification numbers from the card reader at the input end are stored in the computer. Additional information such as tare weights, percent tare, sugar content etc., are stored in the computer as the information is generated. When the cycle is completed all critical information on the sample is transferred to tapes. Upon computer operator command a complete printout can be obtained for a block of samples. At the end of each day, all information on the beet delivery for that day is transmitted to the General Office by data phone for final disposition and contract grower notification.

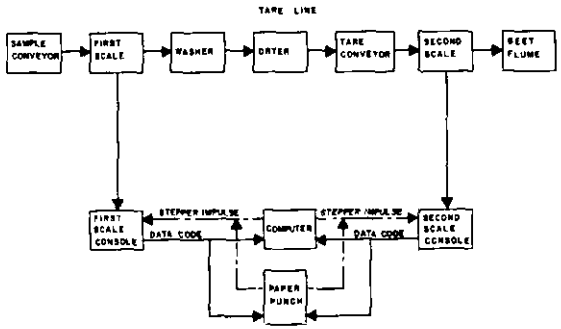

Figure 3-10. Electronic Flow diagram, tare side of the Almalgamated tare laboratory, Mini-Cassia factory, Rupert, Idaho. (Courtesy of The Amalgamated Sugar Company)

During laboratory operation a block of tare or sugar content information can be printed out according to serial number. This information is then compared to the sugar contents determined manually for laboratory control and calibration. Normally ten samples are pulled each hour and compared with the information collected by the automatic system. If variations between the paired samples exceed set limits the automatic equipment is adjusted to bring the laboratory back under control.

Sugar-line memory lights are displayed at critical locations so that operators can readily keep track of the sample flow and location. Serial numbers automatically are assigned to samples as they enter the system. If there is an empty washer compartment; i.e., a skipped compartment, the memory light will not be actuated as the missing sample passes through the various process steps. The various electronic safeguards act to prevent sugar content or tares from being recorded on the wrong sample. If, for example, a sugar content is lost at some point in the line the operator pushes an error button at that point and this information negates any printout for that particular sample.

The laboratory requires twenty-three to twenty-six persons per shift. If beets are excessively muddy, additional help is required on the tare line in order to maintain capacity.

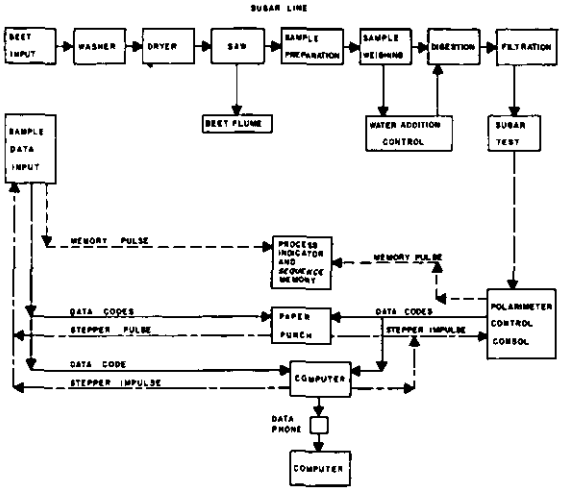

Figure 3-11. Electronic data Flow diagram, sugar side, of the Amalgamated tare laboratory, Mini-Cassia factory, Rupert, Idaho. (Courtesy of the Amalgamated Sugar Company)

Samples are introduced at a maximum rate of four per minute on either line. Nominal capacity is 5 000 tares and 5 000 sugars per twenty four hours. Sample flow rate is controlled by the first scale operator on either line.

The Mini-Cassia laboratory has proven itself to be capable of relatively trouble-free operation. A program of thorough maintenance is carried out each summer. In addition, the maintenance program allows for improvements or *modifications when justified.*

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(13) Resch, W.J., U.S. Pat. 2 162 122 (1939)

Chapter IV

BEET STORAGE*

This review of beet storage has been limited to a description of the current practice in North America. Although the literature on the theory and practice of beet storage in Europe is extensive, their climatic and contractual conditions are somewhat different and, therefore, have not been included in this review. The reader is referred to Vajna (60) for a complete review of beet storage practices prior to 1962. Other more recent extensive reviews have been made by Schalit (52) and Burke (17).

Harvest periods are short in most beet-growing areas of the U.S. and extended storage periods are necessary. In Canada and Northern United States, the content of sucrose in the growing beets increases until sub-freezing temperatures stop photosynthesis. For maximum yield and quality, beets must be harvested before the tops are killed by frost. Therefore, a rapid harvest is essential to remove the beets before they are frozen in the ground. As a result of this short harvest period (3-4 weeks), beets must be stored for extensive periods prior to processing. The factories process beets which are stored for periods up to 180 days, with an average storage period of 45 to 90 days, depending on location. Considerable sugar is lost during these extended storage periods.

Fox (30) estimated that 0.375 lb of recoverable sugar is lost, per ton of beets per day during storage under favorable conditions, but that the loss would increase to 0.45 lb sugar per ton per day or more under less favorable conditions. In commercial storage piles in the Rocky Mountain area, Akeson (4) reported average sugar and recoverable sugar losses of 0.326 and 0.435 lb sugar per ton per day, respectively, whereas Oldemeyer (47) reported sugar losses of 0.31 lb sugar per ton per day in the intermountain area. A 0.4 lb per ton per day loss on 18 million tons stored an average of 60 days would translate into a loss of 432 million pounds of sucrose worth 86.4 million dollars at 20 cents per pound. In California and Arizona there is no freezing weather, and sugarbeets can be successfully overwintered in the ground. Fall rains in Northern California can seriously hamper harvest, and to avoid temporary factory shutdowns, the factories sometimes resort to short-term piling. The intense heat in the Imperial Valley of Southern California requires harvest before July 1 to avoid heat damage to the beets and piling of not more than 20-hours duration is possible under shade. A small storage supply of beets is needed in the far-western factories, and in some factories in other areas during direct delivery, to smooth the flow to the factory during harvesting periods, which must, for maximum economy, operate as close to capacity as possible at all times.

Long-Term Storage

Piling Practices

Receiving Area. Beets are piled at receiving areas located at the factory site or at sites located some distance from the factory (Figure 4-1). In the latter case, the beets are transported to the factory in railcars or trucks when they are reclaimed for processing.

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*Original by R.A. McGinnis and D.L. Sunderland, revised by Walter R. Akeson.

Figure 4-1. Beet receiving at Crookston, Minnesota factory. ***(Courtesy, American Crystal Sugar Company.)***

Piling. The common receiving unit is the beet piler, a mobile unit including a receiving hopper, conveying equipment to elevate the beets to the dirt and trash separating screens, tare sampling apparatus, and a stacker boom which builds the pile of beets (Figure 4-2). The trash-separating screens are usually of the Reinks or grab-roll type (Figure 4-3).

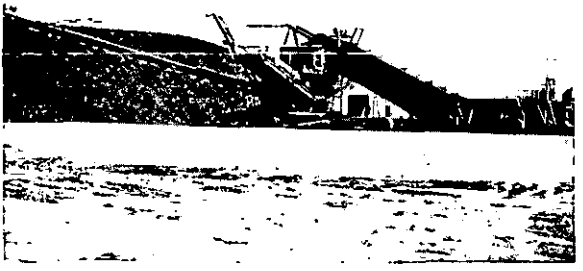

Figure 4-2. Mobile beet piler. ***(Courtesy, American Crystal Sugar Company.)***

Modern pilers rapidly unload trucks, clean the beets over the screens, and stack the beets for storage. Most machines are portable, with the piling boom swinging about a 90-ft radius. The regular portable pilers stack the beets for storage in rectangular piles, with trapezoidal cross sections, and with a base width of approximately 120 ft and a top width of about 80 ft. The pile height varies between 17 and 23 ft, depending on the local fall climate and the type of piler. New "Super-pilers" may build piles with base and top widths of 160 and 120 ft, respectively. The length of the pile depends on the area available and the volume of beets available. The typical pile may be 120 ft wide at the base, 20 ft high, and contains about 40 tons per linear foot. Stationary pilers which build circular or rectangular piles of various dimensions are used at a limited number of factory sites.

The dimensions of beet piles are to some extent a compromise to give a surface-volume ratio which will minimize surface area and yet allow adequate dissipation of heat for pile cooling. Beets in storage give off heat from respiration. Since respiration and resultant heat production are related to pile temperature, sugarbeets must be cooled to about 40 °F as rapidly as possible. The higher and wider the beets are piled, the more difficult it is to cool them by either natural or forced ventilation. Conversely, the smaller the pile, the greater the percentage of beets on the surface exposed to extremes of weather.

Figure 4-3. Beets passing over grab rolls for removal of dirt and trash. ***(Courtesy, The Great Western Sugar Company.)***

The smoothness of the pile surface is also important in reducing the surface area. Some companies level the top of the piles by hand (26), and may use wooden A-frames along the sides of the pile to minimize the surface area (27). These frames are about 5 ft high, and provide an end point for the piler boom swing. When beet-pile coverings are used (described below) these frames, with hinged sides, provide means for opening the covered piles and allowing aeration.

An ideal piler rapidly unloads the sugarbeets, screens out the soil and trash, and stacks the beets in a smooth, even pile with minimal damage to the beets. Piling machines today do not approximate these conditions. The cleaning screens cause the most bruising as beets are dropped over rollers, alternately steel-cage and rubber, which rotate in the opposite direction to the flow of beets (Figure 4-3). This bruising is tolerated to obtain clean beets, since dirt and trash impede air movement through the piles. Bruising also occurs when beets strike other beets and metal objects when unloaded and conveyed along the high speed belts and dropped off the end of the boom. Unfortunately, the rapid rough handling is tolerated because of the shortness of the harvest season and the large capital investment in pilers.

Reclaiming. Beets are normally reloaded from the piles with front-end loading units (Figure 4-4). Scoops on these loaders may hold up to 7.5 tons of beets. Beets are loaded by these units into trucks or railcars for transportation to the factory from distant piling stations. When the pile is at the factory location, the front-end loader may discharge the beets directly into a wet hopper, from which the beets are flumed to the factory.

Figure 4-4. Reclaiming beets with front-end loader. *(Courtesy, Northern Ohio Sugar Company.)*

Factors Affecting Pile Losses

A harvested beet is a living organism which respires, or burns sugar, in order to maintain life as do all living tissues. Respiration can account for 80% of the loss of sucrose and 75% of the loss of recoverable white sugar per ton (62). A second source of sucrose loss is the biochemical conversion to other metabolites, primarily invert sugars and raffinose. Formation of the other metabolites or impurities not only utilizes sugar, but also decreases extraction and thus decreases the percent of sugar in the beets that can be recovered during processing.

Effect of Storage Time. Beets lose sugar continuously during storage with the highest rates of loss occurring during the first 30 days of storage because of harvest wounds and higher pile temperatures and late in the season when piles deteriorate from freezing and thawing (30). Barnes et al. (12) found with multiple regression analysis, that campaign length and rate of harvest, both of which contribute to storage time, are the most important factors affecting year to year variations in storage loss.

Effect of Temperature and the Surrounding Atmosphere. A second important factor influencing sugar loss is beet temperature. As a rule of thumb, the rate of respiration approximately doubles for each 10 °C (18 °F) rise in temperature (62). However, respiration and ultimately sucrose losses, are higher when temperature fluctuates than when held constant (4). Above 0 °C, the temperature extreme is more important than the magnitude of the fluctuations or the mean-treatment temperature. Fluctuations in which the temperature drops below 0 °C drastically increase respiration and sugar loss. Like respiration, accumulation of invert sugars increases not only with increase in temperature, but also as a result of temperature fluctuations (63, 64). Raffinose, on the other hand, accumulates at temperatures less than 4 °C (63).

Rapid pile cooling is essential for minimizing sugar loss and impurity accumulation. Beets should not be piled when initial beet temperatures exceed 50-60 °F (42) or if ambient air temperatures are too high for adequate cooling. Furthermore, since beets generate heat from respiration, natural ventilation is essential. Since trash not only hinders the free passage of air through the pile, but also decomposes and generates additional heat, precautions must be taken to prevent trash

entering with the beets from entering the pile. A pile of clean beets to some extent ventilates itself, because warm air in the center rises, and colder air is drawn in from the surrounding atmosphere, known as the chimney effect. Artificial ventilation discussed later in the paper will increase rate of pile cooling under some circumstances.

The effect of humidity of the air surrounding beets on storage loss, is not thoroughly understood. Dry air cools beets by evaporative cooling, but excessive drying of beets in a pile is injurious (14). Humid air, on the other hand, may promote growth of microorganisms that cause mold and rots.

Mud and Trash. Factories normally do not accept beets with excessive dirt or trash for storage. However, under very dry or very wet soil conditions, removal of the mud or clods is difficult even with the correct and properly adjusted equipment. In such cases, the beets must be accepted, with resultant higher storage losses (38). Adhering clay-type soils may persist through to the factory diffusion juice, and pose difficult problems in carbonation sludge settling, since soil does not respond to settling aids normally used.

Improper Topping and Mechanical Injury. Leaves and petioles left on the beets by improper topping impede the natural circulation of air in the pile. New growth may also start from the crown at the expense of the sugar stored in the beets (Figure 4-5). Historically the storage of beets with intact crowns, as when topped with mechanical "beaters," has not been recommended because of the lower quality of the crown tissue and because of regrowth from the crown tissues. Since the crown, the upper-most section of the beet root, is composed of buds for new growth, it is the most physiologically active part of the beet and has a higher respiration rate than the root (69). However, removal of the crown tissue by topping exposes a large wound which increases the respiration of the root during the early storage period and provides an avenue for infection of rot organisms later in storage. Several recent studies have shown that flailed beets lose less sugar during storage than topped or partially topped beets[1]. Topped beets stored over 100 days can develop extensive mold growth on the cut surface, especially if the hollow area of the crown is exposed, whereas little mold develops on untopped beets (5, 65). Nitrogen fertility has a greater effect upon quality than does crown removal and so, with careful management, beets can be flailed without topping at harvest without sacrificing quality or yield (8). Sprouting, another objection to storage of untopped beets, can be significantly reduced by scalping at least one inch off the top of the crown to remove the main buds.

Beets are handled roughly by the large harvesters and beet-receiving equipment. The beet surfaces are cut, scratched, rolled, gouged, and bruised. Breakage occurs which further exposes open surfaces. The respiration rate of the sugarbeet root responds to injury immediately after harvest and the response may continue throughout the storage period [2]. Although high respiration rates immediately after harvest affect pile cooling and sucrose loss, the major effect of the injury apparently is to facilitate mold growth (46, 65). Small pieces of beets from breakage throughout the harvesting and processing operations are usually lost with the sugar they contain. As much as 3% of the beet weight can be lost in this manner. Each operation – topping and harvest, loading on truck, and piling – causes additional

[1]See refs. (5, 8, 19, 65)

[2]See refs. (5, 6, 19, 23, 50, 65, 69)

damage and thus additional storage loss (69). Development and modification of equipment to minimize damage would significantly reduce storage loss.

Figure 4-5. Sprouting of beets in storage pile caused by high topping. ***(Courtesy, The Great Western Sugar Company.)***

If the temperature of the beets in the pile is greater than 50 °F, bruising will lead to greater rotting. The rate of rotting in one series of experiments was 15 times as great at 65 °F as it was at 45 °F (34). Rotting is of secondary importance to respiration loss at lower temperatures.

Freezing. Serious losses can arise from freezing and thawing of beets in piles. A beet is not killed by moderate freezing, as respiration continues until the temperature reaches −18 °C; however, cell damage, as demonstrated by loss of cellular contents and increased respiration rates, results from exposure to temperatures below −2 °C (63). The damaged tissue respires rapidly when beets thaw, and subsequently decomposes when subjected to increasing temperatures and mold growth. Frozen beets received at a factory should not be put into storage piles but rather processed immediately. Beets which are piled frozen thaw out and rapidly respire. The generated heat increases the respiration rate and mold growth to give high interior pile temperatures and rapid decomposition of beets. Beets frozen in the field which are allowed to thaw and "heal" before harvest store reasonably well. Even though frozen and thawed beets appear to store well, they undergo higher than normal storage losses (1). During piling, the exposed face of a pile may freeze at night and then thaw when fresh beets are placed on the exposed face. These areas may be noted later, as dark and steaming bands across the pile.

The freezing of surface and interior beets of established piles significantly increases storage losses. The surface beets alternately freeze and thaw, contributing to the high sugar losses in rim beets (7). Frozen areas can also develop in the interior of the pile from the sides and top which, upon thawing, produce large areas of decomposed beets. Surface freezing can be reduced by building large piles with a lower proportion of exposed surface beets. Protection of beets with pile covering is discussed in a later section.

Dehydration. Desiccation and wilting of the beets may be a problem in exceptionally dry harvest seasons, particularly on the outside surfaces of the piles. If beets are permitted to wilt in the field, they cannot be stored without excessive losses (34, 35). The rapid harvest and piling with modern equipment normally eliminates

this problem. Desiccation increases respiration, impurity accumulation, and susceptibility to mold invasion (15, 16, 63), and is probably second only to warm temperature as a detrimental factor in storage.

Storage Rot. Storage rots cause significant losses of sucrose by increasing respiration and by increasing levels of impurities such as invert sugar which not only contribute to direct sucrose loss but also interfere with crystallization of sucrose (46). The storage rot problem has been recently reviewed in detail (15). Principal fungal pathogens include *Phoma betae, Penicillium* species, and *Botrytis cinerea.* Bugbee (15) reports that wounds from crowning, harvest and piling, freezing, and dehydration predispose roots to infection. Soil fertility, other diseases, and maturity may also predispose roots to infection. *Phoma betae,* which may be seed borne, infects the growing plant and decays the root in storage. Since fungal growth is related to beet temperature (44), rapid cooling of piles and maintenance of low temperatures reduces storage rot in addition to lowering sucrose loss in absence of storage rot. The most promising methods for controlling storage rots include fungicides applied to harvested beets (44, 46) and development of resistant cultivars (15, 16). Commercial application of fungicides is discussed in the next section.

Beet Culture. The effects that do not arise from conditions during the harvesting, handling, cleaning and storage are grouped under this heading.

a) Cultivar. Large differences between cultivars have been found in respiration rate, accumulation of invert sugars and raffinose, rot and sugar loss in storage[1] and sufficient genetic variability exists within sugarbeet genotypes to sustain a breeding program designed to develop low-respiring breeding lines. Each of the storage loss components — respiration, invert sugar accumulation, and raffinose accumulation — appears to be under independent genetic control (3, 59, 70). A 2.5-fold range in respiration rate was found among 76 inbreds (70). Other studies have shown that cultivars within a test vary as much as 50% in respiration rate, 175% in invert sugar after storage, and 50% in raffinose after storage (3). Significant progress in reducing storage loss can be made through plant breeding and cultivars should be evaluated for storage loss characteristics before being released for commercial use.

b) Nutrition. Larmer (43) found that phosphate fertilization of beets in phosphate deficient soil led to marked reduction of sugar losses during storage. Gaskill (34) has found fertilization in the spring with nitrogen, in certain cases, significantly reduced rot at 45 °F. Recent studies showed that nitrogen fertility levels have a large effect on sugar and purity, but no significant effect upon sugar or recoverable sugar loss (1, 8, 63).

c) Agricultural chemicals. The use of agricultural chemicals — herbicides, nematicides, fungicides — has increased dramatically in the last two decades. Akeson et al. (9) reported that chemicals applied early in the season such as herbicides or nematicides, had no adverse effect on respiration, invert sugar formation, or sucrose loss during sugarbeet storage; however, many chemicals applied prior to or after harvest significantly increased storage loss, no doubt because of phytotoxicity to the beets (23). Watkins (61), on the other hand, reported studies in which varieties not treated with herbicides stored better at 36 °F than the same varieties on which a herbicide was used. Additional research is needed to determine the effects, if any, of commercial herbicides on sugarbeet storage.

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[1]See refs. (1, 3, 15, 16, 59, 63, 70)

Pile Coverings

Experimentation with pile coverings to protect pile surfaces against dehydration and alternate freezing and thawing as well as to reduce the depth of freezing, has been conducted by nearly all U.S. companies storing sugarbeets. Recoverable sugar losses in the outer two feet of sugarbeet piles are three to four times as great as in the interior of the pile. "Rim" losses may account for over 40 percent of the recoverable sugar loss of the pile, although the rim may only account for 17 percent of the pile (7). Within the past decade, pile covering has become a commercial practice for several companies in an effort to reduce this "rim" loss as well as to extend the safe storage period.

Straw and Plastic Covers. Recently, straw and sheet plastic have been used for covering the sides of commercial beet piles[1]. Straw is blown through a regular mulcher-blower, and can be applied to beet piles as high as 35 feet (Figure 4-6). One ton of straw will cover about 1 000 tons of beets when applied 4 to 6 inches deep. The straw is normally applied in two applications, the first layer applied as a thin "sunshade" a few days after piling and the remaining straw applied when the pile is finished. Three men and one truck with a mulcher-blower can cover 50 000 tons of beets in 8 hours at a total cost of 15 cents per ton of beets. Straw covering was reported to reduce recoverable sugar loss by an average of 23.8% in 12 tests conducted over a four year period (7). The most satisfactory covering was a six inch layer of straw on the side and a one to two inch layer on top (4, 7). No benefit was obtained from a thicker side cover while a thicker top cover retards pile cooling. Straw does not prevent freezing but retards alternate freezing and thawing and dehydration of the rim beets.

The straw adheres well to the sides of the pile but tends to erode off at the edges. The straw has caused problems in some factories, but these problems can be overcome by uniform mixing of straw and beets during reloading, improved trash removal systems, and regulation of beet and water flow in the flume.

Sheet plastic materials used for covering pile sides include polyethylene (47), nylon reinforced polyethylene (27), and woven polypropylene (4, 7). A major problem is holding the plastic sheeting on the pile. The polyethylene is easily damaged by wind and must be weighted down with wooden beams, old tires, or other convenient materials (Figure 4-7). The nylon reinforced and woven polypropylene plastics

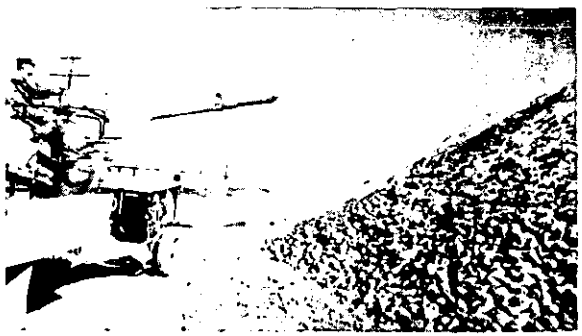

Figure 4-6. Mulcher-blower applying a 6-inch layer of chopped straw to a pile of beets. ***(Courtesy, The Great Western Sugar Company.)***

[1]See refs. (4, 26, 27, 42, 47)

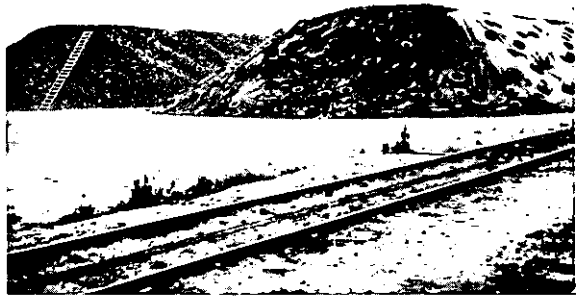

Figure 4-7. Beet pile covered with polyethylene sheet plastic held in place with used tires. ***(Courtesy, The Great Western Sugar Company.)***

are much stronger than plain polyethylene and so can be held down by wrapping edges around wooden boards which are attached to the pile with metal stakes. The plastic is normally applied to the sides with two or three feet left uncovered at the toe of the pile to allow for ventilation (4, 26) and should not be carried over the shoulder of the pile (Figure 4-8). Removal of the plain polyethylene can be difficult if snow is on the pile or if condensed moisture has frozen the cover to the surface beets. The woven polypropylene was equally, if not more effective, than straw in reducing sugar loss in pile rims (7); however, the plastic side cover alters the natural pile ventilation patterns so that deep frozen cones, formed by down drafts, develop from the tops of the pile (4, 26, 47). As the cones thaw with moderating weather, beets deteriorate and rot. The straw covered piles on the other hand freeze in from the side more than the top and the straw retards thawing in this area of the pile.

Figure 4-8. Woven polypropylene cover anchored to the side of beet pile with wooden boards and metal stakes. ***(Courtesy, The Great Western Sugar Company.)***

Canopy, Trench, Bubble, and Rigid Structures. Straw and plastic side covers reduce freezing and thawing of beets and thus reduce sugar loss and extend the safe storage period somewhat; however, they do not give complete protection for safe long term storage of beets. Beet piles must be protected from freezing or must be frozen solid and protected from thawing to give safe storage of sugarbeets past mid-February. Systems tested for protecting beets from freezing included plastic canopies (29, 41, 42), trenches (29), an air supported bubble warehouse (31, 42), and a rigid warehouse (31, 42). The trench effectively prevented freezing only when covered by a plastic canopy. Of these systems, only the plastic canopy has been used commercially. The common feature of each system was a completely enclosing structure with outside cooling air supplied by artificial ventilation. The outside air is closed off or restricted and the inside air recirculated during freezing weather. The plastic canopy (Figures 4-9 and 4-10), bubble warehouse (Figure 4-11), and rigid warehouse were equally effective in reducing sucrose loss, preventing freezing, and extending the time period for safe sugarbeet storage (31). Details of construction, ventilation, and controls have been described and illustrated in detail[1]. The projected cost per ton of beets stored under the plastic canopy in 1974-75 (31) was half that of those stored under the bubble and the rigid warehouse, primarily because of the high capital cost for the latter two. On the other hand, canopies have the following disadvantages: a) extremely high labor input at beet piling time, b) danger of beets freezing before they are covered, and c) susceptibility to damage or destruction by high winds during construction or storage.

Figure 4-9. Construction of canopy cover for beet pile. ***(Courtesy, U and I Incorporated.)***

Forced Ventilation of Beet Piles

In past years much use has been made of forced ventilation of beet piles, to cool the piled beets rapidly[2]. The operational principle is simple — cooling is effected by blowing the cool night air through the pile of beets. Once cooled to 30°-40°F, the beets can easily keep cool, because their respiration has slowed down and they

[1]See Refs. (26, 29, 31, 41, 42)

[2]See Refs. (10, 11, 13, 18, 20, 21, 22, 24, 25, 28, 32, 33, 36, 37, 40, 48, 49, 51, 54, 55, 56)

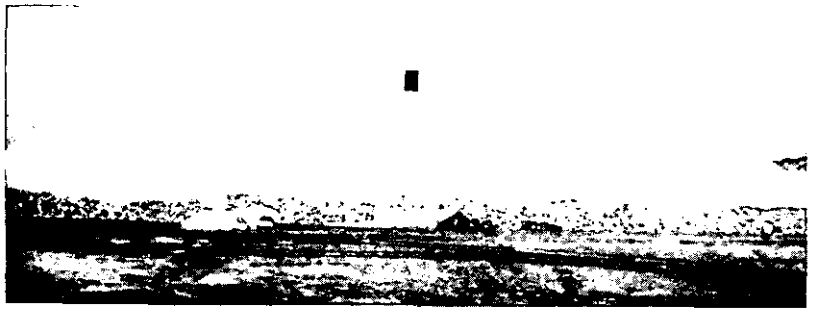

Figure 4-10. Canopy covered beet pile. *(Courtesy, The Great Western Sugar Company.)*

do not produce as much heat as at higher temperatures. Rotting is also not an important factor at the lower temperatures. Forced ventilation of beets has at times been used in nearly all the beet-growing areas of North America. However, forced ventilation of beet storage piles has not been used extensively on noncovered piles in recent years.

Tests with pile coverings and forced ventilation reported by Utah-Idaho Sugar Company research workers (51) indicated that a) side coverings alone prevent severe side freezing, but do not stop vertical convection air currents; b) forced ventilation improves the air currents, but does not totally eliminate hot spots; and c) top and side coverings with forced ventilation accomplish more uniform temperature control. The greatest benefit came from better handling and processing of beets, than from improved purity or extraction.

In earlier years when hand harvesting was practiced and later with simple, smaller beet harvesters, beets entering the storage piles were often warm and wilted. At the present time, with the larger mechanical harvesters, larger truck transport units, high-capacity pilers with grab-roll screens, and continuous deliveries 24 hours a day, beets entering storage are more likely to be turgid and cool, and forced ventilation is considered generally unnecessary. The usual practice in many areas today is not to accept beets for storage when daytime temperatures are high – greater than 80 °F – or when the beet temperature is above 55 °F. Longterm piling is carefully

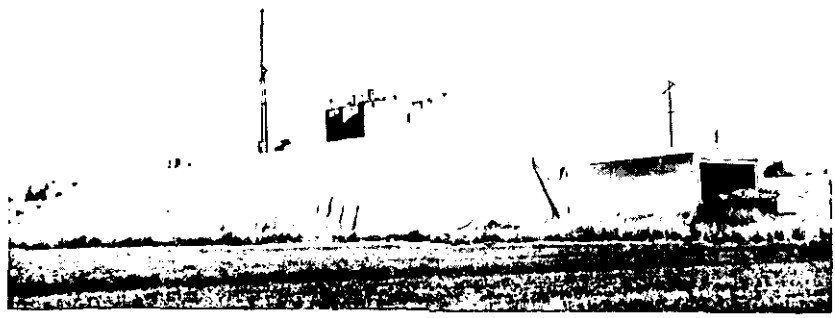

Figure 4-11. Air supported bubble warehouse covering beet pile at Toppenish, Washington. *(Courtesy, U and I Incorporated.)*

avoided in very warm climates, as in California and Arizona. A disadvantage in the use of ventilation has been the development of hot spots over the tubes caused by dehydration of the beets around the ducts or by freezing of the adjacent beets when the fans are operated with very cold, ambient air temperatures.

Forced air ventilation ducts are often made from discarded 55-gal oil drums, which are welded together (Figure 4-12). Gaps are left between the drums, or holes are punched in the drums for air-distributing apertures. Ducts are typically spaced 25 ft apart and are 90 ft long for 120-ft pile width. Axial-flow blowers supplying 10 000 cfm or more against a static head of 3 in. of water are a suitable air source. The tubes are positioned before the beets are piled over them and are held in place by blocking with beets, with small trenches, or by latching to stakes in the ground (Figure 4-13).

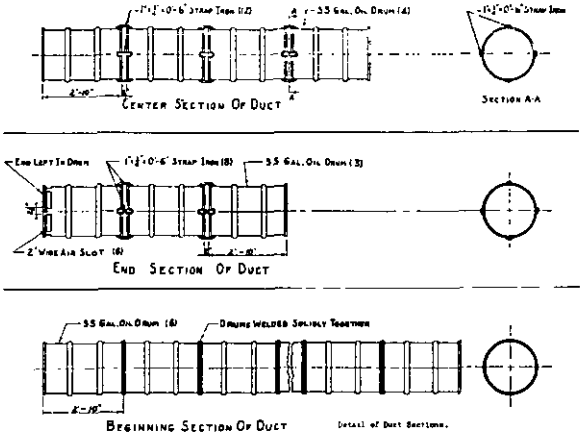

Figure 4-12. Construction details of ventilation ducts made from discarded oil drums. ***(Courtesy, Holly Sugar Company.)***

Recently, 24-inch diameter aluminum culverts with holes drilled for air-distributing apertures have been used in place of discarded oil drums for ventilation ducts (29). The principal advantages of the aluminum culverts were ease of preparation and handling. The culverts can be re-used if care is exercised during the reloading.

Non covered, straw covered, and plastic side covered piles are normally cooled with natural air convection currents; however, the protective structures — trench, canopy, bubble warehouse, and rigid warehouse — require the use of forced air ventilation to cool the pile. Ventilation systems (29, 31, 41, 42) were designed not only to distribute cooling air to the pile, but also to recirculate air from within the structure to prevent freezing during cold weather. Jardine and Stoller (41) described a modified ventilation system for canopies in which hot air was exhausted from the canopy through attic fans. Two-thirds of the ventilating air entered the pile through A-frames at the base. The remainder of the air was supplied to the center of the pile by high pressure axial fans. The axial fan system was controlled so that it circulated outside air only, inside air from the top of the pile or a combination through the pile. This system used about one-half the electrical power as the conventional pile ventilation systems.

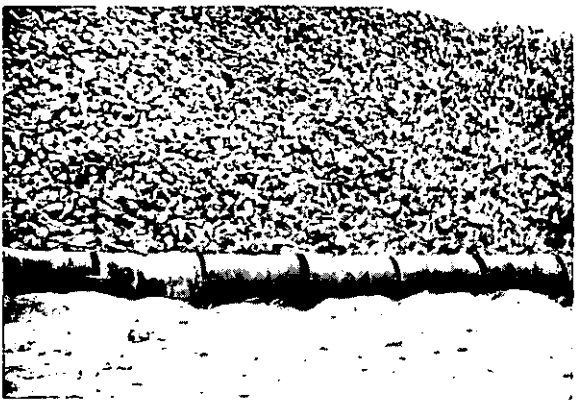

Figure 4-13. Ventilation ducts in place and about to be covered with beets during piling operations. ***(Courtesy, Holly Sugar Company.)***

Wyse and Holdredge (39, 67, 68) recently developed computer models for evaluating the effect of forced ventilation on pile cooling and sugar loss. Initial studies (67) showed 10 cfm/ton which is commonly used in commercial ventilation may not always be sufficient to control pile temperatures, and that 20 cfm/ton may give more adequate cooling. Schmalz et al. (53) confirmed these findings in covered beet piles where ventilation rates in excess of 18 cfm/ton were required to provide adequate temperature control and 35 cfm/ton during the initial cooling period gave most rapid temperature reduction. Wyse and Holdredge (68) found that benefits derived from forced air ventilation varies by geographical area since the cooling capacity of the ventilation system is dependent on ambient cooling air available. The authors proposed ventilating beets at 20 cfm/ton during the first 20 days of storage for rapid cooling and with 10 cfm/ton or no ventilation after that.

Other Methods

Deep Frozen Storage. The complete freezing of sugarbeet piles is currently a commercial practice for long term storage of sugarbeets in the Red River Valley (57, 58). Piles can be frozen solid by artificially freezing the pile with forced air ventilation or by removing and processing the center section of piles (pile splitting) by late November and allowing the smaller piles to freeze naturally. American Crystal employs a large deep freeze commercial application which incorporates ventilation channels through the piles to precool and then deep freeze the beets by using forced air. The ducts are fabricated from 54 inch galvanized culvert cut in half lengthwise and attached to a 2 inch metal base that elevates the culverts above the pile ground and allows the forced air to flow through the stored beets (Figure 4-14). Ducts are spaced eleven feet apart with fans on each end of the ducts forcing 20 cfm air/ton through the beets. Below ground duct air systems installed below a concrete covered area are also used to cool and freeze beet piles (Figures 4-15 and 4-16). The deep frozen beets are sliced during the last part of the campaign. Beet slice, sugar production, and extraction increase while slicing the deep frozen pile. Sugar lost in storage is lower in the deep frozen beets than in the regular piles that had been split and frozen naturally which in turn have lower losses than the partially frozen control piles (57, 58). The frozen storage technique can be used only in areas such as the Red River Valley where temperatures are cold enough to freeze the pile solid and

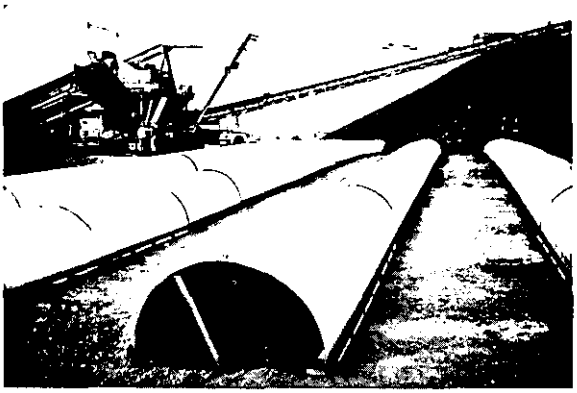

Figure 4-14. Above ground ducts for cooling and freezing beet piles. The ducts go across the width of the pile with fans on each end of the duct. ***(Courtesy, American Crystal Sugar Company.)***

keep it frozen. If all beets in the pile are not frozen, the heat from the respiration of nonfrozen beets can thaw adjacent beets as temperatures moderate. Heat of respiration from thawed beets would then thaw additional areas of the pile which could cause rapid pile deterioration. The factories have problems processing a mixture of frozen and nonfrozen beets but have few problems when all are frozen (57). Wyse (66) reported that the temperature on a pile should be reduced below −5 °C as quickly as possible. Insufficient freezing air could result in cooling only to −1 or −3 °C, causing an increase in respiration rates, and subsequently, increasing the amount of freezing air and time required for completion of deep-freezing. Respiration does not stop until the root temperature reaches −18 °C.

Fungicides. Fungal pathogens, particularly *Penicillium* sp. and *Botrytis cinera,* sporulate in the humid environment of the storage structures. The dry spores are distributed in continually moving air currents and deposited on other roots to renew the disease cycle (15). Thus, these storage structures function as giant incubators for storage rot pathogens and may create major rot problems that would

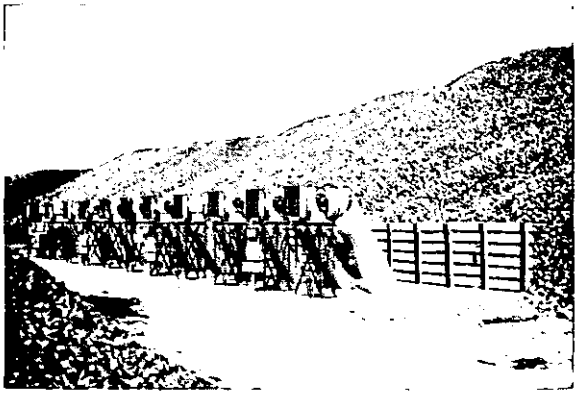

Figure 4-15. Fans and intake ducts which force air through a below-ground duct air system for precooling and freezing beet piles. ***(Courtesy, American Crystal Sugar Company.)***

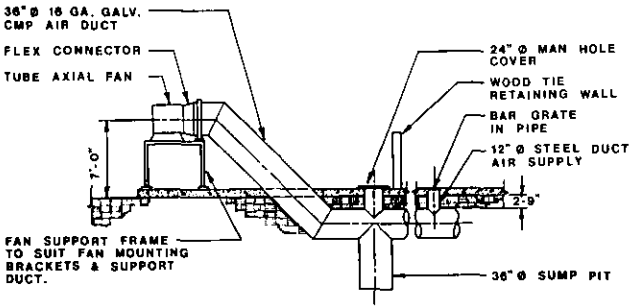

Figure 4-16. Details of below ground air duct system. (Courtesy, American Crystal Sugar Company.)

not be serious in an open pile. Treatment of beets going into structures with thiabendazole has been highly successful in reducing storage rot and thus reducing sugar and quality losses (45). The most successful treatment is a water solution containing 1 500 ppm thiabendazole sprayed onto the beets as they are piled at a rate of one gallon per ton of beets. Thiabendazole, however, did not reduce sugar loss in straw covered piles stored an average of 97.7 days (9).

Washing Beets Before Piling. The Northern Ohio Sugar Company installed a commercial sugarbeet washer as part of a circle piler, to wash beets delivered by growers before piling. The washer was a standard factory type C.F.&I. unit, which also removed trash. The beets were fed from the discharge of the grab-roll screen into the washer and conveyed from the washer to the storage pile, which was ventilated for rapid cooling. Results from two years testing showed that washing did not improve storage and if anything, increased the losses (2).

Short-Term Storage

Short-term storage is used in Northern California, as the rainy season is approached in the fall, to give a buffer supply of beets at the factory. Thus if a brief rainy period temporarily stops the beet harvest, the factory is saved the expense of shutting down and restarting or of running a nearly profitless Steffen campaign.

Most far-western factories have factory receiving bins holding up to a 3-days' supply of beets. When a larger buffer supply is required, a yard pile is started with portable pilers, and a storage pile of the size needed is built. From that time, beets are steadily reclaimed from the initial end of the pile, while at the other end the pilers continue to pile beets. Thus the moving pile is maintained at the desired size, and beets do not remain in storage for more than a given period, say a week or 10 days.

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Chapter V

PRELIMINARIES TO PROCESSING*

Preparation of the beets for processing is the step between the harvesting and slicing operations. In most instances it includes: receiving at the factory site; dirt and trash removal; short-term storage; beet flow control; final trash removal and final cleaning; and elevating to the factory slicers. These steps will be described in this section.

General

Sugarbeets are now usually mechanically harvested and this results in the simultaneous harvesting of trash, weeds, clinging dirt, rocks, mud balls, and metal parts. All of this extraneous material must be removed, damaged beet pieces recovered, and the beets cleaned of adhering dirt. Cleaning before storage is also necessary to remove the smaller materials that would block the ventilation passages between the beets.

In addition to the direct functions of each of these unit processes, there are indirect economic aspects to consider. There can be excessive root breakage in handling, and loss of sugar by leaching in flume and wash waters. On the other hand, the recovered trash, weeds and beet tops represent an economic gain, since these materials are normally fed to cattle at the factory site or mixed in with the beet pulp in the drying operations.

Factory Receiving Equipment

High speed conveying and screening systems are now generally a part of the receiving facilities of the beet-sugar factory. These screens can be of the Reinks multiple-kicker, roller type, the Armer multiple square-roll type (Figure 5-2), or the

Figure 5-1. A spiral roll screen, here used on beet-dewatering service.

*by A. G. Frost

Armer-Silver spiral nip-roll type (Figure 5-3). Each is designed to vibrate the beets to shake dirt loose, to pinch any items such as beet tops and leaves so that they can be separated from the beet roots, and to convey the partially cleaned beets toward the storage area. Large rocks and weed clumps are normally not separated since they are similar to the beet roots in size.

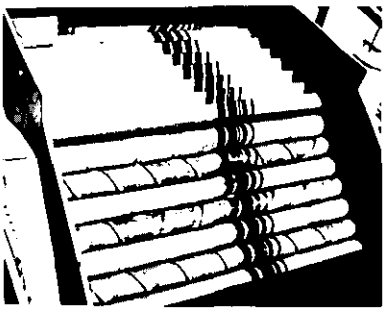

Figure 5-2. Armer square-roll trash screen.

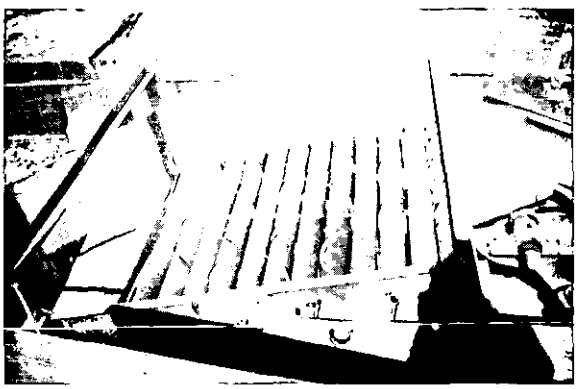

Figure 5-3. Armer-Silver spiral pinch roll on beet trash service.

Storage. Storage facilities are needed to ensure a continuous supply of beets to the factory during the periods when beets are not being received. Long-term storage is the subject of Chapter IV and here will only be covered to the extent of the handling of the beets for processing. There are many types ranging from flat slabs with front-end loader trucks, A-frame bins with flumes (Figure 5-4), to flat slabs with flumes (Figures 5-5, 5-6, and 5-7). Storage capacities will range from under 100 tons to hundreds of thousands of tons. Equipment used to fill the bins or pile the beets can be as elaborate as a fixed overhead structure with movable shuttle conveyors, to a simple beet piler that works along the piling area with beets delivered by truck to the piler hopper. All of the bin filling or piling devices must be designed to minimize breakage of the beets; i.e., there must be short drops to piles and a minimum number of transfer points. "Prod poles," picks, forks, and front-end loaders should be used with caution, since breakage of beets ruptures beet-root cells and increases sugar losses in fluming and washing.

Piles should be built so that the beets can be systematically harvested with no pockets of old beets left behind. Rotting beets can cause contamination of fresh beets, in addition to being a loss in themselves.

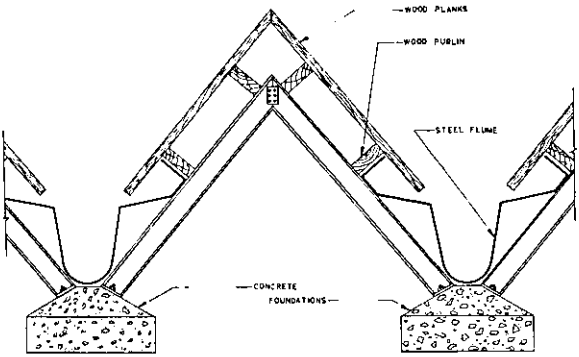

Figure 5-4. Cross section of typical beet flume and A-frame bins, as used in California.

Figure 5-5. Flat-slab beet storage. Elfa nozzles are for wet beet reclaiming and fluming.

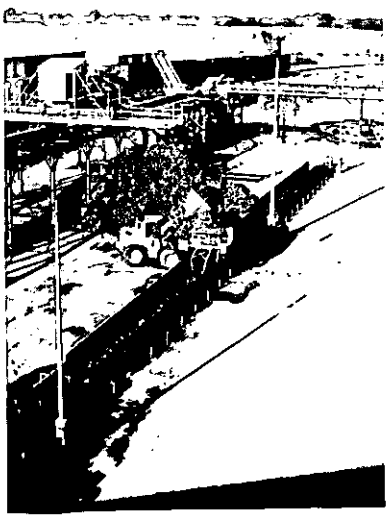

Figure 5-6. Flat-slab storage for dry handling recovery method.

Figure 5-7. Elfa nozzle for wet harvesting of beets.

As a rule of thumb, beets in areas where average daily temperatures exceed 40 °C should be processed with no more than a few hours of storage. In areas where ambient temperatures are 25 to 35 °C, storage should be limited to 2 or 3 days, and where temperatures remain near freezing, beets can be left in large piles up to 2 months. In any case, before storage the beets should be cleaned by passing over the dirt screens so that there will be voids between the beets to permit natural or forced ventilation of the piles.

Railroad Car Receiving Hoppers. Railroad car receiving systems are usually designed to route beets directly into the process without intervening storage. Beets that have been moved by railroad cars normally have spent one to three days in the cars and are sometimes wilted, or frozen. Additional storage could result in considerable sugar losses. Thus, rail car beets are usually placed in wet or dry hoppers and then conveyed directly to the final washing.

A wet hopper is located so that rail cars can pass over the open top, and it will hold one to three carloads of beets. A flume passes through the bottom section, to convey the beets to the factory. Beets are dumped from the car, and any remaining are manually dislodged with wooden poles or streams of water from hoses. This latter method is particularly suited for unloading frozen beets, where strong streams of hot water can be used.

In areas where the beets are firmly frozen into rail cars, the period of time to dislodge the beets is excessive, and special soaking hoppers are required. These facilities utilize hot condenser water to melt the ice and loosen the beets, with a drainage system to reclaim the water which drains from the cars. When the beets will flow, the rail car is transferred to the normal beet unloading and transfer system, usually a wet hopper, from which the beets are carried into the factory (Figure 5-8). Frequently the soaking is done over a part of the wet hopper.

Transport Truck Wet Receiving Hoppers. Recent specialized receiving hoppers have been developed to unload large beet trucks. One method uses a side dump transport truck and dumps directly into an operating flume. Another device cradles the truck trailer in a special tilting frame and raises the front end of the entire trailer until beets flow out the back end. The beets fall into a wet hopper, and a flume carries the beets into the factory.

Dry hoppers are similar to wet hoppers, differing in the use of mechanical conveyors to move the beets to the factory. Dry hoppers are usually adapted to areas with warmer climates and cleaner beets (Figure 5-9).

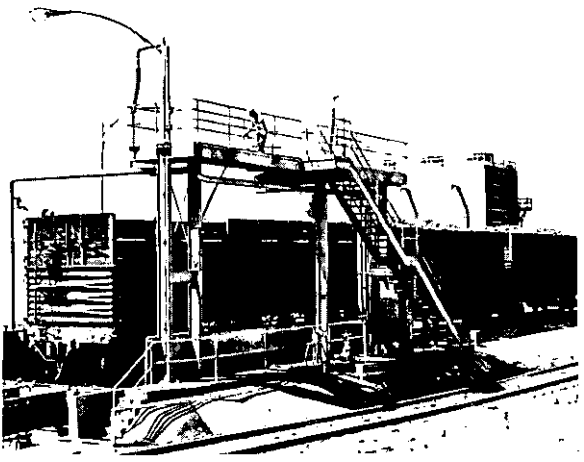

Figure 5-8. Unloading rail car using wet recovery system.

Conveying. Several types of conveying systems are in use to deliver the beets into the final cleaning and trash-removal operation. The most popular method is the beet flume, although dry conveyor belts and even dump trucks are used.

The transportation of the sugarbeets hydraulically by fluming was invented by Riedinger, the master mechanic of a German sugar factory, in 1879. This method is simple and convenient and a considerable amount of the encrusted dirt is washed off during transporting of the beets. Fluming is possible because sugarbeets have almost the same density as water and thus they are readily carried along by the stream of water. The one disadvantage is that some sugar is lost to the flume water by leaching out cut surfaces, particularly if the water is warm.

The flume can be made of steel plate or concrete, and is usually of constant width, with a flat or slightly rounded bottom varying in depth from three feet at the shallowest portion to as much as 12 feet in a long flume. Normal gradients are of about 1.5% for straight portions, and 2% for curved portions (Figure 5-10).

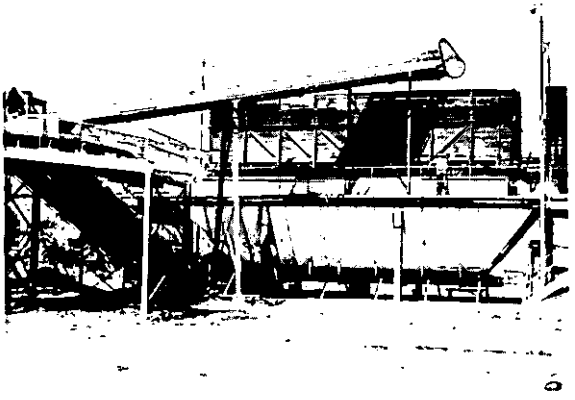

Figure 5-9. Rail car unloading using dry hopper. Beets are conveyed from railroad hopper by conveyor belt.

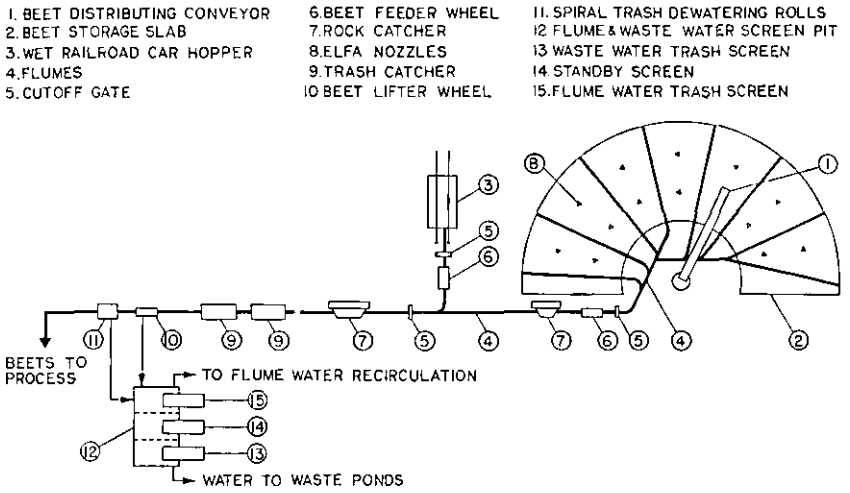

Figure 5-10. A typical California beet storage, fluming, and washing facility.

Most modern flumes are built with "run-arounds" or bypasses on the top edges so that when a plug of beets occurs, the water will flow around the plugged portion. The term "self-fluming" has been used to describe flumes with bypasses, since the flume water, diverted around the beet stoppage point through the bypasses, automatically and steadily washes the beets into the flume on the downstream side of the beet pile, thus tending to remove the stoppage. Such bypasses are also used at beet feeder wheels and shutoff gates in order to permit the flume water to continue around the beet stoppage. This prevents flooding and allows a constant recirculation of flume water when beets are not required in the factory. A variation of the run-around utilizes hold-down bars in the top of the flume to prevent beets from floating out while the water flows through the bypass.

Flumes of earlier vintage or some recent facilities so designed, do not have the bypass feature but are equipped with covers of steel plate, plastic, or wood, easily removed, to permit beets to slide from the storage pile into the flumes. This method requires a considerable amount of labor but some operators feel that plugging is less frequent than with uncovered flumes since it is possible to deliver a steady stream of beets from the pile to the flume.

In areas of high dirt or mud adherence to the beets, special provisions must be made in the flumes to remove the mud which accumulates. One such method utilizes a series of high-pressure overhead nozzles spaced along the flume to periodically, or continuously, flush the mud from the flume bottom. By proper spacing, the nozzles can even assist the beets around a particularly difficult bend in the flume. Bottom-flush nozzles have been used with moderate success in flumes, where the nozzles are embedded in the concrete or welded to the steel flume bottom. Cable hoist-driven plows have been used to manually pull the settled mud along the flume to where it can be flushed or shoveled from the flume.

Separation of beets from flume waters takes several forms. One of the more common methods is with a beet wheel. The beets are lifted from the flume and elevated to a hopper or conveyor. The rim of the wheel is made up of a perforated screen so that the flume water can pass through as beets are lifted. Spiral-pinch rolls are also used because they not only permit the water to pass through but also remove any trash and weeds which are still with the beets. Parallel steel bars, Reinks rolls, vibrating screens, and perforated pan conveyors are additional devices which are employed to separate flume water from the beets (Figure 5-11).

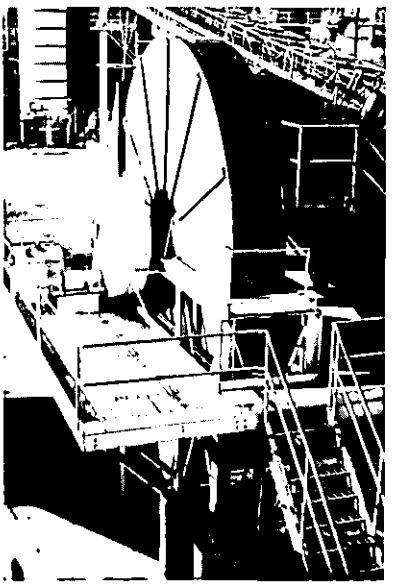

Figure 5-11. Beet lifting and dewatering wheel.

Flume water is generally recirculated many times with a variable amount of water being discharged at each circuit to control the quantities of mud and silt. In recent years, it has been necessary to control the volumes of waste waters genterated so partial or complete flume water recovery systems have been built. Makeup water is generally from condenser seal tanks and house cold water. The flume water temperature should be as low as practical, usually between 30 and 40 °C, unless frozen beets are being handled. Cold water is desirable since it reduces leaching of sugar from the beets. Water should not be run through flumes in storage bins until beets are to be moved as there will be sugar loss into the water. The sugar losses to flume water have been well established at between 0.02 and 0.05% on beets, but may rise up to 0.2% if the beets are wilted or damaged. Thus, the distance of fluming should be kept short and the water temperature low to avoid such losses. The loss of sugar to flume water is appreciable, since a factory slicing 3000 to 4000 tons of beets a day can lose over two tons of sugar a day to the flume water.

A dry handling system has been developed for at least one California factory that moves the beets into the final cleaning operation in a dry state. The losses of sugar to flume water with this method are minimized. It also has the inherent

advantage of keeping the dirt and rocks out of the flume water until just ahead of the rock catcher and beet washer, so that the mud does not settle out and block the flume.

In either fluming or dry conveying, care should be taken not to bruise the beets when moving them from the storage bin into flumes by careless handling. Wooden prod poles should be used instead of picks. Care should also be taken at transfer points to prevent bruising or crushing, and flume walls should be kept smooth, since abraded particles of beet will lose sugar faster than whole beets.

Final Cleaning. Among the types of apparatus which have been developed for metering and finally cleaning the beets are beet feeders, trash catchers, sand separators, stone catchers, magnetic metal separators, picking rolls, picking tables, and beet washers.

Beet Feeders. An essential part of the operations is the ability to control the flow of beets through the final cleaning process and into the slicers. Such control prevents massive plugs, with resulting process delays, which require labor to clear. The final cleaning operation would be impossible with a wide variation in beet flow.

One of the principal means of flow control in flumes is the radial spoke beet feeder (Figure 5-12). This device is composed of a series of radial spokes mounted upon an axis that rotates in the direction of the beet movement. The long steel spokes rotate into the flumes and clear the bottom by only a few inches. The rate of beet flow is controlled by the rotational speed of the feeder.

In one dry handling system the beets are placed into a hopper and a conveyor belt feeds the beets onto another belt conveyor. The speed of the feeder belt is regulated so that the flow of beets is controlled. Beets are finally delivered to a flume which is incorporated into the final cleaning system.

Stone and Sand Catchers. Stones and sand must be removed to prevent damage and wear to the beet slicers and processing equipment. Usually great effort is expended to make this operation effective. Methods which have been developed for separation are generally built around flume systems. The difference in specific gravity of the beets and rocks permits their separation in a water-transport system. In general, all the devices introduce a stream of water through openings in the bottom of the flume. As the beets meet this vertical flow of water, they are buoyed-up and pass over the opening. Rocks and sand fall into the opening and down to a conveyor, which elevates them continuously from the trap.

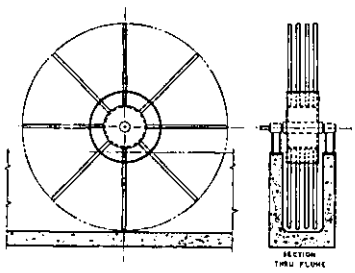

Figure 5-12. Radial spoke beet feeder.

The best known of the older devices is the Franklin type, which uses pockets in the flumes, where strong water pulsations produced by reciprocating pistons force the beets out of the pockets, but stones and sand settle into them. The pockets are manually cleaned.

The Dyer type (Figure 5-13) can operate continuously by using a steady flow of water entering the chute below the flume where the separation takes place. A similar rock removal system utilizes a drag conveyor or bucket and chain rock conveyor in place of the rotating wheel (Figure 5-14).

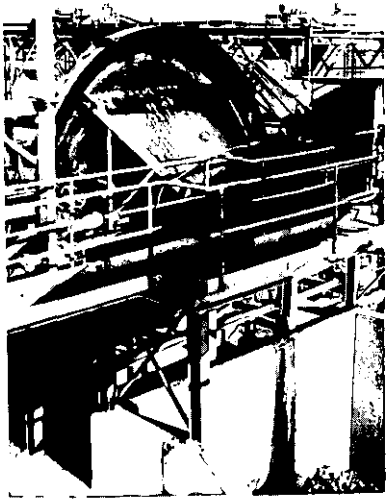

Figure 5-13. Dyer-type rock catcher with wheel and bucket rock elevator in operation.

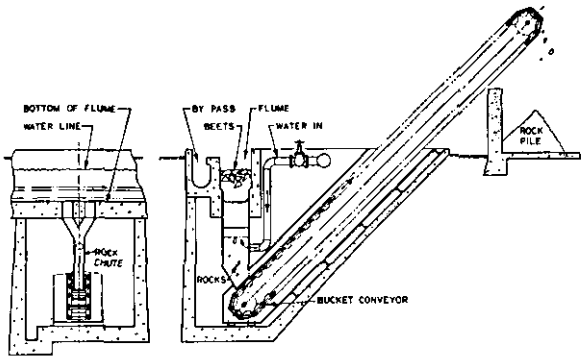

Figure 5-14. Typical flume rock catcher — bucket and chain rock conveyor.

The water used for lifting up the beets is usually a portion of the fresh water makeup, since accurate control of the water flow at this point is essential. In addition, considerable testing is required to obtain the correct upward velocity for the individual flow of beets and rocks involved, because beets can leave with the rocks if they are not properly buoyed by the upward water flow.

Trash Catchers. Trash, in the form of beet leaves, weeds, and grass, clogs the beet cutter knives, plugs the screens in the diffuser, introduces excessive impurities to the process, and carries in dirt which causes wear to the process equipment. Several methods of trash removal have been developed, and among these are: a rake system used in conjunction with fluming operations; a flotation device used with beet washers; and a rotating-roll, dewatering screen.

The simplest type is made by suspending steel bars equipped with barbed hooks into the flume. More in use is the Dalton trash catcher (Figure 5-15), in which the trash is caught on rakes moving countercurrent in the flume. The rakes are removed from the flume and are bounced or struck on fixed or hanging bars to disengage the trash, which drops onto a chute-plate.

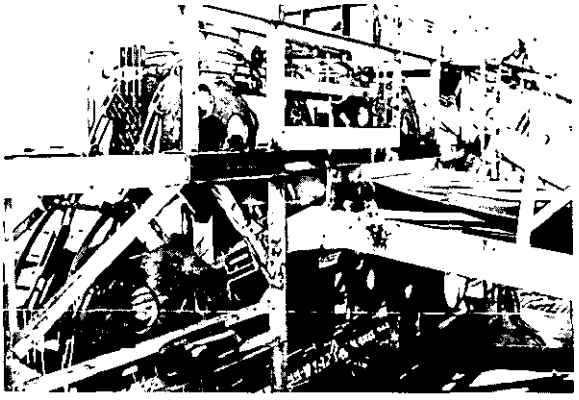

Figure 5-15. Dalton trash catcher. Trash drops to conveyor where beets bounce on sloping surface and return to flume.

A flotation system was developed in conjunction with the beet washer made by Elfa-Apparate (Figure 5-16) which uses a high velocity water stream within the vessel so that the beets are partially lifted. The water carries the trash to the surface where it is collected by spiked discs which are then washed off by the water as it exits the beet washer.

A third system used in flumes comprises spiral, intermeshed pinch rolls for separating the flume water from the beets. The beets pass over the rotating rolls, which are driven externally, while the water passes between the rolls into the recovery system below. In passing between the rolls, any loose trash or weeds are carried into the pinch point of the rolls and pulled through, where they are then carried down with the water. This method has advantages because of its simplicity; however, great care must be exercised in the design of the bearings and driving gears because of the large quantities of dirty water in close proximity to these precision parts.

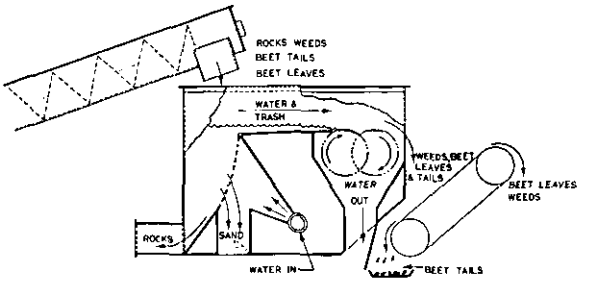

Figure 5-16. Trash separation unit by Elfa-Apparate.

Beet Washing and Magnetic Separators. Of the final beet cleaning steps, the most important operation is the actual washing of the beets before they enter the factory. It is presumed that the bulk of the trash, weeds, rocks, and sand has been removed by processes described above. The final cleaning steps must then incorporate equipment which will complete the cleaning as far as is practicable. This will include any dirt, as well as thermophilic and gas-forming bacteria which it contains. Process equipment wear and destruction from sand and rocks has been described previously but the silts affect the juice purification process and the bacterial growth leads to sugar destruction in diffusion.

An effective beet washer must have: 1) an even, regulated flow of beets in a single-layer distribution pattern, so that the beets can be individually cleaned; 2) clean warm water, preferably at high pressure, so adhering dirt is effectively removed; and 3) a separation section to permit silts, sands, trash, and the cleaning water to be separated from the clean beets. Many designs have been developed which incorporate most of these features, of which four will be described.

The Dyer or arm/agitator and horizontal tank beet washer is the older type (Figure 5-17) and consists of a large water-filled rectangular tank having sand and gravel traps located under the perforated bottom. A large horizontal shaft, equipped with propeller paddles of wood or steel, turns within the tank so that the mass of

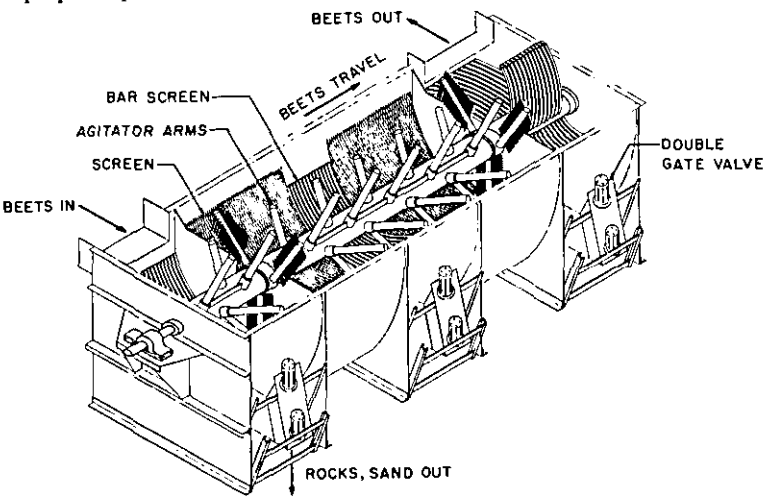

Figure 5-17. Beet washer horizontal tank arm/agitator type.

beets is constantly agitated and moved from the inlet to the outlet. At this point, fingers attached to the shaft move the cleaned beets from the washer.

A beet washer utilizing an oscillating tray with high pressure water sprays has been recently introduced on the North American continent. The unit, long used in Europe is the RT (Raffinerie Tirlemontoise) beet washer, which consists of shaped and perforated lateral metal slats with gaps provided between the slats. In this way the wash water can escape the trays and carry trash, mud and unwanted materials from the beet flow. The oscillating action, along with the shaped slats, rotates and progressively moves the beets so that the high pressure water can clean all surfaces while being transported (Figure 5-18).

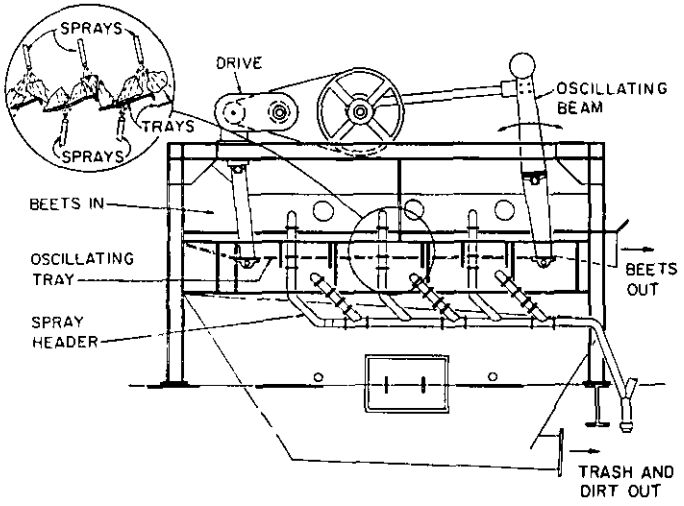

Figure 5-18. Oscillating tray beet washer. ***(Courtesy — Silver Engineering Works.)***

Several innovations have been developed in Europe, of which the B. Brunker unit has some interesting features. This device feeds dry beets into one end of a tank with a parallel-bar grating in the bottom. Large volumes of water, under pressure, are sprayed up through the bottom grating and fluidize the bed of beets. The water also forces the beets along to the end of the washer where they are dewatered. This system appears to be effective in cleaning beets in multiple layer pattern, and can separate the clean beets from the foreign materials.

The Silver beet washer (Figure 5-19) is of more recent design and incorporates many of the desirable features for a washer. It usually consists of 72 transverse rotating rolls of the spiral pinch or raised dimple type. All are mounted on a sloping frame. The beets are delivered at the high end and are moved by gravity, action of the rolls, and by high pressure water sprays, directed along the beet-flow path. Cleaning is mostly by the water sprays, but the spiral rolls pull the trash through, and the button rolls vibrate the beets to dislodge loose dirt and residual water from the beet surface.

Matched in importance with removal of rocks is the removal of metal particles from the beets. This generally follows the washing step and only removes magnetic metal. The magnet can be in the form of a stationary magnetic plate located in the

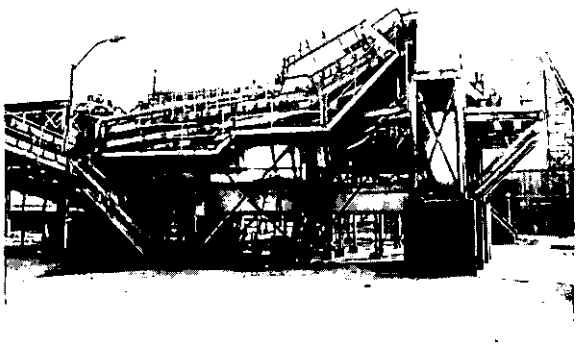

Figure 5-19. Silver beet washer. Unit has 72 rotating rolls with high pressure water sprays (*Manufactured by Silver Engineering Works).*

beet flow, but usually it consists of a rotating magnetic head pulley of a belt conveyor. The metal is held to the belt surface until the belt begins its return and the metal is scraped off or removed from the magnetizing field and drops free of the beet flow.

Occasionally the beet cleaning facilities are unable to cope with unusually large quantities of weeds and trash. During these periods, the recourse is to remove the trash manually from the beet flow. The picking table, which is usually a conveyor belt with positions for personnel to be stationed, is established in the beet-conveying system. Factory personnel pick off the trash for disposal.

Beet Leaves, Weed Trash, and Beet Particle Recovery. In any high-capacity beet cleaning and handling operation, large quantities of beet leaves, weeds, and beet particles are removed. These can be attractive sources of revenue and should be recovered. The beet tops and weeds have feed value for livestock and can be introduced into the wet pulp to be dried and sold, or fed directly as cattle food. Beet particles contain recoverable sugar and can be processed through diffusion. As an example, in a factory processing 4000 tons of beets a day, a daily recovery of 60 to 70 100-lb bags of sugar and five to six tons of dried pulp is possible from the trash and beet particles collected.

A problem is the separation of the beet pieces from the trash. One way is by the flotation device mentioned above, where upward-flowing water separates the trash from the beet pieces. Another device developed by Spreckels Sugar Company (Figure 5-20) uses a rotating steel drum where mixed trash and particles are dropped onto its surface. The wet trash adheres to the drum and is scraped off into a trash compartment. Beet particles are resilient and bounce into a recovery compartment.

Clean Beet Conveying. Transportation of the clean beets from the final washing station to the slicer hopper requires heavy duty equipment because of the great weight of the beets being conveyed. Many methods are used, but the major ways are: 1) inclined drag chain; 2) chain and bucket elevator; 3) inclined belt conveyor; and 4) a beet pump.

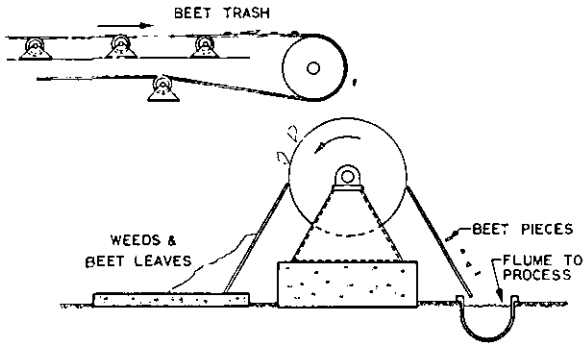

Figure 5-20. Spreckels beet particle and trash separator.

The inclined drag consists of two parallel steel chains with perforated steel plates at alternate links spanning the chains. This equipment is built on an incline and connects the flume dewatering or beet washing section with the beet slicer hopper at the higher elevation. A beet elevator normally uses the same drag chain system, but the drag plates are replaced with buckets and the entire mechanism is enclosed within a vertical housing. A rubber-covered belt conveyor, dished in the form of a trough, built on an incline, comprises the equipment used in the third type of conveyance. To prevent beet slippage, the conveyor slope must not exceed 12°, or be equipped with special slip restraining plates attached to the surface of the belt, and the beets must be dewatered before contacting the belt. Pumping the beets is a rather recent innovation and can be accomplished if the height to which the beets are lifted is limited to 70 feet. Rather large and elaborate dewatering facilities are needed within the factory because usually the entire quantity of fluming water is used for pumping the beets.

THE FUNDAMENTALS OF THE PROCESS*

The progress of the beets has been followed up to the point of entering the factory. Before proceeding further, the reader unfamiliar with the industry may profitably read an orienting summary of the normal beet-sugar manufacturing process, and examine the accompanying composite flow sheet, Figure 5-21. In the following description, only the usual practices and equipment are described, and variant processes and practices will be found in the main text of the book.

The process may be conveniently separated into six stages: 1) diffusion, 2) juice purification, 3) evaporation, 4) crystallization, 5) dried-pulp manufacture, and 6) recovery of sugar from molasses. The first three stages comprise the *beet end* of the factory, and the fourth stage, the *sugar end.*

1) Diffusion. The sugarbeets are run through the beet slicers, from which they emerge as long, thin strips, or *cossettes.* The cossettes, after weighing, are immediately run into a *continuous diffuser,* usually of the slope-type. This consists of a long trough, slanted upwards. The cossettes are propelled up the slope by

*by R. A. McGinnis

scrolls with perforated-plate flights. *Diffuser supply water* enters at the top end, and percolates by gravity through the cossette mass, leaching out the sugar as it proceeds countercurrent to the cossettes. The temperature is raised for better extraction by steam-heated jackets surrounding the trough. The sugar-enriched juice leaving the low end of the diffuser is known as *diffusion juice* or *raw juice.* The sugar-depleted cossettes leaving the upper end of the diffuser are known as *pulp,* and pulp processing is described in Section 5). The diffusion juice contains between 10 and 15% of sugar, which is about 98% of the sugar which was in the beets when sliced.

2) Juice Purification. Diffusion juice contains nonsucrose impurities in both true and colloidal solution, as well as sugar and water. The colloids, in particular, make it very difficult either to concentrate the diffusion juice, or to crystallize pure sugar from it. Thus it is necessary to purify the juice with lime.

The juice is first screened to remove any small particles of cossettes, and then heated, usually to 80-85 °C.

In some factories *preliming* or *predefecation* is used, in which a small portion of the liming agent is added to the diffusion juice, with proper control of time and temperature, to precipitate much of the colloidal matter in a comparatively irreversible form. Whether or not preliming is used, the next step in most factories is the continuous addition of the balance of the liming agent. Carbon dioxide gas bubbles are simultaneously passed through the mixture to precipitate the lime as very small insoluble calcium carbonate crystals. Mixed with and adhering to the precipitated calcium carbonate, the *sludge,* including coagulated and colloidal material, can be settled out in a clarifier. When batch purification systems were still in use in North America, the liming agent was added first, and then carbon dioxide was bubbled through, batchwise.

The lime used is either in the form of an aqueous solution and suspension known as *milk of lime,* or as a slurry of calcium saccharates known as *saccharate milk,* obtained from the Steffen process for extracting sugar from molasses, described in Section 6). A sugar factory using the Steffen process is known as a *Steffen house,* while a factory without such a process is known as a *straight house.* In the liming-carbonation purification, the juices are purified by coagulation and precipitation of impurities, and by adsorption of others on the surface of the calcium carbonate crystals.

The liming-carbonation is divided into *first* and *second carbonation. The most-used system, either unchanged, or with slight modification, is the Dorr system.* In this process first carbonation occurs in two tanks, the primary and secondary. Diffusion juice enters the primary tank, and as it overflows into the secondary tank the liming agent is added. The limed juice flows down through the secondary tank, in which it is treated with carbon dioxide gas. The contents of the secondary tank are continuously recirculated from the bottom of that tank into the bottom of the primary tank at a rate of about 700% on material entering. The pH or alkalinity is kept at the desired point by control of either the carbon dioxide gas or of the liming agent addition. Treated juice overflows from the first carbonation system into a multitray clarifier, where primary separation of the precipitated solids from the juice is effected.

The purpose of second carbonation is the removal of the lime remaining in solution in the juice. Here further gassing with carbon dioxide takes place, and more calcium carbonate precipitate is formed. Since second carbonation is at a lower pH than first carbonation, the calcium carbonate crystals are larger, and are easily filterable. Usually Kelly filters are used for this service. The filtrate is then

treated with a small amount of sulfur dioxide to inhibit color-increasing reactions, and usually an appreciable further reduction in pH results. At this point the juice is known as *thin juice.*

The second carbonation precipitate is usually added to the underflow from the first carbonation thickener, together with the filter cake from the thick juice filters (described below), and is desweetened on the rotary vacuum filters. The filter cake from these filters, sometimes known as *lime flume* when reslurried with water, is discarded. The combined filtrate and wash water from the rotary vacuum filters is known as *sweetwater,* and this is used to make up milk of lime in a straight house, or saccharate milk in a Steffen house. Excess sweetwater is returned to first or second carbonation.

3) Evaporation. Thin juice is heated and sent to the evaporators. Multiple-effect evaporators are used usually with five individual bodies or *effects.* Such an evaporator is essentially a set of single evaporators, in which the steam used for supplying heat to the first effect is drawn from the prime-mover exhaust, but for each succeeding effect the steam used is that formed in the preceding effect by evaporation of water from the juice. Such a system allows multiple use of the same heat energy, and results in decreasing pressures and temperatures as the juice proceeds through the effects. By evaporation the percentage of dissolved solids in the juice is raised from 10-15% to 50-60%. The outflow from the last evaporator effect is called *thick juice.*

4) Crystallization. Crystalline sugars from the second and third boilings are dissolved in thick juice in the *high melter,* and the liquid is then filtered in the *thick juice presses,* usually plate and frame presses, and the filtrate is known as *standard liquor.*

Sugar is crystallized by *pan boiling,* in the *vacuum pans.* The boiling must be at low temperatures to avoid inversion and caramelization, which necessitates low internal pressures. A quantity of standard liquor is boiled in the pan under vacuum, until the liquor is supersaturated sufficiently; that is, until it contains a greater concentration of sugar in solution than the equilibrium solubility of sugar at that temperature. The liquor is then either *shocked* to start crystal formation by the addition of a small amount of powdered sugar, or it is seeded at a lower supersaturation by addition of finely milled sugar in a slurry with isopropyl alcohol. The seed crystals are then carefully grown, through control of the supersaturation by means of vacuum, temperature, feed-liquor additions, and steam.

When the crystals are of the desired size and number, the mass of crystals and mother liquor, known as *massecuite* or *fillmass,* is then discharged from the vacuum pan into a large receiver, the mixer, which is equipped with a slowly moving agitator, and sometimes with a heating unit. From the mixer the massecuite is fed to centrifugals. The sugar centrifugal is, in essence, a perforated basket which rotates around a vertical axis at high speed, within an outer, collector shell. The liquid surrounding the crystals is centrifuged or *spun* off, and leaves the basket through the perforations. Most factories use completely automated, batch-type centrifugals for the first, or *white boiling.*

Following one or two brief washes with pure hot water, the wet white sugar crystals are discharged from the centrifugal basket, and are sent to the drier or *granulator* and the *cooler.* These are rotating cylindrical drums, with interior baffles which pick up the sugar and allow it to fall through a current of moving air. Hot, filtered air is passed through the granulator, and cool, filtered air is passed through the cooler. The granulated sugar is then screened, and either sacked immediately, or

stored in bulk bins to await future packaging or bulk delivery, or is used to make liquid sugars or other special products.

A liquid centrifuged from a massecuite is properly called a *syrup*. A liquid used for a vacuum-pan feed is properly designated a *liquor*. In the first-boiling centrifuging, it is the practice to separate the portion of the syrup that leaves before the application of the wash water, from the syrup that leaves after the addition of the wash water. The former is known as *green syrup*, and the latter as *wash syrup*. If no separation is made, the whole of the syrup spun off is known as *machine syrup*. In the case of the syrups spun from the first or white-boiling massecuite, the syrups are given the designation "high," as *high green* and *high wash*. The high wash is returned to the high melter, a tank equipped with agitator and means for heating, to dissolve remelt sugars in sugar juices.

The high green syrup serves as charge and feed liquor for the second or *intermediate* or *high raw* boiling, which is carried out in the same way as the first boiling. Intermediate or high raw sugar results from the centrifuging of the intermediate massecuite. Most factories use continuous centrifugals for this massecuite, in which separation of the green and wash syrups is not presently possible. The intermediate sugar is dissolved in thick juice in the high melter. The intermediate machine syrup serves as feed liquor for the third boiling.

The *raw* or *low raw* boiling takes somewhat longer than the other boilings, since the material purities (purity is the percentage of dissolved solids which is sugar) are much lower, and the sugar crystallization rates are slower. Following discharge of the massecuite into a mixer, it is run into *crystallizers* and held for periods up to three days, to permit crystallization to proceed as far as practicable. Crystallizers are large, horizontal, cylindrical tanks, ususally equipped with slowly moving stirrers and water-cooled surfaces.

Sugar from the raw centrifuging is known as raw or low raw sugar. This is dissolved in thick or thin juice in the *low melter*, and from there is run into the high melter. The syrup spun from the raw massecuite is *molasses*. Molasses is an end product, and no more sugar can be economically crystallized from it. Molasses is either sold for use as a cattle or yeast food, or it is further desugared by the Steffen process in the same or another factory. Molasses from a straight house is known as *virgin molasses*. Molasses shipped to another factory for treatment by the Steffen process is known at that factory as *foreign molasses*. Molasses which is discarded from the Steffen factory is known as *discard molasses*, or, in the factories of The Great Western Sugar Company, where it was used as raw material for the barium-desurgaring process at Johnstown, Colorado, and for MSG manufacture, it was known as *beneficiated molasses*.

5) **Dried-Pulp Manufacture.** Wet pulp from the diffuser is run to *pulp presses*, usually vertical, single-screw, where the moisture content is reduced from about 95% to between 76 and 84%. The water pressed from the pulp is usually sterilized by brief heating and returned to enter the diffuser at a point near the tail end. After the addition of CSF (described below) or molasses, or both, the pressed pulp is dried to a moisture content of about 10% by hot air in a *pulp drier*, consisting of a horizontal rotating drum. The pulp may then be cooled, and is normally made into *pellets*, in which form it is stored and sold for ruminant animal feed.

6) **Recovery of Sugar from Molasses.** Sugar may be recovered from molasses by the Steffen process. The molasses is diluted with water to about 6% sugar, cooled to under 10 °C, and limed with finely powdered lime in *batch coolers*. An insoluble compound of sugar and lime, called a saccharate, precipitates out, and is filtered off

on rotary vacuum filters. The filter cake is known as *cold saccharate cake.* The cold filtrate is then heated by steam injection to about 90 °C, when another insoluble saccharate precipitates, and is thickened in a multitray thickener. The clear overflow is called *Steffen hot filtrate,* or *Steffen waste,* and is usually carbonated to remove excess lime, and then concentrated, by multiple-effect evaporation, to form *concentrated Steffen filtrate,* CSF, and added to the pressed pulp before drying, for enhanced nutritional value, or it may be the raw material from which monosodium glutamate is produced.

The thickener underflow, containing the *hot saccharate precipitate,* is usually cooled and mixed with the feed to the cold rotary vacuum filter, and the cold and hot saccharates are filtered out together and washed as free of the impure mother solution as practical. The mixed filter cake is then made into a slurry with sweetwater from the carbonation rotary vacuum filters, and is called *saccharate milk.* The saccharate milk is used as the liming agent in first carbonation in a Steffen factory. When carbon dioxide is passed through the secondary, gassing, carbonation tank, the saccharates are broken up, freeing the sugar, and making the lime available for purification and formation of the calcium carbonate precipitate.

Chapter VI

DIFFUSION*

Extraction

The aim of the diffusion process is to extract the maximum amount of sucrose with the minimum amount of impurities. The theories and their applications to obtain this goal in the factory are described in this chapter.

The beets are cut into thin slices or cossettes, which are weighed on belt-conveyor scales before being entered into the diffuser. These scales can serve at the same time to regulate the flow of cossettes to the continuous diffuser. The older type diffuser consisted of a series of tanks or cells, called a "Robert battery." This batch type of diffuser has been replaced by continuous diffusers of various designs in North America. In both batch and continuous systems the diffusion juice moves countercurrent to the cossettes, extracting sugar as it passes. The sugar solution leaving the diffuser is called *raw juice.* Part of the beet juice from the desugared cossettes, *pulp,* is removed in presses. The resulting press water may be returned to the diffuser, either mixed with the fresh water supply, or preferably, to a point within the diffuser. The pressed pulp contains only a small amount of sugar, but enough other nutrients to make it a valuable cattle feed.

The sugarbeet, like any other plant, is made up of a complex system of cells (see pp. 17-19). According to their functions the cells can be classified as parenchyma storage cells and phloem transport cells. Schneider (45) found a 20% higher sugar concentration in transport cells.

In a living cell the sugar-containing liquid is surrounded by protoplasm, a protein substance capable of controlling the diffusion of substances into or out of the beet cells. To permit the extraction of sugar, the protoplasm is made incapable of such control by coagulating the proteins in the protoplasm by heat or coagulating agents. In practice, however, only the high temperature treatment is used. The protoplasm is then said to be denatured.

Denaturing starts at temperatures above 50 °C, the time necessary for complete denaturing depending on the temperature and physical condition of beets. It is interesting to note the different denaturizing rates of the phloem and parenchyma (see p. 25) cells as reported by Schneider (44,45). The slower denaturing rate of the phloem cells, especially at lower temperatures, can adversely affect sugar extraction in practice. Some results of these experiments are shown in Figure 6-1.

Extraction Process Theory

The extraction of sugar follows the general laws of diffusion, which apply to the intermixing of molecules or very small particles in a fluid as caused by the random thermal motion of the sugar and water molecules. These molecules diffuse gradually from regions of higher concentration to regions of lower concentration. The tendency to diffuse increases as the concentration difference increases. The speed with which a particle will diffuse depends on its size and also on the temperature and viscosity of the solution. For simplicity a circular cross-section of the cossette is assumed, and allowance may be made for other shapes. Let the radius of the cross section be r. Assume that initially the concentration, c_0, of sugar will be uniform over the cross section. If the cossette is immersed in juice in which the

*by H. O. Ebell and M. Storz.

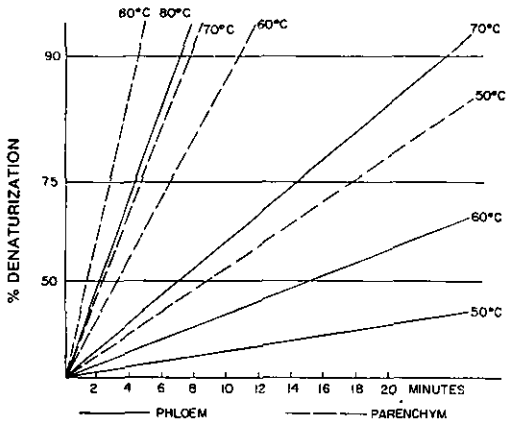

Figure 6-1. Rate of denaturization of beet cells in diffuser (45).

sugar concentration is j_o, and j_o is less than c_o, diffusion immediately starts at the surface and a gradient develops within the cossette along r. It will be considered that the surface film of liquid is part of the cossette and that diffusion and mechanical agitation in the juice is sufficient to keep the concentration on the outside of the cossette at the average of the whole juice concentration. Since time is required for sugar to diffuse from cell to cell in the interior, the concentration gradient will have a gradually diminishing slope, receding from the surface. Thus the concentration in the cossette, following r from the center, will resemble curve 1 of Figure 6-2a. At later times the concentration will resemble, successively, curves 2, 3, and 4. When complete equilibrium is reached, the concentration within the cossette will again be uniform, a flat graph will result and $c_2 = j_2$. This is represented by curve 5. Since complete equilibrium is approached asymptotically, a very long time is involved and it is never closely approached in practical diffusion.

Similarly the rate of diffusion drops from a high initial value at a gradually decreasing rate, until, as equilibrium is approached, it is very low. Thus a graph of the diffusion rate will resemble the exponential curve of Figure 6-2b. Notice in Figure 6-2a that j, the concentration of sugar in the juice, increases as the average of c decreases. The amount of increase depends on the relative volumes of the cossettes and juice.

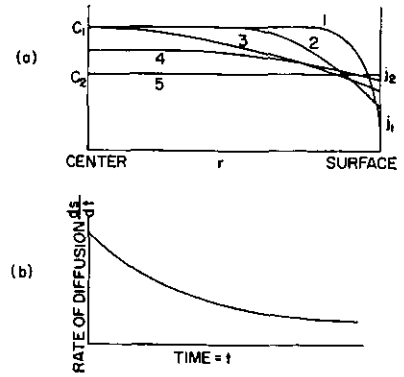

Figure 6-2. a) Sucrose concentration within a cossette at successive times after insertion into juice of concentration j_o. b) Decrease of rate of diffusion with time.

Figure 6-3 illustrates that it is impractical to attempt to continue diffusion until equilibrium is nearly established. Curve 1 shows the change of the average sugar concentration in the cossettes with time, and curve 2, the corresponding change in the juice. After a diffusion time of t_1, most of the sugar has diffused out of the cossettes and into the juice, and little is gained by doubling the time to t_2. The vertical distance between curves 1 and 2 at any time is an indication of the magnitude of the concentration gradient, dc/dr.

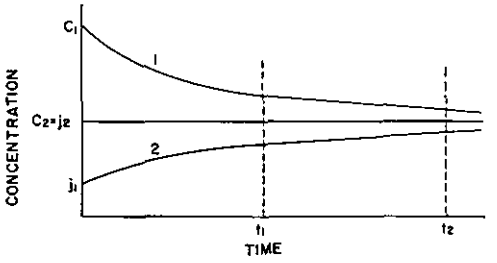

Figure 6-3. Change of average concentrations of sugar in juice and cossettes with time of diffusion.

Mathematics of Diffusion. A considerable advance was made in the mathematical theory of diffusion by Silin (51), when he developed equations relating the various factors affecting diffusion. He started with Fick's law (18), a special case of Fourier's general diffusion law.

$$ds = D_f A \frac{dc}{dr} dt \qquad (1)$$

where ds is the weight of the dissolved substance diffusing through the area A in time dt: and dc/dr is the concentration gradient of the dissolved substance A. D_f is the diffusion coefficient, which depends on temperature according to the Einstein correlation:

$$D_f = \frac{k'T}{\eta} \qquad (2)$$

where k' is a constant for the dissolved substance, T is the absolute temperature, and η the viscosity of the solution. Let c_o be the average concentration of sugar in the juice within the cossettes entering; c_n in the juice within the outgoing pulp; and c, in the juice within the cossettes at any intermediate point. If the assumption is made that the amount of juice in the cossettes and in the pulp is 93% of their weight, the percentage of sugar in the cossettes must be divided by 0.93 to obtain c_o, c_n, and c. Similarly let j_o, be the concentration of sugar in the raw juice, j_n of the diffuser supply water, and j of the juice surrounding the cossettes, in which the concentration is c. All concentrations are expressed as weight percent on juice.

Assuming a steady state, and considering the diffuser as a continuous countercurrent apparatus, the concentration gradient, dc/dr, can be considered proportional to (c − j)/r. The average (c − j) through the length of the diffuser can be determined by calculating the logarithmic mean difference:

$$c - j = \frac{(c_o - j_o) - (c_n - j_n)}{\ln \dfrac{c_o - j_o}{c_n - j_n}} \qquad (3)$$

Also, for a steady state, where $j_n = 0$ the amount diffused, S, is proportional to the time t, because in that case all the coefficients of dt are constants. In a continuous diffuser, all the sugar diffused from the cossettes in the diffusion time is gained by the juice. Therefore, the amount diffused from a unit weight of juice within the cossettes will be $(c_o - c_n)$. Substituting these values and kT/η, for D_f, equation (1) becomes:

$$c_o - c_n = \frac{kTAt}{\eta r} \cdot \frac{(c_o - j_o - c_n)}{\ln \frac{c_o - j_o}{c_n}} \tag{4}$$

If D is the draft, the "draft" based on juice within the cossettes (instead of cossettes) will be, n = D/.93. The amount of sugar diffused, per unit of juice in beets, will then be $j_o n$ and will equal $(c_o - c_n)$ or:

$$j_o = \frac{c_o - c_n}{n} \tag{5}$$

Substituting this in equation (4), and rearranging and changing to common logarithms:

$$\frac{n}{n-1} \log \frac{(n-1)c_o + c_n}{nc_n} = \frac{2k}{2.303} \cdot \frac{T}{\eta} \cdot \frac{A}{r} \cdot t \tag{6}$$

Since T and η are both functions of temperature, the ratio can be set proportional to a new variable, Θ; and since A and r depend on the length of a given weight of cossettes, their ratio can be set proportional to L. Letting $\Theta = T/1000\eta$ and L = the length in meters of 100 grams of cossettes, and combining all the various constants into a single new constant K, equation (6) becomes:

$$\frac{n}{n-1} \log \frac{(n-1)c_o + c_n}{nc_n} = K\Theta Lt. \tag{7}$$

Θ, L, and t form a product which is called P. Note that a change in any of the three can be offset by a corresponding change in any of the others, without altering the value of P. Equation (7) then becomes:

$$\frac{n}{n-1} \log \frac{(n-1)c_o + c_n}{nc_n} = KP \tag{8}$$

Equation (8) can be solved for c_n, from which the losses can be calculated.

$$c_n = \frac{(n-1)c_o}{n\left[\text{antilog}\left(\frac{n-1}{n} \cdot KP\right)\right] - 1} \tag{9}$$

However, K should not be considered a theoretical constant (49). Its value is influenced by the beet material and the diffuser design. A good example is the large difference of the diffusion coefficient in the Robert diffuser and in the various continuous diffusers. More extensive adhesion of cossettes in the battery reduces the

available surface area for diffusion and results in a smaller value for K. However, this is partially offset by the use of finer cossettes in the battery. The type of cossette which can be used is determined by the beet material and the design of the diffuser. To facilitate calculations with the Silin equation, Tables 6-1, 6-2, and 6-3 have been computed. Table 6-1, as calculated by Mück (31) and Dörfeldt (15) shows the amount of pressed pulp obtained with pulp press water return*. The pressed pulp yield has to be multiplied by the percent sugar in the pressed pulp to obtain the pulp sugar loss on beets.

The value of KP can be obtained from Table 2 or equation (8) by using the known draft and the sugar loss in the pulp. Following is an example. Where cossette polarization = 16.0; pulp loss = 0.20; draft = 120; then $0.20 \div 16.0 = 0.0125$, and KP = 5.66. Equation (9) shows that the losses are directly proportional to the sugar content of the beets; therefore the sugar concentration need not be expressed as percent on juice in beets but can be expressed directly on beets.

To recapitulate: n = draft/93; c_0 = weight percent of sugar in entering cossettes; c_n = weight percent of sugar in pulp leaving the diffuser; K = a constant for the diffuser; P = the product of Θ, L, and t; Θ = (average temperature, t °C + 273)/1000 times the viscosity of water at the average temperature of the diffuser); L = length in meters of 100 grams of cossettes; and t = time in minutes, of the diffusion.

Silin conducted experiments to determine if K is actually constant, and its value. In six laboratory experiments, under widely differing but carefully controlled conditions, K averaged 6.55×10^{-5}. In six factory scale experiments K averaged 6.45×10^{-5}, with a range from 6.09 to 6.86×10^{-5}. The validity of the theory was confirmed by Wiklund (63) and Wintzel and Akermark (65), and it was found that a value for K of 6.7×10^{-5} was valid for their conditions.

K can be calculated if the average temperature, the diffusion time, and the Silin number are known. To obtain Θ from the temperature use Table 3. Following is an example. If KP = 5.66, t ° = 70 °, L = 12m/100g, and t = 70 minutes, then K = 8.06×10^{-5}.

Oplatka (35) supplied a formula for the countercurrent diffusion process similar to the Silin formula, but which replaces the Silin number by the expression $12K/r^2$.

$$\frac{C_n}{C_o} = d\,\frac{D-a}{D\cdot e^{\psi}-a}\,;\quad \psi = \frac{12K}{r^2}\cdot t\cdot\frac{D-a}{D} \tag{10}$$

Here C_n is the sugar loss in percent on beets, C_o is the percent sugar of the beets, d is the specific weight of the beets, D is the draft in Kg/Kg, a is the juice volume in the cossettes, K is a diffusion constant, r is the thickness of the cossettes in mm, and t is the active diffusion time after the completion of denaturization of the cells.

Brüniche-Olsen (9, 10) developed a more complicated formula in which he made allowance for the change of the juice concentration in the cossettes during the extraction process. Schneider uses the general formula (46)

$$\frac{C_o}{C_n} = \frac{D\cdot\Psi-1}{D-1} \tag{11}$$

*Theoretical equations give somewhat different values.

Table 6-1 Pressed Pulp Yield, % on Beets

Moisture in pressed pulp, %	Marc content in beets %	Sugar Content in Pressed Pulp 0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0	1.1	1.2	1.3	1.4	1.5
78	4.0	18.7	18.8	18.9	19.0	19.1	19.3	19.4	19.5	19.6	19.7	19.9	20.0	20.1
	4.5	21.0	21.1	21.3	21.4	21.5	21.7	21.8	21.9	22.1	22.2	22.4	22.5	22.6
	5.0	23.4	23.5	23.6	23.8	23.9	24.1	24.2	24.4	24.5	24.7	24.8	25.0	25.2
	5.5	25.7	25.8	26.0	26.2	26.3	26.5	26.6	26.8	27.0	27.1	27.3	27.5	27.7
79	4.0	19.6	19.7	19.9	20.0	20.1	20.2	20.4	20.5	20.6	20.8	20.9	21.0	21.2
	4.5	22.1	22.2	22.3	22.5	22.6	22.8	22.9	23.1	23.2	23.4	23.5	23.7	23.8
	5.0	24.5	24.7	24.8	25.0	25.1	25.3	25.5	25.6	25.8	26.0	26.1	26.3	26.5
	5.5	27.0	27.1	27.3	27.5	27.6	27.8	28.0	28.2	28.4	28.6	28.8	28.9	29.1
80	4.0	20.6	20.7	20.9	21.0	21.2	21.3	21.5	21.6	21.8	21.9	22.1	22.2	22.4
	4.5	23.2	23.3	23.5	23.7	23.8	24.0	24.1	24.3	24.5	24.6	24.8	25.0	25.2
	5.0	25.8	25.9	26.1	26.3	26.5	26.6	26.8	27.0	27.2	27.4	27.6	27.8	28.0
	5.5	28.4	28.5	28.7	28.9	29.1	29.3	29.5	29.7	29.9	30.1	30.3	30.5	30.8
81	4.0	21.7	21.9	22.0	22.2	22.3	22.5	22.7	22.8	23.0	23.2	23.4	23.5	23.7
	4.5	24.5	24.6	24.8	25.0	25.1	25.3	25.5	25.7	25.9	26.1	26.3	26.5	26.7
	5.0	27.2	27.4	27.5	27.7	27.9	28.1	28.3	28.5	28.8	29.0	29.2	29.4	29.6
	5.5	29.9	30.1	30.3	30.5	30.7	31.0	31.2	31.4	31.6	31.9	32.1	32.3	32.6
82	4.0	23.0	23.1	23.3	23.5	23.7	23.9	24.0	24.2	24.4	24.6	24.8	25.0	25.2
	4.5	25.9	26.0	26.2	26.4	26.6	26.8	27.0	27.2	27.5	27.7	27.9	28.1	28.3
	5.0	28.7	28.9	29.2	29.4	29.6	29.8	30.0	30.3	30.5	30.8	31.0	31.2	31.5
	5.5	31.6	31.8	32.1	32.3	32.5	32.8	33.1	33.3	33.6	33.8	34.1	34.4	34.6
83	4.0	24.4	24.6	24.8	25.0	25.2	25.4	25.6	25.8	26.0	26.2	26.4	26.6	26.9
	4.5	27.4	27.6	27.9	28.1	28.3	28.5	28.8	29.0	29.2	29.5	29.7	30.0	30.2
	5.0	30.5	30.7	31.0	31.2	31.4	31.7	32.0	32.2	32.5	32.8	33.0	33.3	33.6
	5.5	33.5	33.8	34.1	34.3	34.6	34.9	35.2	35.4	35.7	36.0	36.4	36.6	37.0
84	4.0	26.0	26.2	26.4	26.6	26.8	27.1	27.3	27.5	27.8	28.1	28.3	28.6	28.8
	4.5	29.2	29.5	29.7	30.0	30.2	30.5	30.7	31.0	31.4	31.6	31.8	32.1	32.4
	5.0	32.5	32.7	33.0	33.3	33.6	33.9	34.2	34.4	34.7	35.1	35.4	35.7	36.0
	5.5	35.7	36.0	36.9	36.3	36.9	37.2	37.6	37.9	38.2	38.6	38.9	39.3	39.6
85	4.0	27.8	28.0	28.3	28.5	28.8	29.0	29.3	29.6	29.9	30.2	30.5	30.7	31.1
	4.5	31.3	31.5	31.8	32.1	32.4	32.7	33.0	33.3	33.6	33.9	34.3	34.6	34.9
	5.0	34.7	35.0	35.3	35.7	36.0	36.3	36.7	37.0	37.3	37.7	38.1	38.4	38.8
	5.5	38.2	38.5	38.9	39.2	39.6	39.9	40.3	40.7	41.1	41.5	41.9	42.3	42.7
86	4.0	29.9	30.1	30.4	30.7	31.0	31.3	31.6	31.9	32.3	32.6	33.0	33.3	33.7
	4.5	33.6	33.9	34.2	34.6	34.9	35.2	35.6	35.9	36.3	36.7	37.1	37.5	37.9
	5.0	37.3	37.7	38.0	38.4	38.8	39.2	39.6	39.9	40.4	40.8	41.2	41.6	42.1
	5.5	41.0	41.4	41.8	42.2	42.6	43.1	43.5	43.9	44.4	44.9	45.3	45.8	46.3

Table 6-2 Pulp Sugar Calculated as Fraction of Sugar in Original Cossettes

Draft	KP										
	0	1	2	3	4	5	6	7	8	9	10
100	1.0000	.2859	.1555	.1012	.0718	.0535	.0412	.0324	.0259	.0209	.0171
102	1.0000	.2816	.1496	.0951	.0657	.0477	.0457	.0273	.0212	.0166	.0131
104	1.0000	.2770	.1442	.0895	.0603	.0425	.0309	.0230	.0173	.0131	.0101
106	1.0000	.2731	.1391	.0842	.0553	.0380	.0269	.0193	.0141	.0104	.0077
108	1.0000	.2694	.1343	.0794	.0508	.0340	.0233	.0163	.0115	.0082	.0059
110	1.0000	.2655	.1297	.0749	.0468	.0304	.0203	.0138	.0094	.0065	.0045
112	1.0000	.2620	.1253	.0707	.0431	.0273	.0177	.0116	.0077	.0052	.0035
114	1.0000	.2586	.1212	.0669	.0397	.0245	.0154	.0099	.0064	.0041	.0027
116	1.0000	.2553	.1173	.0633	.0367	.0220	.0135	.0084	.0053	.0033	.0021
118	1.0000	.2521	.1136	.0600	.0339	.0198	.0118	.0071	.0043	.0027	.0016
120	1.0000	.2489	.1101	.0568	.0314	.0179	.0104	.0061	.0036	.0022	.0013
122	1.0000	.2460	.1068	.0539	.0291	.0162	.0092	.0052	.0030	.0017	.0010
124	1.0000	.2432	.1036	.0513	.0270	.0147	.0081	.0045	.0025	.0014	.0008
126	1.0000	.2404	.1006	.0488	.0251	.0133	.0072	.0039	.0021	.0012	.0006
128	1.0000	.2377	.0978	.0465	.0234	.0121	.0064	.0034	.0018	.0009	.0005
130	1.0000	.2351	.0951	.0443	.0218	.0110	.0057	.0029	.0015	.0008	.0004
132	1.0000	.2326	.0926	.0422	.0204	.0101	.0050	.0025	.0013	.0007	.0003
134	1.0000	.2302	.0900	.0403	.0190	.0092	.0045	.0022	.0011	.0006	.0003
136	1.0000	.2279	.0877	.0386	.0179	.0084	.0040	.0019	.0009	.0005	.0002
138	1.0000	.2257	.0855	.0369	.0167	.0078	.0036	.0017	.0008	.0004	.0002
140	1.0000	.2235	.0833	.0453	.0157	.0071	.0033	.0015	.0007	.0003	.0001
142	1.0000	.2214	.0813	.0339	.0148	.0066	.0030	.0013	.0006	.0003	.0001
144	1.0000	.2194	.0794	.0325	.0139	.0061	.0027	.0012	.0005	.0002	.0001
146	1.0000	.2173	.0775	.0312	.0131	.0056	.0024	.0010	.0005	.0002	.0001
148	1.0000	.2154	.0757	.0300	.0124	.0052	.0022	.0009	.0004	.0002	.0001
150	1.0000	.2136	.0740	.0288	.0117	.0048	.0020	.0008	.0003	.0001	.0001
152	1.0000	.2118	.0723	.0277	.0111	.0045	.0018	.0007	.0003	.0001	.0001
154	1.0000	.2101	.0708	.0267	.0105	.0042	.0017	.0007	.0003	.0001	.0000
156	1.0000	.2084	.0693	.0258	.0099	.0039	.0015	.0006	.0002	.0001	.0000
158	1.0000	.2067	.0697	.0248	.0094	.0036	.0014	.0005	.0002	.0001	.0000
160	1.0000	.2051	.0665	.0240	.0090	.0034	.0013	.0005	.0002	.0001	.0000

Table 6-3 Values of Θ for Various Values of t°C, for Use with Silin's Formula (8)

t°	Θ	t°	Θ	t°	Θ	t°	Θ
20	29.1	40	47.7	60	70.6	80	97.8
22	30.7	42	49.8	62	73.2	82	100.8
24	32.4	44	52.0	64	75.8	84	103.8
26	34.2	46	54.2	66	78.4	86	106.8
28	36.0	48	56.4	68	81.0	88	109.9
30	37.8	50	58.6	70	83.6	90	113.0
32	39.6	52	60.9	72	86.3	92	116.1
34	41.5	54	63.2	74	89.1	94	119.3
36	43.5	56	65.6	76	91.9	96	122.5
38	45.6	58	68.1	78	94.8	98	125.7

Ψ is a function of the extraction process used. Formulas for Ψ are given for continuous countercurrent extraction, discontinuous countercurrent extraction, discontinuous countercurrent with concurrent flow in the single stages, and for discontinuous countercurrent flow with a crosscurrent flow in the single stages.

Other mathematical treatments of diffusion have been published by Dörfeldt (16), Kollman (26), MacDonald (27), Mehrle (29), Rodgers (41), Slavicek (55), and Van Ginneken (60).

Confirmation of Theory

Brüniche-Olsen (9, 10) investigated the diffusion coefficient for cossettes of different thickness over a wide range. He found Fick's law, equation (1) to be valid for cossettes with a thickness of over 4 mm. For thinner cossettes he found decreasing values for the diffusion coefficient. His findings were confirmed by other research (46). This inconsistency of the diffusion coefficient is believed to be caused by the nonhomogeneous nature of the cell structure. The dimensions of the diffusion coefficient, sq cm/min, demand a homogeneous system, in which the resistance to diffusion is uniform over the entire diffusion length. However, sugar and other substances must diffuse first through the very small openings in the cell walls and then through intercellular spaces and the larger openings of the vascular cell system. Therefore, there is a high resistance to diffusion over a short distance and a lower resistance to diffusion over a much longer distance. In thicker cossettes, the diffusion over the long distance is rate-determining, and the diffusion coefficient is constant according to Fick's law. In thinner cossettes the higher resistance to diffusion of the cell wall becomes rate-determining, and consequently, the diffusion coefficient decreases. The rate of diffusion also depends on the beet material. It has been shown that the diffusion coefficient changes with the time of the beet in storage (14, 17). Rathge (37-39) states that diffusion alone cannot completely explain some aspects of sugar extraction. From experiments he concludes that water enters the cells after denaturizing by osmotic force and replaces the sugar juice, which is driven out through the larger cell openings. He, therefore, prefers to call the process of sugar extraction a liquid exchange rather than diffusion.

Schliephake (43) investigated the mechanism of sugar extraction, especially during the initial stages. He defined three basic phases: osmosis, denaturization, and diffusion. During the initial phase water enters the cell by osmosis. The denaturization increases the permeability of the cell wall and the juice-water mixture is forced to flow out of the cell by the sudden change of the cell pressure. Only after the completion of those two initial reactions, can diffusion be considered the rate-determining process. The duration of the initial phase depends on the extraction temperature and corresponds to the time needed for denaturization. During that time, less than five minutes under normal operating conditions, more than 20% of the sugar is extracted.

Figure 6-4 shows the three basic phases, as indicated by the change of the relative concentrations of sugar and water in the beet slices, during the extraction

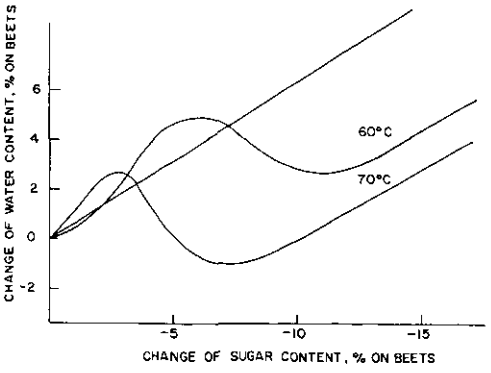

Figure 6-4. Change of water, with change of sugar concentration in the beet during extraction, with water at 60° and 70°C (43).

with water, at two temperatures. The line with the arrow represents the theoretical extraction, during which the water and sugar concentration should remain nearly constant relative to each other throughout the process, if diffusion is the only rate-determining factor. The maxima and the minima of the curves indicate the completion of the first two stages, the osmosis and the denaturization (43).

Elements of Extraction

Cossettes. The size and the physical condition of the cossettes are of great importance for diffuser performance. Since time is required for sugar to diffuse through a given distance, advantage is gained by shortening the distance. The most widely used parameter to measure the size of the cossettes is the Silin number. The values found usually vary between 7 and 20. One factor limiting the size of the cossettes is the diffuser design. The batch-type Robert battery, with no mechanical agitation, can tolerate very fine cossettes, for which Silin numbers of 20 and higher are not unusual. Some continuous diffusers, because of the continual movement of the cossettes, can tolerate cossettes no finer than a Silin number of 15.

In general, coarser cossettes require more heat, longer diffusion time, or a higher draft to obtain the same extraction as finer cossettes. Short cossette particles and fine pulp seriously interfere with the diffuser operation by plugging the diffuser screens. They may also hinder the movement of juice and cossettes by interfering with the permeability of the cossette mass.

Bjerkhög (6) introduced the cossette factor, sometimes called the Swedish number, which is the ratio of the weights of cossettes longer than 5 cm to those shorter than 1 cm. A value between 15 and 30 is considered satisfactory.

Figure 6-5 indicates the rapid decline of the permeability of a cossette column containing cossettes with a cossette factor lower than 10.

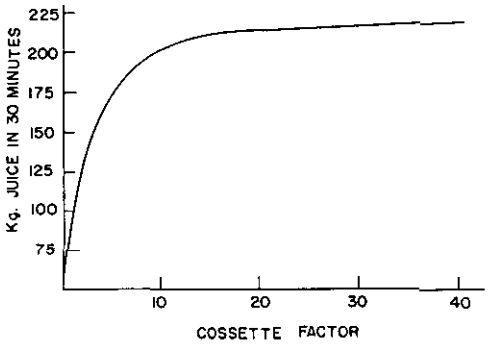

Figure 6-5. Cossette permeability, as shown by juice flow through a column of cossettes of unspecified size, vs. "cossette factor," or "Swedish number" (6).

Diffusion Water. One of the most important factors in the diffusion process is the quality of the diffusion water. Before the introduction of continuous diffusers, this water normally consisted only of condenser water and condensates. Earlier attempts to make use of the pulp press water were not too successful on account of its colloid content, and provisions had to be made to first remove the colloidal matter. For this purpose the Wintzel-Lauritzson process was developed in Sweden (2,62), in which the water is acidified to a pH of 3.2 with sulfuric acid. The flocculated materials are removed by settling and returned to the presses. The acid requirement is approximately 0.025% on beets (2).

There are fewer problems when returning pulp press water to continuous diffusers (64). The higher temperatures during denaturing in continuous diffusers appear to be more favorable for the coagulation of the proteins. No other treatment than screening, sterilizing, and the removal of fine sand are required.

Pulp press water normally contains between 0.5 and 1.5% sugar and should be returned to a point within the diffuser where the juice solids concentration is about equal to that of the press water. Although the purity of the press water is quite low, its effect on the quality of the diffusion juice appears to be small. This is due to the fact that the diffusion process does not have to be carried out to complete exhaustion of the cossettes. The greatest advantage in returning the press water is the considerable decrease of the BOD load of the waste water. In addition one can expect a 10% higher pulp yield.

The amount of fresh water needed to make up the total supply of water is given by the formula:

$$\%\ \text{draft} + \%\ \text{pressed pulp} - 100 = \%\ \text{fresh water (on beets)}. \qquad (12)$$

In most factories, condenser water and condensates are still used as makeup water. This water is always alkaline due to the presence of ammonia. The alkalinity of this water will seriously affect the diffusion process by dissolving otherwise insoluble cell substances, especially pectins (25); see pp. 353–7. Pulp pressing is made more difficult due to softening of the pulp, and problems during filtration and purification can arise due to dissolved impurities. The pH of the water should be lowered to from 5.6 to 6.0 pH. In North America liquid sulfur dioxide is almost exclusively used for this purpose (see p. 729).

In Europe hydrochloric and sulfuric acids are widely used, but have had slight use in North America. Proposals have been made to use cation exchangers to remove the ammonia present (3,4,13,67). However, the use of liquid sulfur dioxide carries the additional benefit of partially sterilizing the water. Condenser water, if not sterilized, can give rise to serious infections in the diffuser. The required usage in Michigan varies between 0.10 and 0.20 lb of liquid sulfur dioxide per ton of beets.

Impurities in the supply water should be kept as low as possible. Although the greater part of the impurities is carried out with the pulp, a certain amount will be transferred to the diffusion juice (22,23,32,48). The amount of the impurities carried into the diffusion juice is related to its concentration in the cossettes and in the water (1). The results of the published investigations indicate partitioning of the impurities between the pulp and the extracting liquid. They also suggest cation-exchange reactions involving sodium, potassium, and calcium ions.

Carruthers (11) explains this ion exchange reaction as due to the presence of polyuronides (polymers of acids formed by the oxidation of the primary alcohol group of sugars, such as glucuronic acid from glucose, and galacturonic acid from galactose). Anion exchange reactions do not appear to take place. The relative concentrations of anions in pulp and juice are due to partitioning, as above.

A theoretical study of the distribution of additives has been made by Stitt (59), who gives the mathematical relations for the partitioning of additives in Robert batteries and in continuous countercurrent diffusers.

Temperature. The diffusion temperature must be selected considering the following factors: denaturization, diffusion, the thermal behavior of the cell wall, enzymatic reactions, bacterial activity, and pressability of the pulp.

Denaturization of the protoplasm takes place at temperatures above 50 °C. At higher temperatures this process is accelerated. The cell wall becomes more permeable and the coagulation of proteins becomes more favorable for the extraction process. At temperatures above 70 °C denaturization is completed in a very short time (Figure 6-1). The rate of diffusion increases directly with temperature. The sugar loss in exhausted pulp is proportional to the product of time, temperature, draft, and cossette size. Higher temperatures in the diffuser make it possible either to lower the draft, or to increase the size of cossettes, or to shorten the contact time between juice and cossettes without increasing the sugar loss. The relation between temperature and diffusion coefficient is given by the Einstein formula (2).

The thermal behavior of the cell wall, especially of its pectic components, is the limiting factor. The saponification of the pectic substance of the cell wall is increased with temperature. The cell wall then loses its rigidity. The results are higher moisture content in the pressed pulp, impaired diffusion inside and outside the cossettes, and plugging of screens through increased fine pulp content and mushy cossettes. Higher pectin concentrations in the diffusion juice can also cause some problems during carbonation filtration and the crystallization of low purity massecuite, requiring considerably more lime in defecation. Enzymatic inversion of sucrose during the extraction process is considered responsible for an increase of invert in the diffusion juice over that in the beets (33,61). This is more evident while processing stored beets. Recent investigations have shown the presence of at least two enzymes with different optimal pH values and temperatures. One enzyme with a pH optimum above 8 and a temperature optimum over 45 °C, is of little importance at the prevailing conditions in the factory. The other enzyme, with its highest activity at pH 5-6 and 60 °C is of far greater significance for the diffusion process. It loses its activity at temperatures above 65 °C, but unless the cossettes are steam treated for about five minutes, some activity can still be observed at temperatures of 70 °C (33). Another decisive factor is the retention time of the cossette-juice mixture in the diffuser. Long retention times will add to the thermal degradation of the cell wall and to the danger of bacterial infection. Differences in the beet material caused by climatic and geographical factors make it impossible to recommend specific temperatures. The most desirable temperature has to be found by observing the condition of the exhausted pulp and by taking the factors mentioned above into consideration.

Draft. The ratio of the weight of diffusion juice drawn from the diffuser to the weight of the cossettes introduced, expressed as percent, is called the *draft.*

$$\text{Draft} = \frac{\text{weight of diffusion juice}}{\text{weight of beets}} \times 100 \qquad (13)$$

Drafts used vary between 100 and 150. Sugar lost in pulp and draft vary inversely. The interdependence of draft and sugar loss can be predicted from the Silin formula, (9), as shown in Figure 6-6.

It is evident from the graph that a considerable amount of water, about 5-10% on beets, has to be added and later evaporated to reduce the sugar loss from 0.25% to 0.15% on beets. Also the purity of the clarified juice is slightly reduced (33). Steam cost and evaporator capacity have to be balanced against the additional sugar obtained (68).

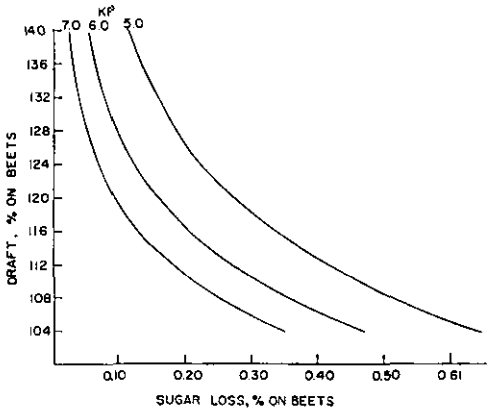

Figure 6-6. Sugar loss vs. draft, according to Silin, for KP values of 5.0, 6.0, and 7.0.

Closely related to the draft is the juice velocity, which in the case of a battery is called the pressure rate (65). According to Wrangel (66), the juice velocity may be calculated by the formula:

$$v = \frac{V}{At}\left(1 + \frac{WD_v}{V - W}\right) \tag{14}$$

where v is the juice velocity in meters per minute, V is the diffuser volume in cubic meters, A is the diffuser cross-sectional area in square meters, t is the time in minutes, W is the weight of cossettes in metric tons per diffuser, and D_v is the draft in volume percent/100. $WD_v/(V-W)$ is the correction factor for the cossette volume. Freund (20,21) determined the juice velocities relative to the cossette movement for different continuous diffuser types. Most of the investigated systems had juice velocities falling between laminar and turbulent flow. His paper also contains an excellent bibliography on this subject.

pH. The cell juice pH of sound beets varies between 6.3 and 6.6. In the absence of a diffuser infection the pH of the diffusion juice is about 0.1 unit lower than the pH of the cell juice. Within the diffuser a lower pH may have beneficial effects. At a pH of 5.5 the diffusion of impurities appears to be at a lower level (24). This might be related to the degradation of the pectins at higher pH. The exhausted pulp is firmer and permits more efficient removal of the water in the pulp presses. Considering corrosion, a pH of 6.0 or slightly below 6.0 within the diffuser appears to be a good compromise. To obtain this, not only the supply water but also the press water may have to be acidified.

Microbiological Action. Before the introduction of the continuous diffusers very little attention was given to the bacterial activity in the diffuser. Any bacterial activity in a diffuser system can lead to a serious sugar loss and lower the quality of the diffusion juice. Bacterial activity in the diffuser can be controlled either by keeping the temperature throughout the diffuser above 75 °C or by the use of bactericides such as formaldehyde, sulfur dioxide, chlorine, and others. In most factories the bactericide is used as a shock treatment. The amount and frequency of this treatment has to be investigated in each individual operation. The necessary addition normally varies between 5 and 15 gallons of 30% formaldehyde per treatment for a 3000 ton diffuser. Please see Chapter XXIII for an extended treatment of this subject.

Diffusion Juice. The juice drawn from the diffuser contains a considerable amount of colloidal matter besides a large number of fine pulp particles, which are difficult to remove by screening. It is desirable to eliminate most of the suspended matter before the juice is sent to the purification process. The color of the juice is usually gray and changes to a dark gray or almost black hue on contact with the air. This darkening is caused by enzymatic reactions and is accelerated in the presence of iron. However, the color of the diffusion juice is of little importance as it is removed without difficulty during purification.

The nonsucrose content of the diffusion juice is related to the quality of the beets and the conditions under which the sugar is extracted in the factory. In some beet-growing areas, storage after harvesting has more effect on the quality of the diffusion juice than normal variations of the growing conditions as experienced from year to year.

Table 6-4 indicates the varying acid concentrations in diffusion juice from different areas of the United States as reported by Stark and co-workers (58) (see also p. 42. The following percentages of the mineral components are extracted from the beet: 80-90% of K and Na, 10-30% of Ca, 60-80% of Mg, and about 80% PO_4^{3-} and SO_4^{2-}. Amino acids and betaine are almost completely extracted and the protein and pectin content of the juice is a function of pH and temperature. At higher diffusion temperatures more pectins are dissolved, but the protein content appears to be at its lowest level at about 70 °C.

Table 6-4 Concentrations of Some Acids in Diffusion Juice, 1950 Campaign, mg/l, at 10% Solids.

Factory	Lactic	Glycolic	Malic	Oxalic	Citric	Chloride	Sulfate	Phosphate
Betteravia, California	1030	215	48	645	592	606	292	651
Brighton, Colorado	82	23	167	267	473	341	262	386
Carrollton, Michigan	775	92	96	221	233	223	211	436
Centerfield, Utah	208	88	260	94	317	392	300	174
Manteca, California	288	nil	191	771	531	365	174	618
Moorhead, Minnesota	1610	26	99	211	621	283	126	331
Mini-Cassia, Idaho	180	nil	145	nil	326	186	147	305
Sidney, Montana	585	39	112	200	744	142	160	434
Toppenish, Wash.	335	175	250	261	702	187	142	369

The following organic nonamino acids are present in larger quantities: citric, oxalic, malic, acetic, and lactic acid. There are only minute amounts of lactic acid found in healthy beets and its presence in the diffusion juice usually indicates a thermophilic infection in the diffuser (Table 6-5) (57).

Table 6-5 Lactic Acid Contents in Cossettes and Diffusion Juice, 1951 (mg/l at 10% sucrose)

Area	Colorado	Michigan	California	Minnesota
Cossettes	5	5	5	6
Diffusion Juice	125	310	412	66

The invert content in the juice normally ranges from 0.4 to 0.8% on solids. Higher concentrations are found during processing of stored beets [Table 6-6. See also (40)]. In northern beet growing areas raffinose and kestose are also present in the juices. They show a considerable increase during the long beet storage customary in these areas. It is not unusual, especially during the later part of the campaigns, to find raffinose and kestose contents of 4.0% and 2.0% respectively, in the molasses, which corresponds to a concentration of 0.2% and 0.1% in the diffusion juice. The presence of these optically-active components makes the purity determinations in the diffusion juice very unreliable.

Table 6-6 Invert, % on Solids in Diffusion Juice, Weekly Averages, Michigan Sugar Co.

	1966				1967			
October	0.68,	0.66,	0.78,	0.61	0.53,	0.76,	0.75	—
November	0.78,	1.10,	1.06	—	0.77,	0.70,	0.71,	0.75
December	1.14,	1.32,	1.08,	170	0.97,	1.23,	1.62,	2.11
January	160,	1.30,	1.63		1.50,	1.35,	1.47,	1.64

The Equipment of Diffusion

The Battery

For almost 100 years the Robert battery had been the principal diffuser to extract sugar from the beet, before its replacement by continuous diffusers, developed to success during the last 20 years.

The Robert battery is usually made of 11 to 14 identical cells, which are arranged in straight lines, or in a circle. Each cell is fitted with a thermometer, pressure gauge, heater (calorizator), water valve, circulation valve, and juice valve. Each cell has a capacity of from 3 to 6.5 tons of sliced beets, depending upon its size and the degree of packing. For identification purposes, the cells are numbered 1 to 14. Operationally, in the circulation series, the last cell filled with cossettes and from which the diffusion juice is drawn is considered to be the first or head cell. Juice circulates through the cell from top to bottom, the cossettes being retained by the screens at the bottom. Chains are hung within the cell to bear part of the weight of the cossettes and to prevent them from packing too tightly in the bottom against the screen.

The cells are connected together in a series, as shown in Figure 6-7, so that the water pumped into the tail cell will pass through the circulating cells in turn, to be drawn off from the head cell. Eleven of the 14 cells usually are in active diffusion, for one of the cells is either "drawing off" or "juicing up," another cell is "washing," and the third cell is "filling." "Washing" means that the cell is being hosed out with high pressure water in preparation to being filled with fresh cossettes. "Juicing up," "coming up," or "diffusing" means that the cell filled with fresh cossettes is also being filled with juice from the previous "first" cell. A cell freshly filled with cossettes is filled with the juice from the bottom up to expel air. When this cell is full of juice, the direction of the juice flow through it is reversed, so that the already enriched juice retraverses the cossettes it has just passed through. This reversal of direction represents both a loss in time and extraction, since a richer juice comes in contact with the cossettes in the bottom of the cell and thus gives up some of its sugar by reverse diffusion.

Normal flow in the cells is from top to bottom, chiefly for mechanical reasons, since a large top door with adequate screen area would be very difficult to handle. It has also been noted that small pieces of cossettes tend to float and would probably plug upper screens if the flow were from bottom to top.

The advantages of this batch-type diffuser come from the fact that the cells are sealed off from air, thus reducing corrosion to equipment, and that the periodic emptying and cleaning reduce bacterial activity. Its chief disadvantages are the high labor requirements and the loss of sugar in pulp water.

The cells in a circular battery are filled with cossettes from a revolvable chute which can be swung from cell to cell; in a straight-line battery they are filled from a belt, or drag conveyor.

After a cell has become successively first, second, etc. and essentially all the sugar has been extracted from its cossettes, it is cut off from the series and its contents dumped into a pit common to all the cells. The pulp water it contains is dumped with the pulp, so that with this type of battery there are losses of sugar in pulp water as well as in pulp. In operating the battery, the cell temperatures are carefully controlled. The juice is heated between cells by the calorizators or by direct steam injection. Equally important is uniformity of draw, with high losses as the penalty for irregularity. Gas formation frequently occurs with dirty beets, from microorganisms carried in with the soil. Defective valves may cause many battery troubles. Leaking water valves dilute the juices, while leaking cell doors are frequently responsible for many so-called "unknown" battery losses, and increase the diffusion loss in factories returning pulp water to the batteries.

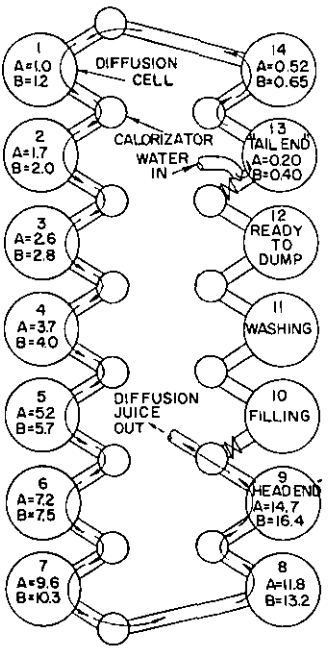

Figure 6-7. Simplified flow diagram of a Robert battery, *with typical analytical figures* for the materials in the individual cells. Note: A = % sugar in juice flowing through cell; and B = % sugar in juice contained within cossettes.

Typical analyses (30) of juice samples taken from the individual cells of an operating battery show that the purity of the juice in the first six cells is fairly constant, but may drop to values below 50 in the last cell. However, the net effect of including this low purity juice is quite small, since the solids content is so low. Table 6-7 lists these data. Note that about half the sugar is extracted in the first cell.

Table 6-7 Juice Characteristics within Individual Robert Battery Cells

Cell No.	Temp. °C	Grams Sugar per 100 ml.	Grams Solids per 100 ml.	Purity	Cumulative % Extracted
1	81	13.92	16.55	84.1	49.6
2	85	7.01	8.32	84.3	63.0
3	85	5.15	6.16	83.6	72.3
4	83	3.85	4.62	83.3	80.7
5	79	2.69	3.28	82.0	87.9
6	69	1.69	2.07	81.6	92.5
7	70	1.04	1.34	77.6	95.7
8	54	0.60	0.78	76.9	98.9
9	55	0.16	0.29	55.2	99.8
10	54	0.03	0.14	23.6	100.0

Continuous Diffusers

The Bergé Continuous Diffuser. The continuous diffuser designed by Julien Bergé is one of the earlier successful types of continuous diffusers. It is a large revolving drum, separated into "cells" by a helix attached to the interior surface. As the drum with its helix revolves, the juice, which stays at the bottom, is transported from the tail end to the head end. Thus the cell actually moves, but it is more convenient to consider the cell as the location of one turn of the drum. If the screw has 30 turns, 30 revolutions of the drum are required to move the juice from one end to the other, and the diffuser is considered to have 30 cells. Fixed to the cylinder are grids, which, revolving with the drum, sweep the cossettes up until they slide off and fall into the next cell. Thus the cossettes and juice travel in contrary directions through the cylinder. The action of the diffuser is shown in Figure 6-8a. A considerable amount of literature exists concerning this apparatus (5,8,12,42,50).

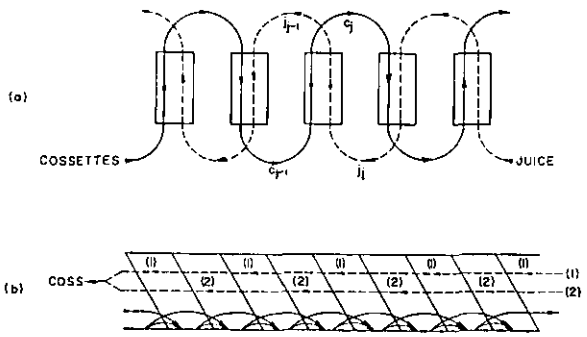

Figure 6-8. a) Flow of cossettes (_____) and juice (....) through a cell-type continuous diffuser. b) Plan for using two streams of juice against one of cossettes.

The R.T.-Smet Continous Diffuser (7,56). See Figure 6-9. This is a development of the Bergé diffuser, which is changed to a double helix, or double-threaded screw, each screw having twice the pitch of the single helix screw. The grids on the R.T. diffusers are basket-shaped and offer a large filtering area. The apparatus is so arranged that the water introduced into the tail end of the drum will reach the other end of the drum after a number of revolutions equal to half the number of compartments along the bottom of the drum. The cossettes again travel in a countercurrent direction, but at half the rate of the juice. The action of this diffuser is shown in Figure 6-8b. The shorter juice retention time is said to decrease sugar loss from fermentation and bacterial action. A diffuser with a nominal capacity of 2700 tons per day has the following dimensions: the scalding tank is 11 ft long, 3 ft wide and slopes toward the 22 in. diameter scalding pipe; the drum, driven by a 60 hp motor, is 89 ft long with a diameter of 17 ft, and contains 27 compartments. Its normal rotating speed is 25 rph, and can be varied from 18 to 27 rph.

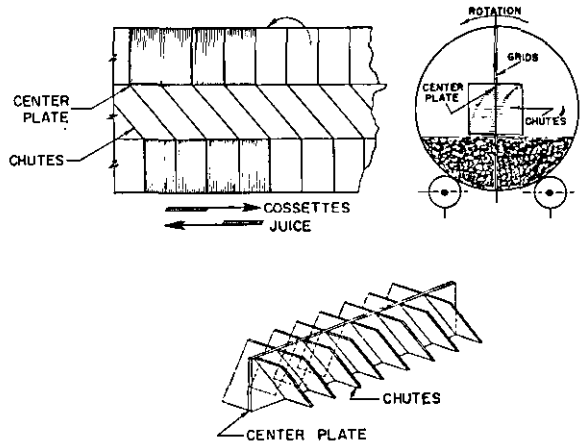

Figure 6-9. Explanatory drawings of the R.T.-Smet continuous diffuser.

The diameter of the drum is enlarged at the cossette inlet side. The enlarged shell is perforated and rotates inside a fixed head, which is not connected with the drum. The cossettes are flushed through the scalding pipe into the mobile head. Two grids are mounted inside this mobile head and carry the cossettes into the drum with each turn. Scalding juice, together with juice from the diffuser, flows through the perforated head into the circulating juice tank. Fresh water is introduced through the hollow shaft into the second compartment and pulp press water is added to the fourth compartment, counting from the tail end. Diffusion juice, corresponding to the draft, is pumped from the circulating juice tank to the process. The remaining juice is heated and used for scalding and flushing the cossettes to the diffuser. As there are no moving parts inside the drum, most mechanical repairs can be carried out without emptying the drum. Recirculation in this type of diffuser is minimal, since there is no mixing other than that caused by the sliding of the cossettes and juice along the bottom, the carryback of juice adhering to the drained pulp, and the juice wetting the walls. The desired temperature in the drum is provided by the heat introduced with the cossettes and the supply water. Additional heat can be

obtained by injecting steam into the drum. The positive and controllable movement of the cossettes and juice make this diffuser extremely adaptable to varying rates of slice.

In 1969, a modified design of the R.T. drum diffuser, the R.T.–4, was first installed in a French factory. The redesigned internal structure produces a more continuous movement of the cossettes during the rotation of the drum. The capacity of the new drum is larger and the retention times of juice and cossettes are shorter (17a, 17b).

The Silver Continuous Diffusers (53)

Chain-Type. The first Silver continuous diffuser, called the "chain-type," consists of a series of U-shaped cells about 2 ft wide and 12 ft deep, and 21 in number. The cells, in effect, form a long bent tube, rectangular in cross section, which doubles back on itself to form two tiers, one above the other. Threaded through the cells are two large drag chains, running over sprockets, held about 12 ft apart by perforated steel-plate flights spaced at 20 in. intervals as shown in Figure 6-10. Overall dimensions of the 21-cell unit are: length about 77 ft; width about 24 ft; height about 30 ft. To reduce chain stress, the drag is driven at a number of points by individual motors. Each cell is set several inches higher than its predecessor, so that the juice may flow through the cells by gravity. Cossettes are fed into the diffuser at one end of the lower tier and are carried by the drag through the entire length of the diffuser and then discharged as pulp. The cells are so connected that juice and cossettes move concurrently within each cell, but countercurrently from cell to cell. Each cell is equipped at its outlet side with a slotted revolving cylindrical brass strainer, through which the juice passes on its way to the next cell. This strainer is continuously cleaned. The first three or four cells are steam-jacketed and are also equipped for direct steam injection into the tops of the compartments.

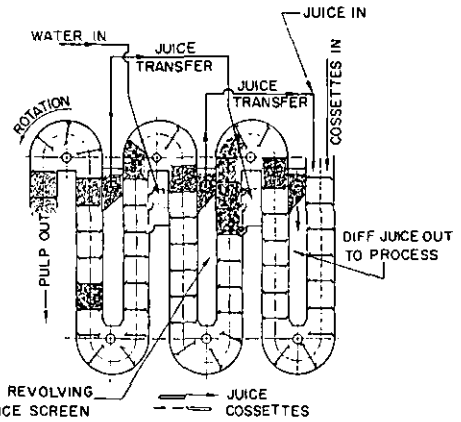

Figure 6-10. Diagram of Silver chain-type continuous diffuser.

For alteration of capacity and adjustment of cell fill, the alternating current motors driving the drag chains can be varied in speed, since the entire diffuser is powered by a variable-frequency, motor-generator unit. The supply water is proportioned by a flow-rate controller which is actuated by a continuous scale, weighing the cossettes into the diffuser. The ratio of supply water to beets can be varied by

the operator to compensate for the difference in the diffusion rates of different cossettes. It is necessary that the desired rate of slice be held as constant as possible to prevent continual adjustment of diffuser speed and possible flooding of the battery. The motors driving the drag, conveyors, and slicers are frequently electrically interlocked, so that when one stalls, all upstream are automatically shut off. In 1969 there were 10 chain-type units in operation in North America.

Scroll-Types. Another type of continuous diffuser manufactured by the Silver Engineering Works, Inc. is the scroll-type, in which the cossettes are pushed through tank-type cells by large scrolls, concurrent with the juice, while operation is countercurrent from cell to cell, as in the chain-type. Cossettes are transferred between cells by a "beet-wheel" type of lift. These two diffusers were the first successful installations in this country.

The Silver D.d.S. Slope Diffuser. This diffuser is the most widely used in this country, there being at least 35 units in operation in 1969. Its design is based on the original Danish Sugar Corporation construction, which was changed by Silver (52) to suit conditions in this country.

Figure 6-11a. Looking into a Silver D.d.S. slope diffuser. Scroll elements with screens. (Courtesy, Silver Engineering Works, Inc.)

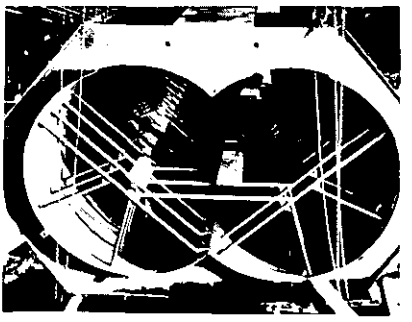

Figure 6-11b. End view of Silver D.d.S. slope diffuser, showing stationary breaker bars. *(Courtesy, Silver Engineering Works, Inc.)*

Two scrolls with two or three intermeshing flights each move the cossettes upwards through a trough which is inclined 11° to the horizontal. The flights of these scrolls consist of aluminum screens bolted to steel frames which are welded to a hollow shaft. The multiple flights are interrupted at intervals of about 20". At these points breaker bars are installed to loosen the pulp and to insure enough porosity for the extracting liquid to pass countercurrent through the cossettes.

Figure 6-12 is a typical flow diagram of the Silver slope diffuser. At the low end of the diffuser the cossettes are forced below the juice level by a double scroll. Fresh water and pulp press water are added to the top end and percolate through the moving cossette bed to the lower end of the diffuser. Screens at this end separate the juice from the freshly introduced cossettes. At the top end of the trough, the exhausted pulp is forced upwards and is removed by horizontal double scrolls to the sides of the diffuser.

The first D.d.S. diffusers were open at the top, and cover was provided through large hinged doors equipped with counterweights. The newer models were

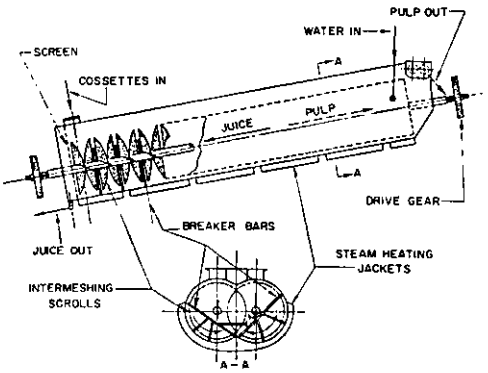

Figure 6-12a. Flow diagram of a Silver D.d.S. slope diffuser. ***(Courtesy, Silver Engineering Works, Inc.)***

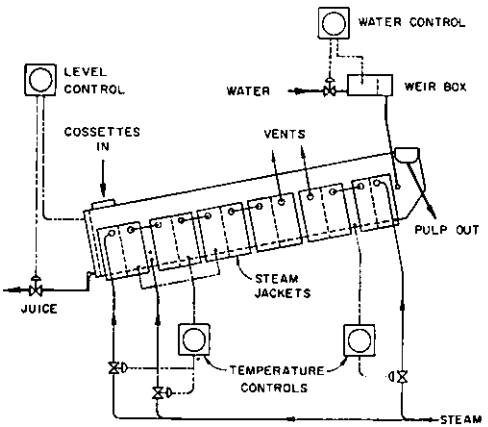

Figure 6-12b. Control diagram for a Silver D.d.S. slope diffuser. ***(Courtesy, Silver Engineering Works, Inc.)***

completely enclosed and the inside can be viewed through a number of Plexiglas windows.

The juice and cossette mixture is heated by steam jackets, which are arranged in sections and which cover more than half of the circumference of the trough. The diffusion juice draft is regulated through the addition of supply water which is controlled by the slice rate. The juice level at the cossette end is kept constant by a level controller.

Table 6-8 shows the dimensions of available D.d.S. diffusers.

Table 6-8 Dimensions of Silver D.d.S. Slope Diffusers

Nominal Capacity, tons per day	1600	2000	2500	3200	4000	5000
Height	26′-3″	27′- 2″	28′- 4″	29′- 8″	31′-10″	35′-10″
Width	18′-0″	19′- 3″	22′- 6″	23′-10″	27′- 6″	28′-10″
Length	68′-2″	70′-11″	72′- 0″	72′- 8″	75′- 1″	87′- 4″
Main Scroll Diameter	80″	90″	102″	115″	130″	144″
Number of flights	2	3	3	3	3	3
Final rpm of main scroll	1.27	.84	.85	.84	.85	.84
Connected hp	72.5	75	97.5	145	195	245

The Oliver-Morton Continuous Diffuser (28). This diffuser in some respects resembles the scroll-type Silver diffuser. The most important difference is in the method of transferring cossettes from cell to cell. Instead of a "beet-wheel" lifter, there is in each cell a series of six sets of fingers at the end of and attached to the scroll shaft. As the fingers revolve, they lift the cossettes from the cell and then pass through a stationary comb of grid bars, which takes the cossettes off the fingers. Beyond and adjacent to the transfer mechanism is a screen, covering the full end area of the cell, through which the juice flows. The cossettes and juice enter each cell at the same end and leave at the other end, but each is transferred to a different cell; the cossettes going one way and the juice the other. Thus the flow of cossettes is countercurrent to the juice from cell to cell, but concurrent within each cell (Figure 6-13).

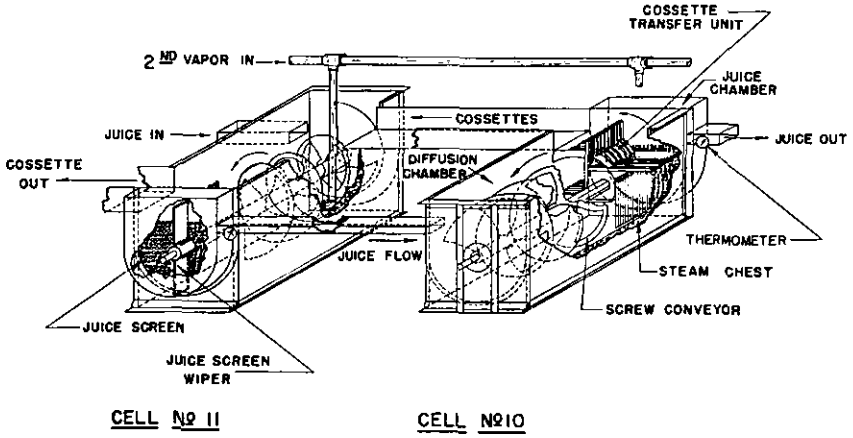

Figure 6-13. Diagram of two middle cells of an Oliver-Morton continuous diffuser.

Each pair of cells has its own driving motor, which cannot be started unless all the subsequent motors are running. The scroll speeds can be varied, but are usually operated at from 3 to 10 rpm, the speeds being adjusted to give the cell fills and uniformity of load desired. The cell fill is in the region of 15 to 20 lbs per cu ft. The draft and water-cossette ratio are controlled automatically when desired. In the Oliver-Morton several cells are steam-jacketed, so that temperatures over the whole diffuser are readily controlled. (See Figure 6-14). There are five units in use, all by factories of the Spreckels Sugar Company.

The B.M.A. Tower Diffuser. This diffuser has been developed from the earlier Hildebrandt diffuser (see First Edition, p. 165), which consisted of two vertical cylinders and a third connecting horizontal cylinder at the bottom. Each of the three cylinders was provided with a perforated scroll to move the cossettes through the cylinders at the desired rate (19). The B.M.A. tower diffuser (Figure 6-15) consists of a single vertical cylinder. Flights on a hollow conveyor shaft move the cossettes upward. A number of baffles are located on the cylinder wall, which are designed to induce an upward flow of the cossettes and to prevent the cossettes from rotating with the scroll. The angle to the horizontal of some of the baffles can be

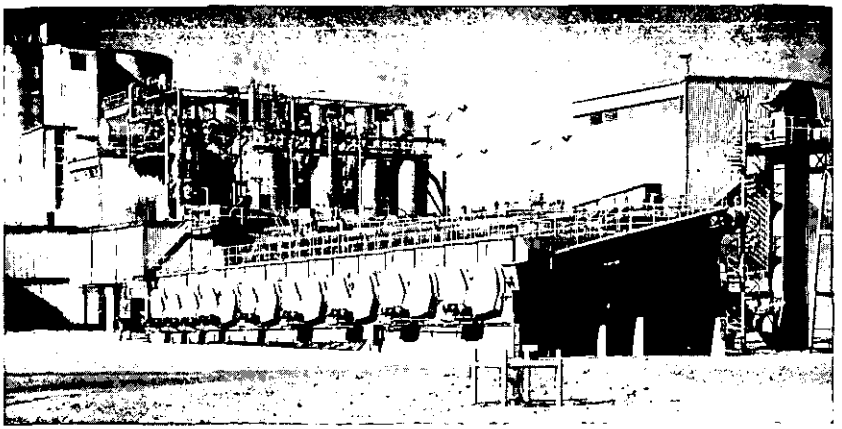

Figure 6-14. Oliver-Morton continuous diffuser at Chandler, Arizona. ***(Courtesy, Spreckels Sugar Company).***

altered during the operation in order to increase or decrease the fill of the tower. A similar effect is obtained by changing the speed of the conveyor shaft, which is usually run at about 0.40 rpm.

The cossettes are preheated outside of the diffuser in a cossette mixer. Two designs are offered: a concurrent flow mixer and a combination concurrent and countercurrent flow mixer. In the concurrent type the cossettes and the heated circulating juice enter the mixer at the same place. Diffusion juice—equal to the draft—and the cossettes-juice mixture are withdrawn from the opposite end of the mixer. The countercurrent mixer consists of a countercurrent and a concurrent flow section. Raw juice is added to the first section, cooled by the cossettes to a temperature which is about 12 °C higher than the cossette inlet temperature, and then withdrawn from the cossette inlet end. The preheated cossettes enter the concurrent mixing section at 65 °C and they are heated further by the circulating juice and then pumped into the bottom section of the tower.

The exhausted pulp is carried out of the diffuser by two scroll conveyors. Fresh water is introduced into the top row of the fixed baffles, approximately 2 ft below the pulp scrolls. Pulp press water is added through the third row of baffles from the top.

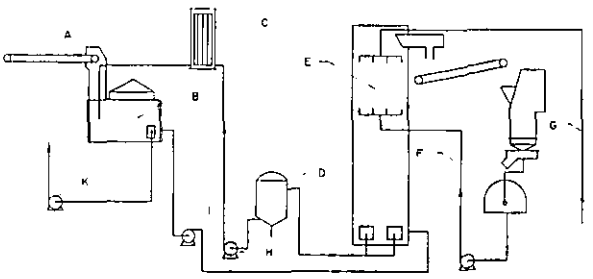

Figure 6-15. Flow diagram of a BMA tower diffuser. a) Cossette belt; b) Cossette scalder; c) Circulating juice heater; d) Sand catcher; e) Diffusion tower; f) Pulp press water; g) Supply water; h) Circulating juice pump; i) Cossette pump; k) Diffusion juice pump.

The circulating juice, which is withdrawn from the diffuser through the bottom and side screens, passes a sand catcher before it is pumped through a heater into the cossette mixer. The bottom screens are kept clean by scrapers, which are hinged to the lower flights of the conveyor.

Diffuser sizes and capacities are given in Table 6-9. The height can be increased by adding additional sections with corresponding increase in capacity.

Table 6-9 Dimensions of Standard BMA Tower Diffusers

Nominal capacity tons/day	Tower diameter feet	Effective diffusion length feet
935	9′ - 10″	45′ - 3″
1100	10′ - 10″	45′ - 3″
1320	11′ - 10″	45′ - 7″
1485	12′ - 6″	45′ - 7″
1650	13′ - 6″	45′ - 7″
1815	13′ - 9″	45′ - 7″
1980	14′ - 5″	45′ - 7″
2090	14′ - 9″	45′ - 11″
2200	15′ - 1″	45′ - 11″
2420	16′ - 1″	46′ - 2″
2750	17′ - 1″	46′ - 2″
3080	17′ - 9″	46′ - 2″
3300	18′ - 1″	46′ - 2″
3850	19′ - 0″	50′ - 6″
4400	20′ - 8″	50′ - 6″
4950	21′ - 4″	54′ - 6″
5500	22′ - 4″	54′ - 6″

The Buckau-Wolf Tower Diffuser. The general appearance and flow diagram (Figure 6-16) of this diffuser resemble the B.M.A. diffuser. Important differences are the cossette mixer and the design of the inside of the tower itself. The cossette mixer consists of a horizontal cylinder which is separated into a larger scroll section and a smaller mixer section. Juice from the bottom screens of the tower passes through a sand catcher and two-thirds of its volume is added directly to the mixer section and is pumped back to the tower together with the cossettes. The remaining tower juice and the circulating juice drawn from the scroll section of the mixer is pumped through a heater and then into the bottom of the scroll section. Part of the juice bypasses the heater and is fed through a foam destruction device into the cossette chute. Juice equal to the required draft is withdrawn from this end of the scroll section and pumped to the process.

The cossette-juice mixture enters the tower through the center shaft and a specially designed distributor which travels over the bottom screens.

The shape of the conveyor flights resembles an aircraft wing and these are designed to loosen and to move the cossettes upwards. All the baffle plates are installed at a fixed angle of 22° to the horizontal and they are secured to the cylinder wall and an inside ring. The exhausted pulp leaves the tower through a series of openings which are spaced around the top of the tower. The pulp is collected in a circular scraper conveyor and sent to a dewatering device.

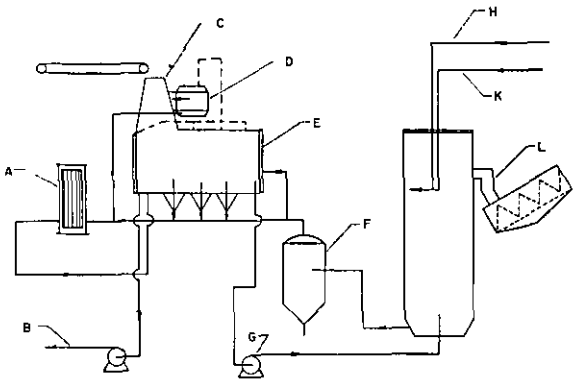

Figure 6-16. Flow diagram of a Buckau-Wolf tower diffuser. a) Circulating juice heater; b) Diffusion juice to process; c) Cossette inlet; d) Foam destroyer; e) Cossette scalder; f) Sand catcher; g) Cossette juice pump; h) Fresh water inlet; k) Pulp press water inlet; l) Pulp outlet.

Fresh water is sprayed on top of the cossette column and pulp press water enters the tower below the pulp discharge through the rotating flights. Table 6-10 gives the sizes and capacities of the various Buckau-Wolf towers available. In 1981 there was one unit in operation in the United States.

The DeSmet Diffuser. This Belgian diffuser of a rather unusual design has been installed in a number of factories in France, Italy, and Spain. A typical flow diagram is shown in Figure 6-17. The diffuser consists of a 100-ft-long horizontal conveyor screen which moves a 2- to 3-ft-thick layer of cossettes below a series of 18 juice distributors from which a curtain of juice is sprayed on the cossettes. The juice distributors are fed from pumps which are arranged in groups of three in one housing on a common shaft, and which are driven by one motor. Each pump receives the juice from the preceding sloped collecting hopper, which is installed between the

Table 6-10 Dimensions of Standard Buckau-Wolf Tower Diffusers

Nominal capacity, tons/day	Tower diameter, feet	Effective diffusion length, feet
880	9′ - 10″	33′ - 6″
1325	11′ - 6″	39′
1760	13′ - 1″	41′ - 5″
1980	13′ - 11″	36′ - 10″
2200	14′ - 9″	36′ - 6″
2650	16′ - 5″	38′ - 2″
3100	17′ - 3″	41′ - 3″
3300	18′	41′ - 4″
3750	19′	42′ - 8″
4000	19′ - 8″	42′ - 8″
4400	20′ - 5″	42′ - 8″
4950	21′ - 4″	42′ - 8″
5250	22′ - 4″	42′ - 8″
5500	23′	42′ - 8″
6050	24′ - 1″	43′ - 2″
6600	24′ - 1″	46′ - 5″

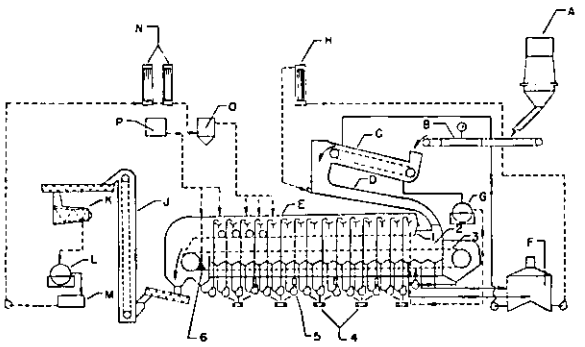

Figure 6-17. Flow diagram of DeSmet diffuser; a) Slicer; b) Scale; c) Separator; d) Flume pipe; e) Diffuser; f) Circulating juice tank; g) Juice screen; h) Circulating juice heater; j) Pulp elevator; k) Pulp press; l) Press water screen; m) o) Press water tanks; n) Press water heater; p) Fresh water tank; 1) 2) Cossette feeding device; 3) Cossette belt; 4) Juice pumps; 5) Juice heaters; 6) Belt spray washer.

upper and the lower belt. Floats are located in every hopper to prevent the intrusion of air and the excess juice is allowed to overflow into the next hopper. The cossettes are heated before they are deposited on the diffuser belt. They pass through an inclined trough and are preheated countercurrently with juice from the diffuser. The cooled juice is withdrawn from this trough and sent to the process. Heated circulating juice flumes the cossettes through a specially designed pipe to the diffuser belt, where the thickness of the cossette layer can be regulated by an adjustable dampening device.

Fresh water is added to the diffuser above the last hopper and pressed pulp water is fed to the cossettes between the third and fifth hopper, where the sugar concentration in the juice is approximately the same as in the pressed pulp water. The retention time of the cossettes can be varied by changing the speed of the conveyor belt. The bacterial activity in this diffuser was found to be quite low even under adverse conditions (47).

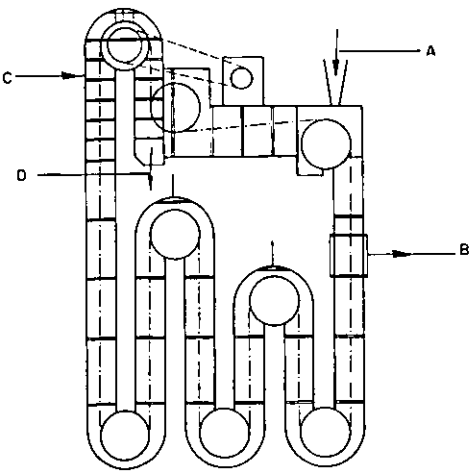

Figure 6-18. Flow diagram of Olier diffuser. a) Cossette inlet; b) Diffusion juice outlet; c) Water inlet; d) Pulp outlet.

The Olier Diffuser (34). This French diffuser was one of the first continuous diffusers put into service. The diffuser consists of four or six vertical pipes, which are connected to each other. Please see Figure 6-18. Perforated steel plates, which are attached to endless chains, drag the cossettes through the pipe system. The cossettes are fed proportionately to the speed of the transport element. A level difference of about 36 ft between the diffusion juice outlet and the water inlet is required to obtain the necessary hydrostatic head for a juice velocity of 4 ft/min. The effective diffusion length is given as 240 ft, which corresponds to a diffusion time of 60 min. It is also possible to add a scalding tank and then pump the cossettes and recirculated juice into the first column at a point below the juice level. The diffuser is jacketed at several points in order to provide the necessary heating, which is regulated by temperature controllers.

The J-Diffuser (36,54). This diffuser was designed by the Hungarian Sugar Institute, and the first full-sized installation was completed in 1957. The name "J"-diffuser was chosen because of its bent rectangular tube, which resembles the letter J. Please see Figure 6-19. The cossettes enter the shorter leg of the diffuser and are moved through the tube by a series of chain-type flights. The flights consist of chains, which are hung in a perforated angle iron frame. These frames are attached to two endless chains. The use of chain flights in place of perforated flights is believed to facilitate the juice flow through the diffuser. The cossettes are scalded at a temperature of 85-87 °C with recirculated and heated juice in the diffuser below the juice outlet. The diffusion temperature is kept at 70-72 °C.

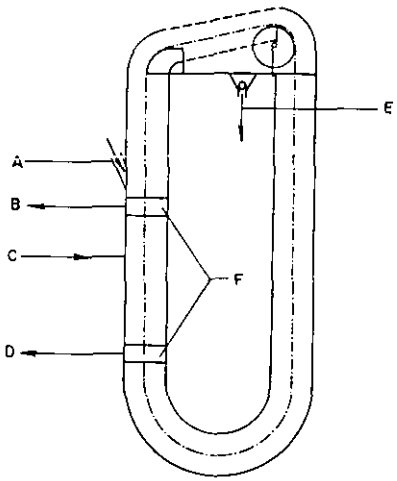

Figure 6-19. Flow diagram of a J-Diffuser. a) Cossette inlet; b) Diffusion juice outlet; c) Circulating juice from heater; d) Circulating juice to heater; e) Pulp outlet; f) Juice screens.

The following operating results were reported with a cossette polarization of 14.78: sugar loss, 0.183% on beets; draft, 125.9% on beets; time of diffusion, 55.8 min.; and Silin number, 21.7 m/100 g.

General Discussion of Continuous Diffusers

In theory the most efficient sugar extraction is obtained by using the countercurrent principle. In practice, very few diffusers are designed for an uninterrupted

The Silver D.d.S. slope and the tower diffusers are designed for a countercurrent flow of juice and cossettes through the entire diffusion process. Although baffling devices are installed in these diffusers to insure a countercurrent movement of cossettes and juice, a certain degree of back-flow and cross-current movement of cossettes and juice takes place. The more compact filling of cossettes in these cellless type diffusers demands a better quality of cossette to prevent plugging.

In cell-type diffusers, the juice and cossettes move countercurrently overall, but concurrently within each cell. In this case diffusion tends toward equilibrium as described in simple theory. After a time (t/N), where t is the time and N is the number of cells, the cossettes are continuously removed to the subsequent cell, while the juice is continuously removed to the preceding cell. This is shown in Figure 6-8a. When the juice and the cossettes travel concurrently in this way, it is probable that the cossettes are subject to extraction by somewhat less than all the juice, since, for example, most of the juice in cell i, at any one instant, probably does not ever contact most of the cossettes that are in the cell i − 1 at the same instant. This assumed reduction of diffusion contact is partially compensated by the greater concentration difference between the cossettes and juice. Recirculation of the juice only adds to the loss. Compensation can come from the use of more cells or from changing other operational variables. Recirculation cannot be entirely eliminated in this type of diffuser, since there is usually agitation within the cells. There is also always "carry-back" of the juice adhering to the cossettes, when the cossettes are transferred to the next cell. Attempts to further increase the rate of diffusion by dividing the juice flow into two streams and passing the streams to alternate cells, may be considered to reduce contact further; that is, to further remove it from a truly countercurrent process, which may again be compensated by a longer diffuser.

Since juice and cossettes are not completely separated by screens when each is transferred to its next cell, there is also a recirculation of cossettes as well as juice. The recirculated cossettes are usually the fines that pass through the screens, and their amount can be increased unnecessarily by too violent agitation. This can only decrease extraction efficiency, since the cossettes, already nearly in equilibrium with the juice, are replaced in juice that is to become enriched, so that reversed diffusion may occur.

The benefits derived from agitation in continuous diffusers are from the increased contact between pulp and juice, since, if the cossettes are left undisturbed, the surfaces of contact between the cossettes cannot act as active diffusion surfaces. Furthermore, if the juice is relatively stagnant, there will be a concentration gradient within the juice, which slows down the rate of diffusion.

Many attempts have been made to evaluate and compare the different diffuser systems. In most instances only the diffusion rate, or a multiple of it, has been used. Factors such as time, temperature, draft, and sugar losses are usually employed in the formulas. Although these operating data are interchangeable in the various formulas, their singular influence on the entire manufacturing process should also be considered.

Corrosion appears to be more severe in continuous diffusers than in the batch type battery. Corrosion is usually caused by the action of acid and oxygen, which is accentuated by continuous friction or erosion caused by the movement of the cossettes and sand which also prevents the formation of a protective oxide film. In some instances, erosion caused by sand in pipes, pumps, and in certain areas of the diffuser, is more severe than the normal corrosive action of acid and oxygen.

With the installation of new continuous diffusers in the last 20 years, the capacity of the factories has been increased without due consideration of the beet washing and slicing capacity. At the same time, the complete adoption of mechanical harvesting has increased the amount of trash and mud entering the factory. Bacterial activity in the diffuser has also been affected. More bacteria are introduced with poorly washed beets, and attempts must be made to avoid long detention times of juice and cossettes. Some diffusers have very short detention times for juice and longer detention times for the cossettes, which will tend to reduce the possibility of a bacterial infection of the juice, but which also will increase the pectin content in the juice which is directly related to the time and temperature. The cost of steam and the evaporator capacity will influence the decision as to the premium to be paid for an installation capable of giving a lower draft.

There is no general agreement among sugar technicians as to the sugar yield which can be obtained from a more extensive extraction of the pulp. More research in this area is desired, especially in regard to the effect of pulp press water return.

Beet Slicing

Beet slicing is one operation which demands the constant attention of the operating personnel. Good cossettes are necessary for all diffusers to achieve best efficiency. Good cossettes should be smooth, of uniform thickness, and with a minimum amount of fines and slabs. Such cossettes can only be obtained from well maintained slicers, using the appropriate knives, which are carefully sharpened and lined up in the slicer.

The Beet Knife. There are two types of beet knives used today in American factories: the splitter knife (Figure 6-21) and the Königsfelder knife. Both of these knives have V-corrugated cutting edges, and the splitter knife has also short, vertical splitters on its ridges (Figure 6-20). Each V-groove in both types of knives is called a division. Other knives sometimes used in other countries are the straight-cut knife, which cuts whole slices and the side-cut knife, which cuts strips.

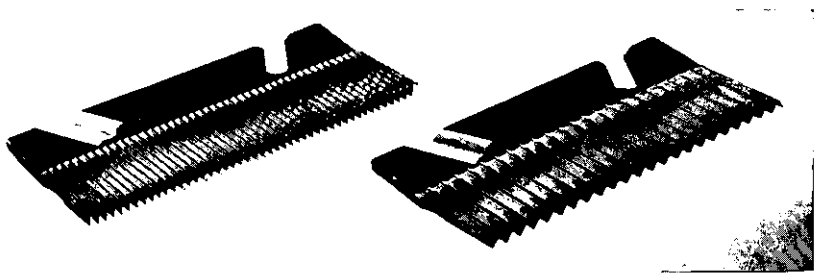

Figure 6-20. Corrugated-splitter and Königsfelder beet knives. *(Courtesy, Henry Disston and Sons, Inc.)*

The splitter knife usually has 23, 36, or 46 divisions for a standard length of 165 mm, which corresponds to a distance of 7.5, 4.5, or 3.6 mm between two ridges. The shape and thickness of the cossettes are determined by the number of divisions of each knife and its position in the knife block. Two knives, called "A" and "B" are available. The "A" knife terminates at both ends with a half V-groove, and the "B" knife terminates at one end with a splitter and at the other end with a

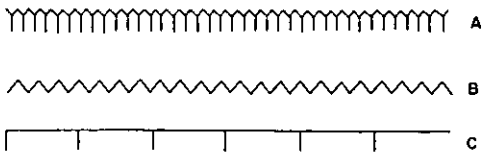

Figure 6-21. Section of the cutting edges of beet knives. a) Corrugated-splitter knife; b) Königsfelder knife; c) Side-cutting knife.

half V-groove. When only "A" or "B" knives are used and the splitters are perfectly aligned, cossettes with V-shaped cross-sections are produced. When the alignment is a half-division to one side, caused by setting "A" and "B" knives alternately in the boxes, cossettes with rectangular cross-sections are produced.

The Königsfelder knife usually is designated by the number of ridges on a knife of 137 mm width (please see Table 6-11). Two knives, called "A" and "B", with ridges staggered by a half-division, are alternately set in knife blocks. This setting will produce cossettes with a V-groove cross-section.

Table 6-11 Königsfelder Knives

Divisions per 137 mm Knife	15	17	19	22	24
Divisions per 165-167 mm Knife	18	21	23	26	29
Distance between ridges, mm	9	8	7.2	6.2	5.7

The quality of cossettes depends on the care taken in jointing, routing, filing, and setting the knives, and in positioning the set knives in the slicer. Jointing refers to grinding the cutting-edge straight by means of a grinding wheel, to remove worn or damaged leading edges in preparation for routing and filing. Routing is the removal of excess metal in each division to thin the metal before filing. With splitter knives, both the V-edge and the splitters are filed to a sharp edge, the former by automatically controlled rotary files, and the splitters by right- and left-hand fraisers.

The sharpening of Königsfelder knives requires fewer operations, and thus less labor is involved. It is advisable to straighten these knives before jointing them as only perfectly straight knives can be restored to original condition. Even new knives may be too thick to make smooth, long slices, and the routing machine is very carefully set, since too thick a knife tends to break the chips off short by bending them back too much when they leave the knife (Figure 6-22). When routed too thin, the edges will break when filed. Splitter knives are commonly routed back about 10 mm and Königsfelder knives about 6 mm from the cutting edge. The cutting edge is, of course, applied very carefully, as dull spots on the edges, known as "shiners," will rupture a great many beet cells and result in ragged cossette surfaces. In addition, dull spots do not cut through trash or fibrous beets, and material will hang on the edges and crush the cossettes. Filing the edge too thin weakens it so that it may turn back and become dull quickly.

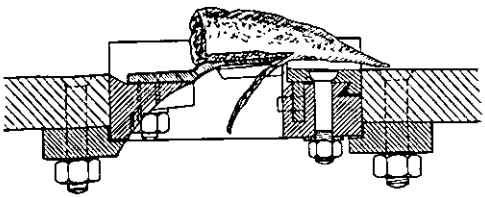

Figure 6-22. The beet knife in action.

The Slicer. The slicer mostly used in the American factories is the rotary-drum type known as the Ogden slicer (Figure 6-23) which is a modification of the earlier French Maguin slicer. There are usually 8 to 10 knife blocks mounted on the rotating drum or "spider." Each knife block holds six knives in three rows. Thus one slicer can contain a maximum of 48 or 60 knives. Beets are fed into the interior of the drum and the cossettes are forced through the outside of the drum. A stationary baffle prevents the beets from riding around with the drum, and is so shaped that the beets are gradually forced out into the knives. The drive usually consists of a motor with an adjustable speed system and a gear-speed reducer which reduces the speed down to between 60 and 120 rpm. The capacity of the slicer depends on the type of knife, its division and the condition of the knife itself. The use of finer knives reduces the capacity of the slicer and increases the power demand for the same beet tonnage. The capacity of the slicer is also directly proportional to its speed. However, slicing at too high speed will increase the amount of fines.

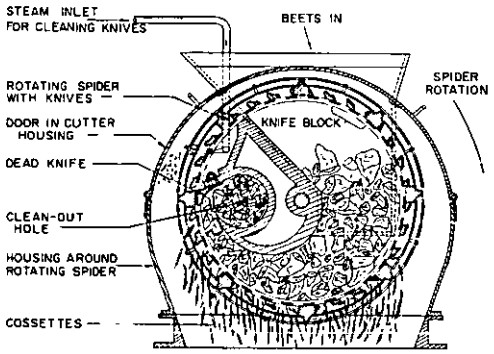

Figure 6-23. Diagram of a Maguin-type beet slicer.

The horizontal type of slicers (Figure 6-24) are mostly employed in Europe, and have almost completely replaced the drum slicer in some countries. During the last few years one of these horizontal slicers, the Putsch, has also been installed in four American factories. The newer Putsch models have 2200 mm and 2320 mm disks. The 2320 mm disk holds 26 knife blocks with one row of two 200 mm knives each, for a total of 52 knives, and the 2200 mm disk holds 22 knife blocks. The wider 400 mm knife blocks are preferred over the former narrower blocks as there is less danger of plugging the beet channel above the slicer disk. One apparent disadvantage of the horizontal slicers is the nonuniform cutting speed across the radius of the disk, which varies from 4.4 to 6.0 m/sec, at 60 rpm for a 2200 disk slicer. The movement of the beets is prevented by baffle plates which are located at the end of each channel. A spring-loaded door at these places will open under pressure caused by stones or other foreign objects.

The drive of this slicer usually consists of a variable fluid drive or other variable motor drive and an electro-magnetic clutch. An auxiliary smaller motor is provided to turn the slicer at a low speed during knife changing. With this arrangement it is possible to change knives while the slicer is full and also to move the slicer in a forward direction. This prevents small stones or other objects from being dislodged and left in the slicer. Only one man is usually required for this operation.

Figure 6-24. Three Putsch vertical-shaft slicers in the factory at Lieusaint, France. The central unit is driven by a d-c motor, vertically placed over the main driving shaft. The outer units are driven by a-c motors with fluid drives. *(Courtesy, H. Putsch & Co.)*

Knife Setting. In setting beet-slicer knives, the metric system is used, with the millimeter as the unit. Thickness gauges are used to set the knives "up" and "back." "Up" is the height of the cutting edge above the guide bar, a removable flat bar located just ahead of the cutting edge of the knife. Each drum slicer block has three bars and, if adjustable, they are known as "rocker bars." Thus, the "up" is the height of the knife exposed to the beet. "Back" is the distance from the knife edge to the guide bar ahead, and this distance is adjusted according to the condition of the beets. *In general, the back setting is a minimum necessary to keep the knives* cleared. Too great a back setting will result in passage of slabs of beets.

Splitter knives are usually set 2 to 3 mm and Königsfelder knives 5 to 8 mm back. The quality of the beets, especially the amount of fibrous material, and the type of diffuser generally determine the exact settings within these limits. When slicing fibrous beets the back setting generally has to be increased. It is then sometimes better to joint the splitter knives squarely without the usual 82 ° angle. Slabs which will pass through the increased opening between the plate and the ridges will be split. Königsfelder knives should be jointed at an angle, which reduces clogging of the knife edges.

In drum slicers the knife blocks are so mounted that all the knife edges are exactly the same distance from the axis of rotation. This is especially important when slicing rectangular cossettes with "A" and "B" splitter knives. To assist in obtaining precise settings, a knife trammer of some type is used to advantage. Knife blocks should be kept as sets for each slicer and each block used only in its individual slot.

To assist in cleaning the knife edges of fibrous material or trash, steam is injected tangentially to the knife edges as the drum turns. Steam pressures of 100 to 150 psi at the nozzle plate are very effective.

Cossette Scales

Continuous weighing of the cossettes is necessary to control the output of the slicers and at the same time to regulate the flow through the diffuser. Please see Figure 6-25. An accurate measurement of the weight of the cossettes is required for general factory accounting.

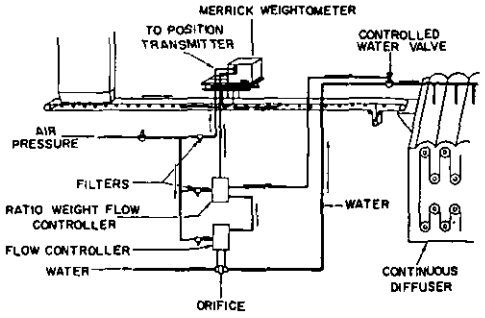

Figure 6-25. Merrick Weightometer, controlling a continuous diffuser. *(Courtesy, Merrick Scale Manufacturing Company.)*

The Merrick Weightometer (Figure 6-26), a belt-conveyor scale, has been commonly used in many American factories. The scale consists of a belt conveyor, part of which is connected to a weighing beam, and the measuring and counting devices. The vertical movement of the belt is transmitted to the weighing beam and is counterbalanced by a float which is suspended from the beam in a pot filled with mercury. Furthermore, the weighing beam is linked to a tilted integrator disk. The position of the disk changes with the load on the conveyor belt. A sprocket chain drive transmits the horizontal movement of the belt to the integrator. The totalized weight of the cossettes is then shown on a counter. The Merrick Weightometer is also designed to actuate a slice-rate controller and the supply-water controller for the diffuser.

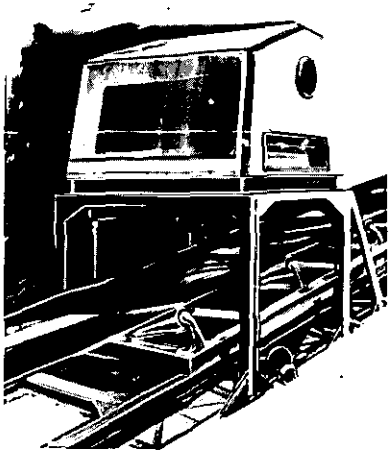

Figure 6-26. A Merrick Weightometer installed.

The weightometer must be checked frequently and adjusted if necessary. The belt is balanced first by running it empty for several circuits. Any gain or loss of weight as indicated by the movement of the integrator disk must be corrected by turning the knurled nut on the float link clockwise, if there is a loss of weight, and counter-clockwise, if there is a gain of weight during one complete turn of the belt.

A roller chain of known length and weight is then placed on the belt center and fastened taut at both ends to a rigid frame. Equal sections of the chain must extend to both sides of the suspended conveyor section. The position of the belt

relative to one point of the rigid frame is marked and the belt is run for three circuits and then stopped as closely as possible to the original position. The over-run or under-run of the belt is measured and recorded on the test sheet together with the weights shown on the counter before and after the test. An example of one test including the calculation is given in Table 6-12.

Table 6-12 A Typical Weightometer Test Record

(1) Length of Conveyor Belt = 140 ft.
(2) Calibration Test Weight per foot = .007500 tons
(3) Weight for one circuit of conveyor = 1.050 tons

	First Trial	Second Trial
No. of Circuits	3	3
Over-Under Travel	—12"	—3"
Reading After Test	8555.212	8558.356
Reading Before Test	8552.060	8555.212
Registered Weight	3.152	3.144
Correction —	—0.008	—0.002
Corrected Weight	3.144	3.146
True Weight	3.150	3.150
Difference	—0.006	—0.004
Error in %	—0.19	—0.13

The error of the weightometer can be corrected by moving the bracket located at the lower end of the integrator link. A movement to front as obtained by a full turn of the lock nuts will cause the integrator to register 1% unit less than before. An inward movement will give the same effect in the other direction. It is important to weigh the chain and measure the length of the conveyor belt before each campaign. The weight per unit length of the chain should be about the same as the average cossette load on the belt. A batch test scale can also be used to check the continuous belt weighing system.

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Chapter VII

JUICE PURIFICATION, I.

History and Fundamentals

History*

In their 140 years of progress up to World War II, juice purification processes have involved few principles that were not well known before 1865. A consideration of the essentials of juice purification history may prevent needless replication of studies that have already been made. Even a superficial examination of the literature shows much duplication of work has already occurred, sometimes in multiple cycles.

The development of methods of processing sugar beets has always been accompanied by a parallel or somewhat delayed development of explanatory theory, and most development has been empirical. Since beet-sugar manufacturing enterprises were relatively small indivdual organizations without heavily endowed institutes for fundamental research, process research has usually been accomplished by the operators themselves, and where small research groups existed, money invested required quick return and justification.

Development in Europe. Until well into the 20th century, juice processing in America was a reflection of that in Europe, although the American philosophy of large scale industrial procedures was becoming evident after 1880.

European factories of the 19th century were of small capacity, mostly cooperatives, drawing their beets from relatively small areas, and usually manned by the farmers themselves. For example, in 1884 there were 849 factories in France and Germany alone, and those in Germany averaged about 150 tons daily slice. Coming from small areas, the beets processed tended to be quite uniform, and consequently the European technologist could tailor the purification process to fit the beets. Since the factories were relatively small, it was not unduly expensive to make equipment alterations. A survey of technical advances from 1850 to 1900 lists approximately 7500 references.

Franz Achard (2), the student of Andreas Marggraf, in his pioneer factory at Cunern, Silesia (1801), first separated extraction and purification into two unit operations. Following rasping of the beets and pressing out the juice, the second process consisted in precipitating a mass of nitrogenous materials, saponins, and other colloids, by acidifying the pressed juice with sulfuric acid to about 3.5 pH, in the cold, and then neutralizing with chalk. Achard did not use lime, since he knew that lime formed compounds with sucrose. The juice was then clarified with cow's milk, a feature carried over from cane-juice processing.

The Colonies Process. About 1811 the so-called West Indies Colonies process for treating cane juice was introduced for beet juice purification. The pressed juice was limed in increments (progressive predefecation) to the "break," with milk of lime, about 0.25% CaO on juice. To this mixture milk or blood was added for clarification, and, after standing, the surface scum was skimmed off, and the clear liquid decanted from the settled sludge. The juice was then neutralized with sulfuric acid. Carbon dioxide was suggested for this purpose in 1813, but was not actually used until about 1840. The pressed juices actually contained more

*by R. A. McGinnis

impurities than diffusion juice, and as a result the purification achieved by the very small amounts of lime used was quite inadequate. Possibly because of the apparent visual completeness of precipitation by liming to the break, almost half of the 19th century passed before any technologist ventured to use major amounts of lime.

Thus there was often too little sugar and too much molasses produced. To improve this incomplete purification, the use of bone char became common after about 1819, and remained an essential part of the juice purification processes until 1880.

Dombasle and Maceration (49). In 1821, the Comte de Dombasle introduced his *maceration* process, in which beet slices 1/8 to 1/4 in. thick were extracted by "successive depletion" with hot water. This was actually diffusion, in open vessels. By this extraction, yields of sugar up to 6 or 7% on beets were possible.

However, the mild purification processes in use could not satisfactorily eliminate the additional nonsugars thus extracted, and his maceration process could not be used, as presented.

While the pressed juice does not contain the leachings of pectins, albumins, and other cell materials that are extracted in a diffusion operation, there are in pressed juices large molecules which are retained in the pulp in diffusion, and more rigorous purification is required, although the greater amount of the lime used is required for removal of lime salts by adsorption on calcium carbonate in first carbonation. The major advantage of the diffusion process is the more complete extraction of sucrose.

In spite of this, modifications of maceration using the successive depletion principle, with cold water and rasped beets, came into considerable use. A typical modification was that of Schuzenbach, used at the Wagheusel factory (3), which was the world's largest in 1837. Here the beets were cut into cubes, which were dried with hot air for preservation. For maceration, the cubes were ground to a powder and extracted with acidified water at approximately 120% draft. The maceration battery was a series of open-topped, agitator-equipped vessels mounted in steps. The liquid was decanted from one vessel to the next. Various ingenious types of continuous macerators were devised, which anticipated later continuous diffusers. For example, Pellatan had a "levigator," a 24-compartment trough set on a slant, which strongly resembled the Raabe "rapid" continuous diffuser of the 20th century. Martin and Champonnois built a tower macerator in 1847.

Juice Purification Catches Up. In 1840 Schatten and Michaelis (130) first used carbon dioxide instead of sulfuric acid for neutralization after liming, an innovation which has since been used without question. Sulfurous acid had some use from 1811 on, although for many decades this was regarded as a trade secret. Sulfur dioxide gas was not used until very much later.

Rousseau (101) in 1847 first broke the "predefecation barrier" and increased the amount of lime used up to 1.5% on juice. He neutralized with one carbonation, and obtained a higher degree of juice purification than ever before. The end point of the one carbonation, however, was difficult to estimate, and a small error could produce a juice very difficult to process.

The final break in the lime-carbon dioxide purification process came when Perrier and Possoz (98) in 1861 used even more lime—from 2 to 5%—with a double carbonation. This was a tremendous improvement, and by making the first decantation and separation at high alkalinity, where the calcium carbonate has a

large surface available for adsorption of impurities, and the second a deliming operation at a low alkalinity, excellent results were obtained.

Julius Robert (4), an Austrian technologist, introduced the diffusion battery at Seelowitz, Austria, in 1864, and combining it with the process of Perrier and Possoz, instituted the beet-juice purification process that, in essentials, is still in use.

At this time, many factories were extracting the beet juices by rasping, and pressing or centrifuging, and other factories by maceration of ground beets in open vessels. The extracted juices were usually limed with a relatively small amount, clarified with some agent, and the scum removed and the juice decanted from the precipitate. The sludges were filtered and desweetened in bag filters. Because of the low degree of purification, bone char treatment was essential for reasonable yields of sugar crystals.

Thus, with the presentation of Robert's combination process, all of the important elements of juice extraction and juice purification were known, if not understood. Grinding and slicing of beets, juice extraction by pressing or centrifuging; solvent purification, precipitation of colloids at either the acid or alkaline coagulation points; the use of lime as a purifying agent in predefecation, progressive predefecation, main defecation, single and double carbonation, clarification with physical "sweeping" agents, activated carbon absorbers, and sulfitation. All were now a part of the technologist's repertory, and since 1864 the only really new principle which has been introduced is ion-exchange purification. While advantages of Robert's combination were recognized rapidly, full adoption by the industry required almost 30 years.

The rasping of beets was gradually abandoned as the diffusion process was adopted, because flow of juice cannot be maintained in diffusers with finely ground materials.

The succeeding 100 years have seen nothing essentially new in extraction processes, excepting the conversion of most of the industry to the use of continuous extractors, after the design and trial of surprisingly large number of machines, the introduction of chemical flocculating and settling aids, and the return of carbonation sludge to the raw juice.

Later European Developments. In the period from 1864 to date, the attention of technologists has been concentrated on improving the juice purification process then introduced. Not only has the method been under direct attack through the proposal or trial of no less than 737 different chemical substances for the purification of the juice—not counting the adsorbing carbons—according to a list of von Lippmann in 1936, but almost every conceivable variation of the liming-carbonation process itself has been considered. So thorough has been this scrutiny, that one can be almost certain that any reasonable variation he may be able to devise involving beet juices, lime, and carbon dioxide, will be found in the earlier literature.

Development in America to World War II. While up to the 20th century the processes used in America were similar to those used in Europe, differences began to appear at that time. American factories were larger, and drew more varied types of beets because of the larger geographical areas served. Thus there has been a tendency to "freeze" the juice purification process with settings of the variables suitable for the average beets encountered.

In Europe, on the other hand, major attention was given to improvement of juice quality and reduction in lime usage.

Surplus of molasses in North America led to the application of the Steffen saccharate processes for its desugaring, which have been relatively unused in their native Europe. This has involved the use of saccharate milk for defecation, with its own specialized techniques.

Separation of the sludge from the juice has been an operation that has often caused delays in production. Types of filters like the Danek, Phillippe, and plate-and-frame presses involved high costs for labor and cloth, and were of serious concern in the United States due to constant efforts to increase factory capacity.

Thus advances in filtration marked the start of a second path for American technology, and commenced in the first decade with the replacement of the older filters with the newly invented Kelly filters for both first and second carbonation.

Shortly thereafter the Oliver rotary-drum, vacuum filter appeared, with its unique ability to filter out solids from a thickened sludge, and to desweeten it with a very small amount of water. Several types of vacuum-filter clarifiers were built to prethicken the sludge for the Oliver filter.

Wide use of these combinations was followed by the immediate acceptance in the third decade of a process designed by the Dorr Company to permit the use of the tray clarifiers they had developed for the cane industry. Fundamentally this is a continuous, simulataneous, mild defecation and first carbonation, which will give reasonable purification of juices from almost any type of beets. In this process, calcium carbonate particles are built up as agglomerates of fine crystals surrounding bits of coagulated nonsugars. The sludge so formed settles rapidly and filters quickly. The use of the new system of continuous carbonation, followed by a tray clarifier for sludge sedimentation and juice clarification, with Oliver filters handling the underflow, gave a process which drastically cut labor and maintenance costs. The savings made with this new process were so attractive, that over two-thirds of American factories installed it, disregarding what appeared to be minor disadvantages.

Economic factors have, properly, dominated in process design, and these factors must be kept in mind to understand the reasons behind the developments. Up to World War II, important differences between these economic factors were, in Europe, the frequently lower labor costs, the higher fuel costs, the necessity of leaving sugar in pulp for cattle, and the strong consumer market for incompletely refined white sugar.

Developments since 1970. Nothing really new has been introduced in the last decade into juice purification in North America. Use of chemical settling and flocculating aids has become general, as has the return of carbonation sludge to the prelimer or raw juice. Processes have tended to firm up into two groups; the classical processes, and the Dorr process. While the total number of operating factories has decreased about 20% due to the increasingly unfavorable sugar market, a number of factories have returned to the Dorr process instead of more complex systems. The number of "package" processes from Europe has increased. There is now one factory which makes only raw sugar. There have been many small process changes made to conserve energy, due to the increasing scarcity and high costs of fossil fuels.

There is a tendency to shut down small factories, and increase the capacity of others, with an undoubted saving in overhead costs.

There is also an increased dependency on European technology for new developments in juice purification.

Finally, the 3 newly constructed factories have all been in the Red River valley, in the states of North Dakota and Minnesota. It is remarkable that this area, almost ideally suited for sugarbeet culture, should have escaped attention for so long.

Chemical Fundamentals*

1. Substances Important in Juice Purification

Water. Water comprises 80-85% of the weight of the beets and the juices, and can thus properly be considered as the solvent, representing 44-47 moles of H_2O per kg of juice. Its simple and small molecule with large dipole moment, high dielectric constant, and the possibility of ionizing both as an acid

$$H_2O \rightleftharpoons H^+ + OH^- \rightleftharpoons 2H^+ + O^= \quad (1)$$

and base,

$$H_2O + H^+ \rightleftharpoons H_3O^+ \quad (2)$$

make it an excellent solvent, facilitating hydration by secondary valences and the electrical dissociation of the solutes. [Recent studies (57) by *relaxation methods* indicate that the proton or hydrogen ion, and the hydroxide ion, probably exist hydrated, with four and three molecules of water, respectively, giving actual formulas: water = $(H_{16}O_8)_n$; hydroxide ion = $H_7O_4^-$; hydrogen ion = $H_9O_4^+$]. It has a very marked tendency to form cybotactic structures, or uniform molecular patterns in long chains, which may serve to "fill up" even very fragile large molecules, making possible the existence of solutions of colloidal substances, such as natural proteins and pectins.

Its own dissociation constant is rather low, and very markedly affected by temperature:
$pKH_2O = pOH + pH = 14.92$ at 0 °C, 13.25 at 50 °C, and 12.24 at 100 °C (Figure 7-1).
It is for this reason that the pH, or pK − pOH, is not a good measure of the pOH at higher temperatures: for a given pH, the pOH is smaller, the higher the temperature.

Water is a sufficiently strong acid,

$$\frac{(H^+)\ (OH^-)}{(H_2O)} = 10^{-15.74} \text{ at } 25°C \quad (3)$$

to influence the dissociation of weak acids and bases, as they are found in beet juices, and it must therefore always be considered in the equilibria established.

*by J. Dedek and Mrs. J. Dedek [Since this definitive text was written in 1951, there have been many advances. The editor has tampered with the original as little as possible. Supplementary passages have been added for very important new work. Smaller advances have been ignored, but many are discussed in Chapters VIII and IX. Sections on various nonsucrose components have been inserted in Chapter II, where they are noted.]

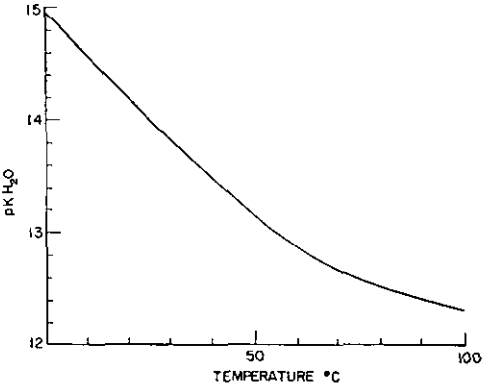


Figure 7-1. pKH_2O and temperature.

The great excess of water in juices enables it to take part easily in hydration reactions, some of which are very important:

$$H_2O + \text{sucrose} \rightarrow \text{invert sugar} \quad (4)$$
$$H_2O + RCONH_2 \rightarrow RCOOH + NH_3 \quad (5)$$
$$H_2O + CO_2 \rightarrow H_2CO_3 \quad (6)$$
$$H_2O + CaO \rightarrow Ca(OH)_2 \quad (7)$$

Lime. (See Chapter XVII) If prepared by the burning or calcination of limestone, CaO is amorphous, highly dispersed, having lost 44% of its weight without a corresponding loss of volume; colloidal (82), since its solubility depends strongly on its method of preparation and the amount of solid phase present (131) (see Figures 7-2 and 7-3); and its solutions in sugar can be ultrafiltered (118). Though its reaction with water is strongly exothermic, it is apparently still able to dissolve in the cold without hydration, and to give more concentrated solutions than those prepared from $Ca(OH)_2$.

Calcium Hydroxide. This is used in the sugar industry in the form of milk of lime—a suspension of $Ca(OH)_2$ in water. Its physical properties depend on the conditions of slaking. Like CaO, it first goes into colloidal solution, in which it is stabilized by sucrose (76,115). Its solubility is therefore not constant, but it is determined by the previous history of the calcium hydroxide, the amount of the solid phase present, the temperature, and the concentrations of sucrose and nonsugars. Lower temperatures and high concentrations of sucrose and nonsugars increase the solubility (66).

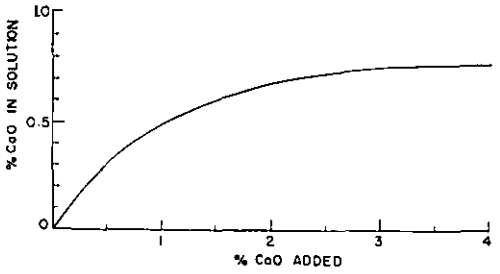


Figure 7-2. Solubility of CaO in sugar solutions. The influence of the amount of solid phase. ***(Waterman-van Aken 1927.)***

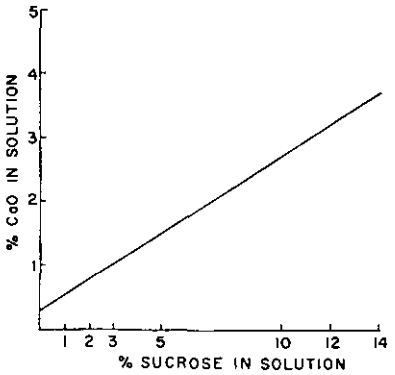


Figure 7-3. Solubility of $Ca(OH)_2$ in sugar solutions. The influence of sugar concentration. ***(Landolt-Bornstein Tables I-752.)***

The influence of temperature is very marked. The heating of a cold sugar-lime solution, such as a juice defecated at room temperature, thus leads easily to a supersaturated solution of calcium hydroxides and a turbidity or precipitate is formed, which generally redissolves on cooling. The composition varies greatly according to details of procedure, the presence of $Ca(OH)_2$ in the solid phase, and other factors (120). Generally more or less sucrose is coprecipitated in the form of saccharates, decomposing only slowly. Still more complicated phenomena take place if such solutions are carbonated.

The second dissociation constant of calcium hydroxide indicates that it is but a moderately strong base: $pK_2 = 12.5$ (15,80). This is very important, since the concentration of Ca^{++} is greatly decreased even at relatively low pH, where the dissociation of carbonic acid and sucrose is still very incomplete. This is another example of the many factors lowering the activity of Ca^{++}.

pCa^{++}. There are no methods to determine it rapidly and with any amount of precision, even though it is enormously important in physiology.

In a 17% sugar solution, containing 0.3 to 0.5% CaO, only from 2-8% of the total amount of lime exists as Ca^{++} (80).

Measurement of the pCa^{++} in sugarbeet juices (14) from thin juice to molasses, using Harnapp's electrode, have given extremely low values, varying from 4 to 9, although the total calcium concentration was far greater.

The existence of undissociated and even of ultrafilterable calcium in biological liquids such as blood serum and milk is a well known fact (67). Many authors have noted the low dissociation constants of calcium salts, especially of weak organic acids and amino acids. Calcium salts of α-NH_2, or α-hydroxy acids are dissociated only 2 to 4%. Amino acids can form chelated, and thus un-ionized, calcium salts. Calcium can also form ions according to the equation (86),

$$Ca^{++} + An^- \rightleftharpoons (CaAn)^+ \qquad (8)$$

and even complex anions (68), of which calcium citrate is important in physiological chemistry (104). This has even found industrial application in the liberation of calcium from cation exchangers, in the form of calcium citrate anion (116). When electrodialyzing sugar-cane juice (111), calcium has been seen moving to the anode, and it has been assumed that it forms a part of the negatively charged colloidal complexes. It appears (16) that in this respect, calcium, of all the alkaline-earth metals, behaves in this very different fashion.

Carbon Dioxide. Carbon dioxide is produced in the sugar factory and applied to carbonation in the gaseous form. Before reacting, it must dissolve in the juice. The absorption of carbon dioxide in alkaline solutions is technically important, and has been repeatedly investigated (110,132). Small bubbles and efficient agitation of the liquid are favorable for better absorption. With all conditions equal, carbon dioxide dissolves more slowly than ammonia, for example.

Its solubility depends on the partial pressure of CO_2 in the gaseous phase, the temperature, and the chemical composition of the liquid phase. Increasing ionic strength lowers its solubility, while the presence of proteins increases it (70). In sugar factory practice it is of less importance than its rate of reaction, as CO_2 reacts immediately with water or the alkalies of the juices.

Carbon Dioxide + Water. Before carbon dioxide can react with alkalies it must become carbonic acid. This very important reaction of hydration has been studied extensively (60,100).

The hydration of dissolved CO_2 proceeds according to two different molecular, and therefore, time reactions:

$$CO_2 + H_2O = H_2CO_3 \quad (9)$$

$$CO_2 + OH^- = HCO_3^- \quad (10)$$

The first reaction, independent of the pH, is positively catalyzed by ions of oxyacids. The second reaction depends strongly on the pH. The total hydration velocity can be summarized as in Figure 7-4 (61). It is very interesting to note that there is a distinct region of pH where the hydration of carbon dioxide proceeds most slowly, this pH being close to that of our second carbonation juices. Higher pH values have a very strong influence; for example, the velocity changes 10^4 times, when the pH changes from 8 to 13. Figure 7-4 is, of course, strictly valid only at 18 °C.

Other substances present influence the hydration velocity. It is technically important (44,45) that sucrose in 0.5 molal or 17.2 ° Brix solution, corresponding to

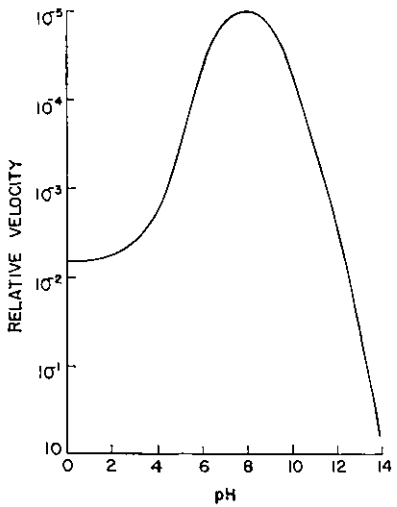


Figure 7-4. Velocity of CO_2 hydration. Influence of pH. Note that relative velocities are plotted with negative coefficients. *(Faurholt, 1924.)*

the approximate density of diffusion juices, slows down the hydration rather strongly (Figure 7-5). There is also a distinct effect from the more negative constituents of the buffer substances used (100), especially on the first hydration reaction.

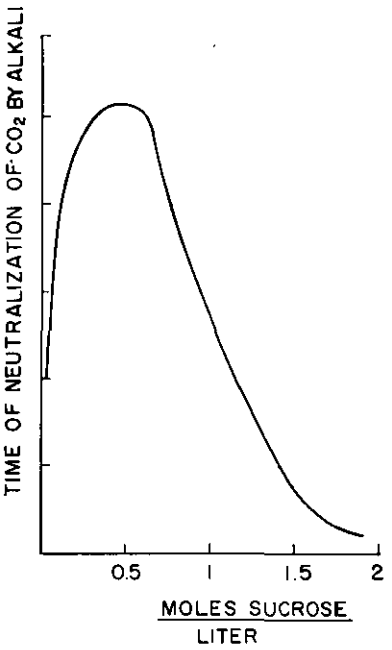


Figure 7-5. Hydration of CO_2. The influence of sucrose. ***(Dedek-Vasatko, 1929.)***

The opposite reactions of dehydration are important to the chemistry of second carbonation when conducted at the boiling point, a type of operation which is said to have been much in favor in the European beet-sugar industry, for the purpose of eliminating bicarbonates formed in second carbonation. The reverse of the first reaction, which takes place in rather strongly acid solutions (pH = 2) is very rapid, and it is complete at room temperature in less than one second. The second reaction is relatively slow in slightly acid solutions. At a pH of 5.5 it is not complete even after 180 seconds (59). This is very important for the effective functioning of carbonation at the boiling point, which can be written as

$$HCO_3^- \rightleftharpoons CO_2 + OH^- \qquad (11)$$

Carbonic Acid. *H_2CO_3 is in reality a rather strong acid. It is usually* considered as a weak one, because it is unable to exist in large concentrations, splitting easily into water and carbon dioxide. At low pH values, say from 0 to 4, there is in addition to 99.88% of carbon dioxide, only 0.112% H_2CO_3 (61). It is difficult to determine amounts of this small quantity with sufficient precision, and therefore usually only the apparent dissociation coefficients of carbonic acid are reported (69,87).

$$K_{(CO_2 + H_2CO_3)} = \frac{a_{HCO_3^-} \cdot a_{H^+}}{a_{CO_2} + a_{H_2O}} \; ; \; pK = 6.32 \text{ at } 30\,^{\circ}C \qquad (12)$$

The true dissociation constant is

$$K_{H_2CO_3} = \frac{a_{HCO_3^-} \cdot a_{H^+}}{a_{H_2CO_3}} \; ; \; pK = 3.7 \text{ at } 20\,^\circ C \qquad (13)$$

The second dissociation constant is

$$K_{HCO_3^-} = \frac{a_{CO_3^=} \cdot a_{H^+}}{a_{HCO_3^-}} \; ; \; pK = 10.2 \text{ at } 38\,^\circ C \qquad (14)$$

The bicarbonate ion is therefore a very weak acid, and, when calculating ionic equilibria, it is necessary to take into account even the dissociation of water. These constants are valid only for pure water solutions; the presence of other ions, or sucrose, and higher temperatures, influence them strongly. Determinations of the values of these constants at high temperatures and in the presence of sucrose are badly needed (13).

The second dissociation constant of H_2CO_3 in aqueous solution and in a 15% sugar solution with an ionic strength of 0.056 at room temperature and at 85 °C is reported (11) to have the following values:

	$pK_{HCO_3^-}$	
	85°C	20°C
Pure aqueous solution	10.25	9.82
15% sugar solution	9.93	9.65

It is very surprising to notice that, according to these data, the presence of sucrose increases the strength of carbonic acid, and thus increases the amount of $CO_3^=$ ions. Higher salt concentrations act in the same way, lowering the value of $pK_{HCO_3^-}$.

Higher ionic strength diminishes the activity, particularly of the carbonate ion (79). This effect, however, seems quite specific, and sodium salts are reported to act distinctly differently than potassium salts (62).

High temperatures (11) such as 85 °C increase the $pK_{HCO_3^-}$, thus lowering the acid strength. This is in contradiction to a number of previous determinations (69) which report an increase in strength, or a decrease of the $pK_{HCO_3^-}$ with increasing temperature. If all other conditions are equal, higher $pK_{HCO_3^-}$ would cause the deliming in second carbonation to be less effective. Further determinations are necessary, which likewise take account of the influence of proteins, pressure, and other factors.

Nothing is known about the possible effect of the carbonates of ammonia or amino acids, which are readily formed when CO_2 and NH_3 or NH_2^- groups react. Their chemistry is very complex, even in pure aqueous solutions (59).

Another, perhaps still more important reaction, is the formation of alkylcarbonates which seems to take place when alkalies are neutralized by carbonic acid in the presence of substances containing alcoholic OH groups. They have the general formula, $CO \begin{matrix} \diagup OR \\ \diagdown OM \end{matrix}$, where R is the radical of an alcohol, and M is a metallic cation. They seem to be primary products which decompose later, rather slowly, into free alcohols and bicarbonates (62,107,108). The presence of even very small amounts of alcohols is sufficient to retard the formation of, e.g., calcium carbonate (24). Alkylcarbonates form colloidal solutions, and amorphous, colloidal precipitates (64,96,126) which are difficult to filter and therefore to analyze. They

are known in the beet-sugar industry as "sucrocarbonates," having first been mentioned in 1868, and their use has even been proposed for the desugaring of molasses by two French chemists (8). [The mechanism of sucrocarbonate formation has been extensively explored by Dubourg (53)]. It is highly probable that sucrocarbonates are more or less normal intermediate products in first carbonation, especially of juices which have been limed at low temperature. The addition of even a few drops of an alcoholic solution of phenolphthalein has been reported to lead to a marked formation of calcium alkylcarbonates (108). It is possible that they are partly responsible for the delayed crystallization of calcium carbonate in second carbonation juices, which has been assumed to be due to its supersaturation (11,26-28).

Calcium Carbonate. Calcium carbonate is a very important substance, and its literature is therefore very extensive (33).

It exists in at least three modifications: vaterite, calcite, and aragonite, besides amorphous and colloidal states. The solubilities are different. The solubility product of calcite in pure water has been reported to be from 3×10^{-9} to 1.8×10^{-8} (mol/l)^2. The hexahydrate is definitely more soluble.

The solubility of the amorphous, unstable form is still greater. It is reported that it is freely soluble in alkaline sugar solutions (127). It is, however, doubtful that the product was really calcium carbonate. The solubility of calcite strongly increases with diminishing particle size (113).

The solubility product of $CaCO_3$ in a pure 15% sucrose solution at room temperature has been given (11) as 1.4×10^{-6} (% CaO)^2, or 4.5×10^{-8} (mol/l)^2 and at 85 °C 0.75×10^{-6} (% CaO)^2, or 2.4×10^{-8} (mol/l)^2. Figure 7-6 shows the curious increase in solubility at higher $CO_3^{=}$ concentrations, strongly reminiscent of the higher solubility of calcium oxide in the more alkaline CaO-sucrose solutions.

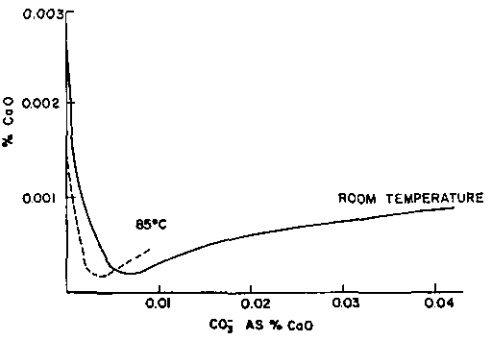


Figure 7-6. The solubility product of $CaCO_3$. (Brieghel-Müller, Brüniche-Olsen, 1949.)

Other salts present in the solution have a very distinct, but also rather individual influence on the solubility of calcium carbonate. Generally they increase the K_{SP} especially if the pH of the solution is altered, as with ammonium salts (58,99).

The effects of amino acids and proteins have been studied in great detail, owing to their importance in physiology (70). The ionic product $(Ca^{++})(CO_3^{=})$ in blood serum is much greater than in salt solutions of the same ionic strength. No

calcium carbonate does, however, crystallize, even in contact with solid $CaCO_3$. Analogous behavior in second carbonation juices has also been reported (11).

Calcium carbonate thus very easily forms supersaturated solutions, even in second carbonation juices. No calcium carbonate dissolves in blood serum, from which Ca^{++} has been removed by oxalate. Nevertheless blood serum can maintain Ca^{++} in solution which has been added in the form of $CaCl_2$. Some similar observations have been made in second carbonation juice (25).

There is a distinct effect from PO_4^{3-}, which seems to inhibit the crystallization of $CaCO_3$ in the blood serum. The stabilization of $CaCO_3$ solutions by hexametaphosphates is well known (77). A precipitate of calcium carbonate does not form in solutions of carbonates immediately after the addition of a soluble calcium salt (44).

Freshly precipitated $CaCO_3$ is very often amorphous, and is changed relatively slowly into a crystalline form. In one case (59) it was not completed at room temperature even after 15 minutes, and the electrical conductivity did not become stabilized until 54 minutes of stirring. Analogous, very slow changes have been observed (19,20,42,81) in sugar-lime solutions to which CO_2 was added in quantities insufficient for complete neutralization. The system did not stabilize after even weeks of continuous agitation at ordinary temperatures.

Calcium carbonate particles in contact with water have a positive electrical charge (94), which seems to play an important role even in practical factory operations (134).

It can be clearly seen (see Figure 7-7) from the dissociation curves of $CO_3^{=}$ and Ca^{++} which widely overlap, that the zone of existence of $CaCO_3$ is rather limited, and that there must always be a large part of $CaCO_3$ hydrolyzed.

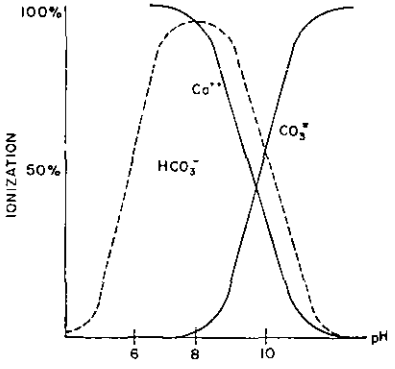


Figure 7-7. Ionization curves of H_2CO_3 and $Ca(OH)_2$.

Lime + Carbon Dioxide + Sucrose. This system, which might serve as a model of carbonation, has been extensively studied (71,129,133) from the very beginning of its industrial application. It can readily be surmised from what has already been said regarding lime, sucrose, carbonic acid, and calcium carbonate that the conditions are extremely complex, and owing to great experimental difficulties, such as in the analysis of the precipitate formed, the system is far from completely understood.

A very instructive picture—simplified but sufficient for a first approach—is given by curve 1 of Figure 7-8 (1,19).

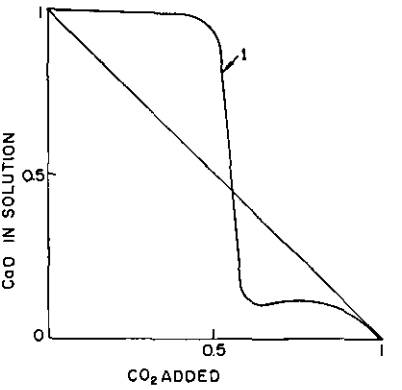


Figure 7-8. Carbonation of lime-sucrose solutions. *(Aten-van Ginnekin, 1927.)*

If a pure, clear, aqueous, sucrose-lime solution is neutralized with measured amounts of carbonic acid, or with any acid forming insoluble calcium salts (35,36,63), there is at first a period during which the juice remains clear and without apparent change. Samples of these clear solutions show a decrease in alkalinity, and an increasing difference between alkalinities at pH = 6 (methyl red). These solutions yield precipitates if their alkalinity has been lowered, either by dilution with water, or by titration with an acid. The electrical conductivity changes at the same time in a very characteristic way, and the polarization drops distinctly (42). This all proves that reactions are taking place involving all the solutes; most probably the formation of soluble sucrocarbonates.

After about half the acid necessary to neutralize all the CaO present has been added, the second period starts, in which a voluminous precipitate is rapidly formed, sometimes preceded by a very marked increase in the viscosity of the solution. The alkalinity of the filtrate drops far under what would correspond to the quantity of acid added, showing clearly that the precipitate must contain CaO, bound otherwise than in the form of $CaCO_3$ (81). The polarization indicates a loss of sucrose, which must thus also form a part of the solid phase.

From this point on in the third period, the alkalinity of the filtrate remains on the whole constant, though further large quantities of CO_2 are bound by the system. Instead of the initial carbonation "in solution," there is now a carbonation "of the precipitate," which must thus continually change in its chemical composition.

There is, however, an abnormality; i.e., a temporary increase of alkalinity in the liquid, which takes place even though the amount of acid added to the system is steadily increased. This curious phenomenon was reported for the first time in 1880 (112), and since that time has been rediscovered a number of times (1,30,35,36,92).

These three periods are the more distinct, the higher the alkalinity of the clear solution, and the more rapidly the acid is added. The second period, involving the instantaneous formation of precipitate, is especially marked (41) if the juice or solution contains large amounts of dissolved lime, and if it has been heated during a certain time to temperatures in the vicinity of 90-95 °C. The precipitate or carbonation sludge in such a case contains considerable amounts of sucrose in "bound" form, which can be washed out only with great difficulty.

The physical condition of the precipitate also undergoes very profound changes, and it can only be easily filtered off after the temporary alkalinity maximum. Its volume decreases considerably with the decrease in alkalinity.

It is important to remember that in the short lapse of time represented by an actual carbonation, these systems are never in real equilibrium. Therefore, very fundamental, and, from the technological point of view, very important changes between the liquid and solid phase are steadily proceeding as long as the juice and sludge remain in contact. Any increase in retention time before filtration, as in the clarifier, or prolonged contact between precipitate and juice as in the filter presses, must necessarily lead to important chemical and physical changes, and may affect the final results of the juice purification.

Curve 1 of Figure 7-8 applies particularly in batch carbonation, where juices limed in the cold are gassed at high temperatures. The lower the initial alkalinity; that is to say, the less the total drop in the alkalinity of the solution during carbonation, the less apparent are periods 1 and 2, and the more the changes occurring in the third period are reduced. By this is meant the carbonation "of the precipitate," which in this case consists merely of undissolved CaO, plus the nonsugars precipitated with lime in case beet juices are involved.

2. The Juice Purification Proper

Fundamental Operations. The literature of juice purification by lime and carbonic acid presents a very great number of different processes. To understand them and in what respects they differ, a clear picture must be given of the possible fundamental operations or reactions between the nonsugars and lime and carbonic acid, which inevitably must take place whatever the details of the particular method.

It is clear that there can be no reaction between lime and the nonsugars or between lime and carbonic acid unless the lime is first dissolved in the juice. When dissolved, the lime must immediately start to react with the nonsugars present. Such reactions are of two types: 1) leading to the formation of precipitates, and 2) giving soluble products.

Thus, the first fundamental partial operation is the formation of insoluble products from nonsugars and lime. With normal juices from healthy beets, these reactions require only very small amounts of lime, of the order of 0.2-0.3% CaO on juice, and this partial operation is thus called *preliming* or *predefecation*. Its aim is to precipitate as fully as possible all nonsugars able to form insoluble products with CaO.

The second partial operation includes those reactions between lime and nonsugars, which do not result in insoluble products. A reaction of this type is the destruction of invert sugar, or the saponification of amides. These are all molecular, organic reactions, requiring appreciable time for their completion. Their velocity, completeness, and even the direction in which they proceed, are functions of the concentration of the lime in solution, and the temperature. They thus take place most fully in the completely limed hot juice. This partial operation is called *liming* or *defecation*, and its chief function is in the *destruction reactions*.

After the liming, when all the possible reactions between lime and nonsugars have taken place, the third fundamental operation is introduced, and it aims to precipitate with carbonic acid the excess of lime added to the juice. It has been found advisable to carry out this precipitation in at least two steps: first and second carbonation.

We therefore call this third operation the *first carbonation.* ["Carbonation" has universal use in America. The longer "carbonatation" is used in England, and in America for cane-juice carbonation, being introduced from England.] Its main purpose is to precipitate the excess of lime and to bring the alkalinity of the juice to a certain low value, of about 0.1% CaO.

The juice, which is normally separated from the precipitate, is now further carbonated, not only to eliminate the balance of the lime left after first carbonation, but also to precipitate the Ca^{++} of the soluble lime salts in the form of calcium carbonate. This fourth operation is called second carbonation, and its primary aim is the final deliming of the juices.

These four fundamental operations are not always found distinct and clearly separated in the actual factory process. They very often take place simultaneously, or are partly suppressed, or even apparently do not take place at all. But in reality they are bound to occur, and they must always be looked for when analyzing any method.

Preliming. The precipitates produced in preliming can be divided into ionic and colloidal types.

The first ionic reaction of lime is the neutralization of acidity. The other ionic reactions have been dealt with in the chemistry of acids giving insoluble lime salts (p. 43). They are commonly quite simple and rapid, taking place as soon as the lime dissolves. As the solubility products of these salts are, by definition, rather low, no great excess of Ca in the solution is required. There may be, of course, many secondary complications and difficulties in the formation of the solid phase. For example, it has been shown that it is better even for these reactions to add lime slowly (6), according to the progressive preliming of Dedek and Vasatko, in order to precipitate calcium phosphates at low alkalinities or pH values where the more crystalline $CaHPO_4$ is formed, and to avoid the bulky, amorphous $Ca_3(PO_4)_2$ which is formed if the lime is added too quickly.

Certain other reactions take place in preliming which do not start until there is a certain amount of lime in the juice. From Vasatko (122-125) we have some knowledge of the precipitation of protein substances, among all the other reactions. This second type of precipitation can therefore be called the colloidal reactions.

The general behavior of protein matter has already been described (pp. 63,64). The added lime seems to have two different functions: first, to create a certain pH in order to give the protein molecule a sufficiently negative charge; and second, to form undissociated calcium proteinates of low solubility. The exact mechanism, which is not yet known, is of course more complicated and certainly involves also purely colloidal functions of the lime, since crystalline CaO prepared from calcium nitrate, behaves in a distinctly different way (125).

This alkaline coagulation seems, according to results of cataphoresis experiments (124), not to be due to an isoelectric state, as is the case in coagulation by acids. There is no doubt that the coagulation is a function of both the OH^- and Ca^{++} concentrations. Conclusive proofs have been given (47) that there is no constant pH, specific for the proteins in question, which would correspond to the maximum precipitation. In juices of similar composition, the amounts of protein precipitated are very different, even though different bases have their coagulation optimums at the same pH, thus demonstrating the dual function of alkalies mentioned above. The precipitation with bivalent cations is far more efficient than by alkali metals.

All this results in a very characteristic and peculiarly shaped coagulation curve I (see Figure 7-9). There is a very distinct maximum for protein precipitation, (a), corresponding to a certain "optimum" alkalinity or pH (b). Such a curve is found most readily when different increments of lime are added quickly, and all at once, to samples of the original diffusion juice. This curve corresponds to an equilibrium state, which can be reached from higher or lower alkalinities, at least with monovalent alkalies. It can be seen from this curve that if the maximum protein elimination is to be obtained in the preliming, and if the lime is added "at once," rapidly, exactly the necessary amount of lime must be used. Even a very small deviation from this optimum addition results in a far less efficient elimination of proteins. In actual factory work, where the composition of the diffusion juice is rapidly and constantly changing, it is practically impossible to ever achieve this maximum effect when adding the lime at one time.

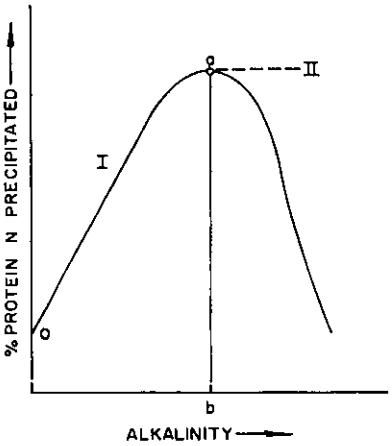


Figure 7-9. Precipitation of proteins in beet juices. *(Vasatko, 1933.)*

The fact that the manner in which the lime is added to the juice exerts a profound influence on the form of the precipitation curve is of considerable practical importance, and still further complicates the conditions. When lime is added very slowly, a certain stabilization of the precipitates, when formed, takes place, which makes it possible to add even a fair excess of lime without losing any of the coagulation already accomplished. In Figure 7-9, the curve from the 0 abscissa point to "a" is followed, and then instead of pursuing the descending slope of curve I, line II is followed, thus maintaining the full effect of preliming. This so-called progressive liming of Dedek-Vasatko[1] is therefore, if carefully executed, fully "automatic," requires little chemical control, and makes certain the full effect of preliming. The fundamental condition is the slow and progressive increase of alkalinity.

The mechanism is not yet understood completely. Two possible explanations have been advanced (21,30). By the slow addition of lime an excessive degree of supersaturation is avoided, and therefore the formation of a fine-grained precipitate

[1]See refs. (5, 7, 21, 22, 46, 73, 74, 121)

is likewise avoided, which would readily redissolve because of its great surface area. This would also explain why the alkalinity can be increased more rapidly at higher temperatures. At the same time an ionic exchange between calcium and the H, Na and K of the colloidal micelles is allowed, thus favoring the precipitation of the less ionized and less hydrated calcium forms. In confirmation of this it is known that a favorable effect is achieved by maintaining for a time a very low alkalinity in which $Ca(OH)_2$ forms practically only Ca^{++} ions.

The importance of this particular condition of progressive preliming was discovered by Brieghel-Müller, and called *stabilization.* It has a very desirable effect on the physical qualities of the precipitate formed, since its volume is smaller and it can be sedimented more rapidly. By this stabilization it is possible to retard the formation of the precipitate even if the alkalinity is increased, making it possible to postpone, or even completely prevent the precipitation of proteins and other colloids in preliming. In such a case, the proteins seem to be eliminated by adsorption on the calcium carbonate during first carbonation.

It now seems generally acknowledged that the effects of progressive preliming are very desirable. The colloids, such as proteins and pectins, are removed as a compact precipitate which settles rapidly and filters well, especially if the preliming is carried out at temperatures above 60°-70 °C. This considerably increases the filterability of the first carbonation sludge, particularly in batch carbonation, making possible savings in the total amount of lime used, filter area required, and sweetwater.

Progressive preliming is an integral part of some of the more recent methods of continuous carbonation, such as that of Wiklund (134) and of Brieghel-Müller (10). In these methods, progressive preliming has been further developed, not only by the stabilization of Brieghel-Müller, but also, in the Wiklund process by the return of slightly over-carbonated unfiltered first carbonation juice to the diffusion juice. Here, the colloidal floc of the preliming is formed in the presence of a mineral skeleton (48) of relatively high specific weight, which seems not only to act in the capacity of seed, but also makes the precipitate heavier and less hydrated (30), and so increases the velocity of sedimentation, besides enhancing agglomeration into large secondary particles (134).

[*Supplement to Professor Dedek's text.* The predefecation process evolved in the 20th century, largely by trial and error. Starting as a single, small lime addition patented by Kowalski and Kozakowski (65) in 1901, it apparently had brief use, and was then forgotten until noted by Naehring (95) and was then studied thoroughly by Spengler and members of the Berlin Sugar Institute, and separately by the Czech Institute at Brno. The latter studies led to the above described developments of Dedek.

Since 1951, the precipitation or flocculation of colloids in preliming has been studied by a number of workers. Dubourg (52) reported on flocculation with other alkaline materials, and found monocalcium saccharate quite effective. He also found no purity increase resulting from predefecation. When preliming was done with calcium saccharate, he noted (55) that flocculation is increased by heating after preliming. Shaposhnikova (106) studied coagulation of colloids in the two pH areas, 2.5-4.5 and 10.5-11.0. In the first region, where the true isoelectric points exist, neutral micelles were found to be formed through adsorption of hydrogen ions, whereas in the alkaline region the micelles are stabilized by adsorbed Ca^{++}.

Significant microscopic studies were made by Kohn (84) of the structure of the colloids coagulated in predefecation. The sludge volume was found to be 70-80 times larger than the true particle volume. The individual particles were found to be rods, and the loose configuration indicated a system of elongated-rotated ellipsoids. Kalab (78) noted that predefecation sludge settles more rapidly when lime rather than calcium saccharate is used as the alkalizing agent. Studies made by Dedek (31) which had indicated the possibility of ion-exchange action of the colloidal micelles, exchanging Ca^{++} from the solution for H^+, Na^+, or K^+ of the proteins and pectins, were not in general confirmed by Tibensky (114), since he observed a small exchange of H^+ ions, but none of Na^+ or K^+.]

Liming. The more important destruction reactions which may take place during liming are saponification of amides such as glutamine and asparagine into the corresponding ammonium salts:

$$RCONH_2 + H_2O = RCOONH_4; \qquad (15)$$

the decomposition of glucose and fructose into colored and acid products; and the formation of oxalic acid from the oxalogenic substances.

Nothing is known of the changes which certainly must occur in proteins and pectins, and in the mother substances of coloring matter. The general effect is, however, in the direction of a repeptization of the precipitate already formed, and an increase in the degree of dispersion; long-chained molecules probably being split into shorter ones.

These destructions probably result in the formation of some acid products, which in the presence of lime must inevitably form lime salts. On the other hand, the use of large total amounts of lime will remove many of these lime salts by adsorption on the large calcium carbonate precipitate in first carbonation.

The effect on color is, on the whole, very favorable. The resulting juices are "thermostable." They will stand further heating in the evaporators without alteration, a treatment which is relatively milder than that which they have already undergone in the energetic liming.

It is, nevertheless, rather difficult to combine energetic liming with good sedimentation and filtration, as all the lime is dissolved, and the heating may easily lead to highly supersaturated solutions, tending toward the formation of sucrocarbonates and saccharates.

As the oxalogenic substances have given off their oxalic acid before first carbonation, the danger of formation of calcium oxalate deposits is greatly lessened.

For the achievement of all these effects it is necessary to use an energetic liming.

Mild liming, on the contrary, avoids all of these destructions. It may therefore be used without hesitation when processing raw juices which are themselves sufficiently thermostable, or with thermolabile juices if other conditions are favorable; for example, if all the following processing is mild as with modern evaporators with short retention times, simple, rapid, boiling schemes, and nonreturning of syrups. If thermolabile juices are still treated with mild liming, some of the destructive reactions will occur during the later stages of processing, leading to losses of alkalinity and finally to inversion of sucrose (23), and even to corrosion of the evaporators, as well as marked increase in juice colors. Such juices are, however, low in lime salts, and permit the use of first carbonation methods giving excellent filtration velocities.

If it is desired to achieve the fullest effect of liming and to complete all the destruction reactions, as much as possible of the CaO must be put into solution in the juice, and it must act as fully as possible. In such a case, large quantities of lime must be added, such as 1.5 to 2.5% CaO, with the juice at a low temperature, such as 20° to 30 °C, in order that it will go completely into solution, and not remain as a suspension of solid CaO or $Ca(OH)_2$ in a juice of only slight alkalinity. Such a strongly alkaline juice must then be heated to a high temperature, such as 85° to 95 °C, and kept for 10 to 20 minutes before carbonation. This is what is termed an *energetic* liming.

In contrast, a *mild* liming is one in which the destruction reactions are limited as far as possible. In this case, the slightly acid diffusion juice is heated, and one of the methods of liming and first carbonation used in which the alkalinity is always maintained at a rather low value, such as when liming and gassing are simultaneous, as in the Dorr system. Specific local conditions determine which of these alternative methods is preferable.

First Carbonation. The main purpose of this operation is to precipitate the excess of lime added during liming, or during the gassing itself, in the form of calcium carbonate. It is clear from the previous sections that this is no easy task, when the many side reactions which are generally taking place simultaneously are considered. To make it more difficult, the precipitated calcium carbonate is used in two different ways: namely, as a reagent for the further chemical purification of the juice, and as a filter aid. These two uses make opposing demands on the physical quality of the precipitate formed. The chemical purification, which seems to be mostly due to adsorption, is favored by a high surface area of the precipitated calcium carbonate, with which sufficient velocities of sedimentation and filtration are hardly compatible.

It can therefore be stated, in general, that any condition which increases the surface of the calcium carbonate precipitate in first carbonation tends to improve the chemical purification of the juices. Lime salts and colors are by far the most sensitive criteria. At the same time the speeds of sedimentation and filtration are lowered, and also very frequently difficulties are observed in the desweetening of the cakes, due to the coprecipitation of sucrose in the sludges.

On the other hand, any increase in the size of the calcium carbonate grains formed, which is equivalent to a smaller total precipitate surface, lowers the effect of chemical purification, but is, of course, favorable both for sedimentation and filtration.

The physical form of the calcium carbonate precipitate depends first on the initial alkalinity before gassing, or better, on the drop in alkalinity during gassing. This is clearly seen from Figure 7-8. The greater this drop, and the more suddenly it takes place, the more rapidly is the precipitate formed, and the finer, in general, its grain, and thus the greater its total surface.

To obtain coarse-grained precipitates, the first precaution is to avoid, as far as possible, high supersaturations of calcium carbonate. This means first of all a low initial alkalinity, which is identified not only with a low concentration of Ca^{++}, but also, through the lowered pH, with a limited degree of ionization of the carbonic acid to $CO_3^{=}$.

This low initial alkalinity can be secured either by liming a hot juice, in which CaO is only slightly soluble; or by using continuous carbonation, where the juice in the tanks is kept constantly at a low alkalinity, which is essentially the final alkalinity of first carbonation; or by recycling several hundred percent of unfiltered first

carbonation juice, which means diluting the limed juice with several volumes of a juice of much lower alkalinity; or, finally, by liming and gassing simultaneously. The recycling of unfiltered first carbonation juice involves also a "seeding" with preformed $CaCO_3$ particles, bringing into the limed juice a previously formed surface of $CaCO_3$ on which it can easily crystallize, instead of forming small, new nuclei.

The second most important condition of first carbonation is its final alkalinity or pH. Since the first carbonation sludges still contain particles of the colloidal matter precipitated during preliming, or at least by the mechanism of preliming, reference to Figure 7-9 shows clearly that if the final alkalinity of first carbonation is lower than the optimum value for preliming, it is possible that redissolving of these particles with resultant juice degradation can take place. This is why Dedek and Vasatko have considered the final alkalinity of first carbonation as, in principle, identical with that of preliming. There is not yet sufficient experimental evidence, however, to be able to judge conclusively to what degree the precipitates formed in a correct preliming are really redissolved in low alkalinity juices. It is probable that the coagulation under such circumstances is to a degree irreversible, even if only a slower rate of re-solution is involved.

Another reaction in overgassing is brought about by the changes in the composition of the lime precipitate itself—especially its free CaO content. Figure 7-8 shows how profoundly the precipitate changes as carbonation proceeds. This lowering of the "free" CaO content of the muds can strongly affect the chemical purification (43). The experiments demonstrated that it is the total alkalinity, to a phenolphthalein end point, of the unfiltered and not the filtered first carbonation juice, which is correlated with the maximum purification effect. In general terms, this means that the free CaO in the sludge plays a definite and important role in juice purification, and that any type of carbonation which leads to "neutral" sludges of low CaO content must necessarily prove less efficient in juice purification, other conditions being equal.

It seems certain, although requiring further experimental confirmation, that adsorption of nonsugars, in particular coloring matter, on the first carbonation sludge is favored by alkalinities slightly higher than the normal alkalinities of first carbonation (0.12 to 0.15% CaO) (91,109). Any lower value, while the sludge is still in contact with the juice, thus involves a freeing of coloring matter, which passes back into the juice—another example of the noxious effect of overgassing. The alkalinities giving the lowest colored juices, however, are often so very high that they cannot be used in factory work, owing to difficulties of sedimentation and filtration.

There are, of course, some beneficial effects from overgassing. Velocities of sedimentation and filtration are accelerated and sludge volume reduced. Such sludges do not normally contain any bound sugar, and are therefore easily sweetened off. Their greater porosity and lower resistance can, however, lead to a less efficient washing.

A rather interesting phenomenon, caused by overgassing, has been discovered by Wiklund (134). The primary sludge particles in such overgassed, unfiltered first carbonation juices easily form large agglomerates, which sediment and filter very quickly. Wiklund believes that through overgassing, "acid" calcium pectates are

formed which are negatively charged, and thus attracted to the positively charged calcium carbonate crystals. Unfiltered first carbonation juices containing these aggregates can be used with great advantage for the seeding of diffusion juices before progressive preliming, serving not only as excellent nuclei for the newly precipitated colloidal substances, but since the agglomerates are cemented together, they can be subjected to even an energetic liming without being again split into the smaller primary particles. Through judicious recycling very large particles may be built up, with excellent sedimentation and filtration rates, together with the full benefits of both preliming and final liming.

It has been pointed out that very often gassing is so rapid that the precipitate is unable to quickly enough adapt itself to the changing alkalinity. Any increased time of contact between the juice and the sludges, especially under conditions of high temperature and agitation, tends thus to further this adaptation, or to bring about further chemical changes in the sludges. It is not possible to tell in advance whether these changes will increase or decrease the total chemical effect of juice purification.

At the same time all the very complex aging phenomena, such as recrystallization, so well known in quantitative analytical chemistry (85), are proceeding, accompanied by great changes in the total surface and in the quantity of adsorbed nonsugars. These changes mostly diminish the effect of purification.

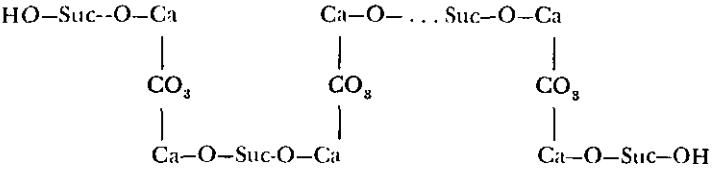
If thermolabile first carbonation juices, which did not receive the benefits of energetic liming, are kept at high temperatures for long times in contact with their sludges, they necessarily undergo destruction reactions, which are under less favorable alkalinity conditions than in an actual final liming, thus giving rise not only to lime salts, but also to relatively large color increases (93). This may explain the many contradictory observations concerning the effects of retention of first carbonation juices before filtration.

The greatest change in the equilibrium between sludges and juice takes place at the moment of sweetening off. The juice is replaced by hot condensed waters, very often containing ammonia and carbonic acid. This is certainly one of the reasons that the purity of the sweetwater is often so low, since quite appreciable amounts of nonsugars—especially lime salts—are repeptized.

From what has been said, it is clear that, on the whole, the use of high temperatures is responsible for many unwanted destruction reactions. This is the reason that rapid, low-temperature processing at one time attracted much attention. It has been shown that considerable gains in purity can be obtained by means of a "cold carbonation" (27,43) with measured quantities of CO_2, and that by the use of mild treatment during the whole purification, very low lime salts are obtained even with very small amounts of total lime use. These methods, however, can only be used in factories where the evaporators, vacuum pans, and crystallizers also permit rapid, low-temperature processing. It is frequently not appreciated how very unstable and chemically delicate are not only the nonsugars of beet juices, but even sucrose. Rapid processing is therefore always the key to success.

[*Supplement to Professor Dedek's text.* The late Prof. Dubourg has made extensive studies of the formation of complexes between calcium, sucrose, and carbon dioxide, and has reported them in a series of publications (51). He postulated the formation of a gel of calcium sucrocarbonates while

passing CO_2 through calcium sucrate solutions. These were compounds of calcium saccharates and $CO_3^=$, such as



or $(-\text{Sucrose}-O-Ca-CO_3-O)_n$

Dorfmüller (50), commenting, doubted if pure calcium saccharates exist, and suggested that the compound obtained is calcium carbonate esters of sucrose. In two other papers, Dubourg further developed his hypothesis (54,56). Tornic (117) described work confirming the existence of sucrocarbonates. Kohn studied the dynamics of the reactions of formation of calcium sucrocarbonates (83).

Dedek himself in 1952 (32) reported laboratory tests showing strong purification effects (as much as 1.5 purity points increase) from adsorption on $CaCO_3$ crystal faces in first carbonation.

That almost all of the lime in first carbonation is present in the form of hydroxides was concluded by LeMaitre (89) on the basis of potentiometric studies.]

Second Carbonation. Whereas the manifold aims of first carbonation make it difficult to obtain every desired result simultaneously, the chief aim of second carbonation is much simpler: the elimination by carbonic acid of all the lime which can be precipitated as calcium carbonate. The chemistry of second carbonation is, notwithstanding, very complex. The importance of electrochemistry in second carbonation has become clear[1].

The fundamental equations may be written as

$$CO_2 + OH^- \rightleftharpoons HCO_3^- \quad (16)$$

$$HCO_3^- \rightleftharpoons CO_3^= + H^+ \quad (17)$$

$$CO_3^= + Ca^{++} \rightleftharpoons CaCO_3 \quad (18)$$

and the corresponding equilibria are

$$\frac{a_{CO_3^=} \cdot a_{OH^-}}{a_{HCO_3^-}} = K_{HCO_3^-} \quad (19)$$

$$a_{CO_3^-} \cdot a_{Ca^{++}} = K_{SP\text{-}CaCO_3} \quad (20)$$

The homogeneous reactions between CO_2 and OH^-, Equations (16) and (17), will first be considered. As the gassing proceeds, the total amounts of bound carbonic acid, or $CO_3^=$ and HCO_3^- are increased, whereas the concentration of OH^-, or the pH, is diminishing. But, with the lowering of the pH there takes place a simultaneous shifting of the equilibrium between $CO_3^=$ and HCO_3^- in the direction of increase of the bicarbonate ion (see Figure 7-7 and Equation (19)). At the same time the velocity of hydration of CO_2 to HCO_3^- will steadily slow down, as is apparent from an inspection of Figure 7-4.

[1]See refs. (11, 12, 26, 29, 38-40, 75, 105, 119)

These two processes; i.e., the increase in the total amount of bound carbonic acid on the one hand, and the shifting of the $HCO_3^-/CO_3^=$ equilibrium towards the bicarbonate region on the other, will result in a maximum amount of $CO_3^=$ in a solution at a certain pH. This pH will, hence, be optimum for second carbonation, allowing reaction **(18)** to proceed furthest to the right, and leading to the elimination of the greatest amount of Ca^{++} from the juice.

If the juice is gassed further, the $CO_3^=$ concentration will decrease steadily, and the Ca^{++}, which, in accordance with Equation **(20)** can co-exist in solution, will increase, with a less efficient deliming.

The total amount of carbonic acid bound is thus a function of the amount of OH^- present in the juice.

The efficiency of the deliming, as measured by the quantity of Ca^{++} left in solution, will, however, depend on the ratio between the total amounts of Ca^{++} and $CO_3^=$. Other conditions being equal, the lower this ratio, the more complete will be the deliming, and the less Ca^{++} will remain in the juice. This follows from Equation **(20)**.

Each CaO molecule dissolving during liming in the form of $Ca(OH)_2$ in the juice, yields not only its Ca^{++} but also the corresponding number of OH^- ions necessary for its precipitation by carbonic acid. Some of the $Ca(OH)_2$, however, gives rise to the formation of soluble lime salts. The very important excess of OH^- which is needed for the deliming of the Ca^{++} of the soluble lime salts having no corresponding OH^-, is the "natural alkalinity" of KOH and NaOH formed through reactions giving insoluble lime salts, and possibly also through some ionic exchange (30). The greater this alkalinity, the more efficient is the deliming in second carbonation.

Actual conditions in second carbonation juices are, however, more complicated. The presence of sucrose, the total ionic concentration, and the temperature have a profound influence on the ionization of HCO_3^-. The sucrose and all the other acids present in the juices, water included, take part in the equilibria, and, strictly speaking, must be considered and added to Equations **(16)** and **(17)**.

It has been shown (119) that the lower the pK of the acids present in the form of lime salts, the less efficient the deliming, other conditions being equal (see Figure 7-10). The very important and unpleasant role of sucrose, which was noted a number of years ago (119) is clearly shown in Figure 7-11 (12). Even at the optimum concentration of $CO_3^=$, it represents, in the form of saccharate ion more anion equivalents than $CO_3^=$ itself.

Buffer substances present in the juice diminish the pH due to the natural alkalinity, and thus shift the $HCO_3^-/CO_3^=$ equilibrium to give lower concentrations of $CO_3^=$ and consequently a less efficient deliming. It was therefore proposed (11,12) to use the so-called *effective alkalinity* instead of the natural alkalinity as a measure of the possible deliming capacity of a given juice. Natural alkalinity is determined by titration to 8 pH, while effective alkalinity is determined by titration to pH 9.25, which is close to the usual optimum for second carbonation.

The very great influence of natural alkalinity is also the reason why it is so important to avoid unnecessary acid formation in the juices before second carbonation. Thus, acid-forming fermentations, such as those in the diffusion battery, and the use of sulfur dioxide before second carbonation, should be carefully avoided.

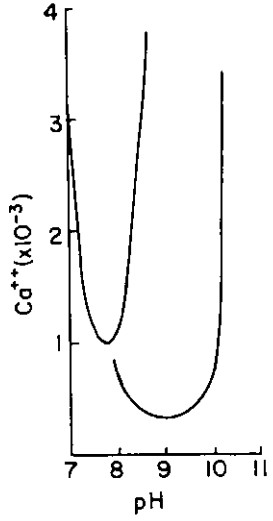


Figure 7-10. Influence of the strength of acids forming lime salts, on deliming. *(van der Heide, 1928.)*

If the natural alkalinity is not sufficient, an artificial one must be added if it is desired to delime the juices. The most popular agent is soda ash, which should be added, at the latest, at the beginning of second carbonation. Various aspects of its use have been discussed several times (29).

Further important complications are due to the fact (14) that the concentration of Ca^{++} ions, which take part in the above listed fundamental reactions of second carbonation, is much smaller than the total concentration of Ca as determined analytically. Several possible forms of un-ionized Ca have already been mentioned in earlier sections.

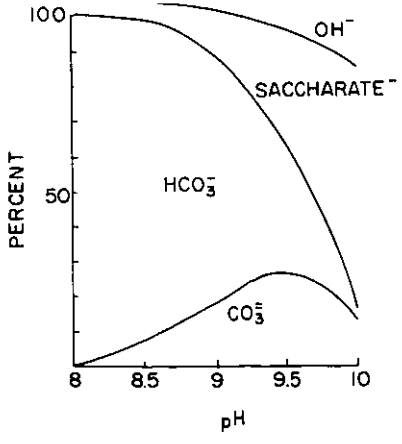


Figure 7-11. Ionic equilibria between OH^-, saccharate, HCO_3^-, and $CO_3^=$. *(Brieghel-Müller, Brüniche-Olsen, 1949.)*

The actual deliming is not completed, however, until all the $CaCO_3$ formed has crystallized out. Sufficient has been said about the peculiarities of this action and the facility with which $CaCO_3$ forms stable, supersaturated solutions, to show that a delayed crystallization may seriously affect the total deliming achieved.

Sulfur Dioxide, Sulfurous Acid. The use of sulfurous acid for juice purification in the beet-sugar industry is very old. It is a far stronger acid than H_2CO_3 ($pK_1 = 1.7$ and $pK_2 = 7.2$ at 25 °C) and can consequently be employed for the neutralization of excess alkalinity in juices and syrups.

Further, it is a well-known reducing agent, and can thus serve to decolorize juices, by reducing coloring substances to their leuco-bases. For instance, colored decomposition products formed by heating glucose are decolorized by $NaHSO_3$ at a pH of 5.5 (90).

Still more important seems the preventive effect of even traces of SO_2 or $SO_3^=$ on many oxidations, described by Moureau as *antioxidative* catalysis. This may explain the undisputably favorable effect of SO_2 on evaporator thin juices.

Sucrose is known to considerably retard the oxidation of $SO_3^=$ to $SO_4^=$ (72,103,128). Calcium sulfate has been reported (102) as being definitely less soluble than calcium sulfite in sugar solutions, and this transformation may, as a result, lead to deposits of calcium sulfate in the evaporators. The solubilities are influenced further by the concentration of sucrose, the temperature, and the presence of other substances, especially ampholytes [amphoteric electrolytes] (88,97).

Important Aims of Juice Purification

The chief aims of juice purification, from the mechanical point of view, are the formation of muds which are readily sedimented and filtered. They are relatively easily measured with the aid of laboratory sedimentation tests (109,134) and measurements of filterability with a microfilter (9,34,37).

The chemical purification is aimed chiefly at forming juices which permit the production of a high quality white sugar, with a minimum loss of sugar in the molasses. It is not easy to determine this by chemical analysis. Owing to the low precision and doubtful interpretation of the analytical methods for the determination of dry substance and sucrose content in the juices, the determination of purities and the calculation of the percent of nonsugars eliminated is rather uncertain. Partial determinations, such as total nitrogen, are of course, inadequate to give a full picture of the results achieved and have not found general application. No chemical analysis of second carbonation juices can give us at present full information as to the quality of white sugar which will eventually be produced from it. There is apparently but one reasonably precise correlation between the loss of sugar in molasses and the second carbonation juice analysis; namely, the relation between milliequivalents of K and Na (plus milliatoms Ca) on one side, and the millimoles of sucrose, which, in the final molasses, tend to bear the ratio 1:1 (9,17,18).

These are some of the reasons why lime salts and colors (135) have become the most widely adopted criteria of the chemical effect of juice purification. They can be determined relatively easily, and with a high degree of precision. They are furthermore extremely sensitive to even small changes in the details of the juice purification procedure, and to differences in the chemical composition of the initial juices used.

Sedimentation and Filtration. Sludges are required which are not easily mechanically deformed or compressible, and as little hydrated as possible. Such muds can be produced if the following conditions are observed (30):

1) The initial diffusion juice must not contain an unnecessarily large amount of colloids, especially pectins, which are introduced mostly through excessive heat or high pH in the diffuser, which dissolves the marc of the beet.

2) All useless high degrees of supersaturation of all substances which are precipitated during the liming and carbonation should carefully be avoided. This means a very slow increase of the alkalinity of the diffusion juice, as in Dedek-Vasatko preliming; low alkalinities during the whole juice purification, involving liming at high temperatures, recycling as in continuous carbonation, and liming and gassing simultaneously as in the Dorr process or in the recent Scandinavian methods (10, 134); and high temperatures to lower $[a_{CO_3^=}]$.

3) Graining during carbonation, by recycling unfiltered first carbonation juice, or the thickened juice from clarifiers.

4) Graining during preliming, or by the same means as in 3).

5) Over-carbonating and recycling to favor formation of agglomerated particles as by Wiklund's method.

6) Provide for ionic exchange of pectins and proteins into calcium-forms, using the stabilization principle of Brieghel-Müller.

Colors and Lime Salts. Low color and lime salts can be obtained frequently at the price of slower velocities of sedimentation and filtration. They are generally favored by:

1) A thorough progressive preliming.

2) An energetic final liming, in which large quantities of lime are added to cold juice, heating to high temperature over relatively long periods.

3) A first carbonation precipitate of great adsorbing power, thus composed of small particles, and created through a high initial alkalinity in first carbonation and rapid carbonation.

4) High final alkalinity in first carbonation.

May it again be emphasized that there are no generally valid rules which guarantee the best effects with every kind of juice. The details of the juice purification to be used must be adapted to the character of the juice to be treated. This is the most important duty of a good chemist in a sugar factory.

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Chapter VIII

JUICE PURIFICATION, II.

Technology of Juice Purification*

Introduction

In 1930, most of the world's beet-sugar factories were using batch methods of juice purification. In broad outline, the next three decades were noteworthy for the conversion to continuous processes, in both Europe and America. At the end of World War II, the majority of the factories in the United States were using continuous systems. The process in general use in Europe in 1945, which was the result of the labors of Schatten and Michaelis, Rousseau, and Perrier and Possoz, has been called the "classic process," with its four elements: preliming, main liming, first carbonation, and second carbonation.

Since that time, European technologists have concentrated primarily on improvements in preliming. The American industry has continued in its conversion to continuous systems of defeco-carbonation, with primary stress on economy of labor and materials, and the capability of processing almost any type of sugarbeets. Each of the unit processes of juice purification making up the classical process will be described, with its respective advantages and disadvantages, and suggestions will be given for its most effective operation.

Unit Operations of Juice Purification

Preliming

Amplifying the definition previously given, the purpose of predefecation or preliming is to precipitate certain nonsugars, among which are acid anion groups with relatively insoluble lime salts such as phosphates and sulfate, proteins and their moieties, and colloidal substances which are not adequately removed in main liming.

Preliming is particularly important in the effective coagulation of proteins. If preliming is properly conducted there will be little peptization of the coagulum at the high temperature and alkalinity of main liming.

While the original Spengler preliming took place in one or two tanks, further multiplication of the number of lime additions was accomplished by adding a tank for each addition. Stepwise systems involving one-, three-, or five-step additions in as many tanks have generally been supplanted with "stepless" systems.

Following the introduction of the multi-stage progressive preliming of Dedek and Vasatko, it was shown that even when the lime addition is made in as many as seven or eight increments, local over-alkalizations somewhat damage the stability of the precipitate, and stepless preliming was introduced.

Brieghel-Müller Prelimer

The Brieghel-Müller unit was the first successful stepless prelimer, and predominates in present installations (103). The mathematical relationships for this preliming were published by the inventor (13).

Referring to Figure 8-1, the prelimer consists of a horizontal U-shaped trough, separated into six compartments, with vertical cross-partitions which do not extend all the way to the bottom of the trough. A shaft extends full-length, to which are

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*by R. A. McGinnis

attached stirring paddles. These paddles serve only to rotate, and not to propel the contents. At the top of each compartment is located a deflecting baffle, which can be adjusted to form different angles with the direction of juice traverse. Diffusion juice is introduced at one end, and flows through the unit by gravity, leaving as prelimed juice at the other end. The liming agent is introduced at the juice outlet end, and is propelled toward the juice inlet end by the influence of the baffles. Careful adjustment of these baffles gives a uniform, stepless increase of lime concentration in the juice under treatment, from zero at the one end, to fully limed at the other end. Any tendency for undue foam formation is controlled by addition of defoaming agents.

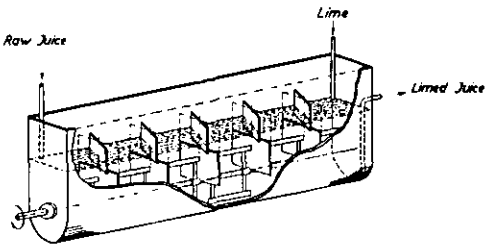

Figure 8-1. Brieghel-Müller Prelimer.

Other horizontal predefecating units followed. The Polish, Wschowa unit, containing six cells, was described in 1960 (69). An East German model was patented by Schuster (80) in 1961, which provided not only for preliming, but contained an additional cell for a "pH-pause" for stabilization, and three more cells for main-liming. A unit which carried the process even farther into defeco-carbonation, was patented by Buckau R. Wolf in 1953 (58). A horizontal unit with a relatively complex system for liming agent addition used at the Polish Goslawice factory (128) was patented originally in 1957 by Zagrodski (129).

Naveau Prelimer. Vertical predefecation units have the considerable advantages of occupying little floor space, and involve less foam formation. The first successful vertical unit was that of Naveau (63).

Referring to Figure 8-2, the vertical, cylindrical tank is separated into a number of compartments. The plates separating the compartments have openings around the central shaft, and around the periphery. Alternate cells contain large paddles, which move the juice centrifugally through the peripheral openings into the cells immediately above and below. The intervening cells contain small stirring arms, and in these cells the flow of juice is centripetal through the openings around the central shaft into the cells immediately adjoining. The main juice flow is from the point of diffusion juice entry at the top to its outlet at the bottom. The reverse flow of lime from bottom to top is obtained by the centrifugal flows, and the amount can be adjusted by the speed of rotation of the paddles, corresponding to the adjustment of the baffles in the Brieghel-Müller unit. Thus a steady decrease of lime concentration is built up between the fully limed juice leaving at the bottom, to the diffusion juice entering at the top.

Following the Naveau, many vertical units appeared. Czech workers developed a vertical unit (94) which was installed and tested (95) with results similar to those obtained with the Brieghel-Müller unit, but with no foam formation. In this equipment the flows were in the reverse direction from the Naveau, the diffusion

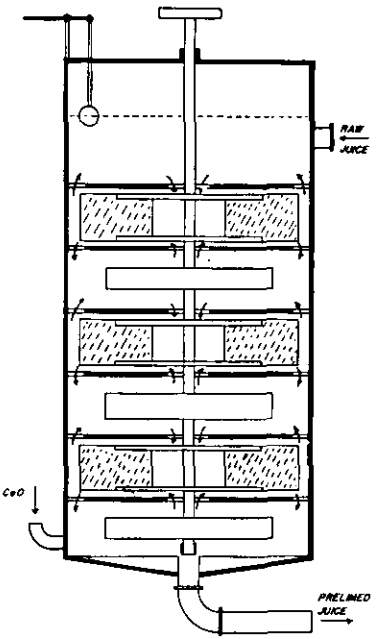

Figure 8-2. Naveau Prelimer.

juice entering at the bottom and the liming agent at the top. Reverse lime flows were obtained by rotating plates with sectors bent up at 30° angles. A Polish patent was issued in 1961 for an apparatus consisting of two vertical tanks, each separated into eight cells (19). The first tank served as a predefecator, in which the opposing flows were obtained by alternating centrifugal and centripetal flows, much as with the Naveau equipment. The second tank, operating on the same principle, was to be used for main liming. The Kollar predefecators, developed in both vertical and horizontal modes in Hungary, apparently operated by means mechanically very similar to others already described (105). An East German patent obtained by Teschner (89) was for a vertical unit which achieved the liming concentration gradient by return of partly limed juices from cells above each individual cell.

In 1966 a patent was issued to Dubourg (29) for a prelimer made of a series of jet ejectors. Each ejector embodied a pressure inlet at one end into which juice was pumped, a jet aspirator inlet at one side for entry of recycled juice, a Venturi tube, and an outlet for mixed juice. A proportion of the juice leaving each ejector was recycled to the preceding (up-stream) ejector, and the milk of lime or defecation juice was aspirated into the last ejector.

Utah-Idaho Prelimer. In 1966 a vertical prelimer, devised by the Research and Development Department of the Utah-Idaho Sugar Company, was installed, and its operation has been very successful (121). Referring to Figure 8-3, it consists of seven cells, with stirring paddles in each cell to give the juice contents a rotary motion. Juice flow is upward, countering the liming agent flow. The metal plates separating the cells have a 2 ft diam hole on one side, and on the other a wedge-shaped segment with a 52° angle has been cut out of the plate and hinged on one side, so that it can be slanted up from the plate to form an angle with the horizontal

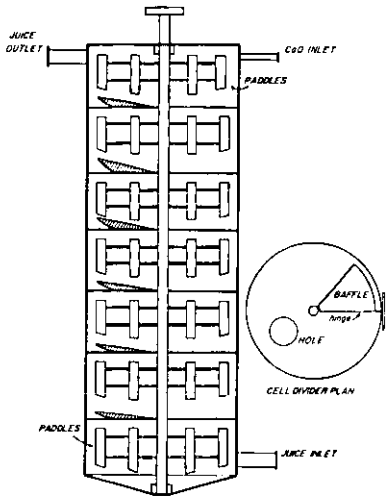

Figure 8-3. Utah-Idaho Prelimer.

plane of the separator. The main juice flow is upward through the circular holes, while the liming flow is downward, the amount depending on the angles of the baffles. The unit has the virtue of great simplicity.

Prelimer Operation. The Amalgamated Sugar Company has made its experience in prelimer operation available, and the following is based on this. The specific values are to be regarded only as typical, as the precise preferred values will vary with the equipment and the materials. The Amalgamated installation uses Brieghel-Müller prelimers, and first carbonation sludge recycle.

Three variables are available for pH gradient adjustment: 1) the setting of the deflector gates on each compartment separator, 2) change of the points of entry of the recycled sludge, and 3) alteration of the liming agent feed addition. Adjustment of the amount of lime feed shifts the curve shown in Figure 8-4 vertically up or down from a curve established by the first two adjustments.

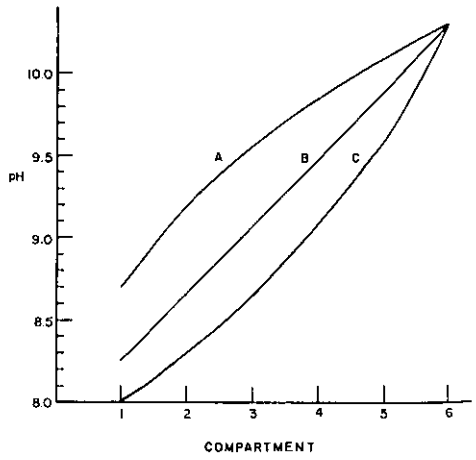

Figure 8-4. Curves showing characteristic pH patterns through a Brieghel-Müller prelimer. ***(Courtesy, The Amalgamated Sugar Company.)***

It is essential, on the basis of both theory and practice, that the pH gradient through the prelimer be such that sufficient time is given for the flocs to stabilize and dehydrate at each point. If the gradient rises too rapidly, these operations will not be completed, and the result will be the production of excessive foam, and a prelimed juice sludge which will have unsatisfactory settling and filtration characteristics.

Curves of the type A in Figure 8-4 can produce a juice with so much foam, and with such inferior handling sludge, that operations are very difficult. A curve of type B is a considerable improvement, but best operation is found with curves which are concave, as type C.

The curve shape desired is attained by deflector adjustment, and, if sludge is recycled, by changing its point of addition. The juice in which the sludge is recycled has a much greater influence on the alkalizing of the juice than the solid sludge particles. The pH at which the finished juice leaves the prelimer is set at an equivalent alkalinity of 0.005% CaO greater than the terminal alkalinity of first carbonation.

Upsets in prelimer operation can occur from unexpected variation of several different variables.

1) The lime addition to the last compartment should be automatically proportioned to the rate of flow of the entering diffusion juice, so that this does not require attention. Sludge recycle, however, may not be so proportioned, and a sudden reduction in the rate of slice without a corresponding reduction in the amount of sludge recycled, can form a convex curve of type A, with its consequent operating difficulties.

2) A change in the apparent Brix of the sludge slurry returned, because of the greater alkalinity of the juice phase, can abruptly change the curve shape. Thus, unusually large amounts of filter sluice water can reduce the Brix and increase the alkaline contribution of the recycled sludge, again tending to production of type A curves.

For its own particular circumstances, Amalgamated has set a maximum pH limit of 8.70 for compartment one (pH measured at the operating temperature), and a preferred operating region of 8.00 to 8.30 pH for this compartment. Gradient curves of type C permit larger pH variations in the individual compartments than curves of the A or B type.

For prelimer operation the following information is considered necessary on a continuously recorded basis: beet-slice rate; milk of lime or saccharate milk Brix; carbon dioxide gas pressure; the pH or alkalinity of compartments one and six of the prelimer; the Brix of the recycled sludge; the prelimer effluent temperature; the first carbonation temperature; and the recycled sludge supply tank level. It is helpful to have automatic control of the prelimer effluent juice pH or alkalinity through the variable liming agent feed rate to compartment six.

Calcium Carbonate Recycling

While the origins of the practice of recycling materials from various stages of the liming and carbonation process can be traced back into the last century (42,88), its intensive study and development in Europe followed shortly after 1945, possibly as a result of the success of defeco-carbonation in the United States. There it produced easily removed coarse flocs, which were stabilized by adsorbed coatings of small calcium carbonate crystals. There followed in Europe a 30-year development period, in which stabilization of predefecation flocs was achieved. There are numerous references to studies of recycling of a wide variety of materials to the dif-

fusion juice or to preliming. Among these recycled materials are: over-carbonated first carbonation juice[1] as in the pioneer Wiklund process; whole, normally carbonated first carbonation juice[2]; under-carbonated first carbonation juice; or normally-carbonated first carbonation juice with added lime[3]; thickened first carbonation sludge[4]; second carbonation sludge (1); sludge from prelimed juice which has been partly carbonated and thickened (127); milk of lime or saccharate milk which is partially carbonated (116); juice from the main defecation (69); and specially manufactured, pure calcium carbonate crystals (104).

When predefecation flocs form in the presence of small calcium carbonate crystals, with gradual increase of pH over a sufficiently long time period, the flocs become quite stable, and the agglomerates formed, with their coatings of calcite crystals, are much more resistant to chemical damage in main liming, although some peptization and juice degradation may still occur. These agglomerates provide good bases for further calcium carbonate crystal accumulation in first carbonation. The final first carbonation precipitate settles relatively rapidly in the clarifiers, and the mass is porous enough to permit efficient desweetening in the filters. Likewise the thesis that the return of carbonation sludge to the raw juice prevents peptization by protein degradation products has been shown to be in error (64a, 111d).

In material recycling at least five actions are occurring. Calcium carbonate crystals are added to the diffusion juice, lime is added which raises its pH, the diffusion juice is diluted (17), the addition raises the juice temperature (24,43), and, from another standpoint, "carbonation" (acidification) of the recycled juice is continued by the diffusion juice acids.

As a whole, all of these actions are advantageous for the purposes of preliming. However, since the calcium carbonate crystals must be readily sorbed to the coagulating flocs, the crystal faces should be clean. Since pure calcium carbonate crystals are too expensive for this use, the return of process sludges is inevitable. The "overcarbonation" or the lowering of the pH by the diffusion juice liberates nonsugars adsorbed on the crystals in carbonation, and the diffusion juice is slightly degraded.

Since the adsorptive power of the crystals, per unit weight, is proportional to the area of their faces, the crystals should be very small. If cost were not a factor, a finely ground, reagent calcium carbonate would be ideal. Next best, but still too expensive, is freshly precipitated calcium carbonate made by partly carbonating the liming agent. The next purest calcite, second carbonation sludge, is not available in large enough quantities. Thus most factories are forced to use either whole, or thickened, first carbonation juice.

The recycling of under-carbonated first carbonation juice, or defecation juice from main liming, constitutes a joint return of liming agent with calcium carbonate.

A second disadvantage lies in the alteration of detention times which may be caused by the recycling of process materials. If the recycling is not accompanied with an appreciable increase in the volume of the system in pipes and tanks, any individual part of the treated juices will have a proportionally decreased retention time in purification. If the volume of the equipment is appreciably increased, the

[1]See references (17,47,62,63,84,90,96,100,101,108,122,123)
[2]See references (14,26,46,54,55,68,81,96,113,115,117,131,133)
[3]See references (11,23,51,57,132,135)
[4]See references (8,20,22,31,40,70,107,114,120)

retention period of the juice under treatment will be correspondingly increased. The net change in detention time resulting from these two factors may explain undesirable chemical effects which have been experienced by certain factories. If juices are returned, the amounts should be kept to a minimum, thus *contraindicating recycling of whole defecation or carbonation juices.*

Precarbonation

Clean calcium carbonate crystals can be directed into the prelimer by partially carbonating the lime in the prelimed juice during the preliming; that is, by applying defeco-carbonation in the preliming stage. There is little waste of lime, since the lime carbonated has already acted as a *purifying or alkalizing agent, perfectly clean* crystals are obtained with no return of previously adsorbed nonsugars, and there are no juices recycled to degrade. This is a part of certain modern juice purification systems (53,78,91). Sometimes the prelimed juices are precarbonated, or a predefeco-carbonation may supplement or replace preliming.

Preliming Sludge Separation

In spite of good stabilization of the predefecation floc, some damage to it still occurs in main liming. The removal of the flocs before defecation improves the purification process, although this advantage is not as definite if defeco-carbonation is used instead of main liming. The first carbonation sludge, freed from substances which reduce settling and filtration rates, becomes very easy to separate. For example, in the Tasco (The Amalgamated Sugar Company) process, one of two formerly used in the United States embodying this separation, the first carbonation sludge was removed by hydrocyclones. Volpino (112) found a purity increase of the thin juices of 0.6 units as a result of this separation. Olyanska and Zhura (64a) found that almost all sludge and juice factors were improved, as well as decreased need for lime. Their findings have been confirmed by numerous other authors and investigators (65a,20a,59a). However, the advantages of the practice become less as the raw juice purity becomes very low (114a).

Claassen (18) noted in 1937 that separated, dried, but not desweetened predefecation sludge contained 1.65% nitrogen, 4.54% phosphate, 36.7% ash, and 35% sucrose. Such material has values for animal feeding, but at that time it could only be separated by filtering with large amounts of filter aid. Because of the interest of Spengler and associates at the Berlin Institute much work was done on its removal by *continuous centrifugal separators (85,98). In 1958 a practical process* was worked out, jointly, by the South German Sugar Company, and the Bergedorf Iron Works (39). In this "Sepa process," the predefecation sludge is first thickened in clarifiers, and then further dewatered for a total concentration of 20:1 in solid-bowl, continuous centrifugals. The dewatered sludge is then mixed with molasses, and made into pellets.

However, the additional juice detention time required by the added clarification and filtration is undesirable, and the cost of the separation equipment is quite high and cannot be justified unless there is a good market for the feedstuff.

Main liming

As described above (p. 171) the advantage of separate main liming, with its conditions of high pH, high temperature, and sufficient time for completion of destruction reactions, is that the purified juices are thermostable. The treatment received in defecation is more vigorous than any that will follow. Usually the

purified juices do not noticeably increase in color or lime salts, and will have less pH lowering or production of excessive scale in the sugar end.

European factories were not usually equipped with evaporator systems with short juice detention times and low pressures, and therefore thermostable juices have been required. Following the major improvement made by sludge return to the raw juice or to preliming, the chief remaining problem in the union of the good juice quality of the classical process with the easily separated, first carbonation sludge of defeco-carbonation, was the inclusion of main liming.

The practicability of main liming depends on the quality of the beets. If the beets sliced are degraded, the amount of decomposition products to be removed by adsorption in first carbonation may be so great that uneconomically large amounts of lime are needed. Thus, beets that contain rot from disease, wilt from long storage, or that have been frozen and thawed, are best processed by mild defeco-carbonation, in which the decomposition reactions are inhibited, but the thermolabile juices produced must be handled with care in the evaporation and crystallization stages. A number of modern "package" juice purification processes permit several degrees of intensity of defecation, invariably with plain defeco-carbonation as the mildest variant.

The union of main liming with defeco-carbonation has been accomplished in some cases by separating the main liming into more than one part, each addition succeeded by carbonations. Sometimes only a portion of the lime is added alone, the balance being introduced in following single or multiple defeco-carbonations. Alternatively, by careful setting of the chemical and physical variables, satisfactorily settling and filtering sludges can be obtained with the essentials of the classical process unchanged.

A "rule of thumb" is that the amount of CaO added should approximate the weight of nonsugars entering defecation in the diffusion juice. With beets of relatively high purity a ratio of CaO: nonsucroses of less than 1 is possible, and with beets of low purity, ratios as high as 1.2 or more may be required.

In the past, many factories have added the lime "dry," to the juice. In such cases the calcined limestone is crushed into small pieces, which are slaked by the juice being defecated, while moved through it by a drag or other mechanical device. The relatively slower addition of the lime, resulting from its gradual slaking, produces a predefecation effect, which is recognized and found desirable. However, higher lime salts are generally produced by dry liming systems (27).

Considerable experimentation was done by Carolan and co-workers (16), who found that limestone dust can be substituted for about one-third of the lime used in defecation without decreasing filtration rates or increasing thin juice lime salts, but there is some increase in colors. According to these workers, if excess limestone dust is used, colors are satisfactory, but filtration of the sludge is slowed. The effect of the length of the liming period was investigated by Fedorova and co-workers (30a) who found, for example, that an increase of the defecation time from 4-5 min., to 11-12 min. improved nonsucrose elimination, and lowered thin juice lime salts and colors appreciably. Golybin and Ivanov (39a), among many others who have studied the subject, showed the advantages of cold followed by hot main liming (5 min. at 50 °C, 10 min. at 80 °C). There were better juice qualities, improved rates of sludge sedimentation, and faster rates of sucrose crystallization.

Defeco-Carbonation

Simultaneous liming and carbonation, or defeco-carbonation, has had use in batch processes. In these cases, the lime is added to the juice at a slow, steady rate, and with continuous carbonation, a relatively constant alkalinity or pH is held until the lime has all been added, at which time the batch is finished.

The introduction of continuous defeco-carbonation by the Dorr Company has been described (p. 157), and the development of methods of operating Dorr defeco-carbonation to produce the best possible juice quality was largely the work of McGinnis and co-workers (59,83).

As Dedek has pointed out (p. 169) while a defeco-carbonation process, such as the Dorr process, does not formally contain preliming and main liming steps, nevertheless both steps are there, even though in brief.

When diffusion juice first encounters the limed juice in the primary tank, it is subjected to an alkalinity which is correct for the end point of preliming. During the short detention time in the primary tank, coagulum formation occurs in the presence of fine crystals of calcium carbonate, such as are introduced in the preliming step by sludge return or precarbonation.

The juice then flows from the primary to the secondary tank, and in transit receives for a few seconds what is, in essence, a very brief, warm, main liming, when the liming agent is introduced. This is terminated on entrance into the lower alkalinity of the secondary tank.

While the elements of preliming and main liming are present in the Dorr system, both unit operations are inadequate. The preliming does not involve a gradual addition of liming agent over a sufficiently long time, with the result that the predefecation coagulum is easily peptized. The main liming is far too short, and is not alkaline or hot enough. The result is that the final first carbonation juice contains undestroyed invert sugars, incompletely saponified amides, potential oxalatae-formers, and still viable thermophilic organisms, which are not present in juices which have received correct main liming. In short, juices from Dorr first carbonation are thermolabile.

On the other hand, such a defeco-carbonation system has real advantages. Partially as a result of the high juice recirculation rate in first carbonation, and of the presence of the small calcite crystals, colloidal flocs which escape peptization, build up into well-agglomerated, filled-up structures, which can be filtered out, or settled out relatively easily and economically. There is also time for near equilibrium to be reached in adsorption of coloring matter and lime salts on the crystal faces. Likewise, beets of low purity, especially when degraded by prolonged storage, or by freezing and thawing, can be processed with this mild method with a minimum of color and lime salts formation in the purification.

However, as a consequence of the lack of thermostability of defeco-carbonation process juices, and in order to avoid color and lime salts development, ammonia evolution, and falling pH values, the juices must be treated gently in later process steps. Essential measures for mild handling are: a short clarifier detention time; a short second carbonation duration; a relatively low second carbonation temperature (contraindicated for low lime salts); no thin juice boiler; rapid, low-temperature evaporation; minimum times in hot crystallization procedures; and avoidance of unnecessary sugar-end material recycling, with prolonged detention times in additional piping and equipment.

Figures 8-5 and 8-6 are photomicrographs of typical first carbonation sludge particles from a defeco-carbonation and from a batch process. The larger agglomer-

ates in the former are clearly seen.

In spite of its disadvantages, Dorr defeco-carbonation has been found to be economically justified in most American factories. The unit operation of simultaneous liming and carbonation has found a place in certain other processes, because of its effectiveness in improving the physical nature of the sludge, both in preliminary and main purification phases, and because it is easily adaptable as a part of a stepwise alkalinity increase pattern.

Following the awakening of European interest in defeco-carbonation, the result of two campaigns during which frost-bitten beets provided severe operating difficulties, many studies were recorded. Vasatko, Tibensky, and co-workers studied the application of defeco-carbonation to processes using Dedek-Vasatko progressive preliming (92,93,109-111). Similar studies were made by East Europeans (25,49,130b).

Convertible Processes and Degraded Beets

As has been noted by Dedek in Chapter VII, "the details of the juice purification to be used must be adapted to the character of the juice to be treated." Obviously, it is possible to adjust the variables in any given juice purification process, and to adapt it, to some extent, to the changing nature of the beets being processed. Such variables as amount of lime, alkalinity, temperature, soda ash addition, and sulfitation, if adjusted intelligently, can go a long way toward remedying the effects of poor beets.

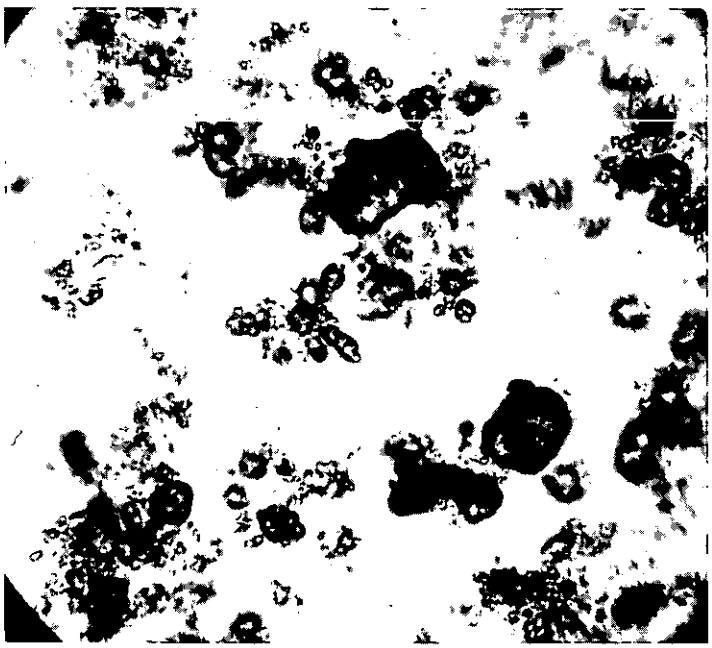

Figure 8-5. Photomicrograph of Dorr system first carbonation sludge, 1000X. Note the clumps of calcium carbonate particles.

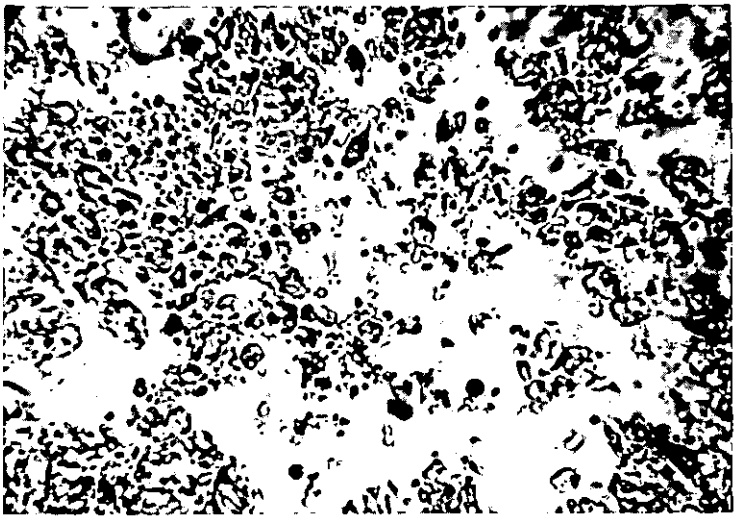

Figure 8-6. Photomicrograph of first carbonation sludge produced in standard batch system carbonation. 1000X.

If supplementary juice purification units such as ion-exchange or activated-carbon systems are available, which can be expanded in scope when necessary, almost any reasonable beet quality can be acceptable. The operation of these supplementary units is expensive, however, and if degraded beets can be processed to make good sugar without such additional expense, through changes in unit operations and flows, it is certainly more profitable.

The wide use in the United States and Canada of defeco-carbonation processes which are nearly the ultimate in mildness, enables the factories to process beets of almost any type excepting the very worst, and to make juices from which saleable sugars can be extracted.

The selection of the proper type of juice purification to use with beets of low quality is a complex problem, involving the particular types of nonsucrose materials which are present in excessive amounts in the diffusion juice, the evaporating and crystallizing equipment available, the quality of the white sugar required, and the costs of lime, of filter aid, and of adsorption-type purification agents needed.

Low-quality beets can be separated into at least three types: 1) beets which have been frozen and thawed, but which have been promptly sliced, and consequently do not contain much invert; 2) frozen and thawed beets which have been permitted to undergo sucrose inversion, or beets degraded in high-temperature storage, or diseased or rotted beets; and 3) sound beets of low purity, usually containing abnormally large amounts of nitrogen.

Beets of the first type, which have been frozen and thawed, but which have low invert content, yield diffusion juices containing the impurities usually found in pressed juices. Such materials are pectic matter, albumens, and fine beet particles. These diffusion juices require greater quantities of lime, whether mild or vigorous purification is practiced. If main liming is used the decomposition products are not unduly troublesome, although additional soda ash is usually required. Filtration of the purified juices is more difficult, and more filter aid may be needed.

Beets of the second type, which contain high amounts of invert sugars, are usually considered best processed by mild processes, as defeco-carbonation. This requires, of course, a gentle handling of the process juices in their passage through the evaporators and the sugar end of the factory.

In certain Western areas of the United States, however, many years of experience with this type of beets has led certain operating firms to the conclusion that vigorous defecation is preferable (10,45,61). The use of defeco-carbonation with the amounts of lime economically practicable, has given juices containing so much coloring matter, coming both from the decomposition of invert during evaporation and from the browning reactions, primarily in the sugar end, that even with the use of activated carbon, the production of satisfactory white sugar has involved too much remelting. With such vigorous treatment much higher soda ash usage is necessary to limit the lime salts and to provide alkali reserve buffer capacity for the juices. This is, nevertheless, regarded as a small price to pay, compared with the costs of the remelted white sugar strikes from the high standard liquor colors.

Representing the opposite point of view, however, other operating firms with factories in the same geographical areas have been able to use defeco-carbonation satisfactorily. Successful operation of defeco-carbonation cannot be achieved if the process is not operated in an optimal manner (see pp. 203-211). This means, essentially, smoothing of all the important variables entering into the process, and maintenance of the optimum alkalinities in first carbonation required for best juice quality, with the use of the necessary settling aids to permit adequate sludge thickening.

Beets of the third type which show no evidence of physical or chemical degradation, but which yield diffusion juices of relatively low purity, are likewise commonly encountered in the Pacific Coast states. Such diffusion juices do not contain appreciable amounts of invert, but are high in nitrogenous compounds. Processing of these juices requires large quantities of lime. It seems almost out of the question to hydrolyze all of the amide-nitrogen, even in a vigorous purification process with prolonged main liming. Thus it appears inevitable that steady lowering of the pH values and evolution of ammonia will occur throughout the sugar end. To purify such materials the use of large quantities of soda ash seems unavoidable, as well as resort to the assistance of activated carbon or ion-exchange purification.

The question of the selection of mild or vigorous processes for low-purity beets must be resolved with the particular circumstances at each factory in mind. The use of mild defecation may be inadvisable if the periods of time, the temperatures, and the pH environments to which the juices are subjected in the evaporation and crystallization procedures are unfavorable.

In Europe, where the more vigorous defecation processes as well as excellent beet quality were the rule, sudden drops in beet quality, due to disease or weather conditions, found many factories unable to make adjustments with sufficient effect. The intensive studies that resulted showed that the preferred treatment for badly degraded beets should embody: predefecation, either cold, or better, eliminated; main liming, cold or better, eliminated; single or multi-stage defeco-carbonation; soda ash or phosphate addition to second carbonation; and mild treatment through the rest of the process.

Many European factories have equipped their first carbonation tanks with lime addition and juice recirculation equipment so that, if necessary, preliming and main liming can be omitted, and the process changed to defeco-carbonation. A number of the modern "package" processes provide several degrees of defecation

intensity, requiring only the changing of a few valves to alter the process.

Chemical studies made in Europe under the impetus of the degraded beets revealed details of interest of the altered character of the juices to be purified. Wallenstein (119) found that lactic, butyric, and acetic acids increase in degraded beets, thus increasing the lime salts in the thin juices. Becker (7) found dextran formed by *Leuconostoc mesenteroides* was a prime cause of difficulty in purifying juices from frozen beets, causing foaming, color formation, slow filtration, and sucrose losses. Duszynska (30) showed that anaerobic organisms in thawed beets decompose polysaccharides, and, to some extent, the albumens and pectins of the beet cell walls. She found that most of the decomposition products were over 5 000 in molecular weight. These are not eliminated in juice purification. Increase in the juice colloids content of degraded beets was found to amount to from 3 to 10% by Orlov (66). Schneider (76) found a levan in degraded beets produced by a rod bacterium. He noticed, however, that extraction of pectin was not appreciably increased unless considerable beet rot was present.

A few of the more important studies of process alterations for processing degraded beets should be mentioned. Pavlas (67) reported that in Czechoslovakia badly deteriorated beets were successfully processed by elimination of preliming and main liming, use of defeco-carbonation in two stages, and return of first carbonation sludge to the diffusion juice. Various modifications of juice purification to process degraded beets were described by Werner (120), Gawrych (32,33), Tibensky (93), Strube (87), Henry (41), Zagrodski *et al.* (131c,131d) and Kolesnikow *et al.* (54a).

Effect of Juice Purification on Certain Specific Nonsucroses

Amino Acids

With normal juice purification, Kubadinow *et al.* (54b) found that almost all amino acids were degraded before the juices reached the evaporators. Thus 70 to 90% of the glutamine, and 40% of the asparagine were rapidly hydrolyzed. Other amino acids were reduced in concentration by adsorption on the $CaCO_3$, or by chemical reactions. Decreases in arginine and histidine were due chiefly to the Maillard reactions. Aspartic acid was removed by adsorption.

Invert Sugars and Color

Vukov has reviewed the status of beet juice coloring matters (118b). Melanoidins are practically irreversibly adsorbed on the calcium carbonate in juice purification. Invert sugar should be largely destroyed. The removal of melanins by adsorption is definitely reversible, and is dependent on the alkalinity. Thus carbonation may not be continued beyond pOH 3.0.

The use of lime added to the second carbonation mud, and its return to the raw juice enables it to adsorb melanins efficiently from the raw juice.

Schiweck (73a) called attention to the effectiveness of bleeding air through the main and preliming tanks. Good results are frequently obtained, reducing the thin juice color by about 15%, using 0.75 Nm^3 of air/m^3. Assuming color formation in juice purification is largely by the Maillard reactions, the results are doubtless due to the destruction of invert sugar with its associated carbonyl groups. Zaorska (131d) reported excellent results using the process in Poland. There have been reports that it is not effective when the defecation process is unusually rigorous.

Havlova (40a) notes that in normal juice purification, if very high invert contents are encountered, it may require main liming of 30 min. duration at 85 °C

and 12 pH to destroy it.

Since the Maillard reactions require degraded proteins or amino acids as well as carbonyl groups, Zhura *et al.* (133a) have pointed out that these protein fragments occur to only a slight extent at temperatures below 60 °C.

A very complete review of colors in beet-sugar manufacture was given by Madsen, *et al.* (56a).

Pectins

Pectins are rapidly dispersed into low molecular-weight fractions, and readily form gels which impede settling and filtration, Volf (111d) *et al.* reports. The addition of calcium carbonate crystals to the juice, before liming, prevents gel formation by adsorbing the pectin fractions; thus giving an explanation of the improved juice filtration and settling achieved by returning carbonation sludge to the raw juice before liming has taken place.

Practically Useful Information of Behavior of Nonsucroses in Juice Purification (9)

The following Tables 1-3 (9) give practically useful information on the nature and behavior of certain types of nonsucroses in juice purification, and, in particular, Figure 8-7 shows the effects of prolonged beet storage before processing on juice purification.

Continuous Carbonation*

Systems Used in North America. In 1920 there were no known continuous carbonation installations in the United States. In 1947, over two-thirds of the operating factories used continuous first carbonation, and over 94% had continuous second carbonation (102). In 1969 there were only six batch first carbonation and one batch second carbonation systems remaining among the 63 operating factories

Table 8-1 Soluble and Semi-soluble Nonsucroses

1. *Soluble Nonsucroses*		
Invert	Raffinose	Organic
Kestose	Dextran	Carbohydrates,
Levan		Non-ionic
Amino Acids	Betaine	Organic, Cationic and Anionic
Sodium	Potassium	Inorganic Cations
Calcium	Magnesium	
Chlorides	Nitrates	Inorganic Anions
Phosphates	Sulfates	
Citrates	Oxalates	Organic Anions
Malates		
Saponins		High Molecular-weight Anions
2. *Semi-soluble, Dependent on Temp., pH, and Salt Content*		
Pectic material		High Molecular-weight Anions
Proteins		

*by R. A. McGinnis

Table 8-2 Soluble Nonsucrose Impurities in Juices, as Carbonation-removable or Non-removable

1. Non-removable nonsucroses	
a. Storage-sensitive non-removable nonsucroses	
Invert	Increase in this class of nonsucroses is usually a function of time-temperature, transport, or storage conditions
Raffinose	
Kestose	
Dextran	
Levan	
b. Non-removable nonsucroses	
Amino acids	Increase or decrease in the quantities of this class of non-sucroses generally comes from agronomic practices — in particular nitrogen usage
Betaine	
Sodium	
Potassium	
Chlorides	
Nitrates	
2. Removable nonsucroses, removed as insoluble calcium salts	
Phosphates	Increases or decreases in this class of non-sucroses may reflect agronomic practices
Sulfates	
Citrates	
Oxalates	
Malates	
Saponins	

Table 8-3 Approximate Percentages of Nonsucrose Classes in Typical Raw Juices

Classification	Nonsucrose	% of Total Nonsucroses in Raw Juice
Non-removable Nonsucroses	Amino acids	40%
	Betaine	
	Potassium	
	Chlorides	
	Sodium	
	Nitrates	
Removable Nonsucroses	Oxalates	40%
	Saponins	
	Citrates	
	Phosphates	
	Malates	
	Sulfates	
Storage-Sensitive Non-removable Nonsucroses	Invert	20%
	Raffinose	
	Kestose	
	Inositol	
	Galactinol	
	Levan	
	Dextran	

in North America. In 1979, with 46 operating factories in North America, there were none.

The Dorr System. Since most factories in the United States use the Dorr system (Table 8-4) it will be treated in greater detail. The following description of a Dorr first carbonation system does not necessarily apply in all details to every installation. Minor differences in design and methods of operation are found in each factory installation, and major variations will be described later.

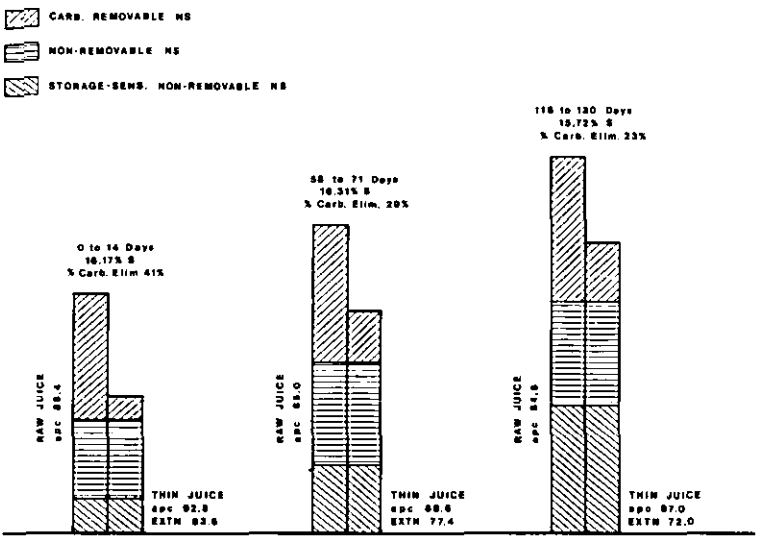

Figure 8-7. Effect of Storage-Sensitive Non-Removable (in Carbonation) Nonsucroses on Purified Juice Quality, % Carbonation Elimination, and % Sucrose Extraction on Sucrose in Beets. The data are from analysis of samples taken from a pile of beets stored in a cold climate, during the same season.

Table 8-4 First Carbonation Systems in the United States and Canada, 1979

	No. of factories
Dorr system, (most with internal sludge recycle)	14
Dorr, Benning carbonator	11.5*
Dorr, G.W. modif., concurrent juice and gas flows	7
Michigan	4
Great Western Multi-tank Systems	5
B.M.A.-65	4
Putsch	0.5*

*(factories concerned have two parallel purification systems)

An original Dorr system is shown in Figure 8-8. The first carbonation equipment consists of two cylindrical tanks with conical bottoms, one for use as secondary tank, and one as primary, the primary being the smaller. Gassing takes place in the secondary tank, while the primary tank receives the diffusion juice and serves as a mixing or circulating tank. For clarity, the secondary tank will be referred to as the "gassing" tank, and the primary as the "mixing" tank. Frequently the primary tank is equipped with gas distributors and the necessary piping, so that the secondary tank may be temporarily bypassed for descaling. When the campaign period is short, scaling may not be severe enough to make it necessary to have a standby gassing tank. Cylindrical tanks have decided advantages over square or rectangular tanks, in the latter of which the chances for "dead spots" are much greater.

Diffusion juice from the juice heaters enters the bottom of the mixing tank, together with recirculated juice from the gassing tank. Flow is upward through the mixing tank, and the liquid overflows from the top into the top of the gassing tank. The liming reagent is added to the overflow pipeline, permitting some mixing before

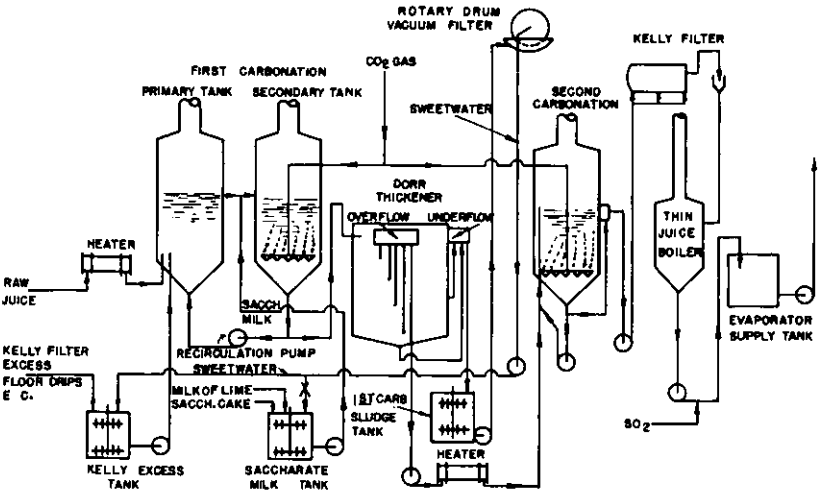

Figure 8-8. Dorr system continuous first and second carbonation, as installed in 1937 in a Steffen factory (Woodland, California). Thin juice boilers are now usually omitted.

entering the top of the gassing tank. Juice travels down through the gassing tank, counter to the direction of travel of the gas bubbles, and out the bottom of the tank to the recirculation pump, and again into the mixing tank. Finished first carbonation juice is sent to the thickener through an overflow line running from the discharge of the pump.

The recirculation pump, which is normally of the centrifugal type, should cause very little mechanical destruction of the flocs. An ideal type of pump for this service is a slow-speed, axial-flow propeller pump. It should have a capacity, such that with normal operation, the volume recirculated equals seven times the throughput of juice from carbonation. The recirculation ratio is important, and should be kept within 5 to 1 and 9 to 1 as outside limits (see pp. 209-210).

Carbon dioxide gas is conducted to the gassing tank through a valve, frequently actuated with the aid of an automatic controller, and to the gas distributors, which are similar to those used in batch carbonation.

The Benning Carbonator. This is a single-tank unit designed to replace the primary and secondary tanks in the Dorr system, with a resultant saving in equipment cost and in space occupied. The primary tank is replaced by an annular addition to the gassing tank. Detention in this mixing section is somewhat less than in the conventional Dorr primary tank.

The gassing tank is cylindrical (see Figure 8-9). The mixing section is about four feet greater in diameter than the gassing tank, and is equipped with perforated baffles. Raw juice enters the discharge line of the recirculation pump, which sends the juice to the lower part of the mixing section. The saccharate milk or milk of lime flows by gravity to several evenly spaced, open troughs above the mixing section. From these troughs it is distributed to the juice through a large number of openings. The gas distributors are straight pipe sections with inverted-U cross sections. The earlier circular cross-section gas distributors caused excessive scaling. The

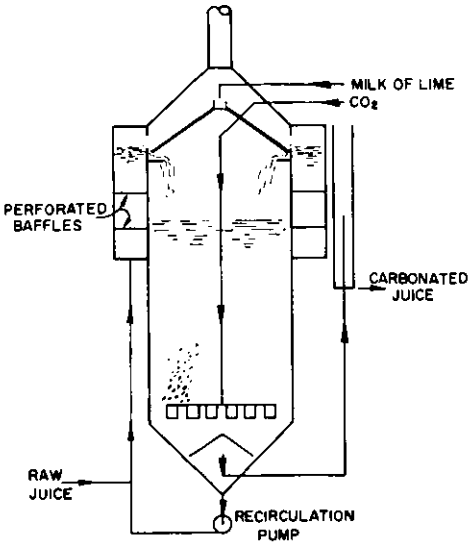

Figure 8-9. The Benning carbonator.

recirculation pump draws the juice from the conical bottom of the gassing tank and discharges to the mixing section, maintaining the standard Dorr recirculation ratio. An overflow box removes the finished juice from a point in the center of the gassing tank near the bottom, where a large hood prevents entrainment of gas.

There appears to be no difference in the quality of the thin juice produced as compared with the standard Dorr arrangement. It possesses what has formerly been considered the disadvantage that if it becomes scaled and requires cleaning, the whole beet end of the factory must be shut down, unless there are two units. Actually such instances seem to have almost completely ceased to occur.

Dorr System, Great Western Modification. Seven of the factories of The Great Western Sugar Company use a modification of Dorr first carbonation. While precise physical details vary from factory to factory, a typical installation is shown in Figure 8-10. The gassing tank contains two vertical partitions, separating it into

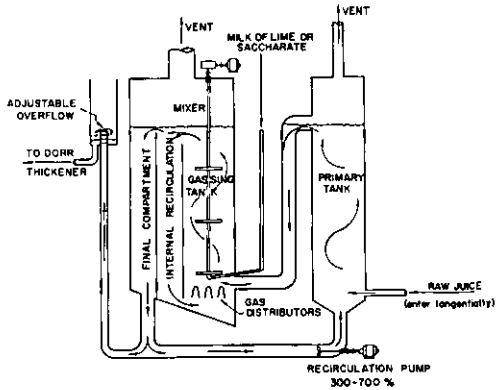

Figure 8-10. Dorr System, Great Western modification. A typical installation.

three compartments. The juice overflowing from the primary tank enters the first, or gassing compartment of the secondary tank at the bottom, slightly above the gas distributors. The liming agent is also introduced separately at this point. Within this compartment the juice travels upward, concurrently with the gas bubbles, and the three-phase fluid is blended by a mechanical agitator. Overflowing the first compartment, a considerable part of the juice travels downward through the second compartment, reentering the first compartment, in an internal recirculation. The other part of the juice overflows into the third compartment, and leaves the bottom of the secondary tank, where it either becomes part of the external 300-700% recirculation, or proceeds to the clarifier.

Operation of Dorr First Carbonation

Automatic Control. The Dorr system customarily involves the use of a continuous indicator of the first carbonation end point, and very frequently automatic control of the end point. Originally, control was by conductivity measurement. This was found unsatisfactory for several reasons. In the first place, the electrodes often scaled rapidly, resulting in a continuous drift in the reading, though this could be allowed for. Actually, conductivity measurements are in general too insensitive for satisfactory first carbonation control. In addition, the conductivity is often affected by variations in concentrations of substances other than hydrogen ion or hydroxide ion. In recent years, through supplemental electric circuitry, sufficiently sensitive conductivity devices have come into use in Europe.

In the early thirties it was discovered that the antimony electrode with calomel half-cell could be used with some success for measuring pH in hot beet juices. Electrode scaling is not as serious as with the conductivity apparatus, but a film does form over the antimony electrode which must be polished off at least every eight hours. Also, the electrode is poisoned by certain compounds which are sometimes present in the juices, and at such times gives false indications. A self-cleaning antimony electrode was introduced by the Holly Sugar Corporation (134) which has had considerable use. Figure 8-11 shows this design. The marble is kept in move-

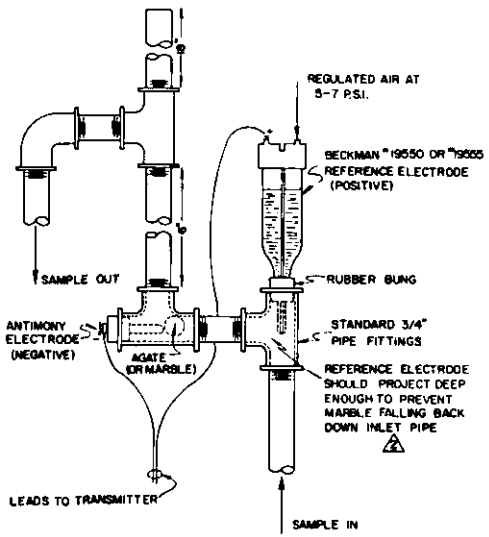

Figure 8-11. Holly self-cleaning antimony electrode. *(Courtesy, Holly Sugar Corporation.)*

ment by the flow of juice, continually burnishing the antimony surface and keeping it free from scale and oxide.

Later, successful high-temperature glass electrodes were introduced, which are far more sensitive than the antimony electrode, and are not easily poisoned. They do, however, require frequent cleaning. The majority of carbonation units in the United States use glass electrodes for pH control. A recently marketed "unitized" form, contains both the glass and reference half-cells in a single unit.

The original conductivity control is now used by only two factories.

Factors Affecting Dorr First Carbonation. The most important factors affecting Dorr first carbonation are; pH or alkalinity, temperature, amount of lime addition, rate of recirculation, retention time, density of diffusion juice, and gas absorption.

Hydroxyl Ion Activity. The control of the end point in first carbonation is normally on the basis of pH. pH is a measure of hydrogen ion activity. It is defined as,

$$pH = -\log a_{H^+} \tag{1}$$

where a_{H^+} indicates the activity of the ion subscript, which at increasing dilutions approaches the concentration in moles per liter. In all aqueous solutions, hydrogen and hydroxide ions are in equilibrium with water:

$$H_2O = a_{OH^-} + a_{H^+}. \tag{2}$$

The ratio of the product of the activities of the dissociated ions to the undissociated water is a constant at any given temperature.

$$a_{OH^-} \cdot a_{H^+} / a_{H_2O} = \text{a constant}. \tag{3}$$

Since the activity of water is nearly a constant it is considered so, and the expression is reduced to

$$a_{OH^+} \cdot a_{H^+} = \text{another constant} = K_w \tag{4}$$

or
$$\log K_w = \log a_{OH^-} + \log a_{H^+}, \tag{5}$$

and
$$-\log K_w = -\log a_{OH^-} - \log_{H^+}. \tag{6}$$

Using the "p" terminology,

$$pK_w = pOH + pH.$$

The precise values for K_w in first carbonation juices are not known. However, they are known for pure water, and the following values may be assumed as first approximations.

Temp., °C	pK_w
70	12.77
80	12.55
90	12.34

Thus, if carbonation is being conducted at 10 pH and 80 °C,

$$12.55 = pOH + 10.00, \text{ or } pOH = 2.55$$

This means that the approximate concentrations of the two ions are,

(H^+) = 0.000 000 000 1 moles per liter
(OH^-) = 0.003 moles per liter

At 90 °C, however,

$$12.34 = pOH + 10.00, \text{ or } pOH = 2.34,$$

and while (H^+) has the same value, $(OH^-) = 0.005$.

While the pH measuring equipment measures the approximate concentration of the hydrogen ions, it is apparent that it is the hydroxyl ions which must be affecting the reactions in carbonation. If the temperature and certain other variables remain constant, a measurement of pH is, for practical purposes, sufficiently related to pOH. If these variables change, however, it is no longer sufficient. Although temperature can be held constant, other affecting factors (86) are not as easily controlled. These are, variations in the nature of the nonsucroses, in the amounts of lime used, and in diffusion juice density.

Baczek and Jesic (5a) introduced a method for the determination of the optimum coagulation point in preliming and 1st carbonation. For the former, a sample of raw juice is prelimed to a number of pH values in the range 10.3-11.8: at each pH value a small sample is withdrawn and allowed to stand, after which the turbidity of the supernatant liquid is measured photometrically at 560 mμ. In 1977 Büsching (15a) modified the method to remove remaining turbidity by filtering through Millipore filters. The optimum is that pH at which absorbancy is a minimum.

For first carbonation the procedure is similar, except that carbon dioxide is passed in, and a flocculent (such as Separan) is added to the sample before it is allowed to stand. This is similar to the traditional American method, in which the lime salts content of the juices filtered after carbonating is measured instead of the turbidity. The Baczek-Jesic method does not take account of the thin juice lime salts (5a,15a).

pOH Measure by Kysilka Electrodes

Kysilka *et al.* (54c) in 1978 introduced a system involving the use of a reference electrode, which, although indirect, may in essence be a reliable method of measuring pOH (Figure 8-12). This differential measurement system incorporates a special type of reference electrode and a dual high-impedance input meter. This is currently being used by American Crystal Sugar Co. to control carbonation pOH. The reference electrode (item B of Figure 8-12) uses a glass pH-electrode as its internal element, and a filling solution having a pH and temperature coefficient similar to the juice being measured. This null-point technique of measurement significantly reduces pOH error in control that is experienced with conventional pH control systems.

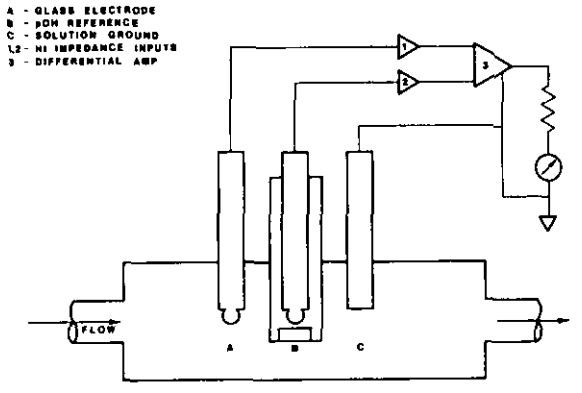

Figure 8-12. Kysilka pOH Reference Electrode System.

Aside from the Kysilka electrodes, there has been no practical method or apparatus for continuously measuring pOH directly. Alkalinity titrations, however, are very close to being proportional to such measurements. The best proof of this is empirical experimental evidence. Figure 8-13 shows the relation between alkalinity, and pH and pOH for a number of first carbonation juices at three different temperatures. Both graphs are plots of the same data (83).

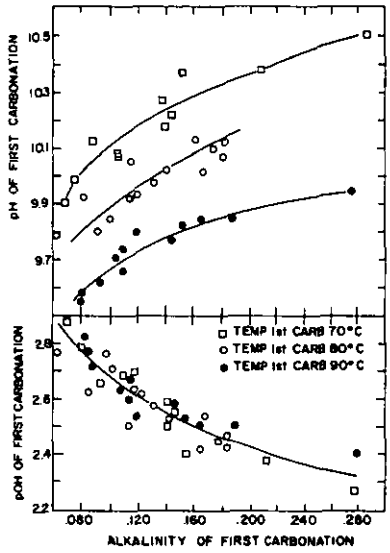

Figure 8-13. Relation between pH or pOH and alkalinity of first carbonation at varying temperatures, with saccharate milk defecation (83). *(Reprinted by permission of the American Chemical Society.)*

Thus, until a practical electrode for measuring hydroxyl ion activity directly is found, titratable alkalinity must be considered the best basic control measure for first carbonation. Since satisfactory automated alkalinity titrators are not common, the most convenient instrumental control is indirectly through a controlling pH meter. Frequent alkalinity titrations are made, and the pH control is reset to correspond with the desired alkalinity.

The second consideration under the subject of alkalinity, is which alkalinity to choose for the first carbonation end point. Experimental work (83) has shown that the higher the alkalinity, up to a certain point, the better is the quality of the thin juice produced, as judged by color, lime salts, and colloid content. The "certain point" for central California juices is about 0.130 for saccharate milk defecation, and between 0.100 and 0.110 with milk of lime defecation (see Figures 8-14, 8-15 and 8-16). On the other hand, the lower the alkalinity, the larger will be the crystalline agglomerates and the more easily the carbonation sludge can be settled and filtered. As a result, the alkalinity chosen must be a compromise, depending on the capacity and efficiency of the sludge separation equipment. Operating end points commonly used vary from 0.060 to 0.120.

Temperature. Settling and filtration of sludge become easier as the temperature of first carbonation is increased, up to a certain point. As before, however, thin juice quality is lowered (see Figures 8-13 and 8-14).

Once again a compromise must be made. Practically, temperatures between 80 °C and 85 °C are generally chosen.

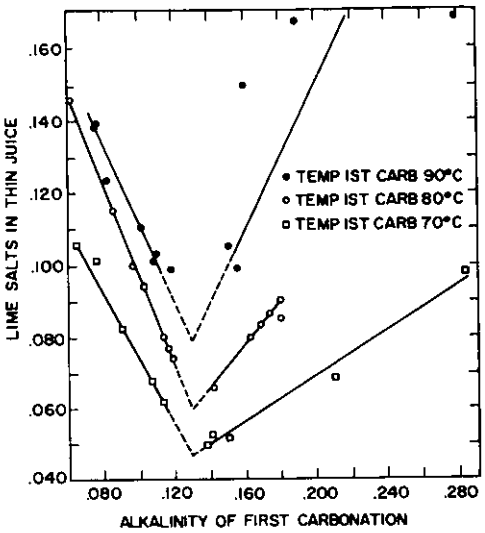

Figure 8-14. Effect of first carbonation alkalinity and temperature on thin juice lime salts, with saccharate milk defecation (83). *(Reprinted by permission of the American Chemical Society.)*

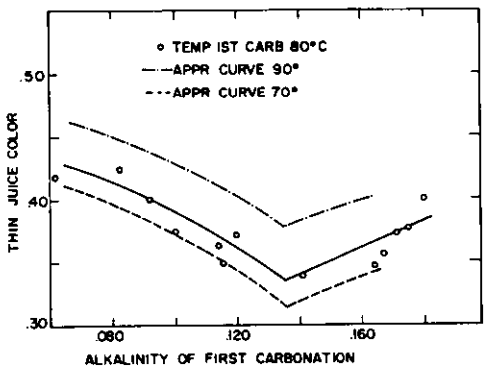

Figure 8-15. Effect of first carbonation alkalinity and temperature on thin juice color, with saccharate milk defecation (83). *(Reprinted by permission of the American Chemical Society.)*

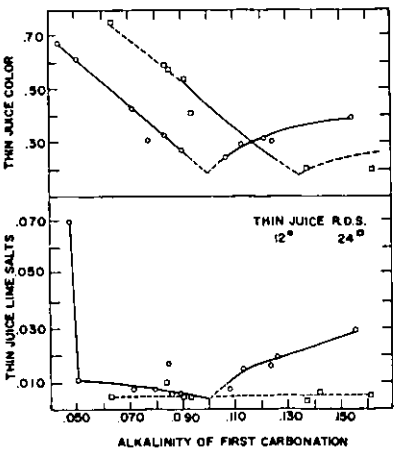

Figure 8-16. Relations between first carbonation alkalinity and thin juice color and lime salts, with milk of lime defecation, at 80°C (83). *(Reprinted by permission of the American Chemical Society.)*

Amount of Lime Addition (59). In a Steffen factory, the amount of lime used is not a free variable, since it is set by the amount of molasses worked; that is, all the lime required by the Steffen process, except for a small amount lost in the waste water, must be added to first carbonation. For this reason, and because of the greater accumulation of nonsucroses due to recirculation, more lime must be used in a Steffen house than in a straight house.

Figures 8-17 and 8-18 show that at normal alkalinities, the greater the amount of lime used, the better is the thin juice quality, up to at least 5.5-6% CaO on beets. An exception is found at very high lime addition with prediscard (see pp 609-610) saccharate milk, where the lime salts content increases. There appears to be no confirmation of the belief sometimes voiced (52) that only very small amounts of lime are required for adequate purification, since the major portion of the lime is only useful as filter aid. Studies by Schoenrock *et al.* (41a,79a) have shown that this is true only for coagulation and defecation. Beyond this lime is effective as an adsorbent, and at the proper pOH values nearly water-white liquids can be obtained with very high ratios of CaO to nonsucroses. The purification efficiency, or amount of purification accomplished per unit of lime used, decreases with large lime additions. This is shown in Table 8-5.

The curves of Figures 8-17 and 8-18 permit comparisons to be made of the purification efficiencies at the first carbonation optimum alkalinities for best thin juice quality, and at 0.080 alkalinity, well below the optimum. Thus for saccharate milk defecation, operation at the optimum alkalinity of 0.130 required only 35.5% of the amount of lime needed at 0.080 alkalinity for the same color of thin juice, and required 63% of the lime needed at 0.080 alkalinity for equal thin-juice lime salts contents.

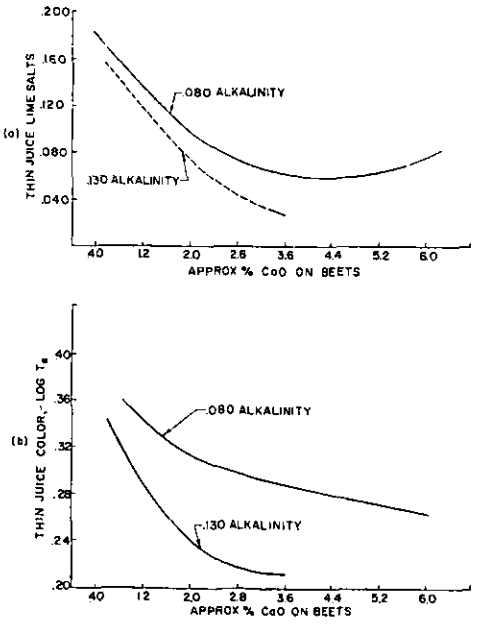

Figure 8-17. Effect of amount of saccharate milk used, on thin juice lime salts and color (59).

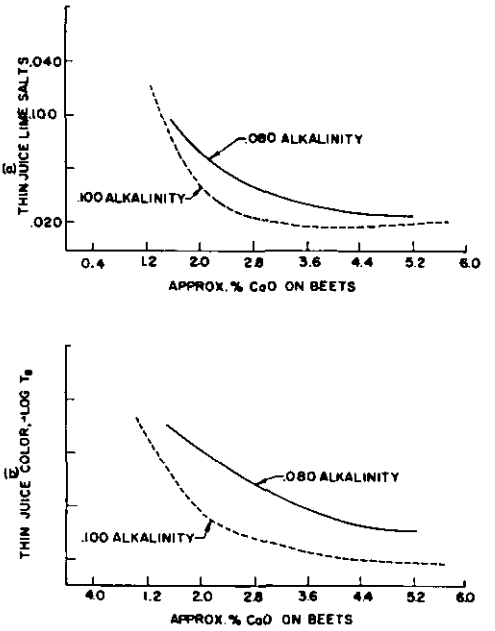

Figure 8-18. Effect of amount of milk of lime used, on thin juice lime salts and color (59).

Table 8-3 Reduction of Purification Efficiency with Increasing Lime Additions

Liming Agent	Alkalinity of First Carbonation	Purification markedly falls off with further lime addition, at about the percentages of CaO on beets indicated, considering: Color	Lime Salts
Saccharate milk	0.080	over 6% CaO	over 3.5% CaO
	0.130	over 2% CaO	over 3.5% CaO
Milk of Lime	0.080	over 3.6	over 3.0
	0.100	over 2.3	over 2.3

For milk of lime defecation, operation at the optimum alkalinity of 0.100 required 59% of the lime for the same color, and 74% of the lime for the same lime salts in the thin juice in operation at 0.080 alkalinity.

The point at which juice quality improvement does not justify the cost of additional lime must, of course, be individually determined for each factory.

The actual amounts of lime required to obtain adequate purification vary with many factors, chiefly the amount and nature of the nonsucroses. There are localities in which the beets are of such high purity that relatively little lime is required. For example, excellent defecation has been secured with lime additions as low as 1.1 percent CaO on beets, with comparatively high-purity diffusion juice. On the other hand, in another territory, 2.75 percent CaO on beets is required, with a comparatively low purity juice. If lime additions are calculated on extracted nonsucroses, comparisons between various factories are more meaningful.

Rate of Recirculation. With the Dorr system, satisfactory settling of the sludge in the clarifier is ordinarily not possible without recirculation in first car-

bonation. By rate of recirculation is meant the ratio of juice by volume recirculated between the two tanks, to the volume of finished first carbonation juice sent from the system, and not, as is sometimes assumed, the volume of the raw juice taken into the system. The volume of the former is greater than the volume of the latter by the amounts of melter returns, excess sweetwater, saccharate or lime milk, and any other additions that may be made. The recirculation rate is fixed by the characteristics of the recirculation pump. This should be 7:1, with outside limits of 5:1 and 9:1.

Density of Diffusion Juice. As the concentration of dry substance in the diffusion juice is increased, the proper end point alkalinity for operation also increases. Thus, a thin juice of very irregular quality will result if care is not taken to keep the diffusion juice density reasonably constant. However, if the optimum alkalinity is maintained in spite of change of density, the defecation is somewhat more efficient at high densities, at least with respect to lime salts. Considering this, and the cost of evaporating water, it is apparent that it is advantageous to carry as high a diffusion juice density as is economically compatible with diffuser losses.

Rate of Gas Absorption. The rate of gas absorption is frequently of considerable importance because of shortages of carbon dioxide gas. The important factors affecting this are the same as in batch carbonation.

Retention Time. There are two retention times to consider, that in the gassing tank and that in the mixing tank. In the usual installation comprising two or more tanks, the retention time in each tank is approximately the same, or about ten minutes in each. In the Benning carbonator, the retention time in the gassing tank is about the same as it is in the usual two-tank system. However, in the Benning carbonator, retention time in the primary or mixing compartment is appreciably less than in the primary tank of the standard two-tank system. The fact that some later Dorr installations have been made with comparatively small primary tanks confirms that reduced detention time in the primary tank may not be a disadvantage. The time in the secondary tank, nevertheless, should not be much less than ten minutes.

Uniformity of Operation. As was stated, the best set point for the operating variables in first carbonation involves a compromise between good thin juice quality, and ease of sludge separation. The best operating alkalinity is one as high as possible, still maintaining good settling and filtration.

Due to irregularities in various factors, it is never possible to maintain an absolutely constant alkalinity. The magnitude of the irregularities determines how high the alkalinity can be carried. For example, if in a given factory the clarifier overflow becomes seriously cloudy at 0.100 alkalinity, and the magnitude of the irregularity is of the order of 0.020 units around the set point, then 0.080 is the highest alkalinity that can be safely carried. If the irregularity is reduced to 0.010, then 0.090 can be aimed for, with a much better quality thin juice produced.

Thus, if first carbonation is to be operated at its maximum efficiency, all the variables must be smoothed out as far as possible. Among these variables are: diffusion juice density, temperature, and rate of flow, carbon dioxide gas pressure and CO_2 content, saccharate or milk of lime density and flow rate, and quality of the calcined lime.

1. Diffusion Juice Density. In order to achieve even density, smooth diffuser operation is essential. Because the juice density changes unavoidably with changes

of juice detention time in the diffuser, there should be enough capacity in the diffusion juice tanks to average out the density fluctuations.

2. Diffusion Juice Temperature. It is possible by careful operation of the diffusion juice heaters to heat the diffusion juice to a fairly uniform temperature. Since the heater tubes are quickly fouled with coagulated albuminous substances, they must be cleaned frequently in order to maintain a high coefficient of heat transfer.

3. Diffusion Juice Flow Rate. This factor is a very important one, and some type of meter or indicator should be used to measure the rate of flow. If the flow rate does vary, the saccharate or milk of lime flow rate should vary with it.

4. Kiln Gas Pressure. This pressure should be constant to within ±1 psi of the nominal gauge pressure for smooth control. When it is noted that most of the pressure is required to overcome the head in the gassing tank, the effect of pressure variations on the small remaining portion of the total gas pressure, and thus on the amount of gas entering the gassing tank, is very appreciable. Since variations of the gas pump speed will frequently interfere with lime kiln operation, some form of pressure controller independent of the gas pump, and close to the carbonators, is indicated.

A good solution, if gas is not in short supply, is to have the gas pump provide excess pressure, and to provide control by venting gas to the atmosphere near the carbonators. In order that such a controller keep in working condition, the gas must be washed reasonably free from tarry substances. With reciprocating gas pumps it has been found useful to discharge from a relief valve back into the suction of the pump itself to smooth out short periods of oversupply.

5. Carbon Dioxide Content of Kiln Gas. The factor is frequently ignored, but it is just as important as the gas pressure. Large drops in CO_2 percentage are commonly due to leaks in the kiln. A common cause is the kiln door failing to close entirely after a charge of rock has been put in. A continuously recording carbon dioxide analyzer is a great help to both kiln and carbonation operators. For the former, it indicates the conditions of combustion or air leakage in the kiln, and for the latter, it permits changes to be made in lime flow, to prevent major changes in alkalinity, when there is a change in percentage of CO_2 without change in total gas pressure.

6. Lime Milk or Saccharate Brix. Unless the density of the milk is constant, carbonation operation will be very irregular. The lime slaking or saccharate mixing station frequently does not receive the attention it deserves, considering the importance of the uniformity of its output. The density should be automatically controlled.

7. Lime Milk or Saccharate Flow Rate. The liming agent flow rate may be controlled with a valve, or with a liquid chemical feeder of the Howard, or bucket-wheel type. Regardless of the type of flow-controlling unit used, it should be supplied by a circulating pipe loop to prevent sedimentation of solids in the pipeline during periods of low demand.

8. Quality of Burned Lime. If the calcined lime rock is not constant in its CaO content, maintenance of a steady addition of CaO will be difficult. Methods of obtaining uniform lime quality are discussed in Chapter XVII.

Other Dorr System Modifications

That many factories (111a) return small amounts of the clarifier underflow to the diffusion juice is motivated by the need to improve the sludge settling rate, or the filtration rate of the thickened sludge, or to lower sugar losses in the waste filter cake *(lime flume)*. Some have noted accessory benefits in less frequent cleaning of the diffusion juice heater tubes.

Seven Great Western factories use a reversed flow direction of recirculation in the primary and secondary tanks, so that the juice flow is concurrent with that of the gas bubbles in the gassing tank (see pp. 201-202).

Michigan Sugar Co. System

First put in company-wide use in 1978-79, this is essentially a "classical system" with ordinary first carbonation replaced with Benning defeco-carbonation with full recirculation. Because of full use of flocculation agents, where formerly it was impossible to use preliming and main liming with Dorr carbonation, it is now possible, with the further use of some first carbonation sludge in the prelimer.

The raw juice at about 65 °C is sent to a Brieghel-Müller prelimer (Fig. 19), which has an additional compartment on the downstream end, into which juice overflows from the prelimer, and in which main liming takes place. Long range plans call for increased slicing rates, and at such a time the additional compartment will become a part of the prelimer. Some carbonation sludge is returned to the prelimer, and the prelimed juice has a pH value of 10.9-11.1. The juice is then heated to 85-87 °C.

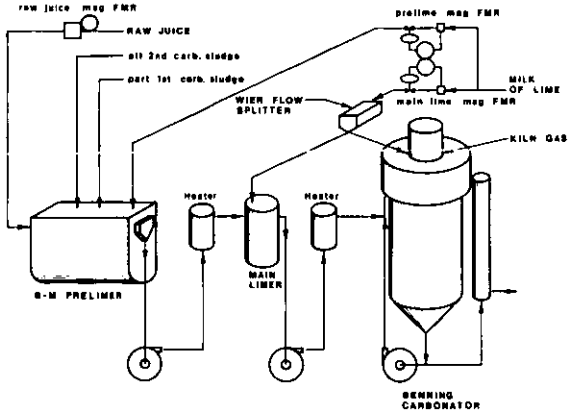

Figure 8-19. Michigan Sugar Co. Juice Purification System.

Standard defeco-carbonation takes place in the Benning carbonator. The milk of lime addition is split with a Putsch lime feeder. In the early part of campaigns, 85% goes to main liming and 15% to preliming. Later in campaigns the split is 50:50.

From the Benning carbonators the first carbonation juice goes to: 1) a Dorr ATV clarifier at Carrollton; 2) a de-airing tank and an Enviro-Clear clarifier at Croswell; and 3) to B.M.A. tray clarifiers at Caro and Sebewaing. The underflow from the clarifiers which is not returned to the prelimer is separated in rotary vacuum filters.

Flocculant addition at all factories varies from 4 to 8 ppm.

The installation at Croswell is similar excepting that: 1) main liming is carried out in two small tanks in series, with slow-speed agitators; 2) the raw juice is heated to 70-80 °C before main liming.

With all installations, preliming and main liming can easily be bypassed, changing the systems to ordinary Dorr-Benning defeco-carbonation.

The Great Western Juice Purification Systems Original, Multi-Tank System

The G-W multi-tank systems are essentially variants of the "classical" system, consisting of preliming, main liming, first carbonation, and clarification (64).

The original installations of the system used batch carbonation tanks throughout. The first tank was separated into three compartments, each containing a stirrer, and to each of which lime was added, totalling about 0.2% CaO on beets. This was preliming. These prelime units have now all been replaced with Brieghel-Müller prelimers. The installation at Sterling is closest to the original. Four other factories use modifications of this system; all of the factories now use Brieghel-Müller prelimers; the Ovid factory has only three tanks as does Goodland; the Greeley and Fremont factories use only two tanks.

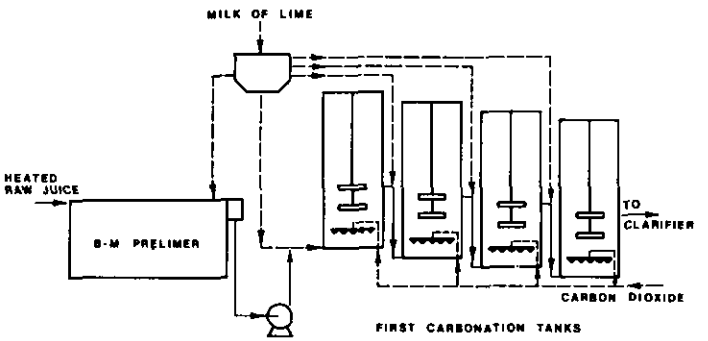

Figure 8-20. The Great Western Multi-Tank Juice Purification System.

Modifications

At Sterling most of the balance of the lime is added to the juice as discharged from the prelimer. Lime can be added to all the four tanks, which are equipped with stirrers, and small amounts generally are. Likewise, gassing can be conducted in any of the four tanks. Presently normal operations at Sterling involve: no gas to no. 1, and small amounts of lime and gas to nos. 2, 3, and 4. Juice flows in all tanks are downward, countercurrent to the gas bubbles. From no. 4 the juice goes to a small de-aerating tank, and then to an Enviro-Clear clarifier.

The Ovid and Goodland factories are using only three tanks after the prelimer, and the no. 1 tanks are used for main liming. As before, all three tanks have stirrers and gas distributors, and in nos. 2 and 3 the juices are carbonated; small amounts of lime may be added. Both these factories use Dorr tray-clarifiers.

The factories at Greeley and Fremont have only 2 tanks after the prelimer. The no. 1 tanks are used for main liming, and the no. 2 tanks for carbonating and liming. The Greeley factory uses gravity-feed Grand Pont filter-clarifiers, while at Fremond an Enviro-Clear is used.

Operations with the Multi-tank Systems

The juices produced by these systems are generally satisfactorily thermostable. Compared to thin juices from the Dorr process, lime salts may be slightly increased, but colors and saponin elimination are appreciably improved.

After the raw juice is heated, it enters the prelimers, where it is held for about 25 minutes. The prelimer deflectors are adjusted in accordance with the pH profile of the six compartments every 8 hours. Most of the remaining lime, about 1.3% on beets is, at Sterling, added to the juice as it leaves the prelimer, and small amounts in the other three tanks. At the other factories the first tank following the prelimer serves as a main-liming tank, with about 15 minutes detention, and small amounts of lime are added in the remaining tanks.

Excepting in unusual circumstances, none of the Great Western factories return carbonation sludge to the raw juice or prelimer.

Dorr-system Modifications

Seven of the remaining factories use variations of the Great Western modification of the Dorr system with no preliming or main liming; defeco-carbonation with concurrent juice and gas flows, and Dorr clarifiers, as described on page 201.

The Scottsbluff factory uses the Dorr system, with countercurrent flows, and a Dorr clarifier.

BMA Juice Purification

The BMA, or "Braunschweig-65" juice purification process is based on the joint development by Schneider and co-workers of the Braunschweiger Sugar Institute (79) and by the BMA (Braunschweigische Maschinenbau Anstalt), and resulted in a "combination process" using first-carbonation sludge addition to the diffusion juice, preliminary defeco-carbonation, main liming, first carbonation with sludge separation by clarifiers and rotary vacuum filters, and normal second carbonation (53,77,91). Studies by Schneider (75) using microscope techniques indicated to him that with this process flocculation of colloids appears to be as complete, and the envelopment of the flocs with calcium carbonate crystals more complete, than with progressive predefecation processes. He found the first carbonation sludge particles to be 30-40μ in diameter, as contrasted to 10-15μ from predefecation processes.

The BMA process has undergone several changes since its first introduction. Figure 8-21 shows it in its current form as "BMA-65."

The process now involves: 1) mixture of the diffusion juice with a portion of the thickened first carbonation sludge; 2) heating of the "mixed feed juice"; 3) defeco-carbonation of the mixed feed-juice by its introduction into a carbonation tank in which the juice is held at about 8.8-9.0 pH (20 °C), into which limed juice from the succeeding "intermediate liming" is recycled at a rate of 800-1000 percent. It is claimed that juice alkalization in this way is much milder than when done with milk of lime, although this would not necessarily be true of stepless progressive preliming; 4) intermediate liming, where about 40 percent of the total CaO is added; 5) main liming with the additional 60 percent of the CaO put into the suction of the pump to the first carbonation heater; 6) residence time in the main defecation tank; 7) first carbonation; 8) heating before entry into the multi-tray clarifier; 9) part of the thickened sludge is sent to the diffusion juice, and the

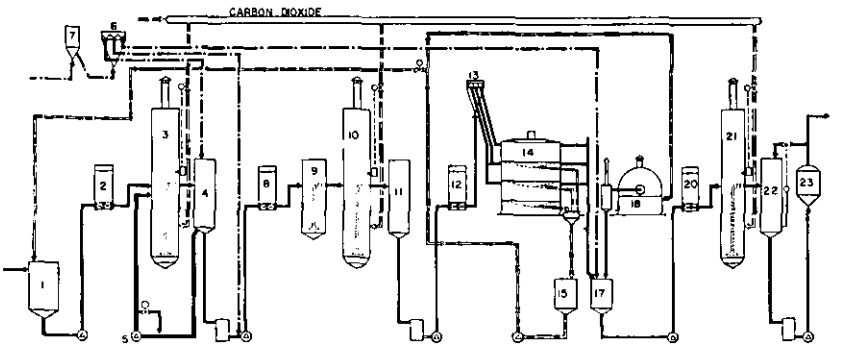

Figure 8-21. BMA-65 juice purification. 1) Feed-juice tank. 2) Feed-juice preheater. 3) Precarbonation. 4) Intermediate liming. 5) Recirculation pump. 6) Milk of lime proportioner. 7) Milk of lime feed tank. 8) Intermediate juice heater. 9) Main liming. 10) First carbonation. 11) Pumping tank—first carbonation. 12) Carbonation juice heater. 13) Carbonation juice distributor. 14) Thickener. 15) Thickened sludge tank. 17) Clear juice tank. 18) Rotary vacuum filter. 20) Second carbonation heater. 21) Second carbonation. 22) Pumping tank—second carbonation. 23) Candle filters. *(Courtesy, Braunschweigische Maschinenbau Anstalt.)*

balance is filtered with rotary vacuum filters, the clear filtrate joining the clear juice from the clarifiers; 10) addition, if desired, of small amounts of milk of lime before the second carbonation heater; 11) second carbonation; and 12) second carbonation juice filtration using candle-element filters. Costesso *et al.* (18a) have calculated the operating factors of the process.

The process can be adapted to use portions of existing equipment, and is available in various modifications, such as for dry liming.

Overall, it is claimed that a weight ratio of CaO used to diffusion juice nonsucroses of only 0.85 is required, and that with the superior sedimenting and filtering properties of the sludge, no filtration of the clear juice from the clarifier is required. There are a very large number of installations of the BMA-65 process throughout the world (6a,21a,72).

Putsch Juice Purification (71)

The Putsch "combination process," of which there are two modified installations in North America, and many in other parts of the world, is unusual in its ready adaptability to diffusion juices from beets of varying quality. Three discrete systems are embodied, any one of which may be put into operation by the change of a few valve positions.

See Figure 8-22. The solid flow line indicates the course of the normal, or A process. The Putsch prelimer receives the mixture of diffusion juice and thickened first carbonation sludge and accomplishes a stepless progressive preliming. The lime for main liming may be added to a compartment in the end of the prelimer before juice heating, so that a vigorous defecation can be accomplished, or the main lime can be added to the main liming tank after the heaters, for a milder defecation. After the juice heating and its residence in the main liming unit, the juice is first carbonated in a carbonation unit with 300-400% external recirculation. Clarification of the finished first carbonation juice is done in Putsch filter-thickeners or tray clarifiers following further heating. Part of the thickened sludge is returned to the

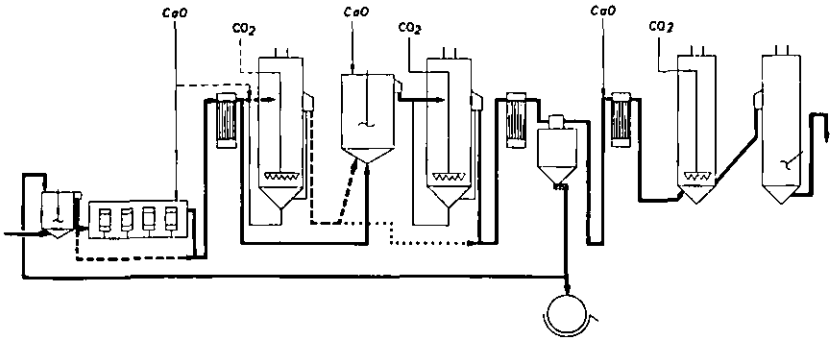

Figure 8-22.
Process A ————; Process B ————, ·····;
Process C ————, ·····, ·····.
(Courtesy, H. Putsch & Comp.)

diffusion juice, but the greater part is filtered in rotary drum filters. The clear juice from the thickeners is mixed with the filtrate from the rotary filters, and proceeds to second carbonation.

The B process is applied to juices from beets which are somewhat degraded from long storage, rot, or disease, and the flows are shown by the solid and dashed lines in the figure. Liming is eliminated, and the diffusion juice mixed with carbonation sludge is treated by a preliminary defeco-carbonation to a suitable pH value, depending on the quality of the beets, and is then main limed and first carbonated to a final pH of about 10.8 (20 °C).

Process C is designed for badly deteriorated beets. Here the flows follow the solid, dashed, and dotted lines. The diffusion juice is again mixed with thickened sludge, both preliming and main liming are omitted, and the process is one of very mild defeco-carbonation with recirculation.

The chief advantages of Putsch juice purification come from its easy variability. Using process A an increase in thick juice purity of 0.8 units over the "classical" process is said to be obtained, and only 1.8% CaO on beets is normally required. Process B uses about 2.4% CaO on beets. The defeco-carbonation process C has a reduced capacity of about 80 percent that of process A.

Barber (6) reported on results with a modified installation of the Putsch system at the Ottawa, Ohio, factory, which gave excellent results with beets which had seriously deteriorated. Performance with normal beets was exceptional.

Continuous Systems Not Used in North America

Novi Sad Juice Purification. The original system was devised by S. Wolf[1], in the Food Industries Institute, Novi Sad, Jugoslavia, and installed in its second and perfected form in the Sremska Mitrovica factory in 1964, and is also used in Austria. The process is presently under the promotion of Maschinenfabrik Buckau R. Wolf.

The purification system, in its final form, is shown in Figure 8-23. The calcium carbonate sludge from the carbonation clarifier is added to the unheated, unscreened diffusion juice, in a Naveau prelimer. In this unit neutralization and

[1]See references 44,98a,99,125,126.

formation of crystalline carbonates are described as occurring (126), as well as ion-exchange type reactions of the colloidal micelles produced, as a result of which potassium, sodium, and magnesium ions are displaced by calcium ions, and stabilized aggregates form around the calcium carbonate crystals. The addition of milk or lime to the mixer is optional, but not usual.

The treated juice enters defeco-carbonation after heating. A recirculation rate of 800% in this unit is said to assist in providing pH changes which help in the dehydration and stabilization of the coagulates. The defeco-carbonation tank is of unusual design, the primary tank being a very small unit contained within the secondary tank. Recirculation is from the bottom of the secondary tank to the bottom of the primary tank. Lime addition is to the primary tank, and carbonation is carried out in the secondary, and sometimes in the primary tank as well.

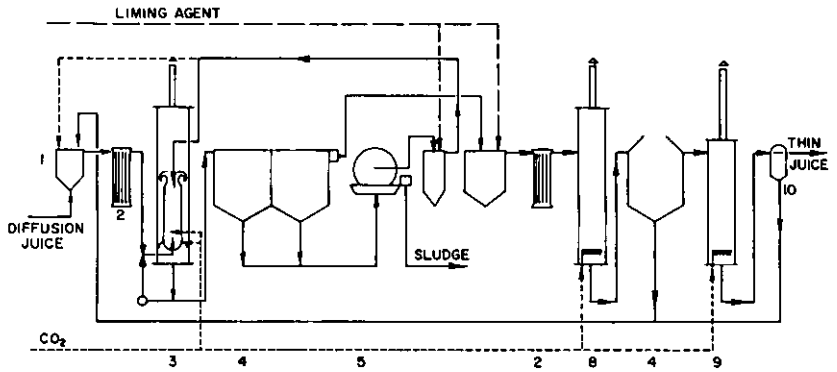

Figure 8-23. Novi Sad juice purification, perfected form. 1) Diffusion juice—carbonation sludge mixer; 2) heater; 3) defeco-carbonation; 4) tray thickeners; 5) rotary vacuum filters; 6) milk of lime mixing tank; 7) main liming; 8) first carbonation; 9) second carbonation; 10) filters. *(Courtesy, Milovan Bosnjak.)*

The effluent from the defeco-carbonation enters a tray clarifier (or filter-thickener to reduce detention time). The clear filtrate (or overflow) is first limed in vessel 6, and then returned to vessel 3 to act as precipitant. The clear overflow from the first clarifiers is main limed in 7 with about 1% CaO on beets, and heated to 94-95 °C after a detention time of about 10 minutes at the high alkalinity. Since the coagulum of colloidal matter has been removed, the vigorous defecation does not peptize any colloidal material. There follows a stage of single-pass carbonation, in which adsorption purification takes place. The precipitate is separated in another clarifier, and all of the underflow is returned to the diffusion juice. Since the second carbonation sludge is returned to the carbonation, all of the calcium carbonate sludges leave the system through the defeco-carbonation sludge system.

The perfected system is said to produce very thermostable juices, with good sludge settling (> 4 in./min) and filtration properties ($F_k < 2$). The thin juices have low colors and lime salts, and the whole process is said to be relatively insensitive to changing beet quality.

The South German Sugar Co.

This process is used in the factories of this company, in the Bavarian region of West Germany. It is typical of the best processes used in Europe, including the

process steps of sludge return to the raw juice, preliming, cold main liming, hot main liming with air injection, 1st carbonation with recirculation, separation of sludge with filter-thickeners, and desweetening with rotary vacuum filters, as well as some more abstruse features (73a).

Raw juice is heated to 50-55 °C, and enters the Brieghel-Müller prelimer. Thickened first and second carbonation sludge is returned to the 2nd or 3rd prelimer cell, in quantity sufficient to give the juice leaving the prelimer a content of 1.2-1.3% on total CaO. The pH at the point of addition must be under 9 "to get a good overcarbonating effect on the sludge."

CaO as milk of lime is added in the prelimer, 0.2-0.3% on beets. In a stirred tank, 1.2-1.4% CaO is added to the still cold juice. The juice then passes through a heater, and is heated to 88 °C before entering the main limer tank. In some installations, finely dispersed air bubbles are injected into the main limer. This is to oxidize coloring matter, which is more completely removed in the oxidized state.

First carbonation is standard with a small primary tank enclosed within the secondary (see Fig. 24) and with 600% recirculation. The first carbonation juice from the recirculation pump enters the line which transfers limed juice to the first carbonation tank. This composite stream enters the cylindrical retention vessel inside the tank. To assure full detention time, this vessel is separated into three parallel compartments, the juice flowing upward in the first, downward in the second, and up again in the third compartment, from which it overflows the brim of the vessel, prior to being carbonated again. Thus the pH value "swings" between 11.0 pH after carbonation and 12.5 after mixing with the limed juice. This exposes the juice to different iso-electric points, and helps to stabilize the floc, it is said.

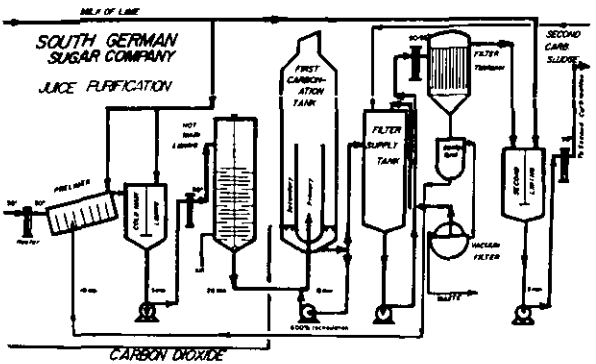

Figure 8-24. Juice purification of South German Sugar Co.

The endpoints of preliming and first carbonation are determined by the method of Bazcek and Jesic, as modified by Büsching (15a).

The first filter-supply tank receives the first carbonation juice, and before passing to the filter-thickeners it is heated, together with the second carbonation sludge, to 90-95 °C and the thickened combined sludges are split between the rotary vacuum filters and return to preliming.

Comparisons made with another factory nearby using an ordinary defeco-carbonation indicated that the new process used 5% less lime and the juices had less color. On the other hand, filtration and settling were slower in the new system.

D.D.S. Juice Purification System

This system, developed by de Danske Sukkerfabrikker (Danish Sugar Company) and installed in a number of factories in Europe, has several valuable features which make its appearance in North America likely (Figure 25) (111e).

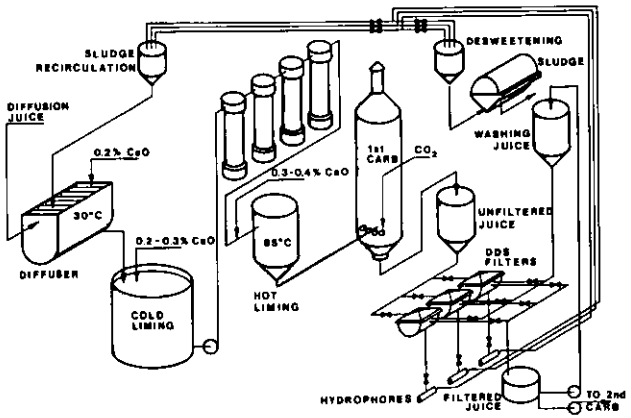

Figure 8-25. D.D.S. Juice Purification.

The most important advantage is the unusually vigorous defecation, and it may be the only system which completely hydrolyzes the amides, thus eliminating pH drop across the evaporators and crystallization processes, as well as ammonia evolution, and the use of thickener-filters with their short detention times.

Probably because of this very complete lime treatment, both first and second boilings produce sugars which are used for first-class product.

The juice from the diffuser is prelimed in a Brieghel-Müller prelimer, to which about 10% of the first carbonation sludge is added. The effluent from the prelimer then undergoes cold main liming, in which the detention time is from 30 to 60 minutes. The very large tank in which this takes place is very valuable also to "take up the slack," in case speed-ups or slow-downs are required by the rest of the factory processes. The juice is then heated and proceeds to a 10-minute hot main liming, and then is carbonated in a single-pass unit. In case the beets require it, small amounts of milk of lime may be added to first carbonation.

The finished first carbonation juice is then clarified with D.D.S. filter-thickeners, and the sludge is separated on rotary vacuum filters, The clear juice is then second carbonated.

Blanke Tube-carbonation. Continuous carbonation using the barometric-leg principle for gassing was introduced by Reboux (73) and Blanke (34). The principle was further developed by Spengler, Bartsch, and Wigand (37,38), by Thieme (36) and Asselbergs (5). Melzwig (60) described the use of such a system at the Walschleben factory.

Kartashov Process. This is a Russian defeco-carbonation process which has gone through evolution, using a varying number of stages of defeco-carbonation, and varying percentages of recirculation[1].

[1]See refs. (47, 47a, 48, 49, 50, 82, 131a)

Brieghel-Müller Process. Originally patented in the United States in 1954 (14) this Danish process has also changed over the years. It incorporates predefecation, return of sludge to the prelimer, and main liming (15).

Alston Processes. P. W. Alston has patented two processes. His A process involves multi-stage predefecation followed by removal of the predefecation floc with low-head, moving belt filters (2). In his B process, the second carbonation juice is passed through the evaporators in the presence of the second carbonation sludge, and the thick juice is further limed, carbonated, and filtered (3).

Dedek-Vasatko Process. What is sometimes known as the Goslawicz process (12,35) involves purposeful fluctuations of the alkalinity in first carbonation for better sludge properties. Dedek and Vasatko proposed a process in which, after preliming, defeco-carbonation was used, holding the alkalinity at the optimum for the whole period of lime addition (21). During this time the alkalinity was permitted to fluctuate, for what were believed to be precipitate-improving purposes (65).

Wiklund Juice Purification. As earlier mentioned, this was the pioneering process to return overcarbonated juice from first carbonation to the predefecation (122,124). Later, the process was altered to return thickened sludge instead of overcarbonated juice (100), and Mottard has reported its modification to use the Grand Pont filter in first carbonation (62).

Oreye Process. Dormal (28) reports a system used at this Belgian factory, requiring only 1.1% CaO on beets. It uses preliming, preliminary carbonation, and removal of the sludge by clarifiers and rotary vacuum filters. The clear overflow juices are main limed, carbonated, and the sludge is removed by clarifiers.

Tasco Juice Purification. This process (9), devised by The Amalgamated Sugar Company's Research and Development department, and installed in its Mini-Cassia factory, was the only process used in North America in which prelimer sludge was physically removed from the juice before main liming. The thin juices produced were of the quality associated with the classical process—slightly higher lime salts, better colors, relatively floc-free sugars, and thermal stability—and there was an additional definite purity increase due to the early removal of the predefecation sludge. Its use was terminated for reasons not related to the method itself, and its future further use by that company is regarded as possible.

See Figure 8-26. Screened diffusion juice was heated to 90 °C and entered the Brieghel-Müller prelimers. Sludge from carbonation was run into the third and fourth compartments, and about 0.25% CaO on beets in the form of milk of lime was added in the last compartment. The compartment deflectors were set to give a concave gradient through the cells, from 6.5 to about 10.0 pH (see pp. 188-191). The final alkalinity of the prelimed juice was held at about 0.005 CaO% alkalinity above the final first carbonation alkalinity or at about 0.085 to 0.090% CaO alkalinity.

The prelimed juice was clarified in a 5-tray clarifier with the assistance of a small amount of polyacrylamide settling aid. The overflow went on to main liming. The clarifier underflow was filtered through belt filters. The desweetened sludge was discharged to waste, and the filtrate was routed to main liming.

The remainder of the lime, about 2.25% CaO on beets, was added in the main liming tank. After a detention time in the main liming tank, the limed juice was submitted to first carbonation in Benning carbonators. The finished first carbonation juice was thickened in 24 Krebs hydrocyclones. The concentrated slurry from the

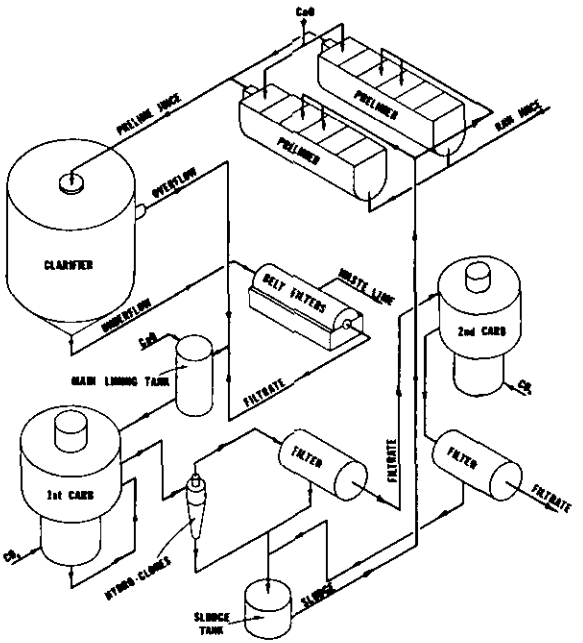

Figure 8-26. Tasco juice purification. *(Courtesy, The Amalgamated Sugar Company.)*

hydrocyclones was introduced into the prelimers as previously indicated, and the slightly cloudy overflow from the hydrocyclones was polish-filtered in Industrial leaf-type pressure filters. The separated sludge was combined with the hydrocyclone underflow, as was the second carbonation sludge, and returned to the prelimers.

Zagrodski Process. A method introduced in Poland by S. Zagrodski Sr. and Jr., in 1971 (130a,131a). The method involves a multiplicity of steps; preliming, precarbonation, 1st liming, 2nd liming, 1st carbonation, 3rd liming, 2nd carbonation. A total of 1.6% lime on beets is used: 0.45%, 1.1%, and 0.1%. It recirculates limed juices and carbonation muds to obtain required pH values.

Table 8-4 summarizes the essential features of the important continuous liming and carbonation systems.

Table 8-4 Continuous First Carbonation Systems in Use in the United States and Canada in 1969, and the Novi Sad Process

			Sludge	
Process	**Pretreatment**	**Main Purification**	**Separation**	**Desweetening**
Dorr	none	2-tank defeco-carbonation with recirculation.	clarifier	rotary vacuum filters
Dorr-Benning	none	Compartmented tank defeco-carbonation with recirculation.	"	"

Table 8-4 (Continued)

Process	Pretreatment	Main Purification	Sludge	
			Separation	Desweetening
Dorr-Great Western modification	none	Secondary tank 3 compartments. Concurrent flow gas bubbles and juice. Internal and external recirculation.	"	"
Michigan	sludge return, B-M preliming	Main liming in extension of B-M prelimer tank. Benning defeco-carbonation.	clarifiers	"
Great Western Multi-tank	no sludge return, B-M prelimer	4, 3, or 2 tanks in series. Main liming in no. 1. Gassing and small amts. of lime in others. Stirrers, single pass.	Dorr or E-C clarifiers. One Grand Pont filter-thickener instn.	"
BMA-65	sludge return, 2-tank defeco-carb. with recirculation	Main liming, single-pass carbonation.	thickener	"
Putsch "A"	sludge return, preliming	Main liming, carb. with recirculation.	clarifier or filter-thickener	"
Putsch "B"	sludge return, defeco-carb. with recir.	"	"	"
Putsch "C"	sludge return	Defeco-carbonation with recirculation.	"	"
Novi Sad	sludge return, 2-tank defeco-carbonation with recircu-lation, sludge sepn. with thickener and rotary vacuum filters	Main liming, single-pass carbonation.	clarifier, underflow to diffusion juice	"
South German	sludge return to 50-55 °C jce. B-M prelimer	Cold main liming. Hot main liming at 88 °. Air injection. 1st carbonation with recircn, pH fluctuation.	filter-thickeners	"
D.D.S.	sludge return to B-M prelimer	Prolonged cold main liming. Hot main liming. 1st carbonation with recirculation. Some liming in filter-thickener supply tank.	DDS filter-thickeners	"
Tasco	all sludge return, pre-liming, coagulum separation with thickener and vacuum filters	Main liming, Benning carbonation with recirculation.	hydrocyclones, underflow to preliming	" in pretreatment

Steffen Campaigns

While the carbonation of Steffen saccharate milk without accompanying beet slicing is only marginally profitable, such operations have been carried out for short periods, up to several weeks, many times on the Pacific coast. The advantage has derived from extraneous factors, depending on keeping the factory running when the beet supply has been temporarily interrupted, due to the interference of weather with harvesting, beet transportation delays, or to lengthy breakdown of beet-end equipment. Continued operation saves shutdown and start-up costs, such as labor expenses and sugar losses, shutting down the lime kiln, and expenses in laying off and rehiring operating crews. Production is necessarily small. One 4000 ton beet-slice factory operates its *Steffen campaigns* at 300 tons of molasses worked per day, which can produce a maximum of about 2000 cwt of sugar a day. The amount of the fixed costs per cwt of such sugar is at least five times normal.

The dilution of the saccharate milk, or the saccharate syrup, is set by the amount of water required by the evaporators to produce the necessary steam for the factory, and has usually resulted in thin juices of a little under 15 rds. Due to the very low natural alkalinity, the alkalinity of first carbonation does not have many of the usual effects. However, since sludge settling is usually retarded a little at this density, and especially since the saccharate is not completely broken up at alkalinities much in excess of 0.060, the alkalinity is usually kept in a low range. In one company the range has been from 0.020 to 0.060 alkalinity.

Thin juice colors and lime salts are very high, and floc very low. Soda ash addition is required. The raffinose builds up rapidly, and it is customary to discard 100% of the molasses produced, to avoid serious interference with crystallization. A major problem is the disposition of the discard molasses, since there is no beet pulp to which it may be added.

Steffen Operations With Thick Juices Reprocessing. Schmalz and Park (74) and Lott and Varney (56) have described the combination of Steffen saccharate and stored standard liquor processing in recent operations in the Northwest.

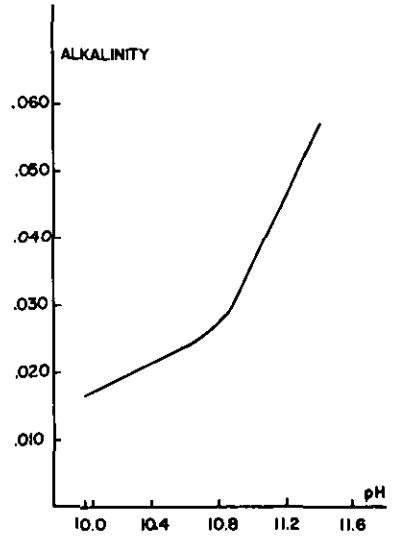

Figure 8-27. Steffen saccharate first carbonation *without beet slicing. Relation between* pH and alkalinity. *(Courtesy, Schmalz and Park, 1970 (74).)*

In this case, lower density (4-9 rds) carbonation juices were indicated. The unusual relationship between the alkalinity and pH was determined, and is shown in Figure 8-26. Operations were carried on in the normal pH range in first carbonation. The optimum pH in second carbonation was found to be 0.2-0.3 units less than normal.

The combination of low juice density and high CaO content yielded very rapid first carbonation sludge settling rates, and the absence of gummy, colloidal material further enhanced filtration rates. To hold the very fine, unagglomerated calcium carbonate crystals, a more tightly woven filter cloth (16 oz cotton twill) was used for both first and second carbonation filtration. The approximatey twice-normal thin juice coloring matter, mostly from invert destruction, was found very troublesome, and required addition of about 5% soda ash on solids, and 300-350 ppm of sulfur dioxide. Because of the relatively low juice flow rates, it was possible to bypass the clarifier, and filter out the first carbonation sludge without difficulty in the rotary vacuum filters.

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B. Clarification*

Clarifying Equipment

Four basic designs of first carbonation clarifiers are in general use in the sugarbeet industry. The first type, the Dorr Multifeed clarifer, normally contains four clarification compartments of which one or two are used to thicken and discharge mud. In addition, this design contains a fifth, or "conditioning" compartment, located above the clarification department.

The second type, of more recent design, is the Dorr ATV clarifier. This clarifier contains four clarification compartments, each of which thickens and discharges mud. The unit has no *conditioning* compartment and contains a simplified feeding arrangement. The basic design principles of this unit are incorporated in the latest designs of many tray clarifiers of European manufacture.

The third type is the B.M.A. unit, which similarly is made of superimposed settling trays. However, the separate tray compartments are not interconnected, and any compartment can be removed from service and emptied at any time.

The fourth type, the Enviro-Clear unit, operates with the use of flocculating agents. These have the advantage of small size and short juice detention times.

Multifeed Clarifier

The Dorr Multifeed clarifier for carbonation juice (see Figures 8-28, 8-29, and 8-30) is enclosed in a cylindrical steel tank, with a flat inverted-cone bottom and a conical cover. The interior is separated into a number of shallow compartments by flat conical steel trays, which are parallel to the bottom and which separate the compartments completely except for a central opening which extends through all of the trays into the bottom compartment.

A central feed cylinder, driven by a mechanism mounted on beams which span the cover, rotates, in this opening. Attached to this central cylinder, are the flocculating paddles and the rake arms mounted in the various compartments, as well as the hydraulic seals and sludge passages which isolate each compartment, and enable it to perform in much the same manner as a separate unit.

In the usual machine, shown diagrammatically in Figure 8-28, there are four trays which separate the clarifier into five compartments. The hot carbonated juice enters tangentially into the upper or *conditioning* compartment, which provides a short period of detention for the release of any entrained gas and for the separation of foam. In this compartment the juice is also subjected to the action of a floc-

*by R. H. Van Note; revised by R. A. McGinnis

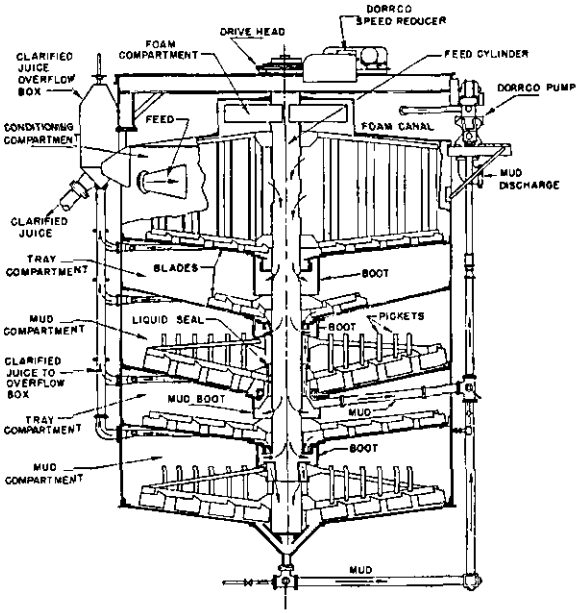


Figure 8-28. Dorr Multifeed clarifier. *(Courtesy, Dorr-Oliver, Inc.)*

culating mechanism, driven from the central cylinder, which is intended to promote coalescence of the fine suspended solids into larger agglomerates with faster settling rates. The foam rises to the surface of the juice into a small cylindrical foam compartment, equipped with rotating blades, which skim the foam into a foam canal (Figure 8-29).

From this conditioning compartment the feed passes into the center cylinder through a number of openings and then flows in parallel into each of the lower compartments where the precipitate settles onto the trays and is collected and moved toward the center of the machine by the raking mechanism (Figure 8-30). The settled sludge from the tray compartments, having been raked to the center of the machine, moves downward through the baffled annular space outside of the center tube and falls into the mud compartments immediately below. From the mud compartments the thick sludge is withdrawn from the machine by means of diaphragm-type suction pumps or air lifts, through suitable piping.

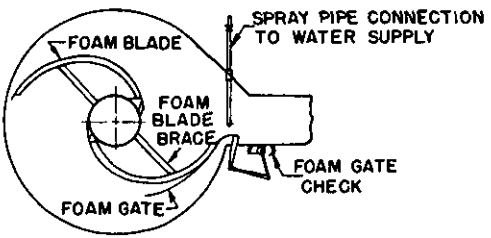


Figure 8-29. Dorr Multifeed clarifier. Arrangement of foam compartment mechanism. *(Courtesy, Dorr-Oliver, Inc.)*

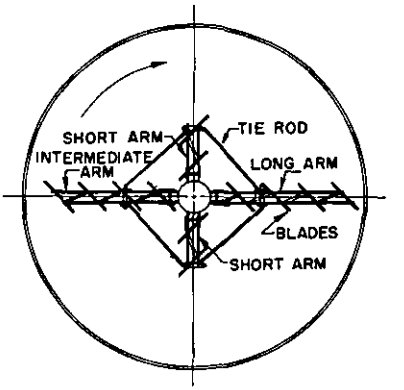


Figure 8-30. Dorr Multifeed clarifier. Arrangement of arms in tray compartment. *(Courtesy, Dorr-Oliver, Inc.)*

The clear juice, from which the precipitate has settled, moves slowly toward the clear juice outlet headers, the intakes of which are located at the highest point of the periphery of each of the four clarification compartments. These clear juice headers lead to the overflow box through individual pipes from each compartment, and the upper end of each pipe is equipped with an adjustable sleeve so that the rate of flow from each compartment can be controlled. From the overflow box the combined clarified juice from all compartments is pumped, or flows by gravity, to the next step in the process.

To prevent heat losses, and the resulting convection currents which might interfere with the settling of the precipitate, the entire clarifier tank and cover is insulated.

The mechanism, with rake arms, rotates at a speed of approximately 1/7 rpm. The diaphragm pumps (see Figure 8-31), removing the thickened sludge are driven at a constant speed of 50 rpm, the volume removed being adjusted by varying the length of the stroke of the pump.

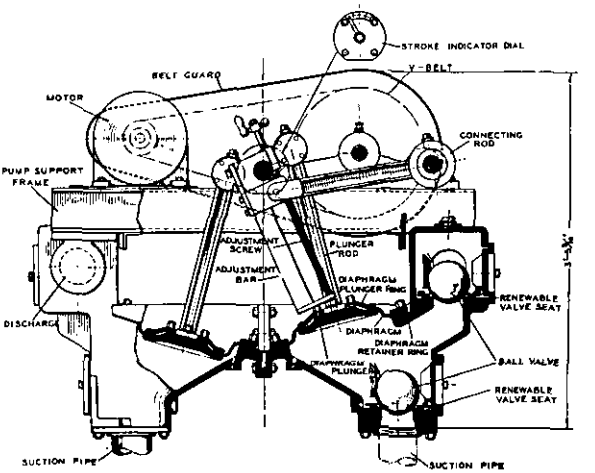


Figure 8-31. Dorrco Sludge Pump. Sectional elevation. *(Courtesy, Dorr-Oliver, Inc.)*

ATV Clarifier

The Dorr ATV clarifier (see Figure 8-32) has the basic tank configuration and drive mechanism structure of the Multifeed clarifier. Arrangement of the foam compartment mechanism is also similar to the Multifeed design.

The machine is separated into four clarification and thickening compartments by three internal trays. The clarifiers might be considered as four separate unit-type clarifiers, each unit stacked one on top of the other and each receiving feed from a common source.

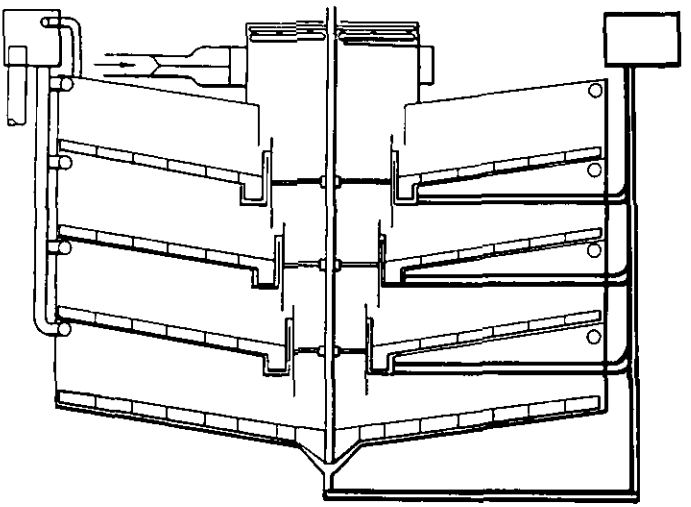


Figure 8-32. Dorr ATV clarifier. *(Courtesy, Dorr-Oliver, Inc.)*

The full-tank diameter conditioning compartment of the Multifeed design has been replaced by a much smaller feed compartment to reduce detention time. Hot carbonated juice enters a specially designed feedwell which splits the flow into equal and diametrically opposite streams. These streams impinge on one another and reduce the initial energy and velocity of the incoming pipeline feed stream to a minimum. This action creates a quiescent effect in the feed compartment, thus providing for the release of entrained gas from the hot juice and the entrance of feed to the clarification compartment without undue turbulence.

Hot carbonated juice enters the four clarification and thickening compartments through centrally located feedwells of gradually diminishing diameters. These feedwells are designed to maintain a uniform and low juice velocity of approximately 4 ft per minute. The feedwells are mounted on the mechanism drive shaft through spiders and rotate with the mechanism. The lower edges of the feedwells are sealed at the level of the trays to form an upcast boot which retains the settled mud in each compartment. Mud is discharged from each compartment by means of individual sludge pumps.

Capacity

An independent survey (14) carried out in 1963 canvassed 22 beet-sugar factories in the Western United States in which gravity settling clarifiers were

employed in the first carbonation systems. Seven of these factories were Steffen houses, and the remaining 15 straight houses. Twelve factories had Dorr ATV clarifiers in operation; 10 factories were operating with Dorr Multifeed clarifiers. A large variation was found in operating conditions at the first carbonation stations and in operating capacities of the clarifiers.

Table 8-5 shows the minimum and maximum values of these operations. Values listed in each column are not related to one factory operation, but rather show the variations found in the industry as a whole.

Results of this survey showed a definite correlation between operating conditions of the first carbonation station and the clarifier capacity. In general, higher lime additions, lower juice alkalinities, higher draft, and lower clarifier underflow solids concentrations resulted in greater throughput rates in the clarifiers.

A compromise is necessary between juice quality, economics of operation and available thickening area that dictates the optimum operating conditions. High first carbonation alkalinities and high liming generally produce the best quality juice. High alkalinities however increase thickening area requirements while high lime additions increase operating costs. Likewise, higher draft and lower clarifier underflow solids concentrations increase first costs and operating costs at the evaporation and filtration stations.

The choice of size of the conventional first carbonation clarifier is therefore critical in obtaining and maintaining a well-balanced plant operation. A conventional first carbonation clarifier designed to operate at a thickening area capacity of around 1.00 sq ft per ton beets per day provides for best operating conditions to produce a high quality juice while operating within sound economic limitations.

Operation

To put the clarifier in operation, the drain valves are closed, the drive unit which rotates the rake and flocculating mechanisms in the various compartments is started, and the feed is admitted to the feed compartment. As the tank fills, the vent lines from each compartment are opened to permit the escape of any trapped air. As soon as the machine is filled, clarified juice will be discharged from the overflow pipes, and the overflow sleeves are adjusted so that flows from each compartment are equal.

Table 8-5 First Carbonation Operations and Clarifier Capacities

Items	Steffen House	Straight House
Lime addition—% CaO on Beets		
Minimum	2.58	1.50
Maximum	3.80	2.80
Alkalinity—% CaO on Juice		
Minimum	0.070	0.071
Maximum	0.088	0.111
Draft-on Beets		
Minimum	120	120
Maximum	145	155
Thickener Underflow—°Brix		
Minimum	33	28
Maximum	45	40
Sq Ft Thickening Area/Ton Beets/Day		
Minimum	0.82	0.55
Maximum	1.04	1.39

In the operation of the clarifier there are two possible adjustments, but the best means of control is generally in the hands of the carbonation operator, as will be explained below. Satisfactory clarifier operation requires a juice containing fast-settling solids which will yield a clear supernatant liquor in the available detention time, and it is at the carbonation station that these characteristics are produced. The amount of overflow from each compartment, and consequently the amount of feed to each compartment, can be adjusted by raising or lowering the sleeves on the overflow pipes. The overflow clarity can sometimes be improved by adjusting flows in this manner so as to obtain the optimum feed distribution. It should be remembered, however, that it is only possible to change the relative flows between the compartments and that raising the overflow height on one compartment, which may be producing a turbid juice, will increase the flow to the other compartments. They in turn may then be overloaded and produce turbid overflows unless the basic cause of the trouble is found, usually at the carbonation station, and is corrected.

The other adjustment is the control of the rate of sludge withdrawal, by varying the stroke of the sludge pump mechanisms. Since the clarifier is a continuously operating machine, the weight of dry solids withdrawn in the form of sludge per unit of time must equal the weight of dry solids in the feed to the clarifier. If insufficient sludge is pumped, the solids will build up in the clarifier until they eventually reach the overflow headers and produce a turbid overflow. Normally it is only necessary to adjust the pumping rate so that a sludge of the desired density is obtained. A sludge of higher than normal density is an indication that solids are building up in the clarifier, and the rate of pumping should be increased, and vice versa.

The underflow density should normally be kept between 40° and 50° apparent Brix at 80 °C, for a Steffen factory, and between 35° and 40° for a non-Steffen factory (see Table 8-5). These figures are equivalent to about 17 to 20% dry mud in the underflow in the first case, and 14 to 17% in the second case. Figure 8-33 shows an approximate curve for this relation.

It sometimes happens, however, that as a result of poorly carbonated juice or because beets of inferior quality are being worked, the clarification rate of the solids is so retarded that insufficient area is available in the clarifier to obtain a sludge of the desired high density. Under these circumstances, it is necessary to increase the sludge pumping rate in order to obtain a juice of acceptable clarity if poly-

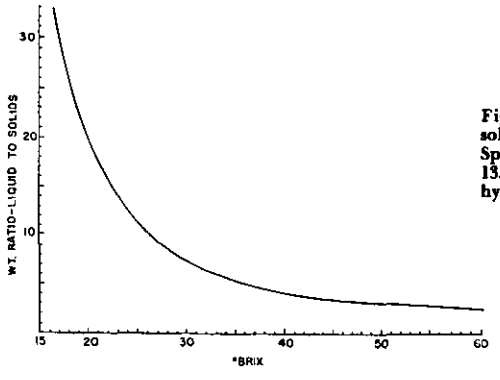


Figure 8-33. °Brix versus ratio of liquid to solids, for Dorr clarifier underflow. Specific gravity of solids, 2.70; juice rds, 13.0. Brix measurements at 80°/ with 80°C hydrometer. ***(McGinnis, 1948)***

electrolytes or other types of flocculants are not added to increase sludge thickening rates. The pumping rate is then controlled to produce a sludge of lower than normal density until corrective measures have been applied, after which it is gradually brought back up to the desired density.

To empty the clarifier, the feed is stopped and the sludge pumps are kept running until all of the mud is out. The remaining contents of the clarifier are then pumped to the second carbonation station.

Factors Affecting Clarification

In addition to the mechanical adjustments listed above, the settling of the first carbonation sludge in the Dorr clarifier is affected by the following variables: temperature, alkalinity of first carbonation juice, amount of lime used, density of first carbonation juice, form in which lime is added, percent recirculation in first carbonation, amount of feed, and other factors not here considered.

1. **Temperature.** This has an important effect on the rate of sedimentation of the mud through its effect on the viscosity of the liquid phase. It is quite an appreciable factor, and it is well worthwhile to keep the temperatures in the range 80-85 °C, both by supplying the juice feed at the proper uniform temperature, and by applying thermal insulation to the tank. Too high a temperature, on the other hand, will interfere with settling through formation of convection currents if insulation is not adequate.

2. **Alkalinity of First Carbonation Juice.** Variation of the alkalinity has a very important effect. It is apparent that in the clarifier, mixtures of mud and juice may be found of any composition between the clear overflow, the dilute mixture entering as feed, and the more concentrated mixture leaving as underflow. Examination of the settling properties of these materials has shown that the clarifier area required is fixed by the largest settling area required by any of these possible mixtures. Thus, in one instance, C, the largest settling area, might be required by the most concentrated mixtures, approaching the composition of the underflow; in a second, A, by the most dilute, and in a third instance, B, the largest area might be required by an intermediate mixture. For convenience in reference, these three cases have been termed types A, C, and B, although strictly speaking, type C is a special case of type B. These type designations are applied to the graphs or curves describing the settling characteristics of first carbonation feed materials, as shown in Figure 8-34.

The types indicated are the results of calculations, and, although dependent on the nature of the materials are not inherent characteristics of the materials themselves. For example, the type settling curve may be changed by assuming another value for the underflow discharge ratio. Hence it must be understood that type references apply only for the specific conditions of operation and assumptions of calculations. With these constant, individual materials will yield curves having the characteristics of the separate types, and the comments in this section apply.

As far as operation is concerned, it is apparent that increasing the stroke of the sludge pumps and consequently lowering the density of the underflow will not increase the clarity of the overflow in the case of type A curves, but that a very positive advantage will be gained in the case of type C curves.

Alkalinity of first carbonation is one of the most important factors determining the type of settling. At low alkalinities with normal juice density and discharge ratios, the material almost invariably gives type A curves. As the alkalinity is raised,

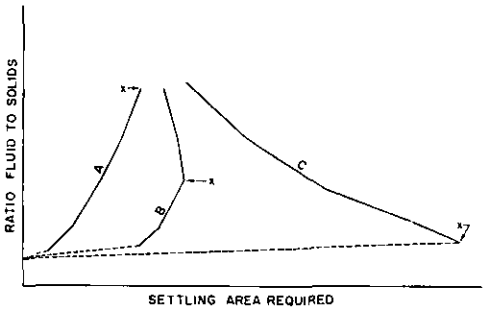


Figure 8-34. Types of settling curves, at constant underflow ratio (37).

the curves go through the intermediate type B, reaching type C in the vicinity of 0.085 to 0.100 alkalinity. As a rule, type C curves involve slower settling than type A curves, so that the overall settling time and the area of clarifier required, increase almost steadily with increase in alkalinity. However, if there is ample sludge filter capacity, and the overflow is turbid, it may sometimes be of advantage to raise the alkalinity to a point at which a type C curve is obtained, and the overflow turbidity may then be decreased by drawing out the underflow more rapidly. Laboratory settling tests, described later in this chapter, may furnish valuable guidance in such cases.

Very frequently a lowering of alkalinity may be noticed during the retention period in the clarifier. At higher alkalinities this effect tends to disappear.

3. **Amount of Lime.** The amount of lime used affects the settling in two ways. First, if the amount of beets sliced remains the same, the amount of mud to be thickened increases as the lime is increased. In the second place, the settling curve type is affected (see Figures 8-35 and 8-36), there being a tendency to shift toward the A type of settling curves with increasing amounts of lime. In general, the sum of these effects is to diminish the effective thickening somewhat with increasing amounts of lime.

4. **Juice Density.** The density of the first carbonation juice has an effect on the settling curve. If the density of the juice is raised, the alkalinities required to give the same type of curve are higher. A second effect is the increase in the juice vis-

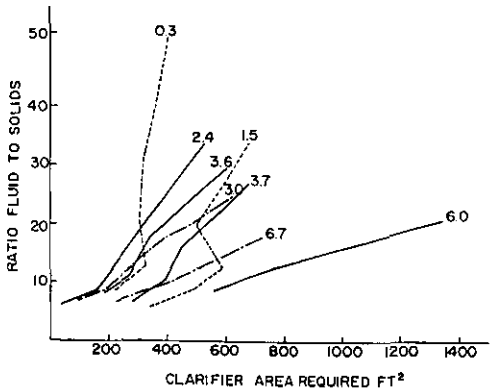


Figure 8-35. Effect of varying amount of saccharate milk on first carbonation settling curves. The numbers on the curves are the % CaO on juice (37).

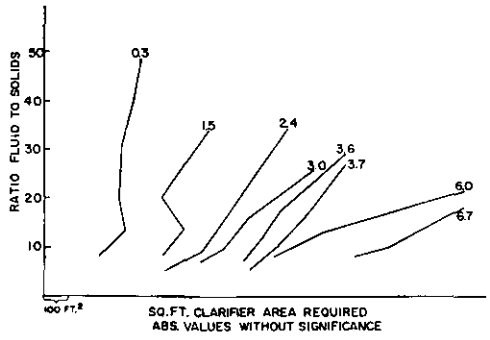


Figure 8-36. The graphs of Figure 8-35 displaced along the X-axis to show the change of curve shape with the amount of saccharate milk (37).

cosity with the density. If the volume flow of diffusion juice is kept constant as its density is increased, larger settling capacity is required. However, if the amount of dry substance entering carbonation in the diffusion juice in unit time is unchanged, settling capacity requirements are somewhat less.

5. **Form of Liming Reagent.** Whether the lime is added to the first carbonation in the form of milk of lime, or as saccharate milk, makes a considerable difference in sludge settling rates. Milk of lime, in general, produces a precipitate that is somewhat slower settling, and that does not thicken quite as readily.

6. **Recirculation Ratio.** As has been pointed out (p. 209-210) the proper recirculation ratio in first carbonation is very important. Without recirculation it is not possible to clarify by settling in a unit of reasonable size.

7. **Amount of Feed.** If amounts of first carbonation juice and mud are fed to the clarifier which are considerably in excess of those it was designed to handle, there is some evidence to indicate that eddy currents within the trays may exert an effect tending to interfere with the settling and thickening. This may add to the extra load already imposed by the increased amount of precipitate to be thickened and settled.

Effect of Dorr Clarification on the Juice

To prevent degradation of the thin juice, the overflow from the Dorr clarifier must be reasonably clear. If it is not, it must be submitted to an auxiliary pressure filtration. Actually, appreciable amounts of precipitate can be tolerated in the overflow without causing noticeable degradation of the thin juice. The process was designed to yield an overflow containing not more than 0.0025% of dry mud. Experiments have shown that at least ten times that quantity can normally be tolerated without producing any noticeable degradation. A better juice may frequently result from operating with a very slight degree of turbidity and a high first carbonation alkalinity, rather than a lower alkalinity and a perfectly clear overflow.

As would be expected, prolonged retention time in the clarifier is damaging to juice quality. A number of investigations have shown clearly that this prolonged storage at low density, high alkalinity and high temperature decreases the effectiveness of the carbonation process. Figure 8-37 shows the results of some tests made on California juices with saccharate defecation. These show an average increase per hour, in color, of about 7% and of about 0.010 in lime salts. Degradation decreases as the first carbonation alkalinity becomes higher, and appears to be less with milk of lime than with saccharate milk defecation.

H. Elliot (19) concluded from plant tests conducted in a first carbonation clarifier in which detentions could be reduced by 59.5% of maximum that "reduction of (detention) time in itself was not the solution to the formation of color." In reviewing the factors influencing color formation in the Dorr system, Elliot concludes, "assuming consistent lime addition, the final alkalinity has more bearing on the color of the juice than any other single factor." Experimental work has shown that the higher the alkalinity, up to a certain point, the better the quality of thin juice produced, as judged by color, lime salts, and colloidal content. At 80 °C the optimum alkalinity was found to be near 0.130% CaO on juice for saccharate milk defecation, and between 0.100 and 0.120% CaO on juice for milk of lime defecation.

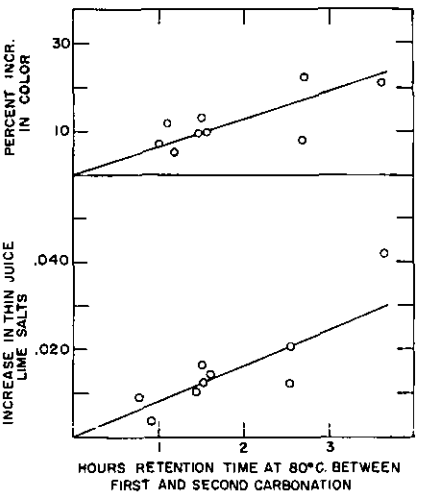


Figure 8-37. Effect of time in clarifier on thin juice quality (60). *(Reprinted by permission of the American Chemical Society)*

B.M.A. Clarifiers

These units, a development of the earlier-appearing Dorr clarifiers, were devised by Braunschweigische Maschinenbauanstalt, and are coming into increasing use in North America. Like the Dorr and its brother descendants they are multiple-tray units and have many points of similarity, but there are important differences.

B.M.A. clarifiers basically consist of a number of settling compartments stacked one over the other; but unlike the Dorr assembly the trays are not interconnected by a central well. Each compartment is a completely discrete unit. Figures 8-38 and 8-39 show the construction.

Each individual tray contains an inner, centrally located truncated cone, inverted, the top of which is fastened and sealed to the roof of the tray compartment. First carbonation juice or other feed material is fed in over a multiple weir which splits the total flow between the individual trays. The feed enters the inner cone tangentially, keeping the "swirling" motion away from the outer settling chamber. After such "cyclone-type action" the preconcentrated juice-mud mixture is discharged through the bottom of the inner, truncated cone into the outer chamber for further

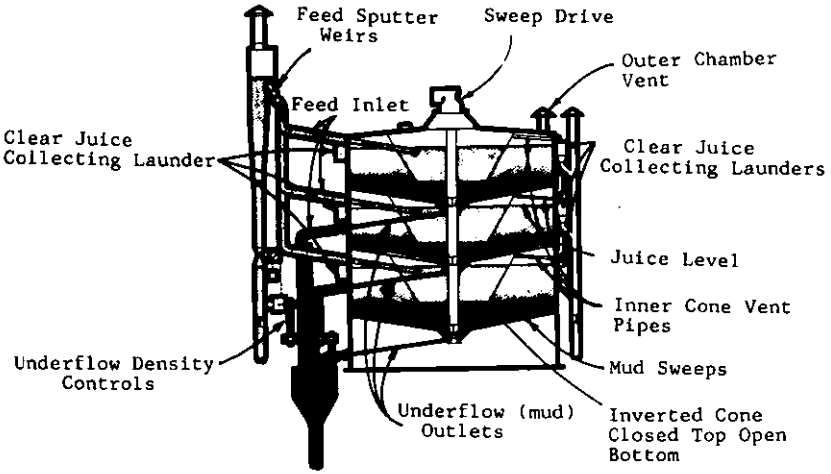


Figure 8-38. B.M.A. clarifier, with three trays, and automatic underflow density control. *(Courtesy, Silver Eng. Works)*

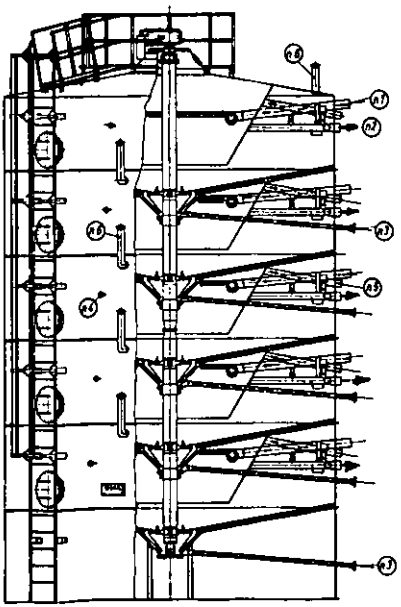


Figure 8-39. B.M.A. clarifier, with five trays, showing details of venting and mechanical construction. 1) Feed line to clarifier; 2) clear juice outlet; 3) underflow outlet; 4) clear juice sampling line; 5) venting of inner chamber; 6) venting of outer chamber. *(Courtesy, Silver Eng. Works)*

settling until the clear juice leaves the individual tray via clear juice collecting launder. This semi-laminar flow arrangement avoids turbulence which might interfere with the sedimentation. As the juice travels out of the open inner cone bottom, the sludge goes to the bottom of the compartment, where it is guided by moving "wipers" or "sweeps" into the central underflow discharge. The partially clarified juice continues its rotary path up the outer compartment of the tray. In its path upward it meets descending particles of sludge which may act to "filter" the juice of inert particles such as field dirt.

An important difference from the Dorr clarifier is that the B.M.A. settling compartments are not filled with pickets and other moving items which might interfere with laminar flow, and are readily broken by encountering any solid item which may have accidentally entered the clarifier.

In a later design clear juice leaves by an outlet header (see Figure 8-40). Skimming devices prevent any floating solids from leaving with the clear juice. The thickened sludge is not pumped out, but leaves by gravity, thus avoiding breaking up flocculated agglomerates. The rate of sludge removal can be controlled by automatic valves activated by hydrometers in the underflow-discharge piping, which can maintain a desired underflow density over a wide range. However, the installations in North America do not use this control. It is rather easy to check the performance of the clarifier in a different way rather than by automatic valves: A small pipeline (see Figure 8-39) located about 8 inches under the clear juice overflow level, runs to the control station, where it passes through a back-illuminated sight-glass tube. If the upper portion of the juice in the compartment becomes slightly turbid, the light behind the sight-glass appears to become extinguished, and the operator remedies the approaching difficulty manually by slightly opening up the appropriate underflow discharge control valve. This advance warning of 8" level difference gives the operator ample time to take corrective action and without foul-up of the clear juice overflow.

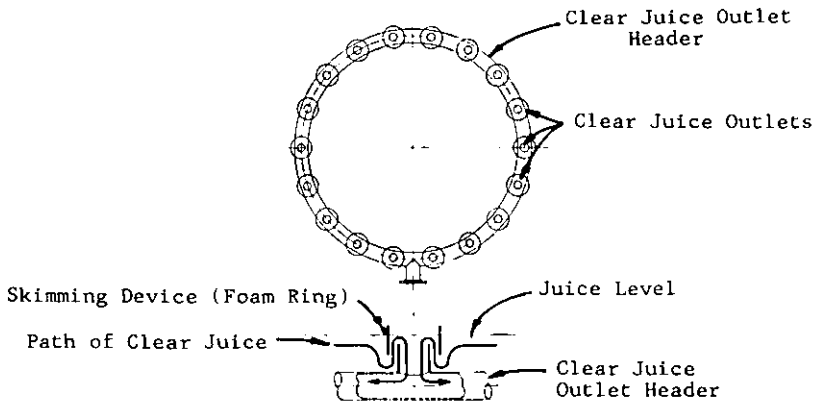


Figure 8-40. Detail of clear juice removal ring of B.M.A. clarifier.

If the rotary vacuum filters are physically located below the clarifier sludge outlet, the absence of sludge pumps permits feeding them by gravity alone, and better sludge filtration results from the large flocs, which have not been broken up by pumping.

A major advantage of the B.M.A. clarifier is that in case an operating speedup or slowdown becomes necessary, one or more of the compartments can be drained and taken out of service, or filled and put in service. This avoids fluctuations in juice quality due to variations in clarifier detention times. The normal detention time is about 40 minutes. Settling aids may be employed by the operating company, if desired.

If the slice rate is to be lowered, the removal procedure to avoid lengthening the detention time is to discontinue the use of the top tray first, or to take any other tray out of service, and run some water into the inactive compartment so that the wipers do not run dry. Any tray can be emptied without any physical problems such as possible structural collapse, as is the case with other units.

References may be made to Figure 8-39 for venting and other mechanical details.

In summary, the B.M.A. clarifier has the advantages that: a) the juice is partially filtered through a layer of sludge; b) if the clarifier is located above the rotary filters, no sludge pumps are necesary, and sludge filtration will be easier; and c) in case of factory variations in operating rates, tray compartments may be put into or taken out of service, avoiding widely varying clarifier detention times.

Laboratory Thickening Tests for Tray-Type Clarifiers

Two batch settling-test procedures have been used, that of Coe and Clevenger (10) and that of Kynch (32).

The Coe and Clevenger test has proven generally successful, in that its use in the last 40-50 years has usually resulted in installation of clarifiers of adequate size. It has two deficiencies, however, the reslurrying of the samples tends to change the physical characteristics of the suspended particles, and it is a complicated procedure for field use. The Kynch batch test is now known to be basically unreliable, so no procedure is given for its use.

Coe-Clevenger test

The determination of the settling curve type of the first carbonation juice is of considerable use in practical operation. The Coe-Clevenger test involves the techniques used. Simple observation of the rate of fall of the mud-liquid interface in a cylinder gives data valid only for the particular dilution of mud and juice, and thus measures the settling rate of the feed mixture only, or in the superior parts of the settling trays. The Coe-Clevenger test measures the rates at a number of dilutions, and by its use a) comparisons may be made of the settling characteristics of various clarifier feeds; b) the clarifier area required to clarify a given feed material may be calculated for a given amount of solids and a fixed ratio of fluid to solids in the underflow; c) the underflow mud ratio to give satisfactory clarification under fixed conditions of clarifier area and amounts of solids may be calculated; and d) the amount of suspended solids that can be settled satisfactorily by the clarifier under fixed area and discharge ratio may be calculated. The method cannot be applied when compression of the mud occurs, but this seldom enters into processing of first carbonation muds (11a,29a). The test as described assumes a sharp "break" in the

settling of the original feed mixture. If this is not so, the procedure must be extended to test mixtures between the consistencies of the clear overflow and the feed.

As mentioned above, in any vessel used for settling, there will be found mixtures of sludge and juice of every consistency between clear overflow and that of the feed and of the discharge, although some of the layers may be infinitely shallow (10). The maximum capacity of the clarifier may be expressed by:

$$C = WR/(F-G)$$

where C is the capacity, in lbs solids per sq ft per hr; W is the weight of clear juice in lbs per cu ft; F is the weight ratio of fluid (clear liquid) to suspended solids in the layer considered; G is the weight ratio of fluid to solids in the underflow discharge; and R is the rate of settling of the mud particles in the mixture of consistency F, in ft per hr.

The capacity of the clarifier for any mixture will be the smallest value of C obtained by applying this formula to a series of tests made on mixtures of mud and juice varying in consistency to cover the range desired.

Test Procedure. A one-liter graduated cylinder of the mixtures is placed within a constant-temperature bath, which has a glass window for observation. An opening in the top of the bath permits stirring and withdrawal of liquid by a suction tube without opening the door. Control of the temperature to ±2 °C is necessary to minimize convection currents which disturb settling. For the same reason, the liquid medium of the constant temperature bath may not be in contact with the test cylinder—fewer convection currents occur if an air space intervenes. The mixture is stirred thoroughly with a large rubber stopper on the end of a glass rod. After the solids have settled about 1/8-inch, the precise level is noted, and an interval timer is started. At the end of, say, 2 minutes, the height of the solids-liquid interface is again noted, and a measured quantity of clear solution is withdrawn from the cylinder. The remaining material is again stirred, and the procedure repeated. A schedule which has been found satisfactory for about 3.5 percent lime addition on beets, involves successive withdrawals of 400, 100, 50, 50, 40, 30, 30, and 25 ml portions. This schedule is changed to meet particular conditions. If the curve is of type A, only four or five test mixtures are necessary—enough to be sure of the type, as in this case the original feed mixture determines the clarifier capacity, providing a sharp mud-liquid interface is obtained. A type B curve may require a greater number of mixtures in the governing region, or the capacity-determining mixture may be missed. Frequently in making tests on unknown materials, a preliminary test is required to determine the proper schedule.

A specific gravity determination is then made with a pycnometer at 20 °C on the original mixture, the contents of the pycnometer are filtered through an asbestos pad in a Gooch crucible, and the solids are dried at 110 °C for 1.5 hours and weighed. The percentage of dry substance in the clear juice is determined with the refractometer.

Further details of the test procedure may be found in the first edition of this book (38a). If chemical settling aids are used, these must be taken into consideration.

C. Water-Soluble Polymers as Settling Aids in the Sugar Factory

Chemical Development*

The main use of water-soluble polymers in the sugar industry is to promote the flocculation and thickening of lime and calcium carbonate sludges. A further use is in the clarification of waste, flume and wash waters. A wide variety of materials has been recommended for this purpose, but only a few are in use. They fall into two classifications: natural, and synthetic.

Naturally-Occurring Agents. Of the naturally-occurring flocculating agents, various carbohydrate materials have been exploited. These include casein from cow's milk (60), algin[1], Irish moss gum, gum arabic, gum karaya (42), starch[2], and lignin (45,46). Various substances chemically derived from natural products are listed as good flocculating agents, among which are carboxymethylcellulose (42), cationic starches[3], oxidized starches (50), cyanoethylated starch (36,67), and collagen derivatives (21).

Probably the most useful for accelerating the thickening of first carbonation sludge has been algin or its sodium salt (38). Algin (Figure 8-41) is a linear polymer of D-mannuronic acid, linked by $\beta-1\rightarrow4$ bonds, with a molecular weight of from 50 000 to about 190 000.

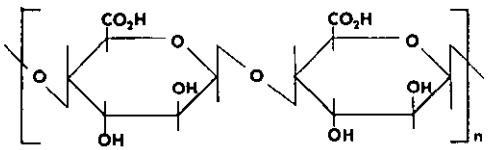


Figure 8-41. Algin, n = 300-1000

Algin was used for many years in sugar factories (25) but is now almost completely supplanted by synthetic materials. The change has been primarily because synthetic agents are more effective, and do not tend to hinder filtration by sliming action when inadvertently used in larger quantities. In uses outside the sugar industry, algin has the disadvantage of being sensitive to bacterial action (65), as are other naturally derived agents. Synthetic materials, produced by carefully-controlled processes, are chemically more uniform, and thus are to be preferred from the standpoint of dependability.

Synthetic Agents. Acrylamide Derivatives. Synthetic organic sedimentation and flocculating agents are all high-molecular weight, water-soluble polymers, produced from low molecular weight monomers. Linear polymers derived from acrylamide, with molecular weights greater than about 300 000, have found the greatest use[4]. Polymerization of acrylamide is normally carried out in aqueous solution by radical initiation (Figure 8-42).

The molecular weights of such products can be controlled from about 30 000 to a maximum of about 10 million. Polyacrylamide can also be prepared by partial hydrolysis of polyacrylonitrile (Figure 8-43). Further hydrolysis of polyacrylamide

[1]See refs. (23, 25, 42, 65)
[2]See refs. (7, 17, 20, 39, 58, 62)
[3]See refs. (9, 44, 51, 59)
[4]See refs. (1, 11, 16, 23, 24, 27, 49, 57)

*by R. M. Sequeira

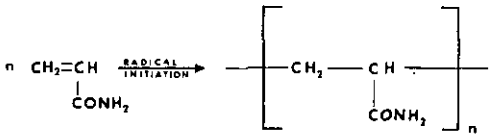


Figure 8-42. Formation of Polyacrylamide

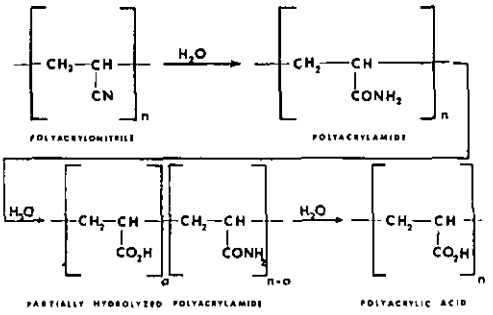
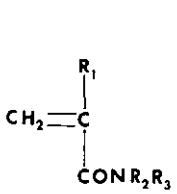


Figure 8-43. Hydrolysis of Polyacrylonitrile

can be carried out to produce partially hydrolyzed material, or, finally, under more vigorous conditions, polyacrylic acid. Saponification of polyacrylonitrile, polyacrylamide, or partially hydrolyzed polyacrylamide with alkali metal hydroxides produces the corresponding carboxylic acid salts. All the hydrolyzed and partially hydrolyzed polymers are water-soluble, and have been mentioned as flocculating agents (2). A wide selection of flocculating and physical properties is obtainable by the partial hydrolysis of polyacrylamide, by controlling the degree of hydrolysis of the amide function and the ratio of amide to carboxyl groups. Further variation of properties is secured by changing the molecular weights used.

Other variations are achieved through copolymerization of acrylic with other monomers. Substituted acrylamides and methacrylamides, which can be copolymerized with acrylamide, to subtly alter certain properties such as solubility, electrophoretic mobility, charge distribution, molecular weight, and other flocculating characteristics are listed in Figure 8-44[1]. Even more materials are potentially available by reactions of individual polymers other than hydrolysis and saponification, but in general, such materials are not commercially available in large quantities (22,55,69).



R_1	R_2	R_3
H	H	H
H	H	CH_3
H	H	CH_2OH
H	H	$CH(CH_3)_2$
H	CH_3CH_2	CH_3CH_2
CH_3	H	H

Figure 8-44. Some Acrylic Comonomers which can be Incorporated into Polyacrylamide Polymers

Other Synthetic Polymers. In contrast to the acrylic polymers, the nonionic polyethylene oxide polymers have been mentioned as useful water-soluble flocculating agents. These are commonly prepared by anionic, cationic, or catalytic

[1]See refs. (4, 6, 8, 30)

polymerization of ethylene oxide (Figure 8-45) and are available with molecular weights up to about 5 million (31,66). Although these polymers are claimed to be useful in the sugar industry, no specific applications have been noted at this time. Polyethylene oxides are somewhat subject to autoxidation in aqueous solutions, and have the further disadvantage that being nonionic, they are less effective than are polyacrylamides in electrostatic coagulation of suspended matter bearing a charge in solutions.

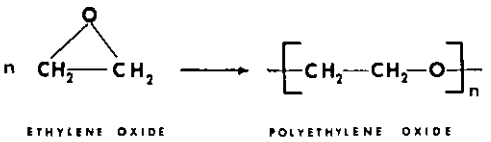


Figure 8-45. Polymerization of Ethylene Oxide

Other synthetic polymers which have been claimed to have utility as flocculants or settling aids are polyethylene imines derived from ethylene imine (aziridine). Polymerization proceeds under acid catalysis as shown in Figure 8-46. The difunctionality of the intermediates involved produces substantial chain branching in the final product, resulting in molecular weights of only about 5000 or less (40,53). Nevertheless, the products are claimed to be useful flocculants, probably due to their high electrostatic charges rather than for viscosity improvement in solution (15). Since higher viscosity, which is related to higher molecular weights, generally improves flocculation, the performance of the polyethylene imines can be raised by increasing the molecular weight through crosslinking reactions with ethylene dichloride or epichlorohydrin. Excessive crosslinking, however, causes insolubility in water due to gel formation.

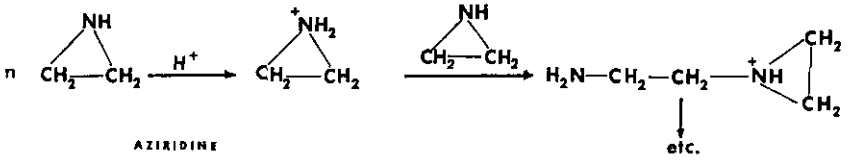


Figure 8-46. Polymerization of Aziridine

Many miscellaneous polymeric materials have been claimed to be of utility in the sugar industry as flocculants. Their practical usefulness has largely yet to be substantiated, however. Among these are included alkylene dichloride-alkylene-polyamine polycondensation polymers and their crosslinked derivatives (54), polyamines (52), vinyl acetate-maleic acid copolymers (29,34), polyampholytes prepared by reacting combinations of copolymers of isobutylene, maleic acid, styrene, methyl maleate, vinyl methyl ether, ethylene, methacrylic acid, and vinyl acetate with dimethylformamide and β-dimethylamino-ethanol (12), condensation products of guanidine and formaldehyde (35), salts of polymerizable acids such as itaconic, ethenesulfonic, and their copolymers with styrene, vinyl esters, vinyl ethers, as well as quaternerized polymers or copolymers of acrylic or methacrylic acid such as β-trimethylammonioethylmethacrylate chloride, vinylammonium chloride, 4-vinyl-*N*-methylpyridinium bromide, vinylpyridinium bromide, or vinyl-pyridinium chloride (56). One interesting polymer claimed in the clarification of cane sugar juice is a liquid polyorganosiloxane of the form $R_nSiO_{(4-n)/2}$ where R is a

univalent alkyl or aryl hydrocarbon and n = 1.99 to 2.01. These polymers are said to aggregate and settle the floc when added before the normal precipitating chemicals (13).

Physics of Flocculation. Basically, flocculation is the agglomeration of small, discrete particles to form larger aggregates. The size of the larger units is such that they are visible to the naked eye and are readily filterable, while the smaller particles are of colloidal or smaller dimensions. In the presence of a settling aid, the smaller entities are held together by macromolecular bridges, forming a loose, three-dimensional network.

The mode of action of a settling aid in promotion of flocculation depends on many factors related to the adsorption of the polymer on the suspended particles. This may take place by chemical bonding or electrostatic attraction. Further, a single, very long-chain linear polymer with high molecular weight may have many adsorption sites and may be able to bind many particles. A typical polymer-solid adsorption system is approximately described by a Langmuir adsorption isotherm as follows (28,33,61):

$$\frac{\Theta}{1-\Theta} = bP \tag{9}$$

where Θ = fraction of adsorption sites on the surface of the particle covered by the polymer, P is the equilibrium concentration of polymer in solution not bound to a particle, and b is an adsorption constant dependent on polymer and particle properties. P is related to the initial, nonequilibrium concentration of polymer, P_o by

$$P = P_o - kw\Theta \tag{10}$$

where k is a constant which depends on the surface area of the particle and the number of adsorption sites per unit area and w is the solids concentration of the suspension. It obviously follows then, that

$$\Theta = \frac{b\,(P_o - kw\Theta)}{1 + b\,(P_o - kw\Theta)} \tag{11}$$

and it is apparent that Θ increases with initial polymer concentration, and with decreasing numbers of particles in suspension.

By a detailed consideration of the rate of floc formation and the rate of floc destruction by desorption, the equilibrium radius of the floc particles (R_e) is shown to be

$$R_e = \frac{k_1}{k_2} N_o^2 \Theta^2 (1-\Theta)^2 \tag{12}$$

where k_1 and k_2 are rate constants related to the formation and decomposition of flocs, and N_o is the (equilibrium) number of solid particles per unit volume. Under these conditions, the extent of flocculation is at a maximum when $\Theta \approx 0.5$. In practical situations, however, a true equilibrium is not necessarily attained, since flocculation rates are also dependent on other factors such as the mode of addition of the flocculant, the degree of agitation, the ratio of particle size to polymer length and molecular weight and size, number and size of adsorption sites, and many other variables.

In colloidal systems, the organic particles in suspension are most often negatively charged. With respect to first carbonation juice, however, the preponderance of a positively-charged inorganic colloidal dispersion of calcium carbonate has a strong influence on the nature of the overall electrostatic character of the colloid. Those polymers, which themselves are positively charged in solution and which have been mentioned as useful in juice flocculation, such as polyacrylamide, guar gum, starch derivatives, and casein, probably react with the organic portion of the colloid to cause flocculation. It may be that anionic types, such as partially hydrolyzed polyacrylamides—Separan, for example—are more effective since they can operate directly on the positively charged calcium carbonate particles.

Considering the complexity of the colloidal system obtained during juice purification, it is likely that successful flocculating agents operate through a combination of mechanisms, such as viscosity effects, hydrogen bonding, metal-ion bridging, and stereochemical entanglement. The viscosity and solution structure of polymers has been found to exert a great influence in formation of floc networks. For instance, polyacrylamide and galactomannan, which is related to algin, have been found to form similarly ordered fibrous structures on colloidal surfaces when examined by electron microscopy (5). Their relative flocculating abilities are strongly correlated with their high intrinsic viscosities and fibrous structure in solutions. An excellent discussion, describing the important chemical and physical mechanisms involved in flocculation has been given by *Attia and Fuerstenau (3a)*.

Use of Flocculating Agents in Clarifiers*

Sedimentation type clarifiers have been in use in the beet-sugar industry for many years for the continuous removal of suspended solids in first carbonation juice (60,68). Many efforts have been made to increase the capacity of existing units.

Although thin juice quality is improved by use of high alkalinity in first carbonation, settling rates of suspended solids in first carbonation juice are better at low alkalinities. Since the clarification operation is often a limiting factor in plant operation, it may be necessary to sacrifice juice quality to achieve increased production.

Since it is advantageous to accelerate settling rates to increase plant capacity and enable use of higher alkalinity in first carbonation, the use of settling aids can be economically justified. The use of algin, which was patented by Moore (42) in 1954, increased settling rates of suspended solids in first carbonation juice by a factor of 2 to 3 and resulted in a much clearer settler overflow. Filtration of underflow was also improved. When settler area was a limiting factor in plant operation, the use of algin was estimated to increase factory capacity up to as much as 50%.

Further improvement in settler capacity was achieved by the advent of synthetic settling aids such as Separan AP30 (43,63). The polyacrylamide type of settling aid was about 3 to 4 times as effective as algin for settling of solids in first carbonation juice. The criteria for a good settling aid are 1) retains effectiveness over long periods of time at high temperatures and alkalinities, 2) no interference with filtration of underflow, 3) no interference with product, 4) produces a favorable ratio of overflow to underflow, 5) produces clear overflow.

*by O. V. Bonney and F. G. Eis

Modern settling aids of the Separan AP30 type met these requirements. The synthetic polyacrylamide type of settling aids have been improved and the newer types may be more than twice as effective as the earlier synthetic products. Normally 2 to 3 ppm results in good coagulation (27,48) of solids but at times higher levels are desirable. The maximum amount which may be used is limited by Food and Drug Administration regulations (3).

For the most effective use of settling aids the compound must be properly prepared in a stock solution and diluted prior to addition to settler feed. The settling aid should be added to the settler feed at the correct place. A typical installation is illustrated in Figure 8-47.

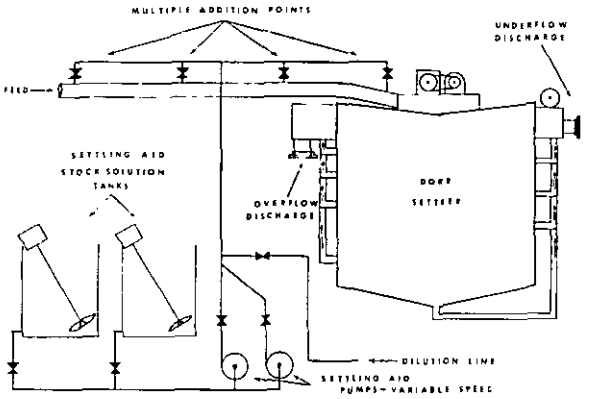


Figure 8-47. Installation for Addition of Settling Aid

Manufacturers specify optimum concentrations of stock solutions, the temperature of water used in preparing the solutions, and the dilutions required. Stock solutions of 0.3% by weight are normally prepared using water at not over 60 °C. The stock solution should be allowed to age for a period of time before use and is then diluted to about 0.05%, prior to addition to settler feed. The point of addition should be considered. For heat economy the dilution liquid should be clarified juice rather than water. A point of addition too close to the settler may result in inadequate mixing and poor coagulation of solids while addition too far from the settler may result in a breakdown of the coagulated particles in the pipeline ahead of the settler. Multiple addition points may be used with good results. Inadequate dilution of settling aid may result in loss of effectiveness by poor dispersion in the feed.

Since efficient operation of the Dorr type clarifier may depend on the proper and continuous addition of settling aid, the installation and equipment must be reliable. A system with dual stock solution preparation tanks, dual calibrated stock solution metering pumps, and flow meters and alarms for various streams and tank levels should be provided.

New Type of Clarifier. A recently developed method and apparatus for separating solids from liquids, marketed by the Enviro-Clear Division of Amstar Corp., utilizes the full potential of modern settling aids (18). A 14 ft diam. single-

tray unit of the new design replaced a 26 ft diam. 4-tray Dorr at the Woodland factory of the Spreckels Sugar Company early in 1969.

In this process, settling aid is added to the feed ahead of the settler inlet to promote initial coagulation of at least part of the suspended solids. Additional agglomeration occurs within the clarifier to produce a rapid-settling and easily-removable, dense underflow. A schematic flow diagram of the process is shown in Figure 8-48.

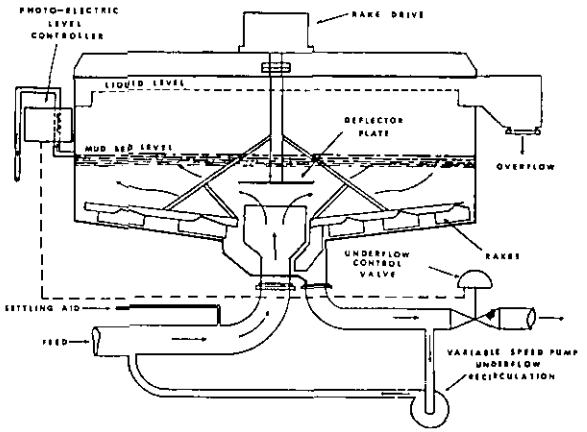


Figure 8-48. Diagram of Settler Marketed by Enviro-Clear and Installed at Woodland, California

The settler depicted is of the bottom entry type. In other, top-entry models, there is a horizontal feed pipe near the top of the settler. This pipe, which enters through the side of the tank shell, extends into a central downcomer arrangement. A deflector plate is located just below the bottom of the downcomer.

The incoming feed, to which settling aid has been added, enters the clarifier through the feed pipe and into the centrally located feed well. The vertical flow of juice is diverted to a horizontal plane by a deflector plate opposite the outlet of the central feed well. The feed is dispersed into a settling bed of previously agglomerated solids and the juice then percolates upward through the sludge bed.

The pattern of flow imparts a gentle rolling action to the sludge bed and provides additional solids contact for further agglomeration of suspended particles and fines. As the particles grow and become denser, they settle at faster rates. The sludge bed has an increasing solids content from the top to the bottom.

The sludge bed level is controlled a short distance above the deflector plate by regulating the underflow withdrawal. A minimum distance is maintained through which solids must settle. The free settling zone, often rate controlling, is avoided by introducing the feed into a zone of compaction or hindered settling. A distinct interface occurs between the sludge bed and the clarified liquor. Maintaining the sludge bed a short distance above the feed distribution point allows a filtering action as the liquid percolates upward through the sludge bed. Conventional sludge rakes aid in sludge compaction while moving sludge to a central mud boot at the bottom of the settler for withdrawal.

Clarified juice is collected at the top of the settler by a launder, and routed to second carbonation in the usual manner.

The sludge bed level may be automatically controlled by appropriate instrumentation. Flow-through cells, sonic interface sensors and float type sensors may be used for this purpose. With flow-through cells, liquid at the sludge level interface is drawn through a transparent cell. The change in light transmission through the cell as the liquid becomes turbid because of the presence of suspended solids is used as a basis to regulate underflow withdrawal equipment. Control systems have also been developed for automatic control of settling aid addition and other variables to optimize settler operation.

Sonic interface sensors utilize high frequency sound. When changes in sludge bed level cause the sensor to be in or out of the sludge, a change in sound transmission occurs, which results in generation of a signal to operate the underflow withdrawal equipment.

A continuous indication of sludge bed level is obtained by use of a float which moves up and down with the sludge bed interface. The specific gravity of the float is adjustable. A varying signal is produced as the float rises or falls to result in an analog output proportional to the float position.

In special cases it may be advantageous to recirculate a portion of the underflow to the settler feed. This technique may be used during short shutdown periods to avoid plugging of the underflow line if the settler is not emptied. During Steffen campaigns, when the settler feed rate may be relatively low and the underflow more dense, recirculation may aid in minimizing the possibility of plugging settler pipe lines. In other applications, such as beet flume water clarification, recirculation may be useful.

The new process maintains short distances for particles to settle, thus allowing a minimum height requirement for clarified liquid. Flow rates of over 5 gpm/sq ft of settler area are readily achieved with retention times of less than 5 minutes. A sludge bed depth of about 2.5 feet allows adequate thickening while a total operating depth of about 4.5 feet allows adequate clarification in a 17 foot diameter settler. Retention times of over 90 minutes are common in conventional sedimentation type settlers.

The short juice retention time minimizes juice degradation. Color and lime salts formation and sugar loss associated with long retention time at high alkalinity and temperature are avoided. The small volumes involved and simplicity of the new type settler also reduce start-up and shutdown times.

Since the introduction, about 1970, to the beet sugar industry, the Enviro-Clear clarifier/thickener has found world-wide acceptance in several other areas, including minerals and coal preparation. Presently, there are 28 units installed in beet sugar factories in the United States, Canada, and Europe ranging in size from 10 feet to 23 feet in diameter. In all, there are over 200 units in operation world-wide ranging in size up to 85 feet in diameter.

Experiences in the use of the Enviro-Clear are described in various publications (4a, 8a, 38b, 56a).

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Chapter IX

JUICE PURIFICATION III

A. Second Carbonation*

The predefecation pH, the amount and manner of reagent lime addition, the detention times, and the carbonation end point alkalinity, all influence the degree of precipitation, adsorption, and removal of several of the raw juice impurities in first carbonation.

With the separation of these precipitated solids and of the impurities adsorbed thereon, a new ionic equilibrium can then be established favorable for additional deliming and impurity removal through a second carbonation and a second solids separation.

For minimizing sucrose destruction at the elevated temperatures of the following evaporator and crystallizing systems and for precipitate filterability, an adjustment of the remaining ionic concentrations and a modification of some of the remaining molecular constituents may be necessary. A pre-second carbonation treatment with a previously precipitated calcium carbonate or a delayed main liming for an adsorptive carbonation, a concurrent addition of alkali cations or a post-carbonation pH reduction by sulfitation are additional considerations of the final beet-end processes.

Basic Considerations in Second Carbonation*

Second carbonation is the second stage of the deliming, with the purpose of removing all the lime possible from the clear first carbonation juice (24,34). This unit process is remarkable for its simplicity of purpose and complexity of chemistry. For a discussion of the basic physical chemistry, please see pp. 176-179.

Several of these chemical formulas, Eq. 16 through 20, are statements of reaction equilibrium. The nonequilibrium dynamics of the on-going, continuously reacting system of the plant process, yield a still supersaturated limed juice, the second carbonation filtrate. Reduction of both the quantity of residual lime and its supersaturation is the basic objective of the total second carbonation treatment (2,38a,38b).

If the lime content of the thin juices is not reduced to a minimum, there may be scale formation in evaporators and vacuum pans, turbidity in solutions of white sugar due to lime salts, and increased loss of sugar in excess molasses formed.

First carbonated clear juice is a rather complex molecular and ionic "soup" of variable composition. The equilibrium constants must be related to their total chemical environment. In the sugar juices of the factory process, the ionic concentrations are not always dilute and many interfering substances are present (1,3,6,17a). Some are simply spectator ions and have little or no influence, while for other ions, particularly nonprecipitating carbonated cations, the common ion ($CO_3^=$) effect is substantial (20a).

The pH and ionic environment established prior to and during first carbonation will have a significant influence upon the effectiveness of impurity removal in

*by R.A. McGinnis; revised by G.W. Cossairt

this, the first precipitation and deliming treatment. Some impurities are completely precipitated at a high alkalinity end point. Others are poorly removed at high alkalinity but are very effectively removed at a lower pH. The process design must be for a high first carbonation final alkalinity, consistent with acceptable settling and filtration rates for a *clear* first carbonation filtrate. The high alkalinity reactions can thus be optimized. This same elevated pH causes more Ca^{++} and OH^- ions to remain dissolved in the clear juice. Fortunately, at a reduced pH, these Ca^{++} ions, in the presence of added CO_3^{--} ions, can be reduced to an optimum low concentration. The total process design is thus made most effective by the two successive and different ionic equilibrium conditions, both of which are optimized at their respective pH levels.

Natural Alkalinity. Assuming no supersaturation of calcium carbonate, the alkalinity of treated juice from which the removal of dissolved lime salts has been completed through vigorous liming and carbonation, is the *natural alkalinity* (19). The OH^- ions making up this alkalinity are ionically balanced against K^+ and Na^+ ions. Other definitions of natural alkalinity: a) it is equivalent to the quantity of lime-precipitable acids, less the lime-nonprecipitable acids, in the diffusion juice; b) it is approximately equal to the equivalents of potassium and sodium carbonates remaining in the thin juice; c) it is equivalent to the potassium and sodium salts in the diffusion juice which have anions precipitable by lime.

In degraded beets (34) there are excessive amounts of acids not precipitable by lime, and their soluble lime salts remain in the finished thin juice as neutral salts. In order to have sufficient thin juice alkalinity to prevent the crystallizing materials from becoming acid, it is then necessary to leave some free lime, with the usual consequences of high lime salts. Therefore it is very desirable to maintain the natural alkalinity against depressing influences.

Substances which are acidic decrease the natural alkalinity. These include (26): a) amides, such as glutamine and asparagine, which form acids with soluble lime salts and evolve ammonia; b) invert sugars, which on destruction in liming form coloring matter and acids with soluble lime salts—mostly lactic; c) calcium and magnesium naturally occurring in the beets, which, on precipitation take hydroxyl groups or their equivalent with them; and d) amino acids, which have soluble lime salts.

Substances which increase the natural alkalinity are: a) compounds with precipitable lime salts, such as oxalates, phosphates, some pectic substances, proteins, citrates, malates, and sulfates; b) alkaline chemicals which are added to the juice, such as soda ash, trisodium phosphate, and caustic soda.

The concept of *alkali reserve* has been introduced (30), which is the difference between the equivalents of alkalinity increasers and alkalinity decreasers. If a juice has adequate alkali reserve, it assures low lime salts, and minimizes pH lowering in the following processing. The natural alkalinity of the diffusion juice can be maintained by preventing fermentations in beet-end juices which produce acids not precipitable by lime, such as lactic, and the lower aliphatic acids; and which invert sucrose, to eventually form more lime-nonprecipitable acids.

A factory incorporating the Steffen process has thin juices with greatly depressed—usually negative—natural alkalinity. Acids of low molecular weight are produced by the decomposition of lime-precipitated substances under the drastic conditions of pH and temperature in the Steffen process, and these reenter carbonation adsorbed to the lime saccharates in the saccharate milk. Beets grown in dry seasons also yield juices with lowered natural alkalinities (31).

The natural alkalinity is customarily determined by subtracting the lime salts content of the juice, as determined by analysis, from its titrated alkalinity to a phenolphthalein end point of 8 pH, the results of both being expressed in % CaO.

Optimum Alkalinity. This is the actual second carbonation alkalinity at which there is a practical minimum of lime salts in the finished juice. This differs from the natural alkalinity, which ignores the presence of bicarbonates, and the effects of ionic strength and of buffering materials. Theoretically, it has been pointed out (32), the optimum alkalinity should be about half the natural alkalinity, and the optimum alkalinity can be determined by titrating the juice to the bicarbonate end point.

The optimum alkalinity differs for juices of different chemical composition, and must be especially determined. At the optimum, there is much more HCO_3^- than OH^- present. This is illustrated by the following data of Dedek (16):

Table 9-1

pH	% CO_2 as $CO_3^=$	pH	% CO_2 as $CO_3^=$
13	100	10	37.0
12	98.5	9	5.6
11	86	8	0.6

At each pH, of course, essentially all of the remaining CO_2 derivatives are in the form of HCO_3^-. From examination of these data, it is apparent that for the best deliming, or the highest ($CO_3^=$), the second carbonation end point should be as alkaline as possible, but the excess alkalinity should be natural alkalinity and not lime alkalinity.

Determination of Optimum Alkalinity. The optimum alkalinity should be redetermined when the type of beets sliced or the molasses worked changes. Following are methods for its determination (29).

1) The standard method is to carbonate a container of clear first carbonation juice in the laboratory, withdrawing samples at frequent intervals, which are analyzed for alkalinity and lime salts. From the results, the alkalinity for lowest lime salts can easily be determined (34).

2) A container of clear first carbonation juice is gassed to a well over-carbonated condition, and then vigorously boiled for several minutes to decompose the bicarbonates. The alkalinity of the final juice is the optimum providing that the original juice was thermostable.

3) Methods are in use in Europe, using as reagents barium or calcium chloride (5,35,39).

4) A double titration with two indicators is made to determine the hydroxyl and carbonate alkalinities. The method is not exact.

5) Schneider (27) has published an automated method using repeated titrations for lime salts with Complexon, a lime-chelating reagent.

6) If sufficiently frequent determinations of second carbonation alkalinity and thin juice lime salts are made for factory control, an inspection of each will indicate the approximate optimum. This method is used extensively in the United States.

7) The classic method is through correlation with the natural alkalinity.

Effective Alkalinity. As a result of previously-mentioned studies of Brieghel-Müller and Brüniche-Olsen (2,3,6,18) it has been found that an *effective alkalinity* which involves titration to an end point of 9.25 pH instead of to 8 pH, is more closely associated with the optimum alkalinity. The former pH is much closer to the normal end point of second carbonation, and does not include the titration of a number of buffer substances, present in variable quantity, which are not usually involved in second carbonation reactions. The effective alkalinity, in % CaO, is obtained by subtracting the ml of soap solution used, or equivalent of chelating reagent, from the ml of *N*/28 H_2SO_4 involved in titrating the filtered first carbonation juice to 9.25 pH. If negative values are obtained, the values afford a rational and accurate measure of the amount of soda ash required to maintain any desired value of effective alkalinity. Figure 9-1 shows the relations between effective and optimum alkalinity.

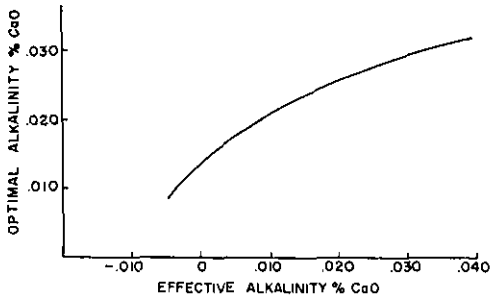

Figure 9-1. Relation between "effective alkalinity" and optimum alkalinity. *(Brüniche-Olsen, 1947)*

The effective alkalinity is, then, a more accurate measure of the deliming ability of a juice than the natural alkalinity, and when one wishes to calculate the amount of soda ash to add for proper deliming, one can use equimolal quantities, instead of using 1.5 to 2.0× molar quantities.

In many areas of North America the beets are of such a nature that natural alkalinities are normally very low, and in all Steffen factories the effective alkalinity is commonly strongly negative. In such cases, addition of enough soda ash to restore a good alkalinity is too expensive to be considered, and it is standard practice to add soda ash, not in the indicated amounts, but in quantities which can be afforded, considering the cost of the chemical and of the extra molasses produced. To this majority of factories, both natural and effective alkalinities are only of academic interest.

Practical Process Alkalinity. Optimum alkalinity, as determined for juices from second carbonation, may not always be the optimum for the balance of the process (8,38a).

Where the natural alkalinity is of moderate value and the juices are reasonably thermostable, the process objective should be the attainment of optimum alkalinity, Figure 9-2. In this instance, it is important that the optimum alkalinity be maintained with as little deviation as possible. If the alkalinity is too low or too high, by definition the lime salts will be too high. Variations on the low pH side are particu-

larly undesirable, since then the excess lime is in the form of bicarbonate, and even already precipitated calcium carbonate can be redissolved. The resultant bicarbonate will decompose to form scale in the high temperature end of the evaporators. Even a very short period of overcarbonation will show up as scale.

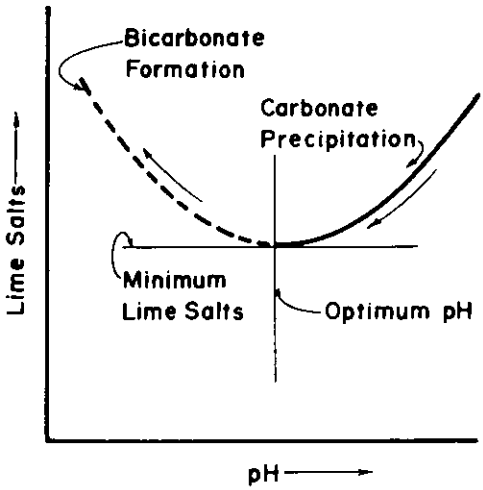

Figure 9-2. Optimum pH.—The pH at minimum lime salts is dependent upon natural alkalinity of the beet. The lime salts at the optimum pH is dependent upon: a) beet quality, b) process quality through diffusion, liming, and first carbonation, and c) subsequently upon temperatures, detention times, agitation, and amount of second liming of second carbonation.

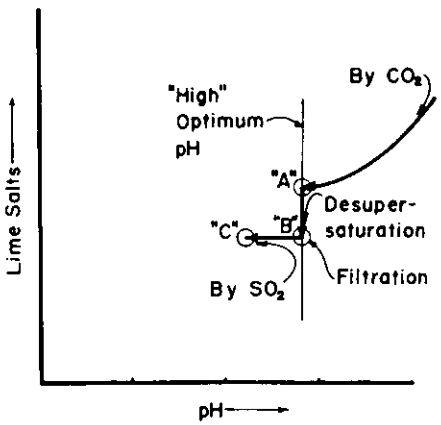

Figure 9-3. Process for High Natural Alkalinity Beets.—Carbonate to optimum pH "A," desupersaturate to "B," filter, then reduce pH of clear filtrate to "C" by sulfitation. pH "C" is selected to yield acceptable pH in evaporators and through white pans with respect to sugar inversion and to saponin suppression.

If the natural alkalinity is high, the pH of the optimum alkalinity is also high, Figure 9-3. This may be too alkaline for good crystallization, which, in turn, is best performed at pH values a little on the alkaline side of neutrality (adjusted for pan boiling and the crystallizing temperatures). In such a case, the objective would be for careful optimum pH control for gassing followed by a pH reduction by sulfitation of the clear thin juice.

If the natural alkalinity is, comparatively, only a little low, the optimum alkalinity may also be only a little low. One might then expect the possibility of mildly acidic crystallizing syrups and massecuites, and of saponins in the sugar. In these cases, the second carbonation pH controller is set to yield an alkalinity somewhat above the optimum and the penalties of high lime salts in the evaporators

would be countered as well as possible. This condition obviously restricts the use of sulfitation for color inhibition purposes. It also establishes the need for an alkalizing agent after the evaporators. Such added alkali (often milk of lime) may be added to the sugar-end boiling systems or crystallizers only as dictated by the sugar-end pH profile, and, preferably as late in the system as possible.

If the natural alkalinity is low and particularly if the beet-end juices are not thermostable, the alternative is to add sodium ion (28) to the thin juice in the form of caustic soda (7) or soda ash to the gassing tank of second carbonation (Figure 9-4).

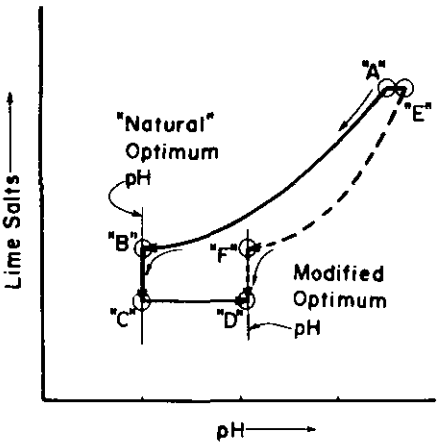

Figure 9-4. Process Options for Low Natural Alkalinity.—Gassing of first carbonation clear juice "A" to its optimum pH "B" would yield second carbonation clear juice pH's too low for evaporators and pans. Addition of soda ash at "A-E" would yield a higher, modified optimum pH "F" for improved thermostability in evaporators. Similar results are obtained by carbonating "A" to "B," desupersaturating "B" to "C," and adding caustic soda "C" to "D" to the second carbonation clear juice.

Estimates of sugar decomposition in the evaporator and sugar-end systems indicate the need for a $pH_{120°} = 7.4$ or greater, where $pH_{120°}$ is the condition existing at a first effect boiling temperature of 120 °C (38a). With sound beets of moderate natural alkalinity, this optimal $pH_{120°}$ for evaporation may correspond to the $pH_{120°}$ of optimum alkalinity for second carbonation. Adjustment of second carbonation actual alkalinity or pH may require sulfitation or alkali addition to assure $pH_{120°} \geq 7.4$.

Equipment for Second Carbonation

The essential elements of a second carbonation *system* include a juice heater, a gassing tank, a detention tank, and a means of separation and removal of the precipitated lime and absorbed impurities. A wide variety of systems has been reported as giving reasonably satisfactory service and that equipment specifications are not critical (20b).

Unclean heat transfer surfaces in the evaporators are significantly related to the quality of first carbonation elimination of scaling impurities and to the optimal deliming and desupersaturation of second carbonation clear juice. Figure 9-5 illustrates a second carbonation system incorporating several of the technical design requirements to achieve such a reduced evaporator and vacuum pan scaling and in some instances, a reduced conductance ash in the final product sugar (24a).

A heater, C, prior to the carbonation tank, elevates the juice temperature to the recommended 95 to 98 °C.

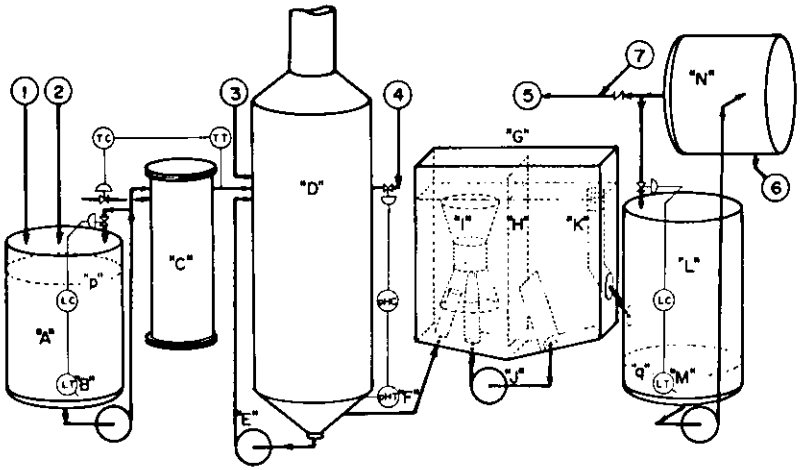

Figure 9-5. Second Carbonation System.—Process flow reactants: 1) First carbonation clear juice, 2) optional milk of lime, 3) optional soda ash solution, 4) kiln gas, 5) clear second carbonation juice, 6) precipitated lime solids, 7) optimal caustic soda solution. See text for process and equipment description.

A gassing tank, D, has a conventional multiple-ring distributor for a reasonably fine bubble dispersion of the CO_2 gas. For effective gas adsorption, the liquid depth, as shown, is in the 13-15 foot range. An axial flow pump, E, provides counter-current recirculation to lessen the possibility of over and undercarbonation and consequently yields a more stable process pH control. The recirculation ratio is in the 5× to 8× range. A detention time of 10 to 15 minutes is required for the precipitate formation reactions.

The detention or "reaction" tank, G, of this system is divided by a curtain partition, H. The first section contains a venturi-shaped, center-well section I, for the rapid and intense mixing of freshly carbonated juice with recirculated, settled precipitate from the second compartment. In the settling section an adjustable overflow weir, K, establishes the level for both the carbonation and detentions. The external recirculation pump, J, operates in the 3× to 6× ratio range. A detention time of 15 to 20 minutes for desupersaturation and adsorption is strongly indicated (12,20,38,38a).

System Control

Temperature. At the turn of the last century, Claassen (14a) reported the desirability of near boiling temperatures for the second carbonation process. Boiling second carbonation would appear to offer a means of "automatic" control of the alkalinity at the optimum, since many lime salts are less soluble hot (15) and since bicarbonates are broken up rapidly (25). Many second carbonation systems were designed and operated on this principle[1]. Carbonation at full boiling is no longer recommended because of the low adsorption of CO_2, and of the very large amounts

[1]See refs. (13,14,17,17a,21,22,23,33,37)

of heat required. Actually the decomposition of bicarbonates is a joint function of the temperature and the area of the liquid-gas interface. There is no magic in the boiling-point temperature.

Various investigators (20b,24a,38a) have confirmed an optimized process in the 92 to 98 °C range. The benefit of these higher temperatures is illustrated in Figure 9-6. The ionic equilibria of the second carbonation reactions are temperature-sensitive. The detention times for precipitate formation and of desupersaturation are temperature-related. Where adsorption carbonation is practiced, the degree of invert destruction, of amide saponification, and of impurity adsorption is time-temperature-related. Heating of the first carbonation clear juice must be adequate and closely controlled.

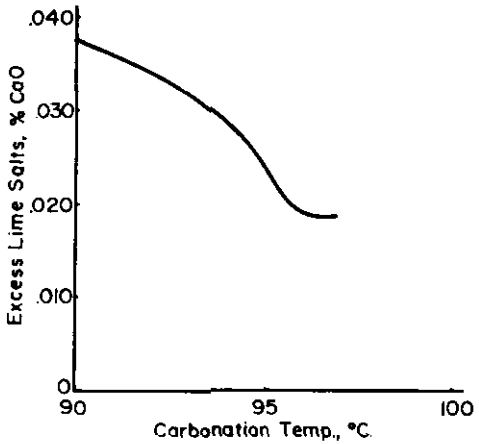

Figure 9-6. Typical effect of temperature during second carbonation on excess lime salts. After Vukov (38a.)

pH Measurement and Control. Several electrode systems are used for pH control. Most are sensitive to carbonate scale coating. Mechanical and ultrasonic cleaning have been employed with varying success. Acid cleaning at scheduled intervals improves the measurement reliability. Self-cleaning antimony electrodes have been reasonably successful.

Whether the objective is optimum alkalinity or a modified higher alkalinity, a system regulation to ±0.1 pH of the selected target is essential for a continuous minimum of excess lime salts in the second carbonation clear juice.

In those growing areas where optimum alkalinity control can be the target, and where there is the need and desire for superior optimizing excellence, flame photometers (41,42), ion-selective electrode systems, or conductivity (4) are among the means of seeking the optimum and then its control.

Solids Separation. The equilibrium solubility equation, Eq. 20, identifies the concentrations of the Ca^{++} and CO_3^{--} ions as the juice leaves the reaction or detention tank. This equilibrium is in the presence of precipitated $CaCO_3$. If the solid phase is removed, then the maximum ionic concentration of Ca^{++} is limited.

A turbid second carbonation filtrate, followed by thin juice sulfitation, results in a partial re-solution of the solid, precipitated calcium carbonate. The scaling potential of the thin juice becomes greater than that of a clear, sulfited juice. A

grossly turbid second carbonation filtrate often becomes a source of filtration difficulty at the standard liquor filters.

A discontinuity of flow or the incorporation of air into the filter feed slurry invariably results in short-term loss of clarity with any pressure filter. The receiver tank, K, Figure 9-5, for carbonated and desupersaturated second carbonation juice, is also the pump tank for the filters. To assure a continuous flow through the filter cake and thus to more reasonably assure a continuously clear final filtrate, filtered juice is returned to the pump tank when its level falls, i.e., just short of vortex and air incorporation into the filter feed pump. The level controller, M, maintains not less than this minimum level q. Provision should also be made to recirculate the first cloudy filtrate as the filter is started up.

Lime Addition to First Carbonation Clear Juice

The addition of a small portion of the total lime, about 0.2-0.25% on beets to second carbonation, has been a fringe, optional accessory to the process for at least 80 years. The practice has been shown to be beneficial at times in completing purification left unfinished in previous steps, particularly with respect to color reduction and saponin removal (36). The variance in the benefit derived has been illustrated by Vukov (38a) in reporting a reduction of minimum lime salts of $38 \pm 17\%$ by a 0.5% CaO addition. The variance in the benefit of lime addition to the first carbonation clear juice is related to the nature of the impurities in the beets as sliced and the purification methods employed prior to and including second carbonation.

Applying the principles of equilibrium, precipitation, colloidal behavior, aging, and interference of foreign substances, as has been carefully developed in advanced studies of analytical methods (20a), the chemistry of second carbonation is still complex but becomes less difficult to understand.

A clear juice being carbonated in a single-pass flow tends toward a homogeneous nucleation, a higher initial supersaturation, and a large population of small crystals of $CaCO_3$. The unsatisfied surface charges on the crystal lattice have a strong affinity for polar, water-solvent molecules. At least temporarily, the water sheath of hydration tends to create a colloidal-type dispersion. The redistribution of surface charges in the $CaCO_3$-H_2O system is time-dependent. The probability of an increased rate of small crystal agglomeration is thereby reduced.

A recirculated juice during carbonation will tend toward heterogeneous nucleation, i.e., growth upon sites already present. A lower supersaturation will prevail. Fewer crystals will form and they will tend to become larger in size. The repeated exposures of the alternating forces of hydration and lattice incorporation provide more opportunity for crystal growth and for small crystals to agglomerate.

Agitation is important in bringing about close proximity of the small particles for agglomeration and for surface area exposure to the still supersaturated "mother liquor." Large size crystals or agglomerates permit practical filtration rates. Large size crystals and a larger-than-normal population of such crystals provide the enhanced solid-surface-area-to-liquid-volume needed for supersaturation reduction and for impurity adsorption.

Continuing investigations have re-enforced the opinion (1,24a,38a,40) that lime addition is desirable: as a) it assists in lowering $CaCO_3$ supersaturation by providing additional crystal surface; b) it adds to the filter cake, and assists in removing fine suspensions; c) Dedek found, at times, a measurable increase in thin juice purities; and d) the purification by adsorption is especially supplemented.

Lime addition in the 0.05-0.2% CaO range has long been found helpful to modify the porosity of the second carbonation filter cake.

A more complete desupersaturation and a stronger impurity adsorption capability is realized with lime additions approaching 0.50% CaO, Figure 9-7. Calcium citrate (Figure 9-8), calcium sulfate, calcium aspartate, saponins, invert destruction products, caramels and melanoidins are among the adsorbed impurities.

While polyphenols and melanins are readily removed in the usual high alkalinity of first carbonation, the results of faulty (low final alkalinity) first carbonation cannot be corrected by a strong second liming and carbonation (38a).

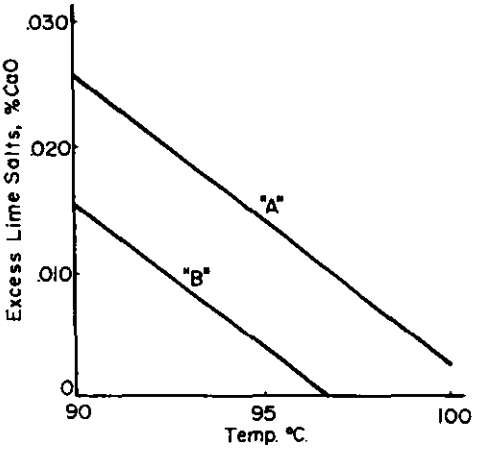

Figure 9-7. Typical effect of temperature and of added CaO upon reduction of supersaturation during postcarbonation reaction period: "A"—without added lime and "B" with 0.3% CaO added lime. After Vukov (38a).

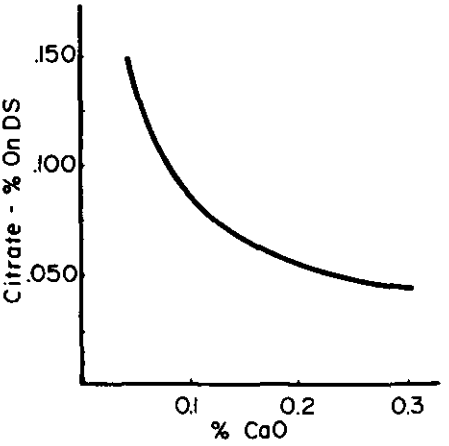

Figure 9-8. Typical effect of added CaO on citrate ion residual in second carbonation clear juice. After Schiweck (24a).

Oldfield, et al., (20c) reported that high concentrations of dextran having a high molecular weight, cause the formation of a very fine crystallinity of precipitate in second carbonation. The addition of up to 1% previously carbonated milk of lime precipitate to the first carbonation clear juice provides a huge population of large crystals in the pre-second carbonation juice. Upon gassing, the supersaturation that

is generated is more easily dissipated by the already present active, calcite-lattice surface area. Those fine crystals that may form are more easily agglomerated.

Elevated levels of invert, pectin, levan, and dextran are often related to beet storage conditions. To attain reasonable filtration rates, particularly at second carbonation, a mild defeco-carbonation may be performed at first carbonation with a satisfactory settling and filter cake formation. In this mild alkalinity environment, the pectin can be flocculated and removed in the first carbonation precipitate. The alkalinity is low enough so that there is little modification of the dextran or invert which passes through with the first carbonation juice. The clear juice is then heated and heavily limed (up to 1% CaO) with a detention time of 10 to 15 minutes. Dextran, levan, and amino acids or amides such as glutamine are partially saponified (24a,38a,38b). The high alkalinity of this second liming (or delayed main liming) will cause molecular modifications of these impurities such that they are more readily adsorbed in the carbonate precipitate. Their influence on the second carbonation precipitate structure is less. The high invert also is, in part, destroyed. With adequate alkalinity present to neutralize the acids formed, subsequent pH stability is improved. More permeable second carbonation filter cakes and more thermostable clear filtrates are attained.

This high CaO addition to second carbonation can be returned to the liming prior to first carbonation and thus can displace an equivalent CaO normally required for first carbonation.

Level control of the first carbonation clear juice receiver tank, A in Figure 9-5, permits process flexibility, i.e., a) at low level, as a simple receiver and b) at a high tank level, as a reaction tank (with an adjustable detention time) for moderate-to-heavy liming for precipitate modification.

Such a lime addition requires an increase in the total amount of CO_2 used, for while less gas is required in first carbonation, gas absorption is much lower in second carbonation, and the total usage is increased. Apparently the lime addition is equally effective whether it is in the form of milk of lime, or saccharate milk.

Lime Salts and Operation

It is an error to group all lime salts in a single chemical class, for operating purposes. There are certain lime salts which, if present in quantities as low as 0.035% CaO, will shut a factory down in a short time through rapid evaporator scaling. This type of lime salts is found in certain of the central areas of the United States, and apparently in many parts of Europe.

This contrasts with a second type of lime salts, found among other areas, in the Pacific Coast states, which can be present in the thin juices in quantities as large as 0.400% CaO, with an accompanying degree of evaporator scaling that can be economically tolerated with regularly scheduled evaporator-body boilouts. Identification of the chemical nature of these two types is needed.

In geographical areas where a) the type of lime salts prevails which does not scale the evaporators rapidly; or b) due to the nature of the beets or to the use of the Steffen process the natural or effective alkalinity of the juices is always low or negative, it is not economical to fully restore the alkalinity; or c) because of the high content of nitrogenous compounds in the beets and the danger of acid crystallizers, as well as to reduce incidence of floc, it is necessary to hold the operating alkalinity of second carbonation well above the optimum for lowest lime salts; operations are under different principles.

In factories in such areas the second carbonation may not be closely controlled, and the end point alkalinity may be set at a value high enough to keep the thick juice alkaline, with the additional help of the soda ash that can be afforded. In such operations, no regard is paid to the natural or effective alkalinities, or to the stoichiometric quantities of soda ash used, and no attention is given the optimum alkalinity for lowest lime salts, since it is usually lower than the operating alkalinity.

Thus the second carbonation tends to assume the status of an automatic process, very desirable from an operating point of view, which is allowed to run with little attention, and with major fluctuations in temperature, detention time, and pH or alkalinity values.

In these areas the beet quality is generally inferior, and processes supplementary to the standard juice purifications are needed to produce white sugars of acceptable quality. Such supplements may be ion-exchange installations, either for deliming thin juice or for mainline juice purification; the use of powdered or granular activated carbon; and recrystallization flow patterns involving many recyclings and prolonged detention times. Under such conditions the juice-degrading effects of less than optimal second carbonation operation may be lost to view in the general lower quality of the juices.

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B. Sulfitation*

The purposes of sulfiting the purified and clarified thin juices are: to control juice color formation, to improve the boiling properties of the juices, and to reduce excess alkalinity.

Equipment for Sulfuring

There are two methods of sulfuring in use, the production of sulfur dioxide by burning elemental sulfur in a *sulfur stove*, and that which uses purchased, liquid sulfur dioxide.

The sulfur stove (see Figure 9-9) is an apparatus in which sulfur is burned to form SO_2, in accordance with the reaction:

$$S + O_2 = SO_2. \quad (1)$$

Unfortunately a second reaction can occur, and always does to a certain extent:

$$2SO_2 + O_2 = 2SO_3. \quad (2)$$

*Original by R. A. McGinnis. Revised by G. W. Cossairt

The two product gasses dissolve in alkaline solution to form sulfite ($SO_3^=$) and sulfate ($SO_4^=$) ions, respectively. It is the sulfite ion which is desired. Sulfate is definitely unwanted, as it is chemically inactive, hinders crystallization, and increases molasses production.

There is also a physical action which may cause trouble: the subliming or vaporizing of the sulfur itself. Generally the gas transport line from the stove to the tower is kept cool to minimize sublimation.

Sulfur dioxide made in a sulfur stove may be introduced in the juice with a steam injector, with the aid of gas pumps, or with blowers and a Venturi injector. The gas is extremely corrosive to most metals in the presence of even traces of moisture, and this can be a source of continual trouble in valves and pipelines. Often the gas absorption apparatus is constructed as a tower with internal baffles, or other absorption aids. In this case, the control of the amount of sulfur dioxide added is quite difficult.

In the rotary sulfur burning system, Figure 9-9, lump sulfur is melted (119 °C) and its temperature is elevated high enough to vaporize the melted sulfur at a rate that matches the need for sulfitation. The oxidation reaction, p. 265, Eq. 1, once started, is exothermic, that is, it gives off heat. The amount of O_2, as primary air, admitted to the rotary drum, establishes the burner's process rate; i.e., the amount of exothermic heat generated and the amount of sulfur melted, vaporized, and carried into the air stream for further oxidation.

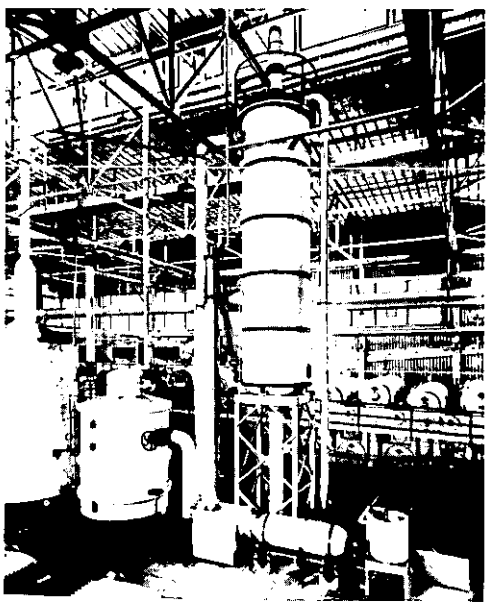


Figure 9-9. Sulfur stove and injector. Carlton, Calif. ***(Courtesy, Holly Sugar Corp.)***

The temperature of this air stream will be in excess of 230 °C [ignition temperature of S_8 vapor (7a)] and, in addition, it will be inversely proportional to the internal surface area of the rotary drum, wetted with melted sulfur. This air-sulfur-vapor-SO_2 stream is mixed with additional air for reaction completion at

900-1100 °C, a temperature range favorable for SO_2 equilibrium and unfavorable for SO_3 formation.

As some excess air will be present, and to avoid or to minimize SO_3 formation, Eq. 2, a rapid cooling of the SO_2-air stream will provide the necessary rapid temperature transition to less than 200 °C.

Every change in air dampers, every batch addition of sulfur to the stove, and every process juice flow rate change, creates or demands a change in the sulfur stove equilibrium. Such changes at the sulfur stove extend over several minutes time duration, thus there will be corresponding periods of unoxidized sulfur vapor moving through and out of the system. A water-jacketed gas duct will not only cool the gas but will also tend to sublime the sulfur vapor on easy-to-clean surfaces.

Athough the process juice equilibrium is quickly achieved and easily sensed as pH, the corrective agent system (sulfur stove) has huge time lags. Such response lags unfortunately degrade the attainable optimum in process flow sulfitation.

As an alternative to in-plant generated SO_2, in some geographical plant locations, liquid SO_2 is commercially and economically available in tank car quantities or in one-ton steel containers. Simple piping and injection control valves permit a very close sulfitation process control.

Two typical and optional liquid SO_2 systems are illustrated in Figure 9-10. For rail bulk delivery, a 40 000 gallon yard tank, A, is used for storage. Two small, water-jacketed measuring tanks, B, are provided in the factory for inventory control.

In some locations, it is more convenient to use steel, ton containers, C. A hot air heater, D, thermostatically regulated, warms the container and its contents to establish the desired vapor pressure (see Figure 9-11). A regulated internal pressure in C moves the SO_2 out of the container through the flexible metal hose, E, and optionally through the steam-heated evaporator, F, and pressure regulator, G. The pressure is adequate to overcome process pipe-line pressure.

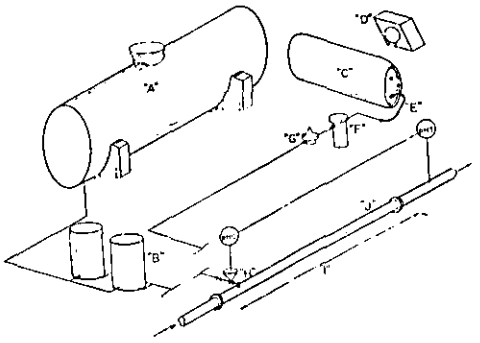


Figure 9-10. Optional Methods of *Handling Liquid* Sulfur Dioxide.—Bulk receiving and storage yard tank with small measuring tanks in plant or ton container system for liquid SO_2 or gaseous SO_2 injection. Process absorption by perforated, stainless tube inserted into section of corrosion resistant process pipe.

The actual injection of SO_2 into the process stream at H may be as the liquid or as the gas. Since the pounds of SO_2 per hour is relatively small, the gaseous injection and its larger specific volume simplifies the injection rate control. Also as a gas, lower pipeline pressures can be realized where elevated points of injection may exist.

Injection, preferably, is into a section of corrosion-resistant pipe, I, fitted with a perforated stainless tube, H, and stationary mixer elements, J, for rapid mixing in pipe section I.

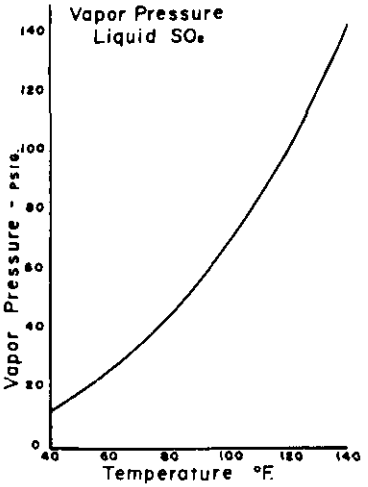


Figure 9-11. Vapor pressure of liquid sulfur dioxide.

Second carbonation clear juice is pumped into the pipe I and flows on to the thin-juice supply tank. A similar arrangement may be used for thick juice or standard liquor sulfitation. The liquid SO_2 systems are much easier to operate, and yield a superior process-sulfiting control than can be achieved with the sulfur stove and absorption tower system.

General Chemical Considerations

The reaction of SO_2 with water is similar to that of CO_2:

$$SO_2 + OH^- \leftrightharpoons HSO_3^- \quad (3)$$

$$HSO_3^- \leftrightharpoons SO_3^= + H^+ \quad (4)$$

$$SO_3^= + Ca^{++} \leftrightharpoons \underline{CaSO_3} \quad (5)$$

The dissociation constants for sulfurous acid and carbonic acid show the sulfurous acid systems to be more strongly acidic (see Table 9-2).

Table 9-2

	Dissociation Constants, 25°C	
	1st Hydrogen	2nd Hydrogen
Sulfurous acid	1.7×10^{-2}	5×10^{-6}
Carbonic acid	3.5×10^{-7}	4.4×10^{-11}

Table 9-3 Water Solubility of $CaCO_3$, $CaSO_3$, and $CaSO_4$ in %, Calculated as CaO

	Temperature, °C	
	50	100
$CaCO_3$	0.008	0.010
$CaSO_3$	0.0080	0.0074
$CaSO_4$	0.0842	0.0651

Data from Silin (11) on the relative solubilities of the calcium carbonate, sulfite, and sulfate, warm and hot, show the sulfite and sulfate, but not the carbonate, to have inverted solubility curves; *i.e.*, to be more insoluble hot, and thus calcium sulfite and sulfate are classed as true evaporator-tube scale formers. See Table 9-3.

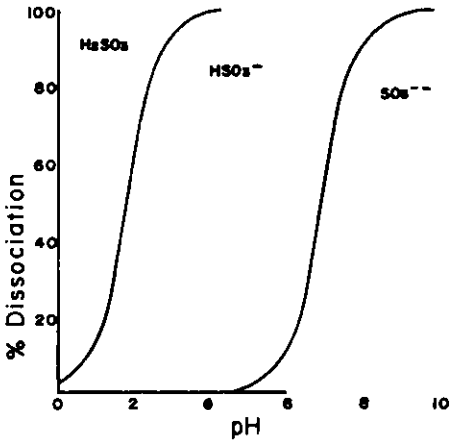


Figure 9-12. Dissociation of sulfurous acid, 18°C.

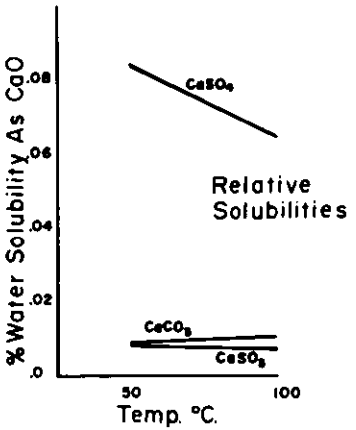


Figure 9-13. Relative solubility of calcium scale forming salts.

The solubility of most calcium salts is decreased by the presence of sugar, as is shown in the following data for $CaSO_4$, at 90 °C (11).

Table 9-4

Sucrose in Solution, %	0	20	42	55
$CaSO_4$ in g of CaO per 100 ml	0.0700	0.0521	0.0300	0.0137

The oxidation of sulfite to sulfate is greatly retarded as the sugar concentration is increased.

Because of the more acid nature of the sulfur dioxide derivatives, sulfitation cannot be carried out simultaneously with carbonation, or in the presence of solid calcium carbonate, as the precipitate will be dissolved, and the amount of lime salts in solution at the end point will be increased.

Use to Control Juice Color

This is the most important reason for sulfitation. Certain groups of atoms having a structural unsaturation, Figure 9-14, and known as chromophores (1a,7c,7e,9c,9d), are normally present in colored organic solutions. The depth of

color increases with the length of chains of alternating single and double bonds (conjugated chains) and with the presence of other specific groups of atoms known as auxochromes, $-NH_2$, $-NHCH_3$, $-N(CH_3)_2$, or $-OH$.

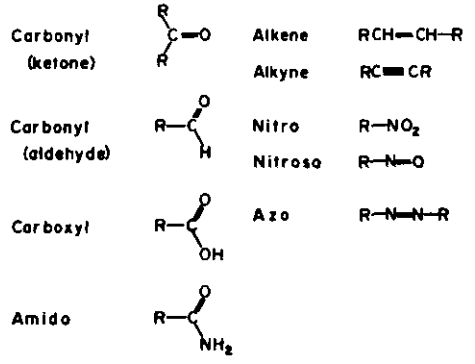
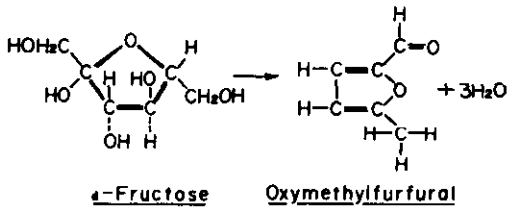


Figure 9-14. Chromophore molecular groups and structural types (7c).

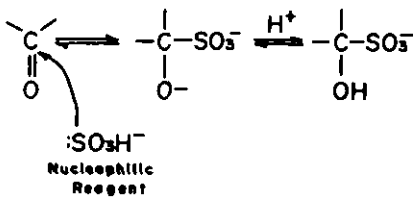
These compounds are characterized by an electron imbalance, an electronically excited state, a molecular resonance, an absorption of specific bands of transmitted light, and to the beholder, color.

Second carbonation clear juice contains invert sugars which continue to split, condense, fracture, dehydrate, and otherwise molecularly rearrange, usually with varying degrees of double bonding and unsaturation. For example:



d-Fructose Oxymethylfurfural

With a vigorous main liming, some of the invert degradation reactions have already been induced. Some of the unsaturated reaction products contain the chromophore groups in a molecular arrangement such that with $SO_3^=$ ion or HSO_3^- ion, a nucleophilic rearrangement can take place and a leuco or non-resonating or colorless form can be established (7e).



Typical of many carbonyl addition reactions, this reaction is reversible. As it is an equilibrium rearrangement, the sustained presence of the SO_3^{--} ion is necessary to maintain the leuco form.

Some ketones (a carbonyl) containing large or bulky groups may not respond to nucleophilic attack by sulfite or bisulfite ion, possibly due to steric reasons. This may account for some of the incomplete juice decolorizing with SO_2.

Some degradation rearrangements must pass through an intermediate reaction before the colored product is formed. If these reactions take place in the presence of SO_2 in solution, the initial substance is modified before it can proceed into the intermediate reaction. Although the basic ingredients for a color compound are present, the key intermediate reaction is inhibited. With oxidation or other loss of sulfite ion the inhibition is no longer effective.

Along with the invert sugars in the second carbonation juice will be significant quantities of impurities containing amino groups. With continued high temperatures beyond second carbonation, additional inversion of sucrose and saponification of amino compounds will release still more of both the carbonyl and amino groups for a sustained Maillard reaction and increased color.

Sulfite ion has been found effective in blocking color-forming Maillard reactions (7b,7d,9c). The mechanisms of some inhibition reactions are well known while others are very complex and require further resolution. Investigations have shown (7) that 20-30 ppm of SO_2 in the thin juice is enough to stop this brown coloring reaction. However, the gradual oxidation of the sulfur dioxide to sulfate, or its inactivation in other reactions tend to steadily decrease its activity. The Maillard reaction can proceed through the various massecuite crystallizations, and to be assured that sugar colors will be at a stable minimum, sufficient sulfur dioxide should be added to the filtered second carbonation juices so that there will be at least 25 ppm in the first-boiling massecuite. This may require from 50-200 ppm in the thin juice, depending on the nature of the juice impurities.

There is general agreement that the most important use of sulfitation is for its color-inhibiting action[1].

Sulfitation should always take place after an ion-exchange treatment, either deliming, or full cation-anion exchanger treatment. It is usually considered that when no sulfitation is used, there will be about 30% color increase during evaporation with comparatively invert-free juices. If there are significant amounts of invert present, the coloration across evaporation is greater. Thus the sulfitation may be considered to permit a color reduction of the thick juices of at least 30%.

Use to Improve Juice Boiling Properties

If the flow of sulfur dioxide to the thin juice is abruptly shut off, in many factories the first pronounced effect is the appearance of refractory boiling properties of juices in the evaporators and vacuum pans. This has been widely attributed to a reduction of the viscosity of the liquors. Nevertheless, it has not been possible to measure differences in the viscosities of sulfited juices (6). Likewise, no differences in surface tension have been found.

Silin has explained that this reduced viscosity is a secondary, apparent, effect. Since massecuites of high pH do not crystallize well due to the large amounts of sucrose in the form of saccharates, the presence of which has been confirmed by the work of Brieghel-Müller and Brüniche-Olsen, it is necessary to increase the density of the mother liquors to maintain crystallization, and this increases the viscosity and makes the massecuites hard to handle. The use of sulfur dioxide to reduce the pH,

[1]See refs. (3,9,9a,9b,13)

reduces the percentage of saccharates and makes it possible to reduce the density of the massecuites, so that the massecuite viscosity is reduced. Silin considers this to be the origin of the attributed viscosity reduction. It does not explain, however, the undoubted effects on the boiling properties of the juices in the evaporators.

Use to Neutralize Excess Alkalinity

If, as is the case with beet materials of very high quality, the natural alkalinity is so large that the crystallizing materials are at relatively high pH values, at which crystallization is slowed, the use of sulfitation is regarded by many as the most convenient means of accomplishing pH reduction. In Europe it is customary to regulate the sulfitation by the thick juice alkalinity, which is kept between 0.010 and 0.020, corresponding to a pH range of 8.2 to 8.6. No more than is equivalent to the natural or effective alkalinity should be added, or acid materials will result (12).

Imidodisulfonic Acid Formation

The presence of this material was first noted by Veibel and Brieghel-Müller (14), and confirmed by extensive studies by Carruthers and co-workers (1,2,5). In these studies, the mechanism of the catalytic inhibitory action of sulfur dioxide on color formation was elucidated. Likewise, the formation of imidodisulfonates was brought to general attention.

Naturally occurring nitrates in beet materials can be reduced by a number of microorganisms in the beet end of the factory, notably in the diffuser, to nitrites. Nitrite reacts readily with bisulfite ion to form imidodisulfonate. Potassium imidodisulfonate, $HN(SO_3K)_2$, can then crystallize out with the product sucrose, increasing the ash of the sugar. The most favorable region for its formation is between 6 and 8 pH. At higher pH values, the salt is more soluble and is more likely to pass to the molasses (4).

At one factory (10) this effect reached alarming proportions, and temporarily shut down operations. Imidodisulfonate formation can be detected in its early stages by comparing the amounts of sulfur dioxide found with iodimetric titration, to the known amounts added to the thin juice. If there seems to be an SO_2-acceptor present; i.e., if the amount of sulfur dioxide found is much too low, imidodisulfonate formation may be suspected.

Carruthers has pointed out (4) that 1 ppm of nitrite ion can inactivate 1 to 1.4 lb of sulfur per 100 tons of beets; i.e., 10 ppm of NO_3^- in the thin juice can waste a considerable part of the sulfur dioxide used. The analytical determination of nitrites in the juice is more direct, but requires a time-consuming analysis which would not otherwise be made.

Usage Practices in Sulfitation

The use of sulfur dioxide in the process goes back to the first part of the nineteenth century. The following listing gives some of the ways in which it has been used, when injected into the juices before the thin juice stage.

a) The juices were simultaneously gassed with both carbon and sulfur dioxide, either during second carbonation, or after second carbonation filtration. b) Sweetwaters have been sulfited. c) Sulfur dioxide has been used in first carbonation or liming in certain purification processes. d) Sulfur dioxide has been used with cossettes and diffusion juice to enhance diffusion rate. e) Diffusion supply water, or returned pulp press water is sulfited for sterilization, and to reduce the pH for better pulp pressing.

Use of sulfur dioxide prior to liming, in small amounts, seems to be without serious disadvantage, since at the high temperatures and lime concentrations of even mild defecations, virtually all of the residues are precipitated as calcium sulfite or sulfate. However, use during juice purification or after defecation and before the end of second carbonation, may increase lime salts in the thin juice, and cause additional scaling in the evaporator first effects.

Common usages, when injected into the process during or after the thin juice stage are listed.

a) Thin juices are sulfured to weak acidity, and then the alkalinity is restored with soda ash. b) The final alkalinity of the second carbonation is left high, at perhaps 0.03-0.04% CaO, so there will be sufficient alkalinity to sulfite heavily. c) The thin juice is sulfited, in amounts not greater than the natural or effective alkalinity. d) "Middle juice" sulfitation may be practiced. Juices at about 45 °Brix, perhaps coming from the third evaporator effect, together with added remelt sugars, may be sulfited before continuing to the next effect. e) The thick juice may be sulfited, as well as the thin juice. f) The thick juice alone may be sulfited.

Increased use of sulfur dioxide in the thin juice, due to a higher than optimum second carbonation alkalinity, means higher lime salts by the amount of lime salts left in the juice through failure to reach the optimum alkalinity. Similarly, over-sulfitation of the thin juice, with alkalinity restoration by soda ash addition, increases the scale forming lime salts by an amount equivalent to the oversulfitation. Both of these practices are attempts to add enough sulfur dioxide to obtain a bleaching effect. As has been pointed out, this is a delusory goal, as any bleaching effect is not permanent.

Since sulfur dioxide is now generally agreed to be most effective as an inhibitor of color formation, and since only small quantities are necessary for this purpose, addition to the thin juice is the usual practice in North America. The advantage over first addition to the "middle juice," or to the thick juice, is that the catalytic inhibitory action is not lost for part or all of the passage through the evaporators. Also, if absorption towers are used, absorption is not as good in the thicker juices.

The addition of sulfur dioxide lowers the whole pH profile across the evaporators. When the nature of the juice impurities is such that addition of enough sulfur dioxide to inhibit color formation throughout the sugar end brings about acid materials, the pH curve may be raised by reducing the amounts added to the thin juice and adding an amount to the thick juice. This will diminish inversion in the evaporators.

In the present state of knowledge, there seems to be no valid justification for the use of more sulfur dioxide than is needed for its action as an antioxidative catalyst. The necessity for pH reduction of very high quality massecuites may be an exception.

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C. Filtration*

Historical

The filter press, either of the plate and frame or of the earlier chamber-press type, was widely used in the early American beet sugar factories for filtration of all factory juices. The Kelly filter was invented in 1905 and gained great acceptance for first and second carbonation juices. This was followed by the development of other pressure leaf filters such as the Sweetland and the Vallez, the inventor of the latter having been with the Bay City Sugar Company. Vacuum filters were being developed by the Hart Brothers and by George Moore, but the first widely adopted drum-type, continuous vacuum filter was introduced by E. L. Oliver about 1908. This could filter the sludge from a carbonation juice thickened by settling, as well as

*by Robert G. Fee

extract sugar from various other solid residues. The tube-type vacuum filters soon followed, developed from earlier ideas and patents of Moore, and were the Oliver-Borden, the Genter (6), and the vacuum-Sweetland. These were combination filters and clarifiers, with the clear liquor going through the medium and on to process, and the deposited cake being blown off the tubes to settle in the hopper bottom for transfer to the drum filter.

In direct filtration of first carbonation juices, the filters were greatly affected by the characteristics of the juice and the solids contained. With the development of the Dorr continuous clarifier, many factories abandoned direct filtration of first carbonation juice and used their filters for second carbonation juice and thin juices. During all of this period, subsequent filtration steps such as of thick juice and standard liquor were, for the most part, carried out with plate and frame filters.

Since then, development of filtration equipment has proceeded in the directions of more effective clarification, reduced cloth consumption, and a lower labor cost for cleaning and operating. These developments have resulted in the wide employment of tubular-element pressure filters and pressure-leaf filters. Both types use a variety of rapid back-wash or sluicing features and have internal provision for cycling and settling.

Theory and Principles

Filtration is a separation accomplished by passing liquid through a porous membrane. Therefore, the filter may be considered to be mainly the device for holding the filter medium. But the filter must also provide the means of distributing the liquid over the surface of the medium, must hold the retained solids during the filter cycles, must provide the necessary drainage of filtrate from behind the septum and out of the filter, and must provide for discharge of the accumulated solids either continuously or at the end of a cycle prior to cleaning of the filter medium.

Filtration may be separated into the following categories:

1. Filter medium filtration. The medium alone removes the particles. The pore size of the filter medium determines the sizes of particles retained. Since the thin layer of collected solids quickly covers the septum, cycles are usually relatively short.

2. Depth filtration. The filter medium alone provides the particle removal, but instead of a thin screen or fabric, the medium is a thick mass of fibrous or filter aid materials. The coarser particles are held on the surface, while the finer particles can migrate down and be trapped in the medium. As particles are retained in the medium, the resistance builds in the form of higher pressures and decreased flows. Also, with continuation of the cycle, some finer particles may start to pass through the filter, and the medium must be replaced.

3. Cake filtration. Most filtrations in beet factory operations are of this type. Here the medium or precoat establishes initial filtration characteristics, but as the solids accumulate on the filter surface, it is the filter cake formed which most influences the operation. The effect of types of deposited solid materials will be discussed later, but special treatment is necessary in the case of extremely fine, slimy, or compressible solids. Very often filter aids such as diatomaceous earth are added directly to the liquid going to the filter. The diatomaceous earth particles, being both porous and rigid, provide passages for fluid flow and give rigidity to the cake, thus prolonging the filter cycle. The filter cycle is also a function of the speed of pressure build-up and corresponding reduction of flow. With very open, porous cakes, the cycle length may be determined by the cake capacity of the filter.

Much of the early investigational work in filtration theory was done by Poiseuille (8) and by D'Arcy (3). The name of the latter is perpetuated as the unit of permeability, the darcy. One darcy unit indicates permeability to pass one ml per second per sq cm of a liquid of one cp viscosity through a cake one cm thick at a pressure differential of one atmosphere.

The primary and secondary influences on permeability, filter performance, and overall filter station operation are listed below, in Table 9-6.

Table 9-5 Primary and Secondary Influences on Filtration

Primary Influence	Secondary Influence
1. Viscosity	Density or concentration of liquor. Concentration of nonsugars. Temperature.
2. Cake thickness	Length of filter cycle. Volume of solids in liquor.
3. Capillary diameters (porosity of cake)	Size of solid particles Compressibility of solid particles. Filter aid addition. Condition of filter cloths.
4. Pressure differential	
5. Filter area	

For more detailed theory along with calculation and extensive bibliographies, please see references[1].

Description of Equipment

Plate and Frame Filters. In the plate and frame press (Figure 9-15), the feed liquor is forced into the hollow frame, either at the top or bottom corner of the frame. The liquid passes through the filter cloth and escapes through drainage channels in the plates. Plate outlets can be individual spouts which provide for observation of individual plate performance. The outlets are sometimes combined in a manifold so that a single stream leaves the filter. Filter outlets can be either from the top corner or the bottom corner of each plate. Top outlet presses have the inherent advantage of providing more uniform precoat and cake distribution.

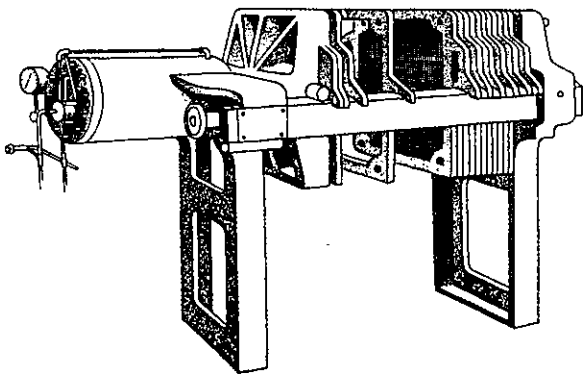

Figure 9-15. Plate and frame press.

[1]See refs. (1,2,4,6,7)

This type of filter has the advantages of low cost, near indestructibility, ease of internal inspection, capability of efficient cake washing, and small amount of residual heel. Since each plate is cleaned separately and the cloths are usually laundered after each cycle, more labor is required for cleaning.

Typical of this type of filter are those manufactured by the D. R. Sperry Company, and the T. Shriver and Company.

Kelly Filter. This is a type of pressure leaf filter, but deserves special mention because of its usage in the industry. It consists of a horizontal pressure cylinder with vertical leaves positioned longitudinally within the tank. The leaves have individual outlets so that the juice flows can be observed. The entire leaf assembly is mounted on a track and will slide out for cleaning and inspection. Please see Figure 9-16.

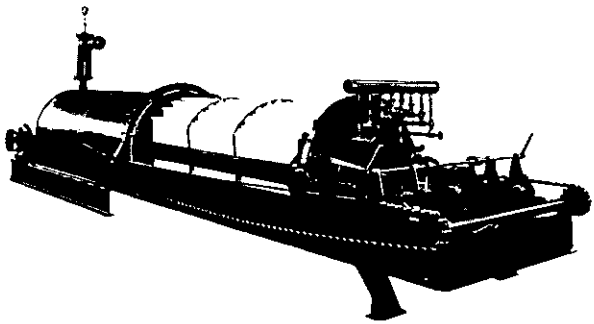

Figure 9-16. The Kelly filter.

Rotary Vacuum Filters. These so-called drum filters have several variations of design. They consist of a rotating drum suspended on its axis in a tank containing the liquid to be filtered. As the drum rotates, vacuum is applied to certain segments across the face of the drum, while segments in other positions may have no applied vacuum, and still other segments may have reverse air pressure applied. The portion immersed in the liquid draws filtrate through the drum, collecting solids on the face of the drum, which is the septum. As the drum rotates out of the liquid, the cake is dried by continuing air suction through the cake. The cake may be washed to remove sugar. The cake is then removed from the drum at another position of rotation by being loosened with air pressure, and sometimes assisted by a scraper.

Certain designs provide for removal of the cake from the drum by means of a series of strings which bear on the drum surface during deposition of the cake and then leave the drum to carry the cake off to a discharge point. A similar type uses a continuous belt which is around the drum for the cake pick-up part of the cycle, and then passes over a series of rollers, sprays, and other cleaning devices for cake discharge and belt washing.

Filters of this type are manufactured by Ametek, Dorr-Oliver, and Eimco.

Rotary Vacuum Precoat Filters. These are similar to the previously described rotary filters except that there is never a reverse passage of air through the drum (Figure 9-17). A thick precoat of several inches of diatomaceous earth is built up from a slurry in clear liquor. After build-up of this precoat, the liquor to be filtered is introduced into the filter. As filtration proceeds, a very thin layer of the filter aid precoat is cut off, along with the solids which have deposited on the sur-

face, by means of a knife set across the length of the drum. This knife is advanced by an adjustable micrometer device which allows for continuous cutting, and thus continuous filtration, for the depth of the precoat layer.

The cycle length of this type of filter is generally longer than in other types of filters, and it is especially adaptable to liquors with high solids content or with solids which may be slimy or otherwise hard to filter. Selection of filter aid for the thick precoat layer is most critical. For instance, if the filter aid is too permeable, solids will penetrate into the cake, cause blinding, and necessitate deeper cutting into the precoat. If the filter aid tends to compress, the cake will pull away from the knife, and become surface-blinded with solids. The ideal filter aid for rotary vacuum precoat filters is incompressible, porous for liquid passage, and will hold solids on the surface for knife removal.

Filters of this type are manufactured by Ametek, Dorr-Oliver, Eimco, and Komline-Sanderson.

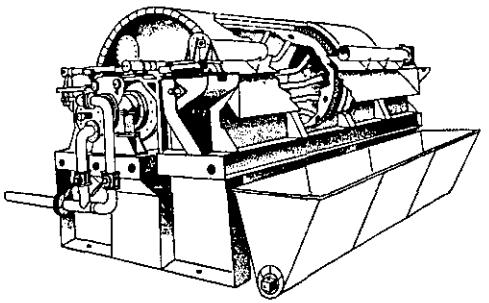

Figure 9-17. Rotary-vacuum precoat filter.

Tubular Element Pressure Filters. The tubular element pressure filter or candle filter consists of a vertical pressure tank with an arrangement of vertical filter tubes through a tube sheet at the top (Figure 9-18). Some have a top or bottom manifold arrangement for the tubes. Some of the filters are designed for rapid backwashing by means of entrapped air in the top portion of the tank above the tube sheet, on the effluent side of the elements. When used for rapid turn-around by means of a quick cleaning feature, the selection of the filter septum is most critical. This is to assure good release of accumulated cake, and to prevent clogging of the septum.

Typical of the above type of operation is that provided by the Hydra-Shoc filter. In this filter the solids built up on the tubular elements are removed by quickly opening the outlet valve at the bottom of the filter shell, thereby allowing the air trapped in the dome to blow the cake loose from the elements and out the drain.

Filters of this type are manufactured by Braunschweigische Maschinenbau Anstalt, De Laval Turbine, Hayward Filters, Industrial Filter and Pump, and Niagara Filters (Ametek).

Pressure Leaf Filters. Pressure leaf filters are made in a variety of designs. Tanks are either horizontal or vertical. In the horizontal designs, the leaves are suspended vertically either crosswise or longitudinally (Figure 9-19).

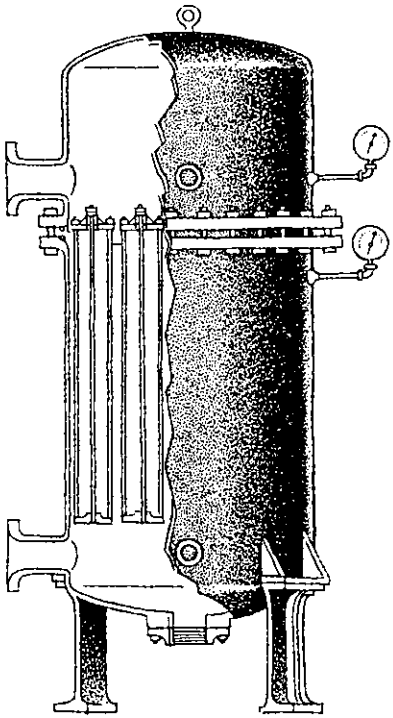

Figure 9-18. Cylindrical element filter.

The filter leaves may emerge from the shell for cleaning, but more often internal sluicing mechanisms are provided. Such sluicing arrangements may consist of an oscillating set of nozzles, nozzles mounted on movable arms, or fixed nozzles with leaves that rotate. Regardless of the particular design, it is of the utmost importance to keep the nozzles from clogging. Clogging may occur by forcing fibrous precoating materials into the openings during precoating, by solid particles in the sluicing juice, or by scale from the sluicing lines. In some cases, sluice lines have

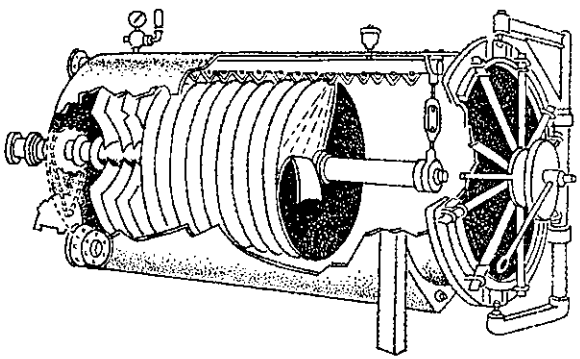

Figure 9-19. Rotating leaf pressure filter.

been replaced with stainless steel to assure that clogging materials will not be carried into the nozzles and strainers have been installed in sluice lines.

In providing for filter sluicing, it is necessary to consider the available quantity of sluicing liquor. To avoid dilution, filter leaves are usually sluiced with thin juice. Effective sluicing requires as much as two gal of sluice liquor per sq ft of filter area and should be applied at 40-60 psi. Experience has shown that if an unusually high volume of thin juice is required at one time, the operation of the evaporators can be upset because of unbalanced flow conditions.

Serious problems occur if the cake spaces are allowed to fill, bridging between the leaves. Warping of the leaves or breaking of leaf rotating mechanisms may result.

In any of the pressure-leaf filters with internal sluicing, adequate provision must be made for the removal of the filter cake, both its drainage from between the leaves and out of the filter shell. In certain designs it is important that the cake be sluiced out of the shell drain area first to avoid clogging the impending flow of the rest of the cake.

Filters of this type are made by many companies, among which are Duriron (Enziger), Hercules, Industrial Filter and Pump, Keene, Niagara (Ametek), Pronto, Sparkler Manufacturing, and U. S. Filter.

Filter-thickeners. These so-called filter-thickeners are very similar in design to the vertical screen or tubular pressure filters. Filtration is accomplished by passing the liquor through a leaf or tube on which the solids deposit. When the cake builds up, it is released from the septum in such a way that it is retained on the bottom of the pressure tank and allowed to settle further in that vessel. At a later point in the cycle, the settled or thickened sludge is discharged out of the filter. The filtered liquor coming through the septum has the same characteristics as with any other pressure filter, but the advantage of the design is that there are relatively short cycles and concentration of the solids for minimum sugar loss.

It is of interest that the Genter and Borden filters were somewhat similar in design and purpose, except that they operated by vacuum instead of by pressure. Representative of these types of filters are those from BMA, Danish Sugar Co., Grand Pont, and Putsch.

Referring to Figure 9-20(a), valve 1 admits unfiltered liquor, generally with pressure provided by an elevated feed tank. Flow is through the elements illustrated and the outlet for the filtrate shown on the right. In removing the deposited cake from the elements, the usual procedure is to open valve 2, which permits liquid to be drawn from the tank, with the loosened cake falling to the bottom of the cone. Filtration is resumed and the operation repeated, allowing the density of the sludge in the bottom of the cone to increase with successive purges. When sludge of the proper density is built up, valve 3 is opened, which allows a complete purge, removing the thickened sludge from the filter shell. The actual mechanics of accomplishing this differs with different equipment, but the process is generally the same; hence, the designation "filter-thickeners."

Horizontal Leaf Pressure Filters. These are very similar to the leaf filters previously described except in the positioning of the leaves (Figure 9-21). Usually the leaves are held together in a stack or cartridge, which is placed in the vertical tank. In many filters of this type the filter septum is paper which is discarded after each cycle. Some designs provide for precoating and internal sluice.

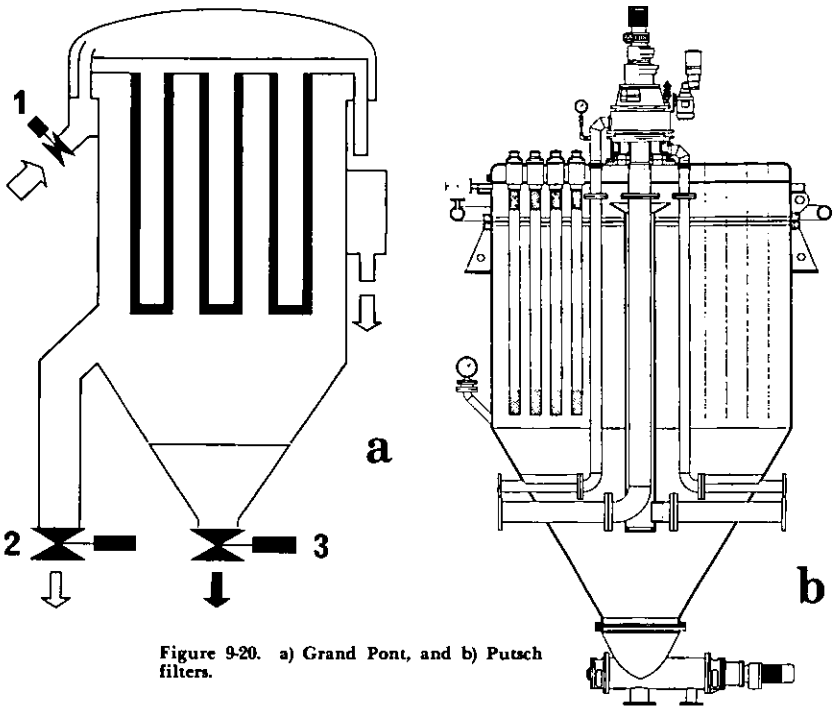

Figure 9-20. a) Grand Pont, and b) Putsch filters.

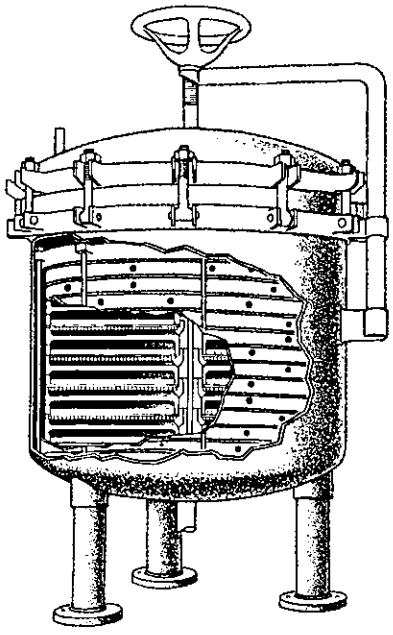

Figure 9-21. Horizontal leaf pressure filter.

Since the precoat and deposited cake are held on the filtering surface by gravity, this filter is useful in operations where the flow may be fluctuating or intermittent. The horizontal leaf filter is usually used in applications where solids loading is low and cycles are relatively long, such as in polishing operations.

Among filters of this type are the Niagara, Shriver, and Sparkler.

Cartridge Filters. These filters are not unlike the oil filters used in automobiles. They are useful as trap or guard filters where the solids to be removed are in a very low range. As described in the section on media, the cartridges are nearly always disposable, and provide the entire filtering mechanisms: septum, backing, drainage area, and support. The element is used in a canister or holder to provide the proper fittings and fluid flow.

Filter Media. The four types of media of interest are, in order of usage: 1) fabric, cotton and synthetic; 2) woven metal wire; 3) paper; and 4) replaceable cartridges.

The filter medium is not only a substantial operating expense, but also its selection is most important for good filter operation. When no filter aid is used as a precoat, the septum must be open enough to allow passage of liquid and, at the same time, retain the undissolved solids. In addition, the septum must provide for stability of cake to resist erosion during slight changes in plant operating conditions. At the conclusion of the filter cycle the septum must release the cake and allow for complete sluicing, backwashing, or other cleaning to prepare for the next cycle.

Following are some of the characteristics of fabrics which affect medium selection (5).

1. Fiber Properties

Cotton: Exposure to high pH causes fiber swelling and reduced flow rates. There is good particle retention because of hairy filaments. It will blind badly.

Dacron: Flex resistance and high initial shrinkage allow the fabric to become quite tight for excellent solids retention, and it has low moisture absorption.

Dynel: Has high dimensional stability with shrinkage of less than 0.5 percent in boiling water.

Nylon: Has smooth surface for excellent cake discharge, good wet strength, and good elasticity.

Polypropylene: Has the lowest density of any synthetic resulting in greater cloth yield per pound of yarn. It has good cake discharge, and resists blinding.

2. Fabric Construction

A. Weave Patterns. There are four basic weave patterns that account for the bulk of woven media: plain, twill, chain, and satin.

1. The *plain weave* is the simplest of all weaves and is strong and durable. A set of threads goes in one direction, and at right angles to the first set a second set of threads proceeds in a one-over-one-under pattern, giving a checkerboard effect. This is the tightest fabric possible to weave. Filter duck is a plain weave.

Its characteristics are: lowest price, least porosity, greatest particle retention, and susceptibility to blinding by solids.

2. The *twill weave* consists of two sets of threads at right angles to each other, the apparent diagonal twill line being caused by moving the filling thread interlacings one thread higher on each successive warp thread.

Its characteristics are: high resistance to abrasion, medium retention and blinding properties, and good flow rates.

3. The *chain weave* is a loose twill weave in a 2/2 reversible broken twill weave.

Characteristics: High clarity and flow rates, and lower tensile strength and retention than plain, but has greater resistance to blinding.

4. The *satin weave* is a smooth surface fabric produced by carrying the warp uninterruptedly on the fabric surface for many filling yarns.

Its characteristics are: superior cake release, best resistance to blinding, and poorest particle retention of the four basic weaves.

B. Yarn. There are three basic types of yarn: monofilament, multifilament, and spun-staple.

1. Monofilament is a synthetic fiber made in a single continuous filament.

Its characteristics are: minimum blinding, good cleaning, and excellent cake discharge.

2. Multifilament yarns are made by twisting two or more continuous monofilaments.

Its characteristics are: greatest tensile strength of any yarn, and good cake discharge.

3. Spun-staple. In natural fibers, spun-staple expresses the length of the fiber in relation to the diameter. In a synthetic, it signifies filaments that have been cut to the length of the natural fibers. Spun yarns are made by twisting staples together.

Its predominant characteristic is the best particle retention because of hairy filaments.

Despite higher prices, synthetic fabrics, especially nylon and polypropylene, are gaining wide acceptance in the industry. This is due to superiority in the areas of chemical resistance, durability, and good solids-release and ease of cleaning. The greatest usages of synthetic fabrics are on rotary-drum filters, tubular-element pressure filters, and pressure leaf filters. Cotton is still widely used in plate and frame filters.

Woven metal wire media are used primarily in filters where a precoat of cellulose or diatomaceous earth is applied. Usually these filters have provision for filter-cake removal by sluicing. The most commonly used metal septum is a single Dutch weave, 24 × 110 with 0.016 in. by 0.108 in. wire. Occasionally a 60 × 60 twill with 0.011 in. wire is used, but this type requires special precautions in precoating and a fibrous material to bridge the larger openings. With selection of the appropriate alloy for both screen and coarse backup grids there is exceptional chemical stability. The metal media have the inherent disadvantages of susceptibility to mechanical damage, and difficulty in cleaning when clogged with solid impurities or scale. Calcium oxalate is a particularly troublesome scale in beet-sugar liquors.

Paper media are in the form of disposable sheets or pads. They have excellent retention properties in solids removal. Because of very poor strength, the paper media, especially the thinner sheets, are supported on a backing of cloth or metal mesh. The paper media are used in special applications which are discussed later.

Replaceable cartridges, like paper, are used in special applications where solids loading is relatively low. The cartridges or filter elements are usually made of yarn or other fiber wrapped on a plastic supporting core. Most of these elements are

Table 9-6 Properties of Fibers

Fiber	Rec. Safe Temp. Limit °F	*Wet Breaking Tenacity	Acid Resistance	Alkali Resistance	Price Ratio to Cotton	SpG	Yarn Forms
Cotton	210	3.3-6.4	Poor	Fair	1	1.55	Spun-Staple
Dacron	300	6.0-8.2	Very Good	Good	2.7	1.38	Multifilament Spun-Staple
Dynel	200	3.0	Excellent	Excellent	3.2	1.30	Spun-Staple
Nylon	250	2.1-8.0	Fair	Excellent	2.5	1.14	Monofilament Multifilament Spun-Staple
Polypropylene	175	3.5-8.0	Excellent	Excellent	1.75	.90	Monofilament Multifilament Spun-Staple

*Tensile strength at rupture expressed in grams per denier.

wound cotton or synthetic yarn, although glass fiber cartridges are coming into greater use. The glass fiber cartridges have greater solids loading capacity and hence a longer life in the filter. In addition to the wound fiber cartridges, molded cellulose or felt have sometimes been used.

Filter Aids

The development of filter aids has greatly improved both the operation and the economics of filtration, and made possible high filtrate quality. To be effective as a filter aid, a material must: have strong, intricately shaped, and porous individual particles; form a highly permeable but rigid and incompressible filter cake; remove even the fine solids at high rates of flow; and be chemically inert and insoluble. Most of the filter aids used in beet-sugar processing are diatomaceous earth, sold under trade names such as Hyflo Super-Cel and Dicalite Speedplus. Diatomaceous earth is the skeleton remains of single-celled aquatic plants called diatoms (Figure 9-22). It is mainly composed of silica and the diatom structures are in a variety of complex forms of different sizes. Diatomaceous earth is processed so that a range of grades is available, offering selection of flow rates and clarities.

Special filter aids are sometimes used for precoating. These may be cellulose, or blends of cellulose and diatomaceous earth. Other materials used as filter aids include various clays, perlite, and carbon.

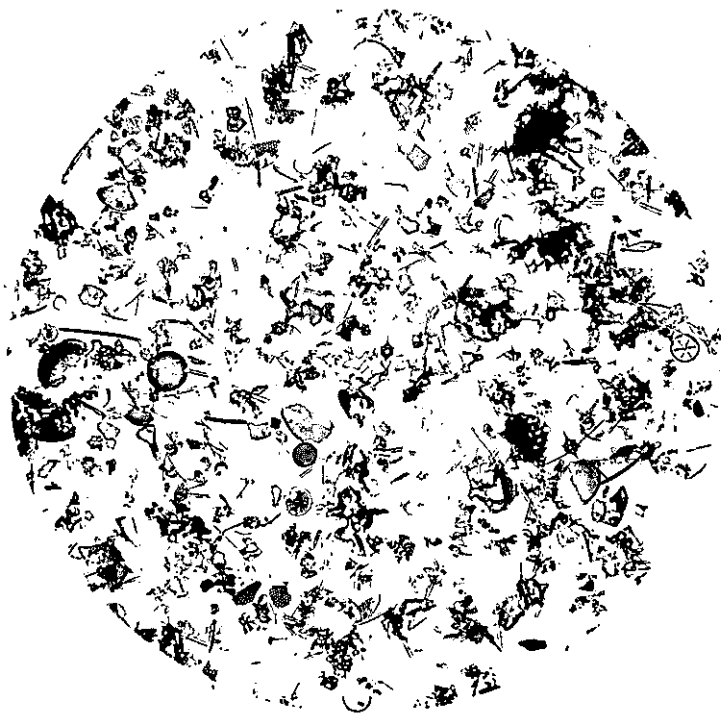

Figure 9-22. Photomicrograph of diatomaceous earth filter aid (× 50). *(Courtesy, Manville Products Corporation.)*

Filter aids are used in two ways. The first usage is as a precoat. The purpose of the precoat is three-fold: 1) to prevent the filter septum from becoming clogged by impurities, thus prolonging its life; 2) to facilitate cleaning the septum at the end of the cycle; 3) and to give immediate clarity.

Precoating is accomplished by circulating a slurry of diatomaceous earth in filtered or clear liquid between the filter and the precoat tank. Since most of the filter aid particles are smaller than the openings in the septum, they must form a precoat by bridging these openings, as shown in Figure 9-23. These bridges can be upset by air bubbles, sudden changes in pressure, or by vibrations; causing the filtrate to become turbid until the upsetting influences are corrected. If distribution in the filter is good, the filter may be filled with clear precoat liquid and a concentrated slurry of filter aid may then be pumped or educted into the filter, followed by recirculation.

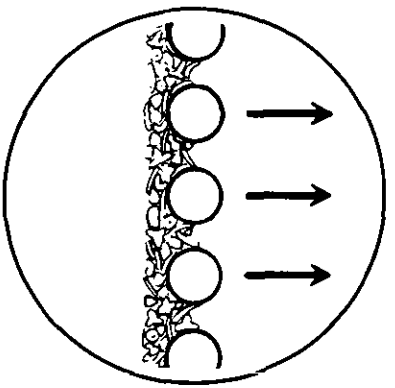

Figure 9-23. Representation of precoating. Filter aid depositing over cloth or wire openings. *(Courtesy, Manville Products Corporation.)*

The amount of precoat should be from 10 to 15 lbs of filter aid per 100 sq ft of filter area, the greater amount being used when the distribution of flow in the filter is poor, or in starting up new filters. If it is uniformly distributed, 10 lbs of filter aid per 100 sq ft of filter area will give a precoat of approximately 1/16 in. in thickness. If distribution is imperfect, 10 lbs of filter aid per 100 sq ft may result in inadequate precoat at the top of the leaves or at the end of the filter away from the precoat slurry entrance. This condition can frequently be remedied by use of baffles.

Precoat slurry concentration will depend primarily on the ratio of filter area to filter and piping volume. The slurries will, however, range from 0.3 percent to 0.6 percent. If they are much below 0.3 percent, precoating may be difficult since the formation of the bridge depends partly on the "crowding" effect of the particles of filter aid trying to get through the septum openings. The precoating rate will depend mainly on the viscosity of the liquid used. The rate should be sufficient to keep all the filter aid in suspension but should not be fast enough to cause cake erosion in the filter. For water, the rate is from one to two gallons per sq ft of filter area per min (gsfm). For viscous liquids, the rate may be as low as five gallons per sq ft per hour (gsfh). A general rule for precoating is to precoat at that rate which gives a differential pressure of about 2 psi. For water, an upward velocity of at least 4.5 ft

per min is required for proper filter aid suspension. The suspension of filter aid can be improved in the tank type or pressure leaf filters by partially recirculating from the top of the filter back to the precoat tank.

A second form of precoating is on the rotary vacuum precoat filter described above. Precoating this filter requires a special technique. This is too involved to describe in detail, but some of the variables are: vacuum, precoat slurry concentration, drum submergence, amount and control of vacuum increase, recirculation (or not) back to precoat tank, and rate of drum rotation. Poor precoating performance may be due to insufficient vacuum, poor hydraulics in the filter, incorrect drum rotation or submergence, incorrect slurry concentration, flooding of the receiver, or a partially blinded septum.

The second way in which filter aids are used is as an additive to the liquids being filtered. This is known as body feed (Figure 9-24).

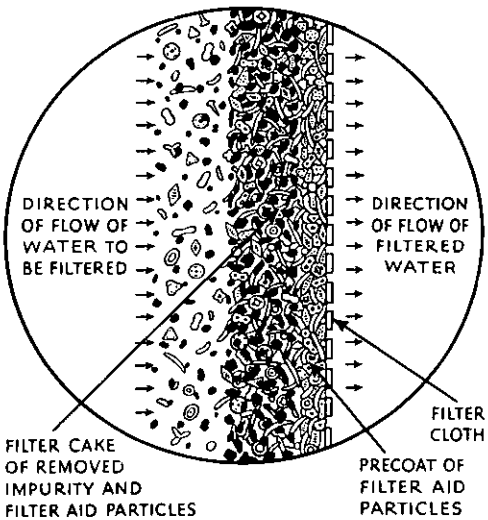

Figure 9-24. Representation of precoat, filter aid as body feed, and solid particles being removed from liquor stream. *(Courtesy, Manville Products Corporation.)*

The effects of varying the amount of body feed addition are illustrated in the graph in Figure 9-25, which shows total throughput on the vertical axis vs. percentage of body feed on the horizontal axis.

As can be seen, the addition of too small an amount of body feed merely reduces the total throughput, since the body feed is completely surrounded by undissolved solids. This only increases the cake thickness without adding anything to its porosity. From this point, as the body feed is increased, the throughput also increases slowly, and then rapidly for a short space of time. The rate of increase then tapers off, reaching a peak, after which it actually decreases again. The decrease in throughput upon larger additions of body feed is the result of turbid particles getting lost in the body feed. In such cases, additional quantities of body feed merely increase the cake thickness. The amount of body feed will also depend on the rate at which the liquid is filtered. That is, the same liquids filtered at higher rates will usually require more body feed for the same throughput or cycle lengths as shown in Figure 9-26.

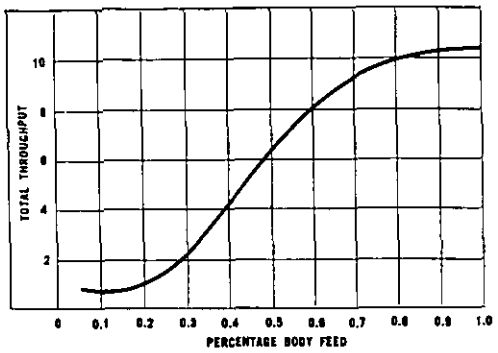

Figure 9-25. Percentage body feed. Rates of flow of same solution with different amounts of filter aid. *(Courtesy, Manville Products Corporation.)*

From the description of body feeding procedure, it is apparent that one of the most important factors for effective and economical filter aid use is a *steady and uniform rate of filter aid addition.* Unfortunately many filter aid feeders in current use are inadequate in this respect and maximum effective use of filter aid is difficult to attain. A second very important consideration is the avoidance of sharp pressure fluctuations while the filter is on stream, as well as avoidance of the inclusion of air bubbles in the filter feed. Both can be very disruptive to the filter cake, and adversely affect filtrate clarity.

Proper use of filter aid reduces operating costs through extended filter cycles and greater cloth life. Also, the filter aid assures removal not only of solid impurities such as carbon particles, but also of mineral salts and many bacteria and other organisms affecting finished sugar quality.

Filtration in the Beet-Sugar Industry

Direct Filtration of First Carbonation. In years past both plate and frame and Kelly filters were used to filter first carbonation juice. Some of the problems encountered have been the high solids loading, on the order of 5% of dry matter. Depending on the variables of carbonation, this can be a granular precipitate, or very fine and hard to filter (see pp. 172, 176-7). The cake has a high degree of compressibility and maximum filter pressure differences are in the range of 25 to 30 psi. The cake before desweetening contains 7 to 8% sugar. This is reduced to a maximum of 0.8% by washing with 125 to 200% of water, based on weight of cake.

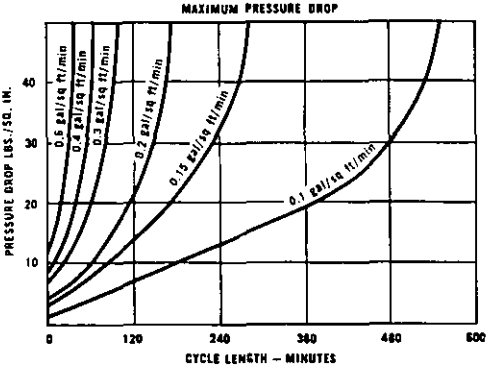

Figure 9-26. Cycle length-minutes. When the maximum pressure drop of 50 lbs/sq. in. is reached, the cycle is ended. *(Courtesy, Manville Products Corporation.)*

More recently direct filtration of first carbonation juice has been carried out using filter-thickener types of equipment with fairly rapid cycling. Also various pressure leaf filters have been used. With pressure leaf filters the cycles have been in the range of 60 minutes and the temperatures of filtration between 80° and 90°C. Depending on carbonation operating conditions and using leaf filters, the surface area required is 1.0-1.8 sq ft per ton of beets processed per day. No filter aid is ordinarily used, since the volume of the precipitate and its filtering characteristics should permit rapid flow and good clarities.

Second Carbonation Filtration. After the second carbonation, the juices are clarified using pressure filters (Figure 9-27). The importance of this step is reflected in subsequent evaporation, and if filtration is not effective, the calcium carbonate particles will dissolve during sulfitation increasing the lime salts content of the juice, and scaling of evaporators will result, as well as difficult standard liquor filtration.

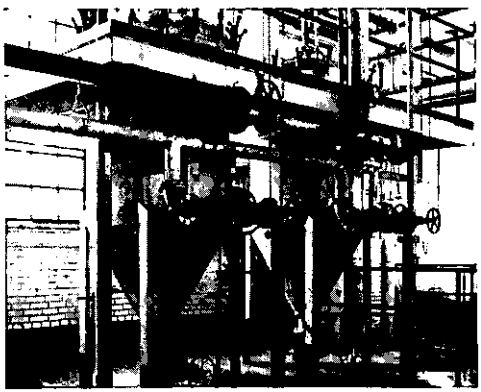

Figure 9-27. Second carbonation station. Hydra-Shoc filter in Fort Morgan, Colorado, factory of the Great Western Sugar Company. ***(Courtesy, The Great Western Sugar Company)***

For this filtration plate and frame, pressure leaf, and tubular element filters are routinely used. Usually no filter aid is used, although in times of processing difficulties some diatomaceous earth has been helpful. This has been used as a precoat to protect the cloths and to effect quick release of cake. Filter aid has also been used as a body feed, but without uniform feeding equipment it has been somewhat inefficient.

One of the problems with any of the filters is the maintenance of the filter cloths. Cloth hardening is a common occurrence and contributing to it are the introduction of steam, air, carbon dioxide, and soda ash to the juice stream or during the ending of the filter cycle.

Carbonation Sludge Filtration. This is carried out on rotary drum filters (Figure 9-28). The sludges originating from Dorr-type thickeners, filter-thickeners, or other first and second carbonation juice filters, sometimes with the admixture of the standard liquor filter cakes, are mixed together and usually passed over the drum filters. Here the carbonation juice is removed and the cake washed to extract all possible sugar. The waste filter cake discharged from these filters should contain no more than 0.8% sugar.

Much thought is being given to disposal of this waste lime cake. In addition to containing about 75% of calcium carbonate and adsorbed impurities from the beet,

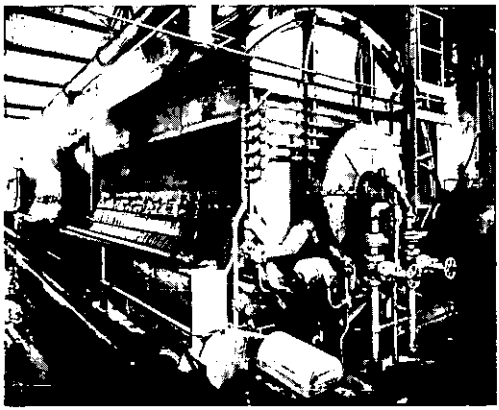

Figure 9-28. Carbonation sludge filtration station. Eimco filters at Woodland, California, factory of Spreckels Sugar Company. *(Courtesy, Spreckels Sugar Company)*

this sludge contains in the range of 6% organic nitrogen compounds, 4% of other mineral compounds, and a fair percentage of phosphoric acid equivalent. The value of this sludge for use as a fertilizer or for soil conditioning is an area of consideration.

Thin Juice Filtration. In past years it has been the practice to filter thin juice after sulfitation. The thin juice was passed through clean plate and frame filters and in many cases it was the purpose to "precoat" the cloths with a small amount of calcium carbonate which had leaked through second carbonation filtration. After the press was used for thin juice for a period of time it was switched over to thick juice. The thin layer deposited from thin juice acted as an aid in clarifying the thick juice. With improved filtration techniques for second carbonation juice and with modern filter aids for thick juice, it is no longer common practice to filter thin juice.

Standard Liquor Filtration. Standard liquor is usually filtered as a combined stream going to pan storage (Figure 9-29). It may be filtered either once or twice. Double filtration is common when powdered activated carbon is used in the process. In either case, equipment and techniques are similar.

In most factories plate and frame filters were used. In most cases, however, these have been replaced with the newer pressure leaf filters with internal sluicing. Probably the greatest reason for this change has been the more efficient use of labor,

Figure 9-29. Standard Liquor filtration station. U. S. Filters in Drayton, North Dakota, factory of American Crystal Sugar Company. *(Courtesy, American Crystal Sugar Co.)*

since a pressure leaf filter station can be automated to greatly reduce labor costs, as only occasional filter dismantlings are necessary for cleaning.

From the standpoint of filtrate quality, this is the most critical filtration in the beet-sugar factory. The liquor from these filters proceeds into the white pans, and whatever solid impurities pass have a good possibility of remaining with the final granulated sugar crystals. It is not only true that the filtrate leaving the filter must be of the highest clarity, but also that it must be protected from contamination *from this point on.* This means protection against solids contamination of press troughs on plate and frame presses, juice lines, and storage tanks.

In order to achieve the high degree of clarity needed, the standard liquor filters are precoated with filter aid. This may be regular diatomaceous earth or cellulose fiber, or a prepared precoat material containing both. During the filter cycle a regular feed of filter aid is maintained into the stream of liquor to the filter. This body feed is regular filter aid with no cellulose or other materials added. A schematic flow diagram is shown in Figure 9-30.

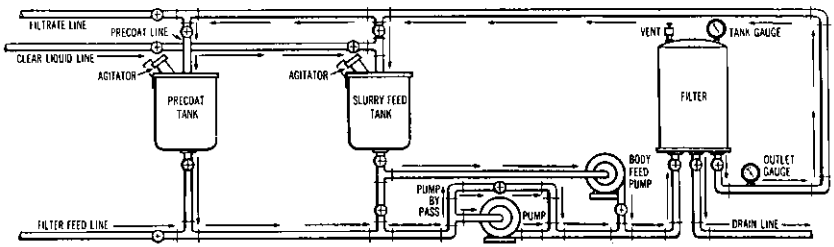

Figure 9-30. Schematic diagram of diatomite filtration system.

The amount of filter aid used varies with operating conditions. When beets are of poor quality and impurities are coming through in the juice stream, it is necessary to use higher rates of body feed. Also cycles will be shorter, and therefore more filter aid will be used for the precoating operations. Filter aid usage usually is in the range of 0.4 to 0.9 lbs per ton of beets. Under unusual conditions, or where filter aid is used on other stations, these amounts will be exceeded. Usage is also somewhat higher in factories including the Steffen process.

When double filtration is used, the second stage filters are often only precoated. Occasionally a body feed about one-third that used for first filtration is used to extend the second filter cycle. This is often advisable during periods of difficult operation, such as with deteriorated beets or other adverse conditions.

Precoating can be done from the troughs of the first filter in the case of plate and frame presses, or through a conventional precoating installation with leaf filters. With either one or two filtrations, one of the most important operating steps is to be certain that clarity is established before a filtrate stream is diverted to the pan storage. In many factories this is done by regular checking with black filter discs as a test for complete precoating.

Standard liquor is filtered at 67-72° Brix and at temperatures of from 90-95 °C. The temperature range is fairly critical, since sugar degradation may occur at higher temperatures, and at temperatures below 90 °C, filtration becomes difficult because of the sharp increase in viscosity.

Thick Juice Storage. At those factories where evaporator thick juice is placed in storage for later processing, the juice is filtered before going to storage. It is felt that good particle removal helps to assure maintenance of good juice quality during storage. Also, it is desirable to remove solids which would otherwise settle in storage and complicate the handling of storage tanks. For the most part the filtration of thick juice in this operation is the same as that described for standard liquor. The same types of filters are used and most of the same operating procedures are followed. Filter aid is used both for precoating and as a body feed as with standard liquor.

Centrifugal Wash Water. Water used for washing sugar in the centrifugal machines needs to be of the highest quality to avoid contaminating the sugar. Evaporator condensates, city water, or softened well waters are used for washing, and are filtered through small plate and frame or horizontal plate filters. The filters are usually dressed with paper sheets, and are sometimes precoated with filter aid to give added retention of foreign particles. A specially treated filter aid such as Sorbo-Cel has been used to selectively remove small amounts of oil which may have found their way into the water supply. These filters run for extended periods of time, since the condensate source of this water is of high quality.

A closely related operation is the treating of wash water for the rotary drum filters. This water is frequently of lower quality, and also may contain small amounts of oil. A major problem is the clogging of spray nozzles, and filtration is sometimes considered to protect the nozzles. The operation and equipment are similar to those used for centrifugal wash water, and the use of treated filter aid for oil removal would be even more important.

Cold Saccharate Filtration. In the Steffen process, the cold saccharate cake is collected on rotary vacuum filters. Here the principal product desired is the cake, and this is removed from the drum and returned to the carbonation process. The filtrate is collected and sent on to the hot stage of the Steffen process. The filter area needed for the saccharate operation is from 8 to 14 sq ft per ton of molasses worked.

Hot Saccharate Filtration. In the hot Steffen process, the slurry is thickened after heating, before going to the rotary vacuum filters. Here again, the cake is the portion retained and it is washed on the filters to remove soluble impurities. Hot saccharate filtration requires from 1.5 to 1.9 sq ft of filter area per ton of molasses worked.

Some years ago plate and frame or Kelly filters were used for this service, and at that time a filter aid precoat was used. No filter aid is used on the drum filters. Because of the corrosive nature of these slurries, filter media have been a problem, but the newer synthetic fabrics have helped greatly in this respect.

Liquid Sugars. Of increasing interest is the production of liquid sugars for industrial users (see Chapter XV). Since liquid sugars are used directly from tanks and often neither filtered nor inspected at the user's location, they must be completely free of all foreign materials. This calls for at least one, and often two filtrations (Figure 9-31).

After the liquid sugar has been manufactured, it is sometimes treated with activated carbon to improve color. Whether or not it is carbon treated, the solution is filtered to remove turbidity, thermophilic spores, and any scale or other foreign particles which may have been introduced at the factory, in storage, in transportation to the liquid station, in the water used for solution, or from lines along the way.

The filters used for liquid sugar are those in which the most stable filter cakes can be maintained. It is for this reason that horizontal-plate filters are often used.

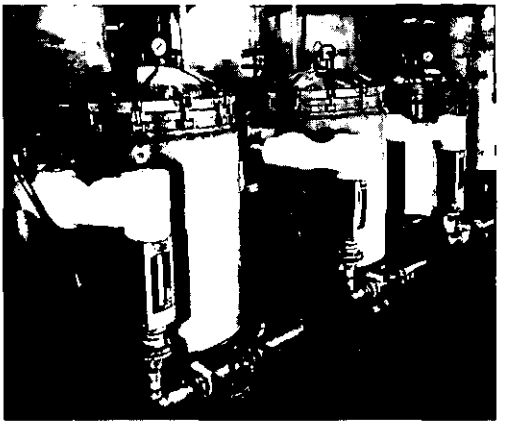

Figure 9-31. Liquid sugar filtration station. Sparkler filters in Brookfield, Ill., plant of The Great Western Sugar Company. *(Courtesy, The Great Western Sugar Company)*

Filters for liquid sugar are usually dressed with filter paper for maximum particle retention, and cake stability. Other types are also used, but with any, the precoat is usually one of the mixtures of cellulose fibers and diatomaceous earth. A body feed of diatomaceous earth is used by mixing it directly into each batch tank of liquid being filtered. This is especially important when activated carbon is used, both to maintain porosity and to assure retention of the carbon in the filter cake.

Filtration is done while the liquid sugar is hot. Again viscosity varies with temperature, but not as greatly in these pure solutions as in standard liquor where many nonsucroses are present.

It is considered advisable in many cases to employ a "final filter" just before the delivery of the liquid sugar. This is of the cartridge type and filter life is extremely long since very little in the way of solids is removed. This is a safety precaution to trap the chance particle of foreign matter which might accidentally have gone into the solution. For maximum protection this unit should be as close as possible to the customer's receiving point. An attractive feature of the cartridge unit is that it can be readily truck-mounted.

Supply Water. There is an increasing concern for adequate supply and quality of the water used by all industrial plants. As it becomes necessary to draw from additional water sources, new treatment facilities will be established. These will be as diverse as the water sources and will include combinations of pretreatment, flocculation and sedimentation, filtration with sand and diatomaceous earth, and chemical treatment. Thus these processes will not be unlike municipal water treatment facilities.

Waste Water. There are continuing moves on the part of various regulatory groups to require industry to return water from process in a pure condition, suitable for immediate reuse. In addition, as water supplies become less available, it is advantageous to reuse water within processing plants to a greater extent.

One of the most important steps in the conditioning of waste water is its clarification. Not only does this assure meeting water standards of turbidity, but at the same time it can bring substantial reduction in both BOD and COD. In this clarification step, several techniques are available. For large quantities of readily

settleable solids, a gravity thickener or clarifier is most practical. After initial clarification, a filter such as a tubular element or pressure leaf filter is used for "polishing" the water with complete removal of solid particles.

The disposal of sludge from a clarifier is a second concern, and usually this is done by passing it over a rotary vacuum filter much as in the carbonation process in the beet factory. When the sludge contains flocculants to aid in settling, filtration properties may be quite poor, and a rotary vacuum precoat filter can best be employed. In either case, the solids from the water end up as a relatively dry filter cake, suitable for land disposal.

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Chapter X

JUICE PURIFICATION IV

A. Juice Storage*

Early in 1944 the Holly Sugar Corporation (9) experimented with storing purified evaporator thick juice for later processing. This early work was very encouraging and was followed by larger and more intensive storage experiments in 1958 and 1959, which proved that carbonated and concentrated beet liquor could be sucessfully stored in quantity for long periods of time without loss or deterioration. The stored juice could also be processed with no more difficulty than with normal day-to-day operation of processing fresh orange juice before storage. The first full-scale installation of a plant utilizing this concept of storing concentrated syrups, such as thick juice, was made in 1960 at Brawley, California (10). The beet end of the 4000 ton Carlton factory was increased in capacity to 6000 tons a day without increasing the size of the sugar end. In 1964 a new factory was constructed by Holly Sugar Corporation at Hereford, Texas, incorporating thick juice storage. Again, part of the thick juice produced by the beet end was stored in large tanks for processing after the normal beet-slicing period.

Due to the high degree of success of the Holly installations, in recent years storage of high-density, purified juices has become popular with many sugar companies, both in the United States and Europe (3,4). This has been either of thick juice or standard liquor. One sugar company in Europe (5) stores high green syrup at one factory and transports it to another factory which has an occasional excess of capacity of the low raw side.

In the past, both The Great Western Sugar Company (7) and the Spreckels Sugar Company (1) had experimented with adding small amounts of lime to diffusion juices, and evaporating the juices for storage. After storage they returned the juice to the plant and carbonated it before concentrating and boiling to sugar. In both cases excessive color formation was found, which caused difficulties in producing sugars of acceptable colors.

Conditions for Successful Juice Storage

In general the storage conditions for concentrated, purified juices are the same as those required for satisfactory storage of liquid sugar. Research carried out by the Holly Sugar Corporation (9) in 1958 and 1959 confirmed that most microorganisms, such as the yeasts and molds normally found in beet sugar juices, will normally not develop or grow in solutions of sugar which are above 67 ° Brix and 8.0 pH. This is not true, however, for unconcentrated thin juices, as these will support growth over a wide range of both temperature and pH. In spite of this normal resistance to microbial attack, all sanitary precautions must be scrupulously observed, since such an infection can start through inadvertent dilution of surfaces by condensed moisture or other mishap, and once started, can have very serious consequences.

Thus, tanks for storage should be clean and well sterilized immediately prior to use, and juice sent to storage should be as clean and sterile as possible. Filtration is necessary, not only to remove spores and organisms not killed by heat, but also particles that might act as nuclei to start crystallization. The density of the juice

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*by Guy Rorabaugh and L.P. Orleans

should be as close to saturation as possible, depending on the purity and storage temperature of the juice. Of course, too high a density or supersaturation, or too low temperatures during storage in cold climates, will result in crystallization during storage. Everything possible must be done to minimize moisture condensation, and the juice should be cooled to as near the ambient storage temperature as possible before being sent to the storage tanks. Storage tanks can be constructed so that a minimum of juice surface is present when the tanks are full. Filtered air circulation should be provided in the storage tanks to prevent condensation on the walls and inside the roof. Several different tank designs and slight modifications of storing purified beet juice have been used. One such method is to use oil on the surface of the juice or liquor to establish a vapor barrier, with no effort to completely fill the tank, and this is described in a later section.

In general, presently the same conditions and practices are followed as were established in the original work (7). During the past 10 years many millions of gallons of thick juice have been stored without any measurable loss or deterioration.

Storage Tank Construction

Tanks used for the storage of thick juice or standard liquor should be constructed, if possible, with a self-supporting roof so that no supporting columns are required, and if structural steel for the roof support is needed, it should be on the exterior of the tank to insure a smooth, unobstructed interior tank surface, to facilitate cleaning and sterilizing. The tank also should have a roof designed to expose a minimum of juice surface to the atmosphere within the tank, as the juice volume decreases due to cooling in storage. One such tank construction (see Figures 10-1 and 10-2) illustrates the ellipsoidal roof construction of the storage tanks used by Holly Sugar Corporation. The cone-shaped roof wth external supporting steel, used at the Bucy-le-Long sugar factory in France, is shown in Figure 10-3. The floor of the tank should be slanted toward the outlet point, to permit complete drainage.

There is a tendency for vapors to escape from the surface of the juice as the tank is being filled. These vapors must be removed from the tank before they can condense on the roof or the exposed walls of the tank and drip onto the surface of the juice. Filtered and sterilized air is used to purge these vapors from the tank. The air is introduced at a central point and exhausted through several outlets near the circumference of the tank. The tank should be equipped with an overflow line and

Figure 10-1. Concentrated juice storage tanks at Hereford, Texas. ***(Courtesy, Holly Sugar Corporation)***

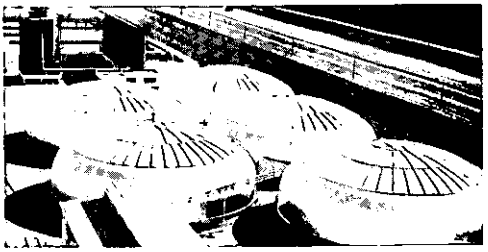

Figure 10-2. Detail of roof of juice storage tanks at Brawley, California. ***(Courtesy, Holly Sugar Corporation)***

should be completely filled with juice to this overflow point. The tanks should also be equipped with sample lines at regularly spaced intervals up the side of the tank. These sample lines should extend at least 12 in. into the tank. Samples should be withdrawn frequently during the storage period to check on the condition of the juice in the tank.

Preparation of Juice for Storage

Thick juice to be taken to storage is removed from the evaporators, and heated to approximately 90 °C, to facilitate the following filtration in pressure filters, to remove insoluble matter and microorganisms. If the filtered thick juice is below the saturation point for the temperature and purity at which the juice will be stored, it must be further concentrated at as low a pressure as possible, to cool the juice, with automatic density control to maintain the desired saturation level.

The concentrated thick juice is pumped to storage, being cooled by passage through a heat exchanger before entering the tank. This heat exchanger also heats the juice as it is removed from the tank and returned to the factory for processing. Care must be taken to prevent cooling the juice below the ambient storage temperature before it is introduced into the storage tank, or there will be problems of excessive crystallization.

Before any juice is pumped into storage the tank should be thoroughly cleaned and sterilized by fogging or cleaning with a suitable bactericide. This process can be continued on a routine schedule as the tank is being filled. The tank should be completely filled, eliminating any void space, or atmosphere, within the tank. Completely filling the tank prevents moisture from condensing on the tank surface and dripping onto the surface of the juice, thus permitting long periods of storage without

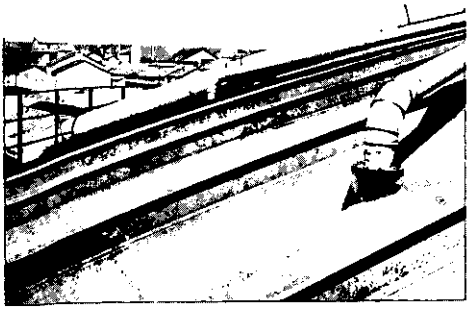

Figure 10-3. Concentrated juice storage tank at Bucy-le-Long factory, France.

deterioration. In the event of juice contraction on cooling, the tank will yield sufficiently without structural damage.

When the juice is required for processing to refined sugar, it is pumped from the storage tanks through a heat exchanger. Heating the juice at the storage tank area will decrease the viscosity and make the juice more easily pumped through the long lines usually connecting the storage area and the factory building. Once the juice is delivered back to the factory building, it is put into the refining process at the normal usage points for the type of juice being stored. The processing of the stored juice is similar to that which was used for the juice at the time it was produced.

Storage Under Oil Barrier

This technique has been used in storing raw cane sugar juices (11), and success has been obtained with it by the Utah-Idaho Sugar Company (7) and The Amalgamated Sugar Company (8). The tanks used in this method are simple, cylindrical, mild-steel units, with roof supported by solid pipe columns welded to the tank floor with continuous welds, so that no syrup can enter the pipes. A layer of mineral oil, 0.25-0.38 in. thick is floated on the syrup surface to provide the vapor barrier. A high-capacity blower equipped with efficient air filters is mounted on the roof at the center, and sufficient air exhaust outlets are welded around the outer roof circumference. Sufficient air circulation is provided in the tank head space, so that the temperatures of the walls and roof never reach the dew point.

The Utah-Idaho Sugar Company stored standard liquor rather than thick juice. In this case it is not necessary to control the density of the juices destined for storage by the installation of a finishing evaporator, nor is an additional set of filters required, since all of the standard liquor is filtered through the standard liquor presses. The portion going to storage is split off, pumped through a flash cooler, the Brix automatically adjusted to 67.5-68.5 ° with thin juice, the pH automatically adjusted to 8.5 with soda ash, and the juice pumped to storage. During two years, this company operated the Steffen process during the stored-syrup processing campaign (see pp. 223-224). The dilute, carbonated saccharate is filtered and the resultant juice pumped to the evaporator first body. Only the first three effects are used. Effects one and two, which evaporate water from the dilute syrup from the Steffen house, supply vapors for the vacuum pans and heaters, and for effect three, which acts as a condenser. The cold standard liquor from storage is pumped to the third effect, where it is heated, and overflows to the high melter. The saccharate syrup leaving the second effect is also pumped to the high melter, or to the high green tank, as required (11).

Advantages of Thick Juice Storage

There are many advantages of storing high-density purified beet liquors.

a) The beet end of a plant can be increased in capacity without expanding the sugar end (6), giving the factory increased slicing capacity without the added cost of increasing the capacity of the sugar end.

b) A factory can produce sugar for longer periods of time, and sugar production is not limited by the availability of beets to slice.

c) Juice storage can provide sugar storage that is both less expensive and, in some ways, safer than bulk sugar storage, and the risk of dust explosions. Sugar production can be planned to fit peak demands and packaging programs (9). These are important in successful marketing programs.

d) Plant capacity and efficiency can be increased, as with purified liquor storage the sugar end and beet end are not as closely dependent. If trouble develops in the beet end, sugar-end operation can be kept at maximum capacity by returning juice from storage. If for some reason capacity is limited in the sugar end, the beet end can continue to operate at full capacity, and the excess juice produced can be diverted to storage.

e) By storing all juice produced and working the sugar end only on stored thick juice, a fully automated and computer-controlled sugar end is possible, receiving as its source of supply a thick juice of even composition.

f) By regulating the amount of thick, purified juice to storage, better control of purity and molasses exhaustion can be maintained in the sugar end, with resulting better factory extraction.

g) Storage of sugar in juice form has been recognized as more economical than storage of refined sugar in bulk or bags. The U.S. Department of Agriculture took note of this fact in 1969 when, after a hearing on the subject, it issued a determination stating that under certain conditions a charge for juice storage could properly be applied to the refined sugar sales expense which is a part of the computation involved in the payment to growers for the beets processed.

Depending on the plant location and its particular circumstances, juice storage can offer many other advantages, some of which are of major economic importance. In the Rocky Mountain area, where beets must be harvested within a very short period of time in the fall, juice storage can reduce piled storage of beets and consequent losses from pile deterioration. Modification of present plants to increase beet-end slicing capacity and storage of the additional thick juice produced for later processing, presents a practical alternative.

Disadvantages of Thick Juice Storage

There are, of course, disadvantages which must be considered, in addition to the installation costs, before installing juice storage. These are such items as a) possible extra costs of sugar-end operation, b) decreased thermal efficiency, c) costs of preparation and maintenance of proper physical conditions of tanks and syrup during the storage period, d) possible higher labor costs, and e) the always present possibility of rapid loss of large quantities of sugar through microbial infection, of which there have been at least two marginal instances.

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B. Ion Exchange

History*

Ion exchange, or deionization, is a technique for removing undesirable impurities from juice, thus increasing extraction and enhancing sugar quality. Nearly all soluble inorganic and many organic compounds are ionized to some degree. In the sugar industry ion exchange is accomplished by passing juices through beds of synthetic ion exchange resins. When properly regenerated in the respective hydrogen and hydroxyl forms, the cation and anion ion exchange resins have the ability to exchange cationic and anionic juice impurities for the hydrogen and hydroxyl ions, respectively.

If salt (NaCl) is assumed as a typical impurity in thin juice, and where R_c and R_a are cation and anion resins, and HR_c and OHR_a are the resins in the hydrogen and hydroxide forms, the following cycles apply.

Cation Resin Service Cycle.

$$Na^+ + Cl^- + HR_c \rightarrow NaR_c + H^+ + Cl^- \quad (1)$$

Anion Resin Service Cycle.

$$H^+ + Cl^- + OHR_a \rightarrow ClR_a + HOH \quad (2)$$

Cation Resin Regeneration Cycle.

$$\underset{\text{Spent resin}}{NaR_c} + \underset{\text{Regenerant}}{H^+} \rightarrow \underset{\text{Regenerated resin}}{HR_c} + \underset{\text{To waste}}{Na^+} \quad (3)$$

Anion Resin Regeneration Cycle.

$$\underset{\text{Spent anion resin}}{ClR_a} + \underset{\text{Regenerant}}{OH^-} \rightarrow \underset{\text{Regenerated resin}}{OHR_a} + \underset{\text{To Waste}}{Cl^-} \quad (4)$$

Thus the typical impurity, NaCl, has been removed from the juice and replaced with water. Many troublesome impurities, including some color bodies, are removed from juices by the use of ion exchange processes.

The principle of ion exchange has been known for over 100 years. A natural sodium aluminum silicate, green sand, has the ability to replace calcium and magnesium in water with sodium, thereby softening water. This material has been used for softening boiler feedwater. Its use on factory juices was of limited value, since calcium and magnesium are replaced with sodium, and elimination of evaporator scaling was the major advantage. In 1935, Adams and Holmes (3a) discovered that certain synthetic resins exhibited ion-exchange properties. Prior to World War II one beet-sugar plant in Michigan installed and operated the process with limited success. After the war, a number of sugar plants, both cane and beet, made pilot-plant tests from which four commercially-sized plants evolved (33,34). By 1950 these plants had all ceased operation because of high costs, primarily due to inefficient regeneration and to resin degradation. After 1950 the resin industry improved resin efficiency and life. Many other industries, such as water treating, organic chemical manufacture, precious metal recovery, and nucleonics, employ the ion exchange process and have contributed much in improving equipment.

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*by J.E. Maudru

At the present time a total of one deionization plant, four Quentin magnesium-exchange plants, and one juice softening unit are functional in the North American beet-sugar industry.

Role of Ion Exchange in the Beet-Sugar Industry

The primary interest of the industry in ion exchange lies in increased extraction, color removal, evaporator scale-prevention, and enhanced sugar quality, all resulting from nonsugar elimination.

In the conventional process the only nonsugar elimination from the juice is accomplished in the beet end. A pound of nonsugar entering the sugar end emerges as a pound of nonsugar in molasses. With 60 purity molasses the elimination of one pound of impurities releases approximately 1.5 pounds of sugar for recovery. Expressed another way:

$$\text{Sugar end extraction} = \frac{S\,(J\text{-}M)}{J\,(S\text{-}M)} \tag{5}$$

where S = sugar purity, J = thick juice purity without remelt, and M = molasses purity.
If S = 100, M = 60

$$\text{Sugar end extraction} = \frac{2.5\ J - 150}{J}$$

At J = 90 Sugar end extraction = 83.3% (Conventional)
J = 96 Sugar end extraction = 93.7% (Ion Exchange).

This is the major return from the use of ion exchange—an increase in thick juice purity without remelt, and the attendant increase in extraction. In a 4000-ton plant, with 15% sugar in beets, such an increase in extraction can increase white sugar production by 1000 bags a day.

Color and ash in sugar-end syrup and in white sugar are reduced, due to removal of ion-exchangeable color bodies. Typical elimination in 100% ion-exchange treatment is: total nonsugar removal, 82%; nitrogen removal, 77%; and ash removal, 98%.

There are several juice and syrup streams in the factory that may serve as a feed for ion exchange and each has advantages and disadvantages.

Diffusion Juice. Ostensibly, ion exchange might eliminate the lime kiln operation with its attendant rock and coke costs (35). Ion-exchange-treated diffusion juice has been the subject of a great amount of study. However, diffusion juice contains dark-colored colloids with isoelectric points at about the pH of cation effluent. The colloid is readily repeptized, and, therefore, is very difficult to process. Another reason for not treating diffusion juice with ion exchangers is that lime-removable impurities may be more cheaply removed with lime.

Thin Juice. This is a good material for ion-exchange feed, and an almost water-white effluent is produced. However, large volumes must be treated because of the low density, and high flow rates, large columns, lines, and valves, are necessary. Evaporator scaling is reduced to a minimum.

Third-Body Evaporator Juice. This is a good feed at about 30° Brix. The volume is about half that of thin juice.

Thick Juice. A partial stream, diluted with thin juice to 30° Brix, is likewise a good feed.

Intermediate Green Syrup. Diluted to 30° Brix with thin juice, and returning the ion-exchange-treated effluent to the evaporator supply tank, this is an excellent material, since volumes and equipment sizes are minimal. Inversion losses are low and nonsugar loading of resins is high, due to the comparatively low sugar:nonsugar ratio.

Although the weights of nonsugars eliminated from the process materials are similar, regardless of the type of feed, other considerations are important. Thin-juice feed, although requiring larger equipment, minimizes evaporator and pan scaling. For maximum economy in plant costs and ease of operation a sugar-end liquor, thick juice, high green, or intermediate green, diluted to about 30° Brix with thin juice, with the effluent returned to the evaporators, makes a very effective system. The exact choice of sugar-end materials depends upon the individual factory and relative sizes of the intermediate and raw equipment. Ion-exchange treatment of a liquor normally employed as a pan feed reduces the amount of massecuite very markedly.

Theory of Ion Exchange

Ion-exchange resins have a certain capacity to remove ionized material from solution, which is stated in terms of millequivalents or kilograins of calcium carbonate per cu ft of resin. A typical resin has a capacity of about 15-25 kilograins per cu ft. A cu ft of resin is measured after backwashing settling. A volume of resin in this state is about 40% voids.

Synthetic ion-exchange resins are cross-linked or polymerized to varying degrees. Normally the degree of cross-linking and the capacity are directly related: e.g., the more cross-linking, the higher the capacity, and the higher the physical stability. The resin must be selected for the specific purpose desired.

Ion-exchange resins are gel-like particles with diffusion capabilities, but actually follow the crystalline mode, in that the molecules are ionized within the gel. The gel particle may be said to have many passages through it. Along each passage active molecular ends protrude which cause the activity of the granule.

Listed below are typical groupings for cation resins:

$$\text{R-SO}_2\text{(H)} \quad = \text{Sulfonic grouping}$$

$$\text{R-}\overset{\overset{\text{O}}{\parallel}}{\text{C}}\text{—O(H)} \quad = \text{Carboxylic grouping}$$

$$\text{R-O(H)} \quad = \text{Phenolic grouping}$$

H in each case represents the H^+ ion which is replaceable by other cations.

The sulfonic type of cation resins is normally employed in beet-juice exchange.

The mechanism of anion resin action is not as well understood as the cation resin exchange reaction. Considering hydrochloric acid as the material to be adsorbed, either of two reactions may occur.

$$\text{RNH}_2 + \text{HCl} \rightarrow \text{RNH}_3\text{Cl} \qquad (6)$$

$$\text{RNH}_2 + \text{OH}^- + \text{Cl}^- \rightarrow \text{RNH}_3\text{Cl} + \text{HOH} \qquad (7)$$

Reaction (6) is adsorption, while (7) is true exchange.

Ion-exchange resins react with anions and cations in a selective manner. Thus, molecules having low molecular weights, and in the case of elemental ions, the lowest atomic numbers, are most loosely held.

Resins and Resin Development*

Synthetic ion-exchange resins are most attractive, as they have relatively high operating capacities, combined with chemical and physical stability for many service cycles. Present costs are dependent on resin type, durability, and capacity.

Synthetic ion-exchange resins are produced by the formation of a water-insoluble polymer into which active acidic or basic groups are introduced by chemical reaction. The synthesized polymer must have an ion-permeable, gel-like, structure, or a macroporous structure in order for ion exchange to take place within the polymer matrix as well as on the surface of the solid. Many synthetic ion exchangers are based on a matrix consisting of styrene and small amounts of divinyl benzene. The divinyl benzene, cross-linked polystyrene co-polymer matrix is converted to a strongly acidic cation exchanger by sulfonation, and to a strongly basic anion exchanger by chloromethylation and amination (14). Figure 10-4 indicates the conventional synthesis of a strong cation, or sulfonic ion exchanger, and a strongly basic, or quaternary amine anion exchanger. Synthetic ion exchangers are also based upon phenol-formaldehyde condensates and epoxy-polyamine condensates (3).

Types of Ion-exchange Resins

Synthetic resins are classified as cation or anion exchange resins, and subclassifications of these include weak and strong cation and anion resins.
The active ion-exchange groups associated with these subclassifications and their respective acidic or basic components are indicated in Table 10-1. The acidic or

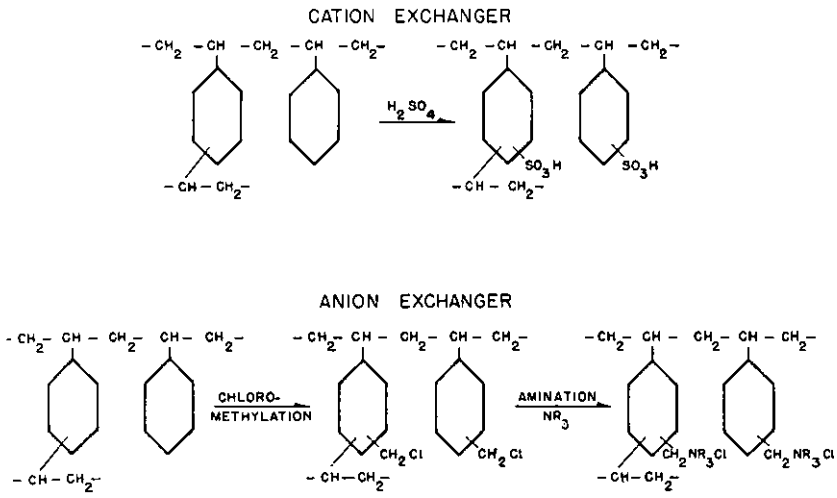

Figure 10-4. Synthesis of cation and anion exchange resins (14).

*by S.E. Bichsel

Table 10-1 Chemical and Physical Classification of Ion Exchange Resins

Chemical Classification	Physical Classification	Active Group	P*	Typical Configuration
Strong Acid	Solid—Chip or Bead	Sulfonic Acid	1	
Weak Acid	Solid—Chip or Bead	Carboxylic Acid	4-5	
Weak Acid	Solid—Bead	Phosphonic Acid	2-3	
Strong Base	Solid—Bead	Quaternary Ammonium	1	
Weak Base	Solid—Chip or Bead	Secondary Amine	6-9	
Weak Base	Solid—Chip or Bead	Tertiary Amine (Aromatic Matrix)	3-5	
Weak Base	Solid—Chip or Bead	Tertiary Amine (Aliphatic Matrix)	3-5	

*P = — log of dissociation constants of acids and bases

basic exponents, P, are calculated as the negative logarithms of the dissociation constants of the acids and bases in question. Figure 10-5 shows the typical titration curve of a strong sulfonic acid resin, IR-120, and a weak carboxylic acid cation resin, IRC-50 (6). Figure 10-6 shows the titration curves of a strongly basic quaternary amine, IRA-400, and a weakly basic secondary and tertiary amine, IR-4B, (6). Strong cation exchangers are capable of adsorbing weakly basic ions such as amino acids and betaine. The neutral salts, sodium and potassium chloride, are converted to hydrochloric acid due to strong cation exchange of hydrogen for sodium and potassium ions. The titration curve for a strong cation resin indicates that ion exchange may take place over a pH range from 0 to 14 pH, while the weakly acidic resins have effective operating ranges from 5 to 14 pH. Weak cation resins are generally capable of splitting multivalent cations of alkaline salts. Strongly acidic cation exchangers, by and large, are completely regenerated with 300% to 400% and weakly acidic cation resins with 110 to 115% of the theoretical acid requirements. The titration curve of the strong anion resin indicates an ability to adsorb weak acids and split neutral salts. The effective strong anion-resin operating range is from

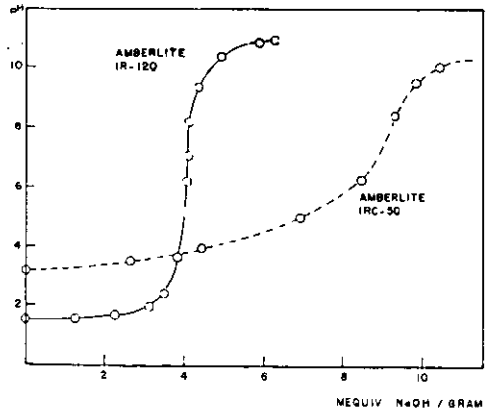

Figure 10-5. Titration curves of cation-exchange resins (6).

0 to 14 pH. The weak anion exchange resin operates effectively from 0 to 9 pH. The weak anion resin is capable of adsorbing strong acids from solution. It is general practice in the beet-sugar industry to follow treatment with a strong cation resin by treatment with a weak anion resin to adsorb the strong acids generated by the salt-splitting capacity of the former. Strong anion resins require 300% to 400% and weakly basic resins 110 to 115% of the theoretical base requirements for complete regeneration. Weakly basic resins may be regenerated with a strong base such as sodium hydroxide, or a relatively weak base such as ammonium hydroxide. Strong base anion exchanger regeneration can only be done with strong bases.

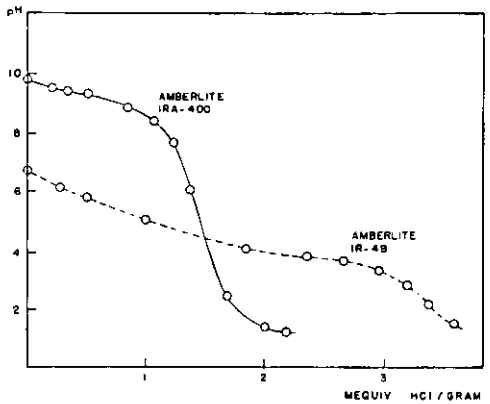

Figure 10-6. Titration curves of anion-exchange resins (6).

In relatively dilute aqueous solutions the affinity of ions for ion exchangers may be described in terms of the lyotropic [salting-out efficiency] series based on increasing valence and atomic number.

Strongly Basic Anion Exchangers. The order of affinity is sulfate > bisulfate > nitrate > nitrite > chloride > bicarbonate > fluoride > hydroxide.

Weakly Basic Anion Exchangers. The order of affinity is hydroxyl > aromatic sulfonic acids > citric > chromic > sulfuric > tartaric > oxalic > phosphoric > arsenic > molybdic > nitric > hydriodic > hydrobromic > thiocyanic > hydrochloric > hydrofluoric > formic > acetic > carbonic.

Strongly Acidic Cation Exchangers. The order of affinity is barium > lead > calcium > nickel > copper > iron > magnesium > potassium > ammonium > sodium > hydrogen.

Weakly Acidic Cation Exchangers. The order of affinity is hydrogen > barium > calcium > magnesium > sodium > potassium. Affinity data for weakly acidic cation exchangers is limited.

Physical Stability

The physical stability of the ion exchanger resin bead or chip is affected by mechanical attrition, chemical oxidation, osmotic shock, thermal shock and microorganism growth within the resin matrix. Mechanical attrition may be significant in continuous ion-exchange systems where resins are subjected to continual physical transfer. Fixed-bed ion-exchange units do not generally have resin loss problems from mechanical attrition. Oxidative resin de-cross-linking has not been a great problem in treating clarified process juices. Treatment of juices or liquors downstream of sulfitation insures that the resin is exposed to a reducing environ-

ment. Cation resins with percentages of cross-linking as low as 5% have been used successfully in deionization service on cold-sulfited feed juice (7). Osmotic shock induced by exposure of the resin to wide variations in electrolyte or sucrose concentration can cause bead or chip fragmentation due to contraction and expansion. Thermal shock may also produce fragmentation. Physical losses of strongly acidic cation exchangers range from 1 to 5% per 130-day deionization campaign. Weak anion-resin, osmotic-shock losses range from 15 to 25% per 130-day deionization campaign (9). Resin loss due to osmotic shock is a function of the resin type—gel or macroporous, the number of resin service cycles, the ionic strength of the stripping solution, the Brix change during water displacement, the temperature of the feed juice, and the ratio of the fixed resin column diameter to the bed depth. Thermal shock fragmentation is caused by high temperature differences between the resin and the juice feed to the column, regenerants, and rinse water. Microorganism growth in ion exchange resins, due to trace amounts of sugar or other organics remaining in the matrix of the ion-exchange resin, can fragment the resin or reduce the operating capacity of the resin. Microorganism infection occurs primarily during intercampaign storage periods. Storage of anion resin in the chloride form and cation resin in the sodium form, in a 5% salt—0.5% formaldehyde solution eliminates the possibility of such attack.

Resin Fouling

The long-term operating capacity of cation and anion resins is related to the resin classification, the amount and type of feed juice or liquor impurities, the resin stripping and regeneration procedures, and the operating temperatures. Under normal deionization service conditions cation resin capacity declines are associated with precipitation of calcium sulfate within the resin matrix. This fouling effect is avoided if proper sulfuric acid regeneration procedures are observed (30).

By far the most critical capacity decrease problem encountered in treating sugar beet juices and liquors is organic fouling of weak and strong anion resins. Irreversible fouling can usually be traced to sorption of high-molecular-weight organic acid or high-molecular-polymeric type molecules with polyfunctional groups (1). Melanoidin color bodies in beet juices have long been suspect. Irreversible fouling causes decreased total capacity, decreased operating capacity to a given pH breakthrough, extremely sensitive operating capacity—feed-flow rate relationships, and high rinse requirements to reach a specific conductivity after caustic regeneration. Oxidation of secondary or tertiary weak-anion, amine groups results in a weakly acidic exchange group. By the same token, incorporation of a macromolecular foulent into the weak anion resin matrix may also give the weak anion resin weakly acidic capacity, with the resultant alkali retention, and the long rinse requirements after caustic regeneration. Regeneration of weak anions with ammonia characteristically reduces high weak anion rinse requirements. (15).

New Resin Types

Resin developments of significance include two-phase, macroporous-structure resins, which are generally more resistant to osmotic-shock fracturing and oxidative de-cross-linking. These advantages are partially negated by lower specific gravity, higher expansion volumes, lower capacity, and slightly higher costs when compared with competitive gel-type resins. In some industrial applications where severe chemical and physical conditions exist, only macroporous resins can be used. In sugar treatment the catalytic inversion rate of a macroporous resin is usually significantly less than those of competitive gel resins under the same conditions (39).

A new type of weakly acidic cation resin based on an acrylic matrix with a gel or macroporous structure, is capable of eliminating significant amounts of calcium, sodium, and potassium from sugarbeet process juice and liquor streams (2). These resins, utilized in the hydrogen form, offer the advantages of high total capacity, regeneration with only 110% of theoretical acid requirements, and negligible sucrose inversion in the 15°-20°C deionization service ranges. Disadvantages are high-swelling values—65% from the hydrogen to the sodium form—high leakage sensitivity to feed-flow rate variations, difficulty in using over 50% of the total resin capacity due to high sodium and potassium leakage, and high initial cost. In the area of polymeric and resinous sorbents several new materials have been produced, which are based on cross-linked polystyrene polymers, characterized by uniformly macroporous porosity (49), and on highly porous hydrophilic resins with phenolic hydroxyl and methylol groups for hydrophilic properties. Generally these synthetic sorbents are most effective in eliminating sugar-juice color bodies of high molecular weight, of the caramel or melanoidin type (13). When used in conjunction with activated carbon, the resinous adsorbents are most effective as the primary color-removing units. Activated carbon may be used as a "polisher." Regeneration of resinous sorbents is accomplished with a dilute solution of sodium hydroxide and a dilute sulfuric acid rinse to pH 4.

Equipment and Equipment Development

Proper design and sizing of ion-exchange equipment is essential for efficient and profitable ion-exchange plant operation. Deionization of beet-sugar juices differs significantly from water deionization. Impurity loadings are much higher, most systems operate at an economically controlled leakage, and provisions must be made to cool the juice to minimize catalytic inversion. Service cycles are comparatively short, and dilution from column sweetening-desweetening ("sweetening-on," "sweetening off") can become an economic problem. Maximum utilization of the costly acid and base regenerants is very important. These problems can be minimized effectively by proper design (30).

A standard, fixed-bed, ion-exchange column consists of a column container, internal distributors for the liquid entering or leaving, a resin-bed support, usually of graded quartz gravel, and adequate empty space or freeboard above the resin bed so the resin can double its volume when backwashed. Figures 10-7(a), (b), (c) indicate standard ion-exchange equipment, both fixed and continuous bed, in use at present. Ion-exchange columns, transfer lines, and valves are usually food-grade, rubber-coated, or lined with polypropylene to protect against corrosive acidic, basic, or salt solutions. In addition to columns, transfer lines, and on-off valves, an ion-exchange plant must have provisions for liquid-flow control, density control, pH and conductivity read-out, timed-cycle control, and feed-juice, temperature control. Absolute control of column-juice feed, volume, Brix, purity, and temperature is a necessity in juice deionization. Similarly regenerant concentration, flow rate, and time of use must be controlled if maximum economic usage is to be assured. In most cases, corrosion-resistant storage tanks lined with epoxy or rubber must be provided for once-used regenerants and cation-stripping solutions. Bulk-regenerant storage tanks are usually extra-thickness mild steel for concentrated sulfuric acid; stainless steel, high-pressure, rail- or truck tankers for anhydrous ammonia; rubber-lined tanks for concentrated sodium hydroxide; and mild steel for 24-25% aqua ammonia tanks.

Automated control is necessary in a modern ion-exchange plant. Utilization of proper instrumentation, alarm systems, and visual readouts can reduce labor re-

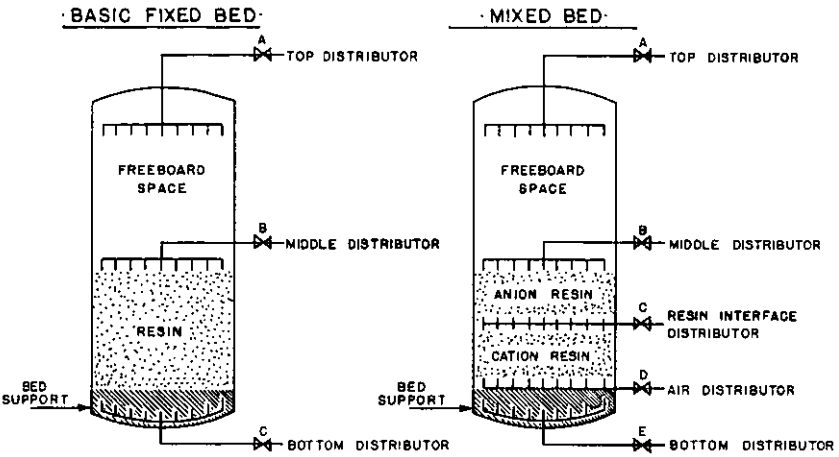

Figure 10-7a. Basic fixed- and mixed-bed ion exchange equipment.

quirements in the most complex deionization plant to one operator per shift. In addition automation contributes to optimum plant performance. Figure 10-7(a) shows a flow diagram for basic fixed, and mixed-bed ion-exchange columns.

Fixed Beds

In a basic fixed-bed system, process juice is admitted at constant flow rate, density and temperature through valve B. The juice is discharged through valve C after percolating uniformly through the fixed resin column, and the inert, resin-support bed. Regeneration and resin-bed rinse and desweetening liquids are carried out in the same manner—in through valve B and downflow out valve C. During resin backwash, water flows in valve C at a controlled rate and out of valve A. The resin bed is expanded to twice its fixed volume.

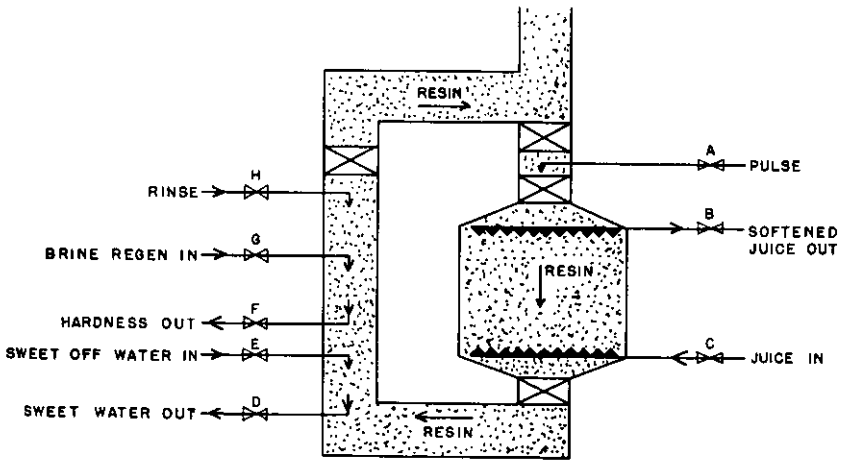

Figure 10-7b. Continuous-loop type, moving-bed, ion exchange contactor.

Fixed, Mixed-Resin Beds

The mixed-resin, fixed bed differs from the basic fixed bed in that it contains both cation and anion resins, which, by virtue of different specific gravity values, can be hydraulically separated during backwash. Separation of the anion and cation resins is necessary for regeneration. A basic regenerant and an acidic regenerant flow downwards through valve B, and upflow through valve E, respectively. Waste regenerants are combined and flow out the resin interface distributor through valve C. The rinsed, regenerated resins are then mixed by admitting air through the sparger distributor valve B. The thoroughly-mixed anion and cation resins are then ready for the downflow service cycle.

Continuous Beds, Loop-Type

Figure 10-7(b) shows a simplified schematic flow diagram for a continuous loop-type contactor (11,12,14) and Figure 10-7(c) a continuous, countercurrent ion exchanger (5,37,55) operating in the thin-juice softening mode. Both these continuous systems operate counterflow—juice, rinse water, or brine regenerant flowing continuously in one direction, and the resin moving in the counter direction.

The continuous, loop-type, moving-bed contactor consists of a hydraulic-pulse chamber, a service section, and desweetening, brine regeneration, and resin-regenerant rinse sections. The pulsing action responsible for periodic resin movement requires 5-10 seconds and resin pulses may occur every 2-15 minutes. The resin movement displaces a length of 6 to 18 inches. Juice enters valve C and percolates counterflow, through the resin bed and out valve B. Rinse, brine regenerant, and desweetening-water function in the same manner.

Continuous, Countercurrent Beds

The continuous, countercurrent ion-exchange process consists of a service column, a rinse column with attached resin hopper, a desweetening column and a regeneration column. Juice to be treated enters the service column through valve H,

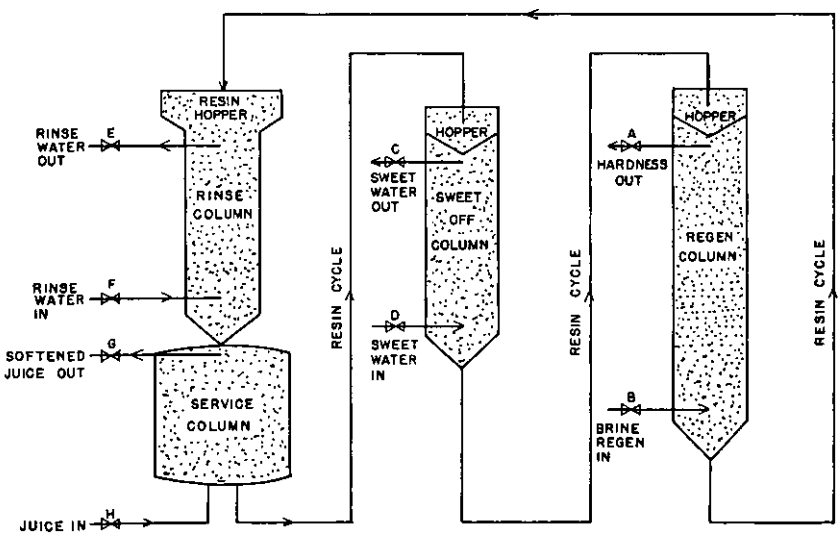

Figure 10-7c. Continuous, countercurrent ion exchange process.

and percolates upward through the resin bed. The softened juice is discharged through valve G. At time intervals dependent on the juice-impurity load, a slug of resin is admitted to the service column from the resin hopper feeding the rinse column. Simultaneously, exhausted resin is discharged downflow from the service column, through the resin-cycle line into the desweetening column. Desweetening water is admitted on a continuous basis through valve D and out valve C. The resin is transferred to the regeneration column, and regenerated in a counterflow manner with brine through valve B. Hardness ions are discharged through valve A. The brine-regenerated resin is transferred through the resin-recycle line to the resin-rinse column, where residual brine and hardness is rinsed from the resin countercurrently. Rinse water enters through valve F and leaves through valve E. The regenerated, rinsed resin is transferred to the service column and the cycle repeats itself. Several other continuous systems such as the *CCIX* and the *Vat-type* countercurrent contactor all rely on countercurrent flow to increase operating efficiency (4,23).

Comparison of Fixed and Continuous Beds

The following economic advantages are claimed in comparing continuous ion-exchange systems with conventional fixed-bed units (4,31): a) lower capital costs, 25-35% less than a conventional fixed bed; b) decreased use of regenerant chemicals, resulting in lower operating costs; c) lower resin inventories; d) higher effluent purity and more uniform effluent quality; e) more efficient usage of regenerants resulting in less waste disposal; f) greater operational flexibility; g) significantly less treated juice dilution, and catalytic sucrose inversion in the case of deionization; h) less maintenance and lower labor costs; and i) space savings of approximately 20-30%. These claims have yet to be realized in the beet-sugar industry.

It should be pointed out that continuous countercurrent units have operating problems in the following areas: premature failure of resin-transfer valves; incomplete resin exhaustion, regeneration and rinsing, and high resin-attrition rates due to continual movement in the resin circuit. Economic comparisons of fixed-bed versus continuous ion-exchange equipment have usually ignored the basic advances which have increased fixed-bed operating efficiency (35). Such advances include counterflow regeneration to decrease impurity leakage in strong cation and anion fixed-bed columns: "merry-go-round" anion and cation-column operating flows to insure complete resin exhaustion, and weak cation or weak anion resins overlaying strong cation and anion resins. This arrangement extends the strong cation and anion-bed cycles and can reduce regenerant requirements by more efficient use.

Operational Procedures

Precampaign Preparation

The success or failure of an ion-exchange plant start-up is dependent on adequate intercampaign maintenance of instruments, valves, pumps, juice-cooling units, and other auxiliary equipment. Initial prestart-up efforts should be concerned with rinsing the intercampaign preservative solution from the resin beds, and thoroughly backwashing all beds. Juice flow through the entire ion-exchange system should be simulated with water several days before beet slicing starts. All control instruments and flow-control valves must be checked for calibration. Particular attention should be directed to accurate volume calibration of the meters measuring juice into and out of the ion-exchange plant. Errors in either of these matters make ac-

curate material balances difficult. Automatic cycle-control timers should be cycled on an accelerated basis to completely check for proper functioning of all automation and control-panel circuitry. Start-up inventories of acid, base, and brine should be measured. Delivery schedules with regenerant suppliers must be confirmed. Alternate means of regenerant delivery should be established in case of truck or rail strikes. The water testout period serves not only to test the equipment but to reacquaint operators with the operating cycles and procedures.

Cooling to Avoid Inversion

Adequate cooling of deionization plant feed juice is necessary to minimize heterogeneous and homogeneous catalytic sucrose inversion. Heterogeneous inversion is induced by sucrose contact with sulfonic acid groups on a regenerated, strong cation exchanger. Homogeneous inversion is caused by molecular contact of sucrose with hydrogen ions in solution. Hydrogen ions are released by the strong cation resin, in exchange for cationic juice impurities in the process-juice stream. Heterogeneous inversion and homogeneous inversion rates are influenced by the temperature of the juice. Figure 10-8 shows the effects of pH and temperature on the homogeneous inversion of sucrose (27). Figure 10-9 shows the sum of homogeneous and heterogeneous inversion expressed as percent of the total sugar treated (8). This plant data was obtained at three different juice temperature values. The decrease in inversion values as bed volumes increase, clearly indicates the diminishing effect of heterogeneous catalytic inversion as the strong acid resin, Duolite C-25-D, becomes exhausted. The treated juice was an 80-82% purity, 30° Brix mixture of intermediate green syrup and thin juice. The flow rate approximated 0.20 gal/cu ft resin/min. Normal total inversion losses for a deionization

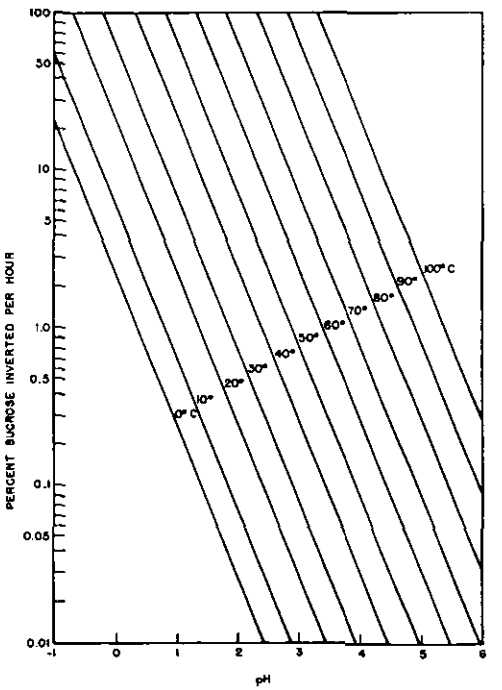

Figure 10-8. Homogeneous inversion (data of Sadtar).

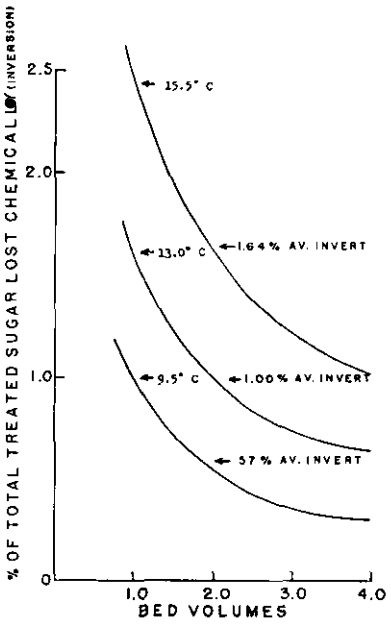

Figure 10-9. Homogeneous and heterogeneous sucrose inversion in ion-exchange beds (8).

plant operating with juice cooled to about 10 °C are from 0.3 to 0.6% of the total sugar treated. These values increase or decrease in relation to a) the resin percentage of divinyl benzene cross-linkage; b) the regeneration level of the resin; c) the average juice-resin contact time; and d) the juice temperature, degrees Brix, and purity. These conditions must be considered when ion-exchange plant, juice-cooling capacity is under study.

Juice cooling is generally accomplished in one of three ways. a) Juice is initially cooled by passing through a high-efficiency, plate-type heat exchanger, counterflow, to cool deionized juice. Further juice cooling is accomplished by passing the juice through a compressor-type mechanical refrigeration unit of the kind used to chill brine continuously. b) Juice is initially cooled as mentioned in a) and final cooling to the desired temperature is accomplished by passing juice through a high-efficiency, plate-type heat exchanger, counterflow to chilled brine or ethylene glycol solution, recirculating through a mechanical refrigeration unit. c) Juice is initially cooled as in a) and chilled to the final temperature by vacuum-induced flash evaporation. Vacuum is produced by steam jet evacuators. Juice cooling by methods a) and b) require large volumes of water, preferably below 23 °C to cool the mechanical refrigeration units. Power requirements to drive the mechanical refrigeration units are significant. Juice cooling by method c) requires a substantial amount of high pressure steam. The judicious selection of juice cooling equipment will vary according to the availability of large quantities of cold water, industrial power rates, and high-pressure steam.

Operating Cycles, Sweetening

Operation of the ion-exchange plant begins with the *sweetening cycle*. Cooled juice flows through the middle distributor, located several inches above the fixed,

cation-resin bed. The juice level within the column dome is controlled by air pressure, or by a noncompressible water dome. In the case of the latter, since the sugar-bearing juice has a higher density, it forms a juice layer below the water dome. Water-juice mixing is minimized by low-turbulence distributor outlets. Water is displaced during desweetening as indicated in Figure 10-10, curve B or A (32). Perfect displacement does not occur, as in curve C, due to imperfect sweetening-juice distribution, juice-water backmixing, and nonequilibrium, water-juice diffusion conditions within the resin bed. This nonequilibrium, tailing effect is particularly apparent after the first bed volume. The time, in terms of bed volumes of sweetening juice, to reach diffusion equilibrium or constant effluent Brix, is decreased by 50% with a juice-temperature increase of 10 °C, or a flow-rate decrease from 1.0 to 0.5 gpm/cu ft. During the sweetening cycle, water is generally diverted to the drain until a 1.0 ° Brix effluent is indicated. This is shown by a sharp increase in the cation-column effluent conductivity, due to effluent breakthrough. A Brix spindle may also be used to determine the proper sweetening, cutoff-Brix point. The 1.0 ° Brix cation effluent is diverted to the regenerated, weak-anion column, and the anion-sweetening cycle continues until a 1.0 ° Brix anion effluent occurs. Conductivity, Brix spindle, or the measurement of the volume of anion-sweetening effluent may be used to determine the exact time required to reach a 1.0 ° Brix anion column effluent. At this time the alkaline, pH 9.0-9.5, deionized juice is diverted to the factory juice evaporators, or ion-exchange effluent concentrators.

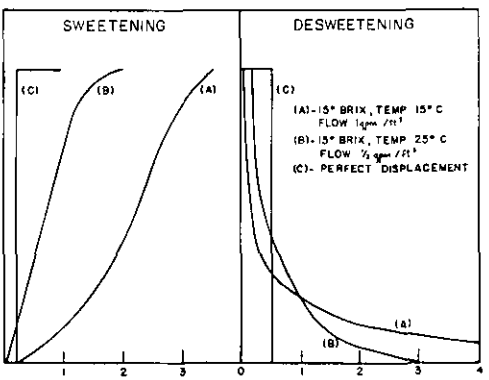

Figure 10-10. Ion exchanger bed displacement curves (plant data).

Service Cycle

During the service cycle the cation-column, effluent pH is 1.5 to 1.7, and the anion effluent approximates 9.0-9.5 pH. The duration of the service cycle is dependent upon the type and amount of impurity loading/min/cu ft of anion and cation resin, and the regeneration level of the strong-cation resin. It is assumed that the weak-anion resin is completely regenerated. The economics of strongly acidic cation resin regeneration are generally most favorable at a level of 70 to 80% of the total resin-column capacity. When the paired cation-anion column pair process scheme is used, cation-anion resin volumes must be balanced so that both columns are exhausted at approximately the same time. This process scheme requires careful maintenance of cation-anion resin volume ratios and cation regeneration levels.

Recent trends in fixed-bed ion exchange technology favor operating a primary cation-anion resin pair in series with a secondary cation-anion pair (29). The

primary cation-anion pair is operated to complete exhaustion, and rotated out of service to the desweetening cycle. The secondary cation-anion pair is rotated to the primary juice receiving position. The secondary cation-anion pair is replaced by a regenerated cation-anion pair. Three cation-anion pairs are rotated in a merry-go-round fashion from standby—to primary—to secondary—to desweetening, backwash, and regeneration—and to standby. This process scheme uses cation and anion capacities completely.

Another scheme operates the cation and anion column in an independent mode (7). Three strong-cation columns are operated with one regenerated column in service. When the service cation column reaches exhaustion it is desweetened into the next regenerated cation cell in standby position. The regenerated cation cell in new service temporarily receives the desweetening flow from the exhausted cation unit, and the service juice flow. This operating procedure, in conjunction with counterflow cation regeneration, insures complete utilization of cation capacity with minimum impurity unloading or leakage. The cation-column, effluent juice flows through two, weak-anion columns in series, in primary and secondary positions. When the primary weak anion effluent juice pH or conductivity approximates the decationized primary anion feed value, the partially exhausted secondary anion exchange cell is rotated in a merry-go-round fashion to the primary position. Predicating secondary anion rotation to the primary position on the basis of total primary anion exhaustion eliminates nonsucrose leakage associated with rotating the secondary anion on the basis of pH decline. The completely exhausted, primary, weak-anion cell is rotated from the service position to the desweetening cycle. A regenerated, weak-anion column in standby position is rotated to the secondary service position. This operating scheme insures complete exhaustion of cation and anion resins and eliminates interdependence of the cation and anion service columns.

Strong-cation operating-capacity breakthrough is most easily monitored by observing the decreasing cation-effluent conductivity. Weak-anion capacity breakthrough is easily determined by anion effluent pH decrease. Table 10-2 shows deionization plant nonsucrose and color elimination values. The juice treated was a 30° Brix, 80-82 purity, mixture of intermediate green syrup and thin juice (7).

Table 10-2 Ion Exchange Nonsucrose Elimination (7)

	Influent			Effluent			Percent Nonsugars Eliminated		
Run[1]	1	2	3	1	2	3	1	2	3
Purity A.P.	81.6	80.8	81.0	95.1	94.0	93.6	-----	-----	-----
N.S./100 Sugar	22.54	23.76	23.36	5.15	6.38	6.84	77.2	73.2	70.8
Organic (N)/100 Sugar	1.27	1.38	1.17	.29	.30	.29	77.2	73.2	75.2
Total Amino (N)/100 Sug.[2]	.31	.39	.24	.06	.06	.06	80.7	79.5	75.0
Betaine (N)/100 Sugar	.36	.36	.39	.15	.16	.14	58.3	55.6	64.1
Unk. Organic (N)/100 Sug.	.60	.63	.54	.08	.06	.09	86.7	90.5	83.3
Calcium/100 Sugar	.08	.08	.09	0	0	0	100.0	100.0	100.0
Sodium/100 Sugar	.97	1.00	.89	.05	.04	.03	94.9	96.0	96.6
Potassium/100 Sugar	1.49	1.55	1.59	.03	.04	.03	98.0	97.4	98.1
Chloride/100 Sugar	1.01	.95	.90	.01	.01	.01	99.0	99.0	99.0
Color Index[3]	2523	2420	2348	51	49	64	98	98	97
pH	8.9	8.7	8.8	7.2	7.1	7.3	-----	-----	-----

[1]Eight Hour Composite
[2]Included Amino Acid (N) Plus PCA (N)
[3]ICUMSA color method at 560 nm.

Desweetening

Termination of the anion-cation column service cycle is followed by the desweetening cycle. Figure 10-10 indicates that perfect displacement of the juice with water is not possible. As noted in the discussion of sweetening procedures, increased sweetening juice temperature and decreased flow rates reduce the amount of desweetening water or sweetening juice needed to reach a 1.0° Brix column effluent. It is general practice to reclaim the last fraction of the desweetening effluent ranging from 5 to 1.0° Brix, and averaging 2° Brix, for use in place of water during the initial part of the next desweetening cycle. This technique can reduce total ion-exchange dilution by 30%. An economical compromise must be established when considering dilution from sweetening, desweetening, and the percent of total sugar physically lost.

Figure 10-11 defines the operating parameters of percent total dilution, percent total physical sugar lost, and the percent sugar or Brix of the sweetening effluent—desweetening cutoff point (7). Dilution is calculated from plant data. Total dilution is expressed as the percentage difference in (tons of water/day out of the ion exchange plant) - (tons of water/day into the ion exchange plant). No correction has been made for water formed due to reaction of H^+ and OH^-. As an example, assume a sweetening cutoff point of 1° Brix, or 1% sugar. This corresponds to a total dilution of approximately 100%. If sweetwater recycle is employed, the dilution decreases to 70%. Further decrease in dilution may only be accomplished at the expense of increases in sugar losses.

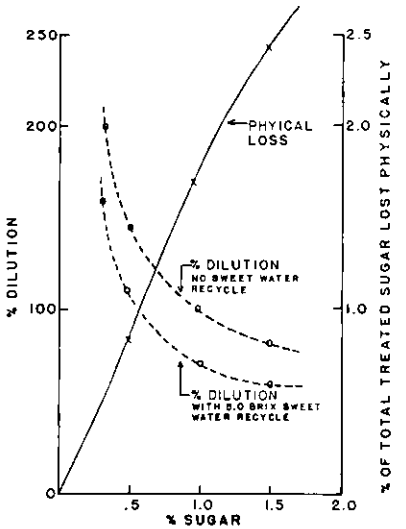

Figure 10-11. Ion exchanger dilution and percent total sugar loss, as a function of the Brix of the effluent at the cutoff point (7).

Backwashing

The desweetening cycle is followed by the backwashing cycle of the anion and cation resin columns. Raw water or recycled backwash water may be used to backwash cation or anion resins. If recycled backwash water is used as a means of minimizing water usage and subsequent discharge, the recycled backwash water must be free from resin fines as a result of settling or filtration. In addition, recycled backwash water must be dosed periodically with an FDA-EPA approved biocide to

minimize the possibility of bacterial infection. Periodic blowdown of recycled backwash water or continual bleed-off of recycled backwash water with equal volume of raw water makeup is a requirement for successful reuse of backwash water. Backwash water is introduced through the bottom distributor, with sufficient velocity to expand the resin bed 100%. Backwashing accomplishes the following objectives: a) gas bubbles, particulate matter, and resin fines produced by osmotic shock are eliminated from the resin bed; b) the resin is classified with respect to particle or bead size. Resin classification prevents regenerant and juice channeling through the resin bed. Fines are separated from the resin and discharged to waste through the top column distributor. Figure 10-12 shows the percentage of bed expansion, versus the backwash rate in gpm/sq ft of bed area (17). The graph shows the resin-bed expansion is a function of resin-mesh size, resin form, and resin type, as well as backwash flow rate/sq ft of bed area. DOWEX-1, and 21K are strong anion-exchange resins, and DOWEX-50 is a conventional strong anion resin. Backwash water temperature also has a great effect on the percentage of bed expansion. A decrease in the water temperature from 30° to 20 °C can increase bed expansion by 20%. The duration of the backwash cycle usually does not exceed 25 minutes. A backwash cycle is terminated on a time basis or by visual examination of the backwash water clarity. When backwashing is complete, the resin bed is allowed to settle. A short, fast downflow rinse insures resin-bed compaction in preparation for the regeneration cycle.

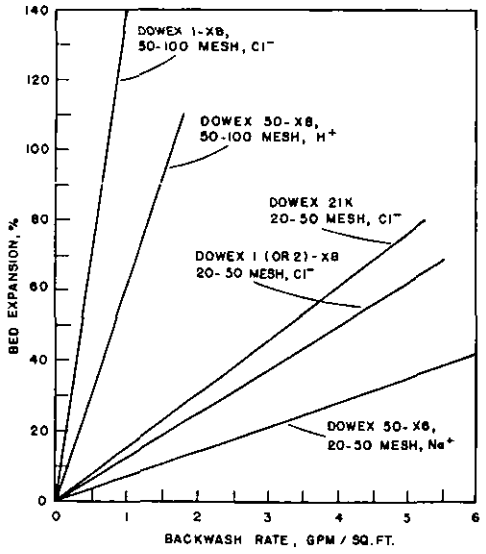

Figure 10-12. Backwash characteristics at various flow rates, 25°C (17). (Courtesy, Dow Chemical Company.)

Weak-Anion Resin Regeneration

Weak-anion resin regeneration is customarily accomplished with ammonium hydroxide, ranging from 3 to 6% ammonia concentration. The dilute aqua ammonia solution is produced by diluting 24% to 25% aqua ammonia with decationized or softened water. Downflow regeneration rates may vary from 0.25 to 0.50 gal/cu ft/min—the higher the regenerant concentration, the lower the flow rate.

Regenerant-resin contact time must be adequate for maximum utilization of regenerant (51). In some cases it is advantageous to inject concentrated sodium chloride brine into the ammonium hydroxide regenerant stream, to form a 5% solution with respect to sodium chloride. Plant tests indicate that brine plus amonium hydroxide regenerant effectively strips sorbed color bodies from weak-anion resins (8). The combination of regeneration and simultaneous anion-resin stripping retards weak-anion fouling. Weak-anion regeneration with aqua ammonia equivalent to 110 to 115% of the theoretical resin base capacity is sufficient for complete weak-anion regeneration.

The weak-anion regenerant waste stream, consisting of ammonium salts of organic and inorganic anionic impurities from the juice, may be quantitatively reclaimed for use as a strong cation-resin stripping agent prior to cation acid regeneration. Weak-anion regenerant rinse must be carried out with decationized or soft water, due to possible magnesium and calcium hydroxide precipitation within the resin beads. These precipitates can reduce weak-anion operating capacity. If this occurs, operating capacity may be restored by rinsing the resin with a dilute hydrochloric acid solution, followed by a conventional sodium hydroxide or ammonium hydroxide regeneration and rinse.

Strong-Anion Resin Regeneration

Sodium hydroxide must be employed to regenerate a strong-anion resin. Complete strong-anion regeneration requires 300 to 400% of the theoretical strong-base, resin capacity. Two-stage regeneration is used to insure maximum use of sodium hydroxide. Strong-anion regeneration is followed by a slow, regenerant-water rinse, and a fast rinse, to a given column effluent conductivity.

Ion Exchange Operation

For reasons of operating economy and waste-regenerant disposal, ammonium hydroxide is used in many beet-sugar juice deionization plants employing weak-anion resins (32). The cost differential, per equivalent, between sodium hydroxide and ammonium hydroxide makes the use of sodium hydroxide prohibitive under normal circumstances in most geographical areas in North America.

Cation Resin Regeneration

Maximum utilization of sulfuric acid regenerant during strong-cation regeneration is of prime economic importance. Sulfuric acid usage can account for 35 to 50% of the total deionization-plant operating costs (8). The first stage of strong-cation resin regeneration consists of the *cation resin stripping cycle,* using weak-anion regeneration, alkaline waste. This waste consists of sodium or ammonium salts of anionic juice impurities. Cation stripping accomplishes the following: a) calcium is exchanged for ammonium or sodium ions, thus eliminating the possibility of calcium sulfate precipitation during strong-cation sulfuric acid regeneration; b) color bodies sorbed on the cation resin are stripped by the weakly alkaline salt solution; c) ammonium or sodium ions are easily exchanged for amino acids. The opposite is true when direct acid regeneration of a strong-cation resin is employed. It is not surprising that acidic regenerants are not particularly efficient in elution of amino compounds, since amino acids are highly ionized in acidic media and are essentially non-ionic in an alkaline solution. A high degree of ionization is associated with effective amino-acid bulkiness and slow diffusion from the resin matrix during acid regeneration (36); d) cationic and anionic juice impurities are recombined to form a 5.0 Brix RNS (reconstituted nonsugar stream), which may be

concentrated in a manner similar to hot Steffen filtrate for addition to beet pulp in conjunction with molasses, prior to drying. This approach to the disposal of the high-BOD, deionization-plant waste, converts a potentially noxious waste stream into a valuable addition to dried pulp (10). RNS is also sold in the concentrated form as a high protein animal feed supplement (11). Protein content of RNS approximates 60% on a dry solids basis.

Cation resin stripping is followed by a slow and fast raw-water rinse. The strong-cation acid regeneration cycle, for efficient regenerant use, is conducted as a two-stage process. The first stage involves regeneration of the stripped cation resin primarily in the ammonium form, with once-used sulfuric acid obtained from the last half to third of the fresh, sulfuric acid regeneration effluent, plus the first half of the slow, raw water rinse after fresh acid regeneration. The once-used acid cation column effluent may be combined with waste lime from the carbonation vacuum filters, or neutralized with anhydrous ammonia gas to produce an ammonium sulfate by-product fertilizer stream (9a, 10). Alternatively this fertilizer stream may be concentrated, spray dried and granulated to produce a homogeneous product with a fertilizer analysis of 17% nitrogen, 0% phosphorus pentoxide, 5% potassium oxide, and 23% elemental sulfur (11). If a complete homogeneous granular fertilizer is desired, phosphoric acid in conjunction with sparged anhydrous ammonia to produce mono or di ammonium phosphate may be added to the reactor prior to spray drying.

Utilization of eliminated nonsucroses (RNS) and waste regenerant streams to produce a high-protein feed supplement and a fertilizer by-product resolve a basic waste disposal problem and significantly enhance the economic return of deionization. The production of a fertilizer containing nitrogen and sulfur also gives the processor an economic hedge against increases in anhydrous ammonia and sulfuric acid regenerant cost due to the direct value relationship between sulfuric acid, anhydrous ammonia and ammonium sulfate fertilizer.

Fresh sulfuric acid, ranging in concentration from 6 to 10%, is introduced through the cation-resin bed at a flow rate ranging from 0.25-0.50 gal/cu ft/min. The fresh acid effluent from the regenerant column is reclaimed for reuse as previously indicated. Fresh acid regeneration is followed by a slow and fast raw-water rinse to a given acidity. The cation column is now in a partially regenerated condition suitable for the juice service cycle.

It is not economical to regenerate a strong-cation resin column completely. By judicious use of a two-stage regeneration, 70 to 75% of the total column resin volume may be completely regenerated with the use of acid, equivalent to 100% of the total theoretical resin requirements. Figure 10-13 graphically relates lbs of 66° Bé sulfuric acid regenerant used, per cu ft of cation resin, with the solid line (tons of nonsugar throughput to the strong-cation conductivity break), and the broken line (tons of nonsucrose eliminated by the cation and anion columns) (7). As an example, at a strong-cation regeneration level of 8.0 lbs 66 °Bé sulfuric acid per cu ft of resin, corresponding to complete regeneration of 80% of the total cation-column resin volume, the cation-column effluent would indicate conductivity breakthrough, after two tons of nonsucroses has passed into the cation resin bed. The actual cation plus anion column elimination is 70% of the two tons of nonsucroses to which the cation column resin was exposed. In the stated example, only 1.4 tons of nonsucroses were eliminated by the ion-exchange plant per cation service cycle. This elimination is equivalent to a resin, nonsucrose-capacity loading of 8.9 lbs/cu ft of resin. The fact

that 70% of the total nonsucroses entering the ion-exchange plant is eliminated is indicated by the difference between the nonsucrose throughput to the conductivity break, and the actual nonsucrose elimination. The difference, in Figure 10-13, is indicated by B.

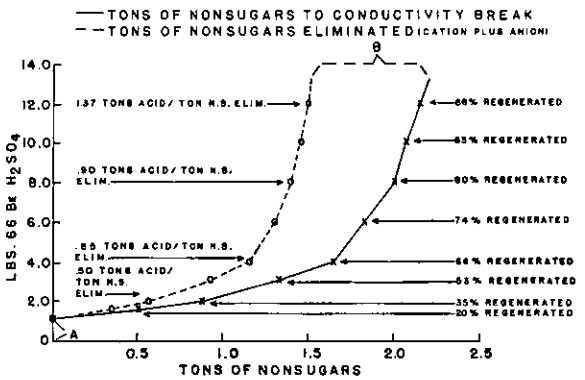

Figure 10-13. Cation exchanger regeneration levels, vs. tons of nonsucroses eliminated (7).

The 8.9 lbs 66 °Bé sulfuric acid per cu ft regeneration level corresponds to an elimination of 1.0 ton of nonsucrose impurities, per 0.90 ton of .66° Bé sulfuric acid used in regeneration. Intersection of the capacity curves at point A with the regeneration level ordinate indicates the minimum amount of total cation regenerated volume at which instantaneous conductivity breakthrough occurs. In this example, the regenerated volume at which instantaneous breakthrough occurred was 64 cu ft of resin. These plant data were obtained from a strong-cation column, containing 315 cu ft of Duolite C-25-D resin rated at a regenerated capacity of 1.7 meq./ml. The column feed juice was a 30° Brix, 81-82% purity mixture of intermediate green syrup and thin juice, feeding the column at 0.20 gal/cu ft/min.

The economic fallacy of complete cation resin regeneration of a strong cation resin is evident from Figure 10-13. Fresh sulfuric acid usage, far exceeding economical acid utilization, is necessary to obtain total resin regeneration. At the other extreme, lower acid regeneration rates severely limit cation-column capacity as well as return on equipment capital-investment costs. The best practical compromise seems to be regenerant usage of between 6.0 and 7.0 lbs of fresh 66 °Bé sulfuric acid per cu ft of resin, plus once-used acid from the previous regeneration. At these regeneration levels, the ratio of fresh acid used to nonsugars eliminated ranges from 0.75 to 0.90 tons of acid per ton of nonsugars removed.

Strong cation stripping and regeneration may be accomplished either by upflow or downflow. Upflow is advantageous with respect to cationic leakage during the service cycle and sharp conductivity breakthrough. Upflow stripping and regeneration does, however, require careful stripping-solution and regenerant-flow control to prevent resin-bed levitation, and subsequent loss of stripping and regeneration efficiency. The regenerant solution must always flow through the resin and not around it. When the cation regeneration and the slow and fast raw-water rinses are complete, the column is ready for the juice-service cycle. The time involv-

ed in desweetening, backwash, stripping, regeneration, and rinses must not exceed twice the juice-service cycle time, beginning with sweetening and terminating with desweetening, when three cation or anion columns are used. If four cation or anion columns are used, the column turn-around time can be extended to three times the juice-service time. This simple turn-around time rule assumes that only one cation and one anion column are in the juice-service cycle at one time.

Shutdown Procedures

Intercampaign, ion-exchange plant shutdown procedures consist of complete rinsing of all cation and anion columns until the effluent is negative for sugar by α-naphthol test. The rinse is followed by complete regeneration of the cation column with 300-400% of the theoretical acid requirements, and 150% of weak-anion theoretical base requirements. It is important to remove all traces of amino acids and organic acids from the cation and anion resins. If not removed, these organics within the resin matrix can supply the necessary nutrients for growth of microorganisms. After complete resin regeneration, the cation columns are completely exhausted to the sodium form with 3 to 5% sodium chloride brine solution, containing 0.5% of formaldehyde. The hydrochloric acid effluent from the cation resin is diverted to the weak-anion resin in the regenerated-hydroxyl form. Sodium chloride brine feed to the cation resins is continued until complete brine breakthrough is accomplished, as indicated by the reading of a Brix spindle in the column effluent. The brine is allowed to flow through the anion columns until complete brine breakthrough is noted in the anion-column effluent. The final cation and anion effluent should have a noticeable formaldehyde odor. The anion and cation resin is stored during intercampaign in the exhausted sodium and chloride forms, respectively. Both anion and cation resins are stored in a 3 to 5% brine solution which contains 0.5% of formaldehyde (8).

Operating Problems and Solutions*

[In this section, a few subjects treated in earlier sections may be reiterated for completeness.]

Flow Control

For the uniform and effective operation of an ion-exchange system, flow-rate control is a key problem. At the same time, the uniformity of Brix and the purity of the feed stream affect the flow control through viscosity and the nonsucrose load.

In instances where either dilution or stream mixing are applied to the feed stream, suitable mixing-equipment and density-control instrumentation must be employed, and several satisfactory types are available. Where stream mixing is used, such as a green syrup and a thin juice, only Brix control is necessary, provided the individual component streams are relatively constant. A constant flow of the heavier syrups, diluted to controlled Brix with the thinner stream, has proven satisfactory.

Since the pressure drop across each piece of equipment in the series is an important factor in flow control, this must be considered in equipment design. Furthermore, during operation, the pressure drop should be continuously monitored. A sudden change in pressure drop is a warning of trouble.

A well-designed heat exchanger will require a minimal pressure drop at the rated flow. An unusual pressure drop in this equipment generally means some kind

*by L.W. Norman

of blockage. This can, of course, be caused by foreign objects, and with dilute sugar juices it can also mean serious bacterial growth of the *leuconostoc* type, usually on the cold side of the heat exchanger. To avoid this, regularly scheduled dosages of the juice stream with bactericide are desirable. It is also desirable to provide means of steam cleaning and sterilization of heat-exchanger equipment. Where refrigeration is used to cool juices to the vicinity of 10 °C, viscosity may become a major factor in heat-transfer rates. This further amplifies the necessity of accurate Brix control, and may impose a maximum-Brix limit for an individual piece of equipment. Use of a secondary heat-transfer medium, such as a water-ethylene glycol system, allows greater flexibility.

Because an ion-exchange bed provides numerous but relatively small passages for juice travel, the pressure drop and flow control through the bed are critical. Precise Brix and temperature control must be maintained. A proper backwash cycle is also important. If accumulated resin fines are not properly eliminated each cycle, the pressure drop across the resin bed will slowly build up to intolerable proportions. On the other hand, it is costly to wash out whole resin beads or particles in the backwash step.

In the case of air-dome operation, level control becomes an integral part of the flow-control problem through the bed. As pressure drop becomes greater, the level will tend to rise. This must be compensated by greater air pressure in the dome, so that the level is maintained. This may be automatically achieved by means of level-sensing devices connected with an air-supply and vent system. For instance, two such devices positioned in the cell at the allowable high and low level points can hold the level by admitting air when the high point is reached, and by venting air when the low point is reached.

The sweetening curve is much steeper than the desweetening curve (see Figure 10-10). Thus, when an exhausted cation bed is taken off stream and a regenerated one put on stream, juice sufficiently concentrated to put through the anion column is available from the fresh bed before the juice from the exhausted bed is low enough in sugar content to divert to waste. During this short interval of time, the effluent from two cation resin beds must be put through the anion resin at double the normal flow rate. While this does cause pressure-drop and flow-control problems, careful coordination will minimize the trouble.

Systems which employ two anion or cation columns in series, and random cycles; i.e., cation-anion resins not balanced as to capacity, can encounter even greater short-term flow increases if both the cation and anion column are taken off stream simultaneously. This can readily be solved, however, by judicious foreshortening of one of the cycles without serious loss in column-capacity utilization.

Physical Losses

In ion exchange losses are potential in two forms, physical losses due to leaks and to sweetening and desweetening, and chemical losses due to inversion. The complex valving system in ion exchange processes requires use of the best quality valves to minimize leakage losses. The Saunders type has proven satisfactory for the service. Other physical losses, such as leakage at flanges, must be minimized by adequate installation and maintenance procedures. Sweetening and desweetening losses must be controlled by careful attention to the point of diversion to waste. The choice of this point is dictated by evaporator capacity and by economic considera-

tions of the value of the sugar sent to waste versus the cost of recovery. Cutoff at 1 °Brix is generally considered a reasonable compromise.

Inversion Losses

Heterogeneous and homogeneous inversion losses have been described (pp. 310-312)(32). Temperature and time are important in both cases, and pH, of course, in the latter. While the choice of cation resin strongly affects the inversion rate, in any given system inversion is minimized by the use of the lowest practical temperature and the highest practical flow rate, or shortest contact time. In systems employing strong acid cation resins the temperature is normally kept in the 10-12 °C range.

As there is little or no control of juice pH, flow rate and temperature are again the important control factors. If temperatures are kept low, the homogeneous inversion will be quite low relative to the heterogeneous, and the total can be kept to tolerable proportions in the neighborhood of 0.3% on sugar in the feed stream.

If the juice-treating system includes a recycle, such as the return of treated, diluted machine syrup to evaporator thin juice, invert and other nonionized nonsucrose substances increase in concentration in the part of the sugar end within the recycle. In this case, the increase is noticed in the white and intermediate massecuites. The effects are increased color, poor boiling characteristics, and decreased crystallization yields. The system may be purged of these objectionable materials by changing the ion-exchange feed to thin juice without recycle, or to diluted thick juice with limited recycle. The impurities are then eliminated with the molasses.

Nonsugar Leakage

In order to make ion-exchange treatment of sugar solutions an economically feasible operation, an optimum balance must be attained between the efficiency of elimination of nonsugars, resin-capacity usage, and regenerant levels. This generally means that some nonsucrose leakage is allowed because it is too costly to remove all potentially removable nonsucroses, and in particular those which are most weakly retained by the resin. Flow rate also affects this leakage, and a compromise must be reached for optimum economy. Under given equipment limitations the point at which the resin is considered exhausted, or the service cycle end point, is important. Ordinarily leakage will start at a low level and slowly increase. With some resin systems, generally the strong ones, a fairly sharp breakthrough of nonsucroses may occur, but with others the breakthrough may be diffuse. The breakthrough, and thus the cycle termination for a strong acid cation resin, may be easily detected by the rapid increase in ionic strength of the effluent, as indicated by conductivity. Likewise, the end of the anion resin cycle, which follows a strong cation resin, may be detected by a rather sharp pH change. However, with some systems, the cycle termination may necessarily be put on an arbitrary basis of pH, conductivity, or other measure.

Backwashing Problems

Backwash not only eliminates the resin fines to prevent excessive pressure drop, but also reclassifies and redistributes the resin bed to help prevent channeling, and resultant poor capacity usage. This step must be carried out properly after each cycle or trouble will eventually occur. Water flow rate and temperature are important factors. Each resin type and resin particle size are also determinants, as is resin

depth and column geometry. The proper backwash procedure must be determined for each individual system. Precaution, particularly with a lower-density anion resin, must be taken to prevent levitation of the entire bed with resultant resin loss. Where this occurs, the situation can generally be helped by starting backwash at a low rate and increasing to the desired rate. Sometimes a jolt of backwash followed by a short settling period is helpful, as has been loosening of the bed by means of air.

Waste Disposal

The spent regenerant streams may be a disposal problem, since they are unusable either in the sugar factory or the ion-exchange process. When mixed, the combined stream consists of all the cationic and anionic nonsucroses eliminated from the juice stream, plus any excess acid or base and possibly a trace of sugar. The salt load is high, the BOD is high, and generally the stream is decidedly acidic. One possible scheme for turning the waste streams into valuable by-products provides for first utilizing ammonia as the anion regenerant. The spent regenerant from the operation, containing ammonium cations and nonsucrose anions removed from the juice, is further used to strip the exhausted cation bed. This leaves the cation in the ammonium form with a spent effluent containing both the cations and the anions removed from the juice. These reconstituted nonsucroses may economically be dried on pulp (10). When the cation resin is then regenerated with sulfuric acid, the spent regenerant will consist of ammonium sulfate plus some excess acid. This regenerant waste contains both nitrogen and sulfur—both of potential value as fertilizers. (10).

Color bodies are eliminated by both cation and anion resins, the cation resins being more effective in this respect, with color-removal efficiency dependent upon the particular resin type. Although this is an important beneficial result of ion exchange, it is not without drawback.

Color Problems

Color bodies are held on the anion resin far more strongly than their weak ionic nature warrants, presumably by adsorptive forces. Thus, complete removal by the regeneration cannot be effected and irreversible fouling may result with corresponding capacity loss. Use of supplementary treatment with salt is particularly effective in removing color from the resin and maintaining capacity, but fouling remains a serious problem in effective resin life (8). The use of carbon adsorbents to protect the resins has been reported.

In early ion-exchange installations in North America, the common practice was to treat all the juice to the sugar end (12). This, of course, eliminated nearly all ionic species from the juice and rendered it highly unbuffered and subject to pH instability. Present practice tends toward treatment of intermediate sugar-end syrups, as well as bypass of a portion of the nonsucrose load to maintain adequate buffer capacity for stable pH conditions.

The pH of unbuffered juices tends to decrease to alarmingly low levels through evaporation and pan boiling, causing inversion. The Maillard, or browning reaction, in which the reactants are invert sugar and amino acids, produces a very viscous, dark-red colored after-product which very seriously inhibits crystallization. The Maillard problem may be alleviated by bypassing 15-20% of the juice around the ion-exchange unit. The buffers in this untreated fraction are sufficient to stabilize the pH of the entire juice flow.

Economics

Certainly the addition of an ion exchange demineralization or purification system to either an existing or new beet sugar plant must be economically justifiable. However, because economic considerations are variable both with geographic location and with time, no specific values can be stated which will have general application.

Rough approximation of overall economics can be made by comparison of the difference between the income and cost factors. Income may be estimated by multiplying the amount of expected additional sugar to be recovered by the unit difference in its value as sugar and as molasses. All applicable cost factors must also be estimated and compared with income to determine whether a favorable relationship exists.

Such estimations and comparisons may be as rigorous or as rough as the needed accuracy of the data require. In addition to capital and operating considerations any applicable cost and/or income associated with pollution control and by-product recovery should be included.

Miscellaneous Ion-Exchange Systems

Because demineralization via series-bed operation typifies most of the important concepts in ion exchange, represents one of the systems that can achieve the greatest increase in sugar recovery, and is the only demineralization system in commercial use at the present time in the North American beet-sugar industry, it has received the most attention. Continuous demineralization systems have the appeal of steady-state operation with a potentially lesser regenerant usage, lesser dilution and lesser resin inventory, though control problems are greatly increased. Among such systems are the Higgins loop (12), the Degremont-Cottrell system (42), and the CCIX system (4).

Mixed bed processes which combine cation and anion resins mixed together in one column for deionization offer advantages in several respects (20). The mixed resins allow less leakage of weak ions prior to breakthrough. Since the acidity due to cation exchange is immediately taken up by the adjacent anion resin, inversion is less and somewhat higher temperatures may be employeed, though most anion resins in the hydroxyl form have poor stability at high temperatures. A serious deficiency is the requirement for resin separation before regeneration.

Reverse deionization, which calls for passage of influent first through a strong anion resin and then through the cation-resin bed, is claimed to cause less inversion, since the cation resin contacts alkaline material which it neutralizes. However, the anion resins are not generally efficient salt splitters, so leakage may yield an acid cation effluent anyway, and efficiency, both of ionic elimination and of regeneration, is impaired.

The Vajna process is a demineralization system in which the juice cations are converted to ammonium ions and the anions to hydroxyl ions. The ammonia is then driven off by evaporation, leaving the juice demineralized (54). The Moebes process employs conversion of juice cations to the ammonium form and anions to the carbonate form (38). This is followed by liming and carbonation to precipitate the carbonate and drive off the ammonia, leaving the juice demineralized. The system employs mixed cation and anion resins in a single bed with regeneration of both, simultaneously, by means of ammonium carbonate solution.

Processes are also in commercial use, both in the United States and in Europe, which employ cation exchange only.

Deliming or Softening by Ion Exchange

Calcium compounds which are not removed in second carbonation, the so-called lime salts, create processing problems such as: scaling of heating surfaces, sediment or turbidity in final sugar, poor juice-boiling characteristics, and added molasses if soda ash has been employed. (see pp. 253-258). While elimination of the invert and the carboxylic acids which cause lime salts is the best solution, this is not always possible. Hence, juice softening by ion exchange can become an attractive alternative.

As ion-exchange processes go, softening is one of the simplest. A single-bed system is used, in which a strongly acidic cation resin, one of the most stable, is used to exchange the unwanted calcium ions for sodium ions (45). This is not without disadvantages, however, since the sodium ions introduced are considered more melassigenic than the calcium ions replaced. Relatively cheap salt brine is used for regeneration.

Normally, fixed-bed column operation has been employed in the United States (44,21,40). Since the cycle length in juice softening is relatively long, two columns may be simultaneously on stream in parallel, with a third column under regeneration (18,21). Cycles must be staggered to provide continuous operation. If columns are of sufficient size so that the entire throughput can be handled by a single unit, a two-column system may be used.

In fixed-bed softening, the on-stream or service portion of the cycle is followed by conventional off-stream desweetening, backwash, regeneration, and rinsing. For regeneration, about 12-15 lbs of salt at about 10% brine concentration may suffice (44,21). Optionally, a portion of the once-used regenerant may be recovered and reused, although care must be exercised in the practice to assure both satisfactory concentration of monovalent ions, and sufficient freedom from contaminating divalent ions. Sweetening, desweetening, backwash, and rinse steps are carried out in essentially the same manner as in demineralization. For detailed discussion of fixed-bed operation (see pp. 310-320).

Filtration of the carbonated juice, and also of the regenerant brine solution must be clean, if problems with foreign particulate matter trapped in the beds of resin are to be avoided. Although finely divided, the calcium carbonate precipitate from second carbonation is difficult to remove by backwashing. If hot condensate is ued for backwashing, this problem is made worse by virtue of density difference, and higher flow rates are required than would be necessary with ambient-temperature water. While either upflow or downflow regeneration may be acceptable, the general practice has been to use upflow. This method may result in less calcium leakage, since the more thoroughly regenerated portion of the resin bed is at the bottom of the column to act as a scavenger. On the other hand, flow rates with upflow regeneration must be well controlled to prevent bed levitation, and reduced regeneration efficiency.

Leakage of calcium in softening systems occurs at low levels rather early in the cycle, continuing thereafter at a nearly constant rate until the rather diffuse breakthrough occurs. It has been found that complete decalcification of the juice is undesirable (21,40,44). Both foaming and corrosion have been attributed to overly softening juices. Control may be obtained by deliberate bypass of a portion of the untreated juice, so that the recombined stream is maintained at 0.02-0.03% lime salts. Optimum conditions should be sought for each individual case.

Ion exchange softening has been shown to effectively reduce lime salts in carbonated beet juices, and to reduce evaporator boilouts due to calcium scale deposits (21,40). However, the cost of this process has not been covered by the savings of boilout labor and material alone. The principal advantage of the process has seemed to lie in the additional evaporator capacity allowed by cleaner tubes and the resulting additional slice capacity, although there may be other advantages in specific instances.

Of considerable interest is a recent idea for regeneration which uses the softened juice itself as the regenerant (18). Upon concentration to thick juice, the combined monovalent ion concentration becomes sufficient to provide effective conversion of the resin bed from the divalent to the monovalent form. The process, in essence, merely bypasses the divalent cations around the evaporators, where they cause scaling problems, returning them to the juice thereafter to utilize their lesser melassigenic effect, as in the Quentin system.

Continuous systems for deliming beet juices have been reported in Europe (55) but none are presently being operated in North America. In general, it has been shown on both continents that juice softening is a technically feasible process. Its utilization depends on the severity of the lime-salts problem, and the economics of the specific plant installation. Four factories in the United States are presently using ion exchange thin juice deliming.

Quentin Process*

The highly melassigenic effect of the salts of sodium and potassium was suspected as early as 1843 by Hochstetter (25). Many attempts have been made since that time to eliminate these salts from beet liquors by ion exchange, in which sodium and potassium were replaced by magnesium or calcium ions. The earliest recommendations involved the application of soils to the juices.[1] Later, natural or synthetic zeolites were used (19,22). However, the practical commercial use of cation exchange awaited the development of efficiently operating synthetic cation-exchange resins.

Successful ion exchange results in a significant reduction of sucrose solubility in the syrups, and thus increased sugar extraction.

Quentin (47) placed the influences of the various cations on sucrose solubility in the following order:

Mg^{++}	$< Ca^{++}$	$< Li^{+}$	$< Na^{+}$	$< K^{+}$	(ion)
0.61	0.66	0.73	0.94	1.00	(relative sucrose concentration in molasses: potassium molasses = 1.0)

Figure 10-14 shows typical solubility curves for sucrose in beet molasses, a) before ion-exchange treatment, and b) after 30% of the total sodium and potassium ions have been replaced with magnesium. Thus, sucrose extraction can be increased by about 0.5% on beets.

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[1]See ref. (24,50,53,56)

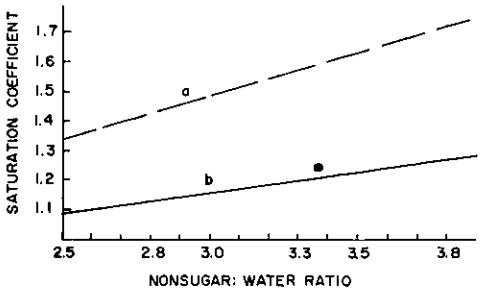

Figure 10-14. Change of sucrose solubility in molasses after exchange of 30% monovalent cations for magnesium. Example: At nonsucrose: water ratio of 2.73 and at 50 °C, potential molasses purity 57.15% before exchange and 51.39% after exchange, potential additional extraction through Mg- exchange 0.484% on beets at 1.75% nonsucroses on beets; saturation solubility: ratio of saturation solubilities for impure solution/pure solution.

Extensive application of the Quentin process has been made in Europe, and in four factories in the United States, in which part of the sodium and potassium ions are exchanged for magnesium ions. To be profitable, a source of inexpensive magnesium-salt regenerant must be available, frequently as a by-product of an inorganic chemical industry. Such sources exist in Central Germany, and in the vicinity of the Great Salt Lake. Magnesium chloride brine from Utah, for example, is the final residue solution from the salt evaporation of deep-well brines, or Great Salt Lake surface water.

Mechanism of Exchange.

In the Quentin process, a strongly-acidic resin with sulfonic acid groups, loaded with magenesium counter ions (ions with opposite electrical charge to the overall resin matrix charge, attached to the matrix), is used to treat intermediate green syrup of 68-70% solids, at 83-90 °C.

$$R-(SO_3^=)_2-Mg^{++} + An^= - K^+ (\text{or } Na^-) \rightleftharpoons R-(SO_3^=)_2-K^+ (\text{or } Na^+) + An^= - Mg^{++}$$

where R is the resin matrix and $An^=$ the syrup anions. Due to the high concentrations of Na^+ and K^+ in the syrup, they displace $Mg^=$ in the exchanger matrix, even though the exchanger has a stronger attraction for divalent cations.

Since complete removal of sodium and potassium cations is not attempted, a relatively large leakage of these ions through the exchanger beds is tolerated, and more than 80% of the potential exchanger capacity is used. With flow rates through the beds of not more than two bed-volumes per hour, a cumulative 30-50% of the alkali ions in the syrups is normally replaced with magnesium. Exchange efficiency and operating capacity increase with a decrease in dissolved solids concentration from 70 to 60% and a reduction of the flowrate from 2 to 1.5 bed volumes/hr (30a).

The syrup pH must be kept below 9.0 to prevent resin fouling with precipitated $Mg(OH)_2$. Inadvertent fouling requires extensive acid stripping to restore activity.

Because of the high osmotic shock inherent in the process, gel-type resins cannot be used. Even highly cross-linked macroporous resins break down prematurely if the syrup density exceeds 70 °Brix. Pressure drop through the resin bed, and desweetening economics also become unfavorable over that density. On the other hand, dilution below 68% solids leads to increased fuel costs to evaporate the water.

Regeneration with magnesium chloride brine proceeds as follows:

$$R-(SO_3^=)_2-K^+(\text{or } Na^+) + Mg^{++} - Cl^=_2 \rightleftharpoons R-(SO_3^=)_2\,Mg^{++} + K^+-Cl^- + Na^+ + Cl^-$$

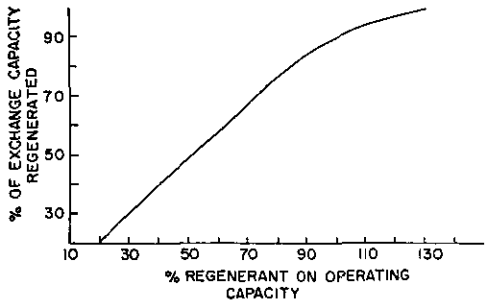

Figure 10-15. Exchange profile for a strong cation resin in the K^+/Na^+ form during regeneration with a 1.4 *N* $MgCl_2$. Temperature 80°C; flow rate, two bed-volumes per hour.

Because of the resin preference for multivalent cations, less than 130% of the theoretical regenerant is required. Figure 10-15 details the course of regeneration.

The diluted magnesium chloride brine used for the regeneration of the exchanger to the magnesium form should have a pH of less than 6. A pH above 7 indicates contamination with $Mg(OH)_2$ which can lead to progressive fouling of the exchanger. Acidification of the brine with HCl is indicated under these conditions.

Oldfield et al. found Kiserit ($MgSO_4 \cdot H_2O$) with less than 1% monovalent cations to be a suitable regenerant for conversion to the magnesium form (43a). The effect of background limesalts in the greens to be treated on the fouling of the exchanger with precipitated $CaSO_4$ was not reported.

Fractionated desweetening is usual in the Quentin process. Sweetwater of about 25° Brix is used to displace the syrup after resin exhaustion. Sweetwater of 5-10° Brix is used to dilute intermediate green syrup to 70° Brix prior to resin treatment, and to displace the more concentrated sweetwater during desweetening. This technique also lessens osmotic shock. With proper operation dilution can be limited to less than 3% of the syrup treated.

Countercurrent operation for a Quentin process was achieved with a dual column design (52a) at the Nampa factory of The Amalgamated Sugar Company. The countercurrent flow pattern has inherent advantages relative to increased exchanger utilization and reduced regenerant usage. Additional benefits accrue from reduced product dilution, increased service cycle length, and reduced process water demand. The major drawback of such systems is their sensitivity towards suspended solids, and resin fines which can generate an intolerable pressure drop. Their effect on the pressure drop is greatly increased because of the increased cross-sectional flow velocity for such systems.

Kammerer (29a) applied a column of bi-level construction to increase operating capacity, reduce regenerant consumption, condensate requirements, waste water production, and non-productive cycle length.

Integration with Sugar End Flows

Intermediate green syrup purities must be held below 75% to allow molasses exhaustion to below 55% purity.

Quentin massecuite with purity as low as 75% centrifuges well, depending somewhat on the exchange rate. Since sugar is shifted in the direction of the white boiling, raw massecuite volumes are decreased with magnesium exchange, thus permitting more equipment capacity and time for raw pan boiling, crystallization, and centrifuging.

It is sometimes necessary to alter boiling practices when the magma is rich in magnesium, because of the altered sucrose-solubility relations. The lower density syrup eliminates the normal need for water addition during raw boiling, thus largely offsetting the dilution associated with magnesium exchange.

Magnesium molasses presents no difficulties in Steffen processing, actually permitting an increase in Steffen house capacity of 15-20% over nonmagnesium molasses. Somewhat slower first-carbonation sludge settling and filtration rates may be noticed, however, due to the increased nonsucrose recycle load.

The technology and economics of magnesium exchange are thoroughly treated in the literature.[1]

The economic balance between the extraction gain, and the countering investment and operating costs depend on many variables, such as the cation concentration in the syrup to be treated, the ion exchange rate, the brine cost, and process optimization.

A flow diagram for magnesium exchange of intermediate green syrup is shown in Figure 10-16. Table 10-3 lists performance criteria:

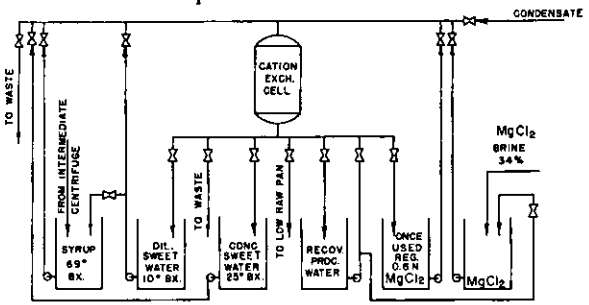

Figure 10-16. Flow diagram for magnesium exchange of intermediate green syrup, by the Quentin process.

Table 10-3. Economic Target Guidelines for Magnesium Exchange

Equivalent $Na^+ + K^+$ per 100 g nonsugars	0.37-0.55
Exchange Rate	30-55%
Extraction Gain	0.3-0.6% on beets
Brine Consumption (30% $MgCl_2$)	0.5-0.8% on beets
Dilution	0.2-0.6% on beets
Sugar Loss	0.01-0.05% on beets
Waste water	6-10% on beets

Rearick, et al. (48a) have shown a positive effect of magnesium salts in Quentin molasses on the refractometer dry substance. The true dry substance as determined by the Karl Fischer method can be derived from the refractometer dry substance accordingly.

[1]See ref. (26,28,41,46,48,52)

True ds (KF)* = 0.093 rds + 0.581. A correlation coefficient of 0.928 was found in this study. The effect of magnesium ions on the refractometer dry substance of molasses must be considered when comparing molasses purities based on rds.

Environmental pollution is not a serious factor with the Quentin process, since the salts to be discarded are inorganic.

Related Purification Systems*

Systems related to ion exchange, but not actually involving an exchange, have also been investigated for potential use in sugar processing. One such process is *ion exclusion* (43). In this process a bed of cation resin, exhausted in the nonsugars of the stream being treated, is contacted alternatively with the juice and with water. During juice contact the non-ionic sugar molecules penetrate the pores or interstices of the resin particles, while the ionic species are rejected or excluded and a separation of the two is accomplished. When water is then passed through the system, the ionic sugars are immediately displaced from the voids between the resin particles, and the sugar is then recovered in a diluted, but purified form as it diffuses back out of the resin particles.

Ion retardation employs a special resin containing both cationic and anionic exchange sites (16). As in ion exclusion, the resin is alternately contacted by sugar solution and water. However, in the case of ion retardation the ionic nonsucroses are held, though weakly, the sugar being recovered ahead of the nonsucroses in a chromatography-like fraction. The water acts, in essence, as the regenerant.

Another system involves the use of semipermeable ionic membranes and an electric current to selectively remove the ionic species in solution through the appropriate membrane, leaving the purified sugar solution behind. In this instance, of course, electric power can be compared with the chemical regenerant of ion exchange.

*Karl Fischer determinations

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C. Activated Carbon*

It has been known for several hundred years that wood char or charcoal has the ability to remove tastes, odors, and colors from a variety of liquids. However, activated carbon in the modern sense of the term was not invented until about 1900 when a Russian scientist, Raphael Ostrejko, obtained patents (14) for the activation of chars of vegetable origin. Ostrejko found that the adsorptive properties of a char could be enhanced tremendously by heat treatment in the presence of steam or carbon dioxide. Alternately, the activation could be accomplished by the carbonization of vegetable matter impregnated with various salts. Modifications of these processes are still used today for the production of activated carbons from such diverse raw materials as coal, lignite, peat, pulp mill black ash, and by-product acid sludge coke.

Although the structure of activated carbon has not been resolved with certainty, there is evidence (6) that the principal units in the structure are randomly dispersed, graphite-like crystallites of submicroscopic dimensions. Each crystallite is composed of a small number of layers made up of carbon atoms arranged in a pattern identical to that in a polynuclear aromatic compound. The distances between the layers are greater in activated carbons than in graphite. A structure of this type leads to a complex network of very fine pores, and consequently a very great internal surface area. It is concluded that as much as one-half of the total surface area of the carbon atoms in the structure is exposed and available for adsorption. The surface areas of sugar-treatment carbons range from 600 to 1200 sq m/g. This means that one pound of carbon can provide more than 100 acres of internal surface. It is the magnitude of the surface area available to the adsorbate, the adsorbed substance, that is primarily responsible for the adsorption ability of the activated carbon. Unbalanced electrostatic forces on the internal surface become satisfied when other molecules in the vicinity become attached to the surface or adsorbed. The forces involved are the van der Waals[1] forces and the phenomenon is termed physical or van der Waals adsorption. The activated carbon surface is nonpolar in nature, that is, it is neither positively nor negatively charged. Nonpolar adsorbents in general attract nonpolar adsorbates and have little or no affinity for either positively or negatively charged ions. For this reason, activated carbon preferentially adsorbs organic molecules and does not adsorb inorganic ions. The larger the organic molecule the more strongly it is held on the surface. Thus the larger molecules present as impurities in sucrose solutions will be preferentially removed by carbon treatment and in fact will displace adsorbed sucrose from the surface.

Effects of Activated Carbon Treatment

In the treatment of cane and corn sugar liquors activated carbon is used primarily for the adsorption of color. In beet sugar processing, however, equal importance is attached to the removal of other impurities such as colloids, floc precursors, and compounds leading to foam formation as well as color. Furthermore, activated carbon treatment can achieve significant increases in sugar purity if a sufficient quantity is used.

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[1]van der Waals forces are defined as the attractive forces exerted by adjacent molecules due to the interaction of electrostatic charges.

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*by R.S. Joyce

Color in beet sugar may be due either to substances extracted from the beet along with the sugar or to degradation products formed during processing. Both types of color are removed by carbon. The adsorption of caramel on the sugar crystal and its interference in crystallization has been investigated (4,23). Caramelization products in thick juice can be effectively removed by activated carbon treatment (10). Colloids, because of their high molecular weight, molecular complexity, and minimum solubility are readily adsorbed by carbon. Harris (5) has observed that the percentage of colloids adsorbed from beet sugar juices of high and low purity is much greater than the percentage of color removed. Several investigators have claimed that the presence of colloidal material depresses the surface tension and increases the viscosity of beet sugar liquors, leading to impaired rates of filtration, evaporation, and crystallization (12, 17). Improved rates of evaporation and crystallization have reportedly been effected by carbon treatment (9,11).

Activated carbon is also effective for the removal of compounds which upon acidification produce a flocculant precipitate. These compounds are generally termed floc and consist in part of oleanolic acid and its glycosides (beet saponin) (3) see pp. 53-55). Removals of up to 80% saponin by the granular carbon treatment of 70° Brix pan feed liquor have been achieved (16). The ability of carbon-treated thick juice to produce essentially floc-free sugar has been demonstrated (2,18). Similarly, marked improvements in the foaming characteristics of carbon-treated liquors have been reported (2,22).

A study of the compounds extracted from used beet sugar carbons was made by Walker and his co-workers (21). In addition to sucrose, reducing sugars, predominantly glucose, and trisaccharides such as kestose and raffinose were extracted from the carbon. Organic acids, natural proteinaceous material, some saponin, and an unidentified carbohydrate polymer were noted among the nonsugars in the eluates.

Types of Carbon and Points of Application

There are two forms in which the activated carbon can be used in sugar treatment, namely, pulverized and granular. The primary difference is in the particle size, the former being a fine powder having an average particle diameter of 40μ or less, while granular carbons for beet sugar treatment are in the 12×40 U.S. sieve series range and have a mean particle diameter of approximately 1 mm. The procedures and equipment for the application of the two forms differ considerably. In the first case, the finely powdered carbon is slurried with the liquor to be treated and subsequently filtered out. In the second case, the liquor is percolated continuously through a bed of carbon granules.

At the time that the first edition of this book was written, essentially all of the carbon used in the beet sugar industry was pulverized. The carbon was used at one or more points in the process where it could be conveniently added and subsequently filtered out by existing equipment. This situation has been reversed, however, first with the advent of high capacity, abrasion-resistant granular carbons. Secondly, within the last decade, unit processes have been developed for the effective use of these adsorbents.

The greatest advantage of granular carbon is that it can be repeatedly regenerated and reused with only a small percentage of makeup to replace the handling losses. Thus the material cost of equivalent carbon treatment is far less than with powdered carbon which is usually discarded after only one use. A further advantage is that granular carbon can be used in a countercurrent treatment system

which makes more efficient use of its adsorptive capacity than the concurrent flow methods commonly used with powdered carbon. Furthermore the use of a granular carbon bed provides reserve adsorptive capacity to cope with unanticipated increases in impurity levels of the juice. Finally, the use of granular carbon eliminates the waste disposal problem associated with the powdered material.

Pulverized carbon has the advantage that it can be conveniently used in the existing factory process with little or no equipment modifications. Methods are available for improving the capacity utilization. A semicountercurrent application can be used, wherein a batch of liquor is treated with once-used carbon and the filtrate is treated with fresh carbon. The latter is recovered by filtration and reused on a second batch of untreated liquor. The powdered carbon savings effected by countercurrent usage can be calculated from the results of relatively simple experiments [See the publications of Sanders (19) and Helbig (7)].

Alternately, one-half of the total powdered carbon requirements can be used at each of two points in the process: for example, standard liquor and second carbonation juice. In a modification of this approach all of the carbon can be used at one point, recovered by filtration, and reused at a second point where the impurity level is higher. The two-point approach takes advantage of the increased capacity with increasing residual impurity. For the most part, granular activated carbon has been used for the treatment of thick juice, and, more recently thin juice. Pulverized carbon on the other hand has been used at a number of points in the process as outlined below (13).

Filtered First Carbonation Juice. The carbon is added to clear, first carbonation juice, carried through the second carbonation tank, and filtered out on the second carbonation filters.

Second Carbonation Juice. Carbon may be added to the overflow from second carbonation and removed at the second carbonation filters.

Thin Juice. In this case the point of addition may be after the second carbonation filters prior to the sulfitation step or to a point in the thin juice line after this step. In either case the carbon is removed in the thin juice filtration.

Thick Juice. The carbon addition is made to the thick juice line leaving the last evaporator effect and removed in the thick juice filtration.

Standard Liquor. Carbon is added generally in the high melter but can be added to the low melter if raw sugar becomes a component of standard liquor. In either case the carbon is removed by double filtration at the standard liquor presses. Carbon treatment of standard liquor is not recommended where double filtration is not practiced.

Laboratory Evaluation of Activated Carbons

The Adsorption Isotherm. Regardless of whether pulverized or granular carbon is under consideration, relatively simple laboratory tests can provide considerable preliminary information on carbon performance. Such tests are of value in determining the effect of carbon treatment on a particular liquor, in comparing the relative merits of different carbons, as quality-control tests to insure a uniform source of supply, and in evaluating the quality of reactivated carbon.

The tests are normally based on the adsorption isotherm, which is a plot of the amount of impurities adsorbed against the amount of impurities remaining in solution at constant temperature. Generally, straight line plots can be obtained by mak-

ing use of the empirical Freundlich equation which relates the amount of impurity in the adsorbed phase to that in the solution phase by the expression

$$x/m = kc^{1/n} \quad (1)$$

where x = amount of impurity adsorbed; m = weight of carbon; x/m = concentration in the adsorbed state: i.e., the amount of impurity adsorbed per unit weight of carbon; and c = equilibrium concentration in solution after adsorption. k and 1/n are constants. Taking logarithms of both sides:

$$\log x/m = \log k + 1/n \log c \quad (2)$$

Equation (2) represents a straight line whose slope is 1/n and whose intercept is log k when c = 1. Therefore if x/m is plotted against c on log-log paper, a straight line should be obtained. This is true for most sugar solutions where relatively low concentrations of color and other contaminants are encountered.

Data for plotting isotherms are obtained by treating fixed volumes of liquor with a series of known weights of carbon. The carbon-liquid mixture is agitated for a fixed time at constant temperature. After the carbon is removed by filtration the residual color, or the concentration of another impurity, is determined. From these measurements all of the data required to plot an isotherm may be calculated.

Detailed procedures for isotherm determinations have been published (1,15). This discussion is limited to the considerations that affect the selection of the experimental variables.

Carbon Particle Size. Powdered carbons are tested as received. Granular carbons should be pulverized so that 90 wt. % passes a 325 (U.S.S.) screen. This increases the rate of adsorption which is intraparticle-diffusion controlled. The total capacity of the carbon is unaffected.

Contact time. For powdered carbons the point of carbon addition and removal in the factory will in many cases fix the contact time. Where granular carbons are being evaluated in an isotherm test, contact time should be sufficiently long to ensure a reasonable approach to equilibrium. This can best be determined by a preliminary test in which identical weights of carbon are contacted with equal volumes of the test liquor for various times. Generally, contact times ranging from one to two hours suffice.

Temperature. Similarly, in case of powdered carbons, the temperature will often be fixed by the process conditions. For granular carbons the temperature should be as high as possible without adversely affecting sugar quality. Evidence has been cited (3) that inversion of thick juice in contact with granular carbon occurs above 95 °C. Temperatures in the 80-95 °C range are usually employed. Microbiological inversion may occur below this range.

Carbon Dosage. This will depend on the particular liquor treated. As a general rule carbon weights ranging from 0.5 to 10% of juice solids will suffice. Subsequent adjustments in dosage can be made to obtain an adequate range of experimental points.

The pH of the Test Solution. Carbon may affect the pH of the liquor it contacts, particularly at the higher dosages. Accordingly where color measurements are being made, the filtrate should be adjusted to the original pH level to help compensate for the indicator effect. A Freundlich plot of an isotherm for the carbon decolorization of thick juice is shown in Figure 10-17a. A considerable amount of information can be obtained from the isotherm plot. In the first place, one can tell immediately whether or not the desired degree of purification can be attained, and the carbon dosage required to achieve any degree of purification in a batch system

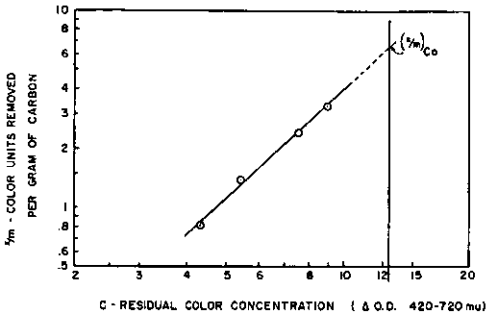

Figure 10-17. Isotherm for the decolorization of thick juice. (65°Brix) at 80°C.

can be read from the plot. Secondly, the adsorptive capacity of granular carbon in a column system can be estimated. By drawing a vertical line from the point on the horizontal scale corresponding to the initial concentration C_0, and extrapolating the isotherm to intersect this line, the x/m value at the point of intersection can be read from the vertical scale. This value, termed $(x/m)_{C_0}$, represents the amount of color adsorbed per unit weight of carbon when that carbon is in equilibriuim with the initial or influent concentration. Since this equilibrium should be closely approximated, at least in the upper section of the carbon bed during the column treatment, it represents the ultimate capacity.

From the $(x/m)_{C_0}$ value, the capacity in terms of volume of liquid treated or pounds of sugar solids treated per unit weight of carbon may be calculated. These figures can be obtained by dividing $(x/m)_{C_0}$ by the concentration units removed per unit volume. From the capacity figures an approximation of the cost of carbon treatment may be obtained. It should be noted, however, that capacity values for granular carbon, estimated from an isotherm, may not be attained in practice. It may be impractical to operate the column in such a manner as to ensure complete saturation of the carbon at the inlet end.

Before a full-scale adsorption system can be designed certain additional information must be obtained. The operating capacity of the carbon as well as the optimum combination of flow rate and bed depth must be established. These factors will depend upon the rate of adsorption which must be determined by dynamic column tests.

Column Tests. Full-scale columns for beet sugar treatment are 9 ft or more in diameter and 25 to 40 ft in depth. Laboratory columns can be scaled down to 1 or 2 in. in diameter and the flow rate reduced to simulate plant conditions. It is difficult, however, to extrapolate data obtained in a shallow bed to predict performance in a deeper bed. Accordingly, it is recommended that the depth of the laboratory columns be the same as that anticipated in the plant. Theoretically, both the column depth and the flow rate can be reduced to obtain an approximation of the full-scale contact time. However, this may introduce experimental difficulties in accurately metering low flow rates and ineffective column packing leading to wall effects.

The connection of a number of column sections in series is a convenient way of obtaining the required bed depth in the laboratory. This arrangement facilitates the collection of effluent samples at various bed depths, thus evaluating the effect of contact times. Further, a series of sectioned columns is essential for determining the effects of countercurrent operation. Columns for laboratory or pilot-plant tests can be constructed from plastic or glass tubing, or pipe. Constant temperatures may be obtained by using jacketed columns and by circulating water from a constant

temperature bath through the jackets. The transfer lines between the columns should also be jacketed for best results. Sampling ports should be provided between columns. Gauges may be installed to obtain measurements of the pressure drop across the system. In order to obtain constant flow rates a small positive displacement pump is desirable. A stainless steel screen at the bottom of the column makes an effective bed support. (Figure 10-18).

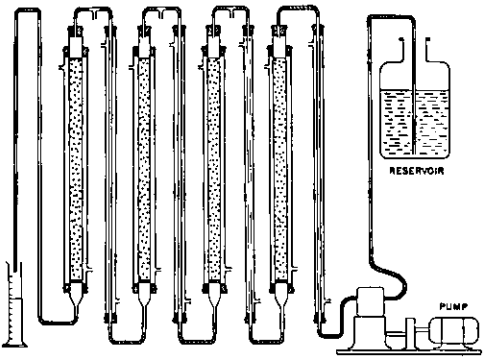

Figure 10-18. Laboratory columns.

The carbon should be charged to the columns in a manner that will avoid air entrapment. This may be accomplished by boiling the carbon in water and charging the hot slurry to the column at a sufficiently slow rate that a layer of water is always maintained above the bed. Alternately, if it is desired to avoid a "sweetening-on" or *sweetening* step, the carbon may be charged to the column in the hot sugar liquor.

After charging is completed the liquor is pumped through the column system at a predetermined flow rate. For the treatment of thick juice the flow rates investigated should be in the range of 0.5 to 1.0 gpm/sq ft of cross-sectional bed area. For less concentrated liquor the flow rate can be increased in inverse proportion to the solids content of the liquor.

When the column system is in operation effluent samples should be collected at regular intervals from the various bed depths for analysis. Feed samples should also be collected regularly. A series of curves can be constructed showing either color, or an alternate criterion of impurity concentration, as a function of throughput volume. These plots are commonly called breakthrough curves. Examples are shown in Figure 10-19 for the column treatment of thick juice. From the curves, the volume of acceptable effluent collected from each bed depth to a predetermined breakpoint can be determined and compared. From these figures the carbon dosage or the weight of carbon used per weight of sugar solids treated can be calculated. Ideally, during the adsorption cycle in a column process the carbon at the inlet end of the bed will be practically saturated with impurities, the carbon at the outlet end will be relatively free of impurities, and between these two extremes there will be a zone in which the bulk of the adsorption is taking place. The latter is referred to as the adsorption or mass transfer zone. This zone will move downward through the bed as the carbon becomes saturated and can be regarded as an adsorption wavefront moving through the column (20).

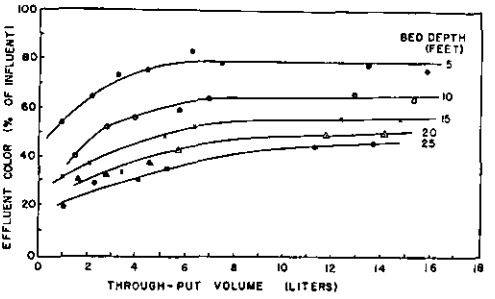

Figure 10-19. Breakthrough curves for the treatment of thick juice (65°Brix) at a flow rate of 1 gpm/sq ft.

The shape of the breakthrough curve provides an indication of the depth of the adsorption zone. A sharply defined and steep breakthrough curve is indicative of a relatively shallow adsorption zone. If, however, some breakthrough occurs immediately and considerable time elapses before the effluent concentration approaches the influent concentration, the adsorption zone is deep relative to the overall bed depth.

Invariably, for the treatment of sugar liquors, particularly for concentrated liquors such as thick juice, deep adsorption zones are encountered. Where this situation prevails, and a single fixed bed carbon column is used, only the carbon at the inlet will have a high adsorbate loading when the effluent from the outlet reaches an unacceptable level. The impurity loading on the carbon will decrease progressively from the inlet to the outlet end and thus most of the carbon will still have usable capacity. It would obviously be uneconomical to discharge all of the carbon for reactivation at this stage. Therefore, countercurrent usage of the carbon should be considered.

Countercurrent operation may be achieved by using a number of columns in series. When the effluent impurity concentration reaches an unacceptable level, the first bed is removed from service and replaced by a fresh bed of carbon at the outlet end of the system. This procedure is carried out indefinitely, and ensures that only highly saturated carbon is removed from the system. The external piping arrangement must be such that any one of the beds can occupy any position in the series. This can lead to complex piping arrangements where more than two beds are used.

An alternate approach to countercurrent operation that has found acceptance in the beet-sugar industry is the moving bed system. In this case, the liquor is passed through the column in an upflow direction. Spent carbon is withdrawn from the bottom of the bed either continuously or intermittently. Reactivated carbon is simultaneously added to the top of the bed.

Laboratory evaluation of the effects of countercurrent operation can be determined in the sectioned column system described above. This system can be operated for a number of cycles in a countercurrent fashion until relatively steady state conditions are established.

At this stage the carbon dosage can be calculated from the volume of liquor processed per cycle and the weight of carbon removed from the system in each cycle. In some cases, pilot moving-bed columns, 8 in. in diameter and 40 ft high have been used to determine countercurrent dosages.

Granular Carbon Systems in the Beet-Sugar Industry

Several systems are in operation in the United States and British factories. All of these are moving bed systems. Typically they each consist of two or more carbon adsorbers, a reactivation furnace, and auxiliary equipment for hydraulic transport and dewatering between the adsorbers and the furnace. The installation previously used at the Carlton plant of the Holly Sugar Corporation (18) was evolved after considerable operating experience with the various alternate system components at other sugar factories. It was a pulse-bed system in which the changing and withdrawing of carbon were performed intermittently rather than continuously.

In the Holly system, Figure 10-20, two columns were used in parallel, each 10 ft in diameter and having a straight side height of 35 ft. The columns (Figure 10-21) were fitted with a conical bottom and a dished top and each contained 2960 cu ft of carbon. This installation was originally designed for a thick juice but has also been used for thin juice treatment.

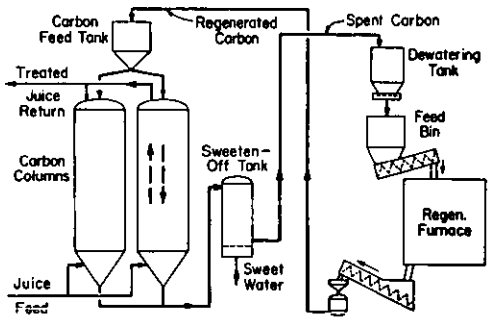

Figure 10-20. Flow sheet of granular carbon system at Holly Sugar Corporation, Carlton Factory, Brawley, California.

In the case of thick juice the products from the evaporators or storage juice tanks flowed through a heat exchanger to a surge tank feeding the column system. In the heat exchanger the temperature was raised to 85 ° C. The feed to the columns was maintained automatically at 64 to 65 ° Brix. The flow from the surge tank was divided equally between the two columns by a flow controller. Juice was introduced at the base of each column through six equally spaced inlets protruding into the conical section and fed by a manifold pipe encircling the column. The juice flowed upward through the carbon bed at a flow rate of 80 gpm. Purified juice flowed from the top of the column through eight tubular screen elements into launders, and then by gravity to the surge tank supplying carbon-treated juice to the factory.

At intervals of about four hours, a "slug" of spent carbon, approximately 160 cu ft, was discharged from one of the columns into a desweetening tank. At the same time an equal volume of reactivated carbon, slurried in thick juice, was introduced into the top of the column. During the slugging operation which required 15-20 minutes, the flow of juice to the column was stopped. The surge tank and the second column temporarily handled the total flow.

The slug of spent carbon in the desweetening tank was desweetened with hot water at a flow rate of 16 gpm. The sweetwater was returned to the column feed

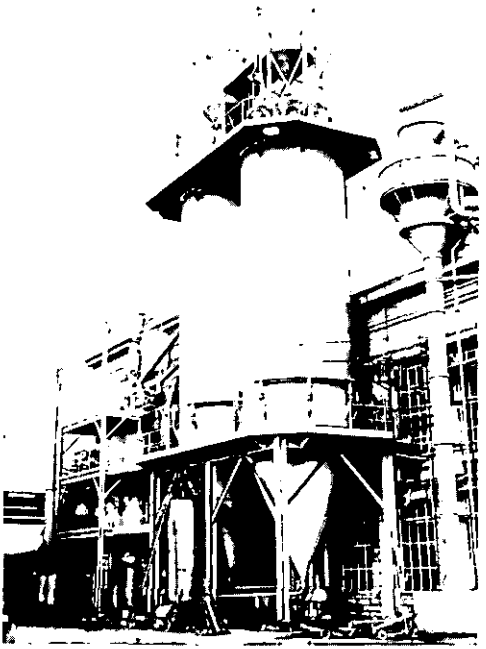

Figure 10-21. Typical moving bed system.

tank until the concentration reached 40° Brix. From 40° to approximately 0° Brix, it was returned to the plant raw juice supply. At this point the stream was diverted to waste and the washing was continued for an additional 30 min.

The carbon was then transported hydraulically to the dewatering tank for the removal of excess water by drainage, prior to reactivation. It then dropped into the furnace feed hopper at an average moisture content of 38%, wet basis. The carbon was fed to the multiple-hearth reactivation furnace described below, by a variable speed scroll.

Reactivated carbon dropped out of the furnace through a chute equipped with internal water sprays. The chute extended below the surface of the water in the quench tank, sealing the furnace from the atmosphere. The quenched reactivated carbon was conveyed up an inclined scroll which had a screened bottom for dewatering purposes, into the blow case feed hopper. The carbon containing about 48% water was slurried in treated thick juice, fed into the blow case and transferred by air pressure up to the carbon feed tank above the columns where it was stored until the next slugging operation.

The granular carbon installations at the five factories of Spreckels Sugar Company incorporate the same essential features of the Holly system, except that the adsorbers and the desweetening columns are designed for continuous rather than intermittent carbon flow (2). The carbon adsorption columns utilize the patented CAP (continuous adsorption process) principle (Figure 10-21). In these columns the syrup is fed to the bottom of the column at a rate such that the carbon bed is maintained in an expanded but not fluidized state. In an expanded bed the particles do not have sufficient freedom of movement to alter their position relative

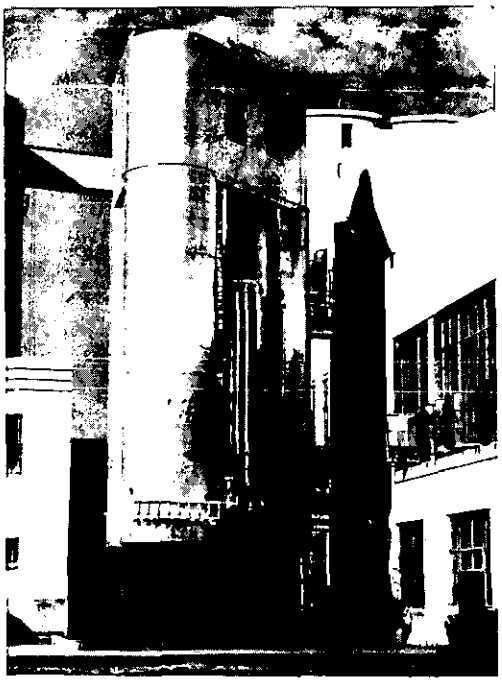

Figure 10-22. CAP columns at the Woodland factory of Spreckles Sugar Company.

to other particles. Consequently, a plug flow of carbon downwards through the column is achieved and countercurrent efficiency maintained. By contrast, in a fluidized bed individual particles can move in random fashion relative to each other, there is considerable mixing of fresh and exhausted particles, and countercurrent efficiency cannot be maintained.

In a typical installation, three adsorption columns are used in parallel, each 9.5 ft in diameter and 47.5 ft high. They are constructed of mild steel, epoxy-coated in the cylindrical section, and stainless-steel lined in the conical bottom. Each column is steam traced, insulated, and covered with an aluminum jacket. Each column contains 62 000 lbs of carbon with an expanded bed height of 31 ft. There is an annular carbon feed well at the top extending 3 ft below the liquor level and an 8-ft conical bottom for carbon collection and withdrawal. Feed liquor is admitted immediately above the conical bottom through nozzles in removable spargers or feed pipes. The treated liquor is collected in a launder at the top of the bed and returned by gravity to the sugar end of the factory. The spent carbon is withdrawn to the top of a central desweetening column 24 ft high and 18 in. in diameter, which operates similarly to the adsorption column. The carbon passes downflow through the column and is withdrawn continuously. Hot water is admitted at the bottom and the sweetwater emerging from the top is returned to the factory for purification and evaporation.

Plant Performance. In a test conducted in 1965, the effectiveness of the Holly granular carbon system described above was evaluated (18). In this plant-scale test the sugar obtained from thick juice after carbon treatment was compared with that obtained from non-carbon-treated thick juice of otherwise identical quality. During the test period carbon treatment effected a consistent upgrading of sugar quality, as shown in Table 10-4.

Costs are summarized in Table 10-5 which indicate that there was a 4% increase in the extraction of sugar fed, corresponding to a 5.5% increase in sugar bagged. The net operating cost of achieving this added extraction was only 0.675 per 100 lbs of sugar produced, since the costs of the carbon treatment were largely offset by savings made in other parts of the process.

Table 10-4 Effects of Granular Activated Carbon Treatment on Thick Juice and Sugar Quality (Carlton, 1965)

A. Average Thick Juice Results	Influent	Effluent	% Removal
Thick juice apparent purity, %	85.78	86.30	
Specific color [1]	493.3	342.8	30.5
pH	7.3	7.4	
B. Average White Sugar Results	**Treated**	**Untreated**	
Solution grade [2], color	92.4	91.0	
Solution grade, turbidity	98.5	99.8	
Floc test	0	+ 2.2	
C. Molasses Produced			
Molasses true purity	60.4	61.2	

[1] Adsorbance at 425 nm on filtered juice divided by the product of the cell length in cm and the concentration in g/ml.

[2] Transmittances of a 50° Brix solution in a 5 cm cell at 425 nm for color and 650 nm for turbidity.

Table 10-5 Cost of Granular Activated Carbon Treatment (Carlton, 1965)

	Treated	Untreated	Difference
Cwt sugar produced/cu ft juice	0.4008	0.37590	0.0248
Lbs carbon used/cwt sugar	0.1526	0	0.1526
Cost of carbon — $/cwt sugar produced	0.04822	0	0.04822
Added sugar loss " " " "	0.00244	0	0.00244
Cost of labor " " " "	0.19591	0.24076	—0.04485
Cost of fuel " " " "	0.14480	0.15648	—0.01168
Cost of other materials " " " "	0.07108	0.05846	0.01262
Total cost " " " "	0.46245	0.45570	0.00675
Extraction — % of total sugar fed	76.42	72.43	3.99

It was concluded that the extraction gain was not entirely due to the increase in juice purity but also to the considerable decrease in reboiling. It is also apparent from Table 10-5 that due to the decreased reboiling, the costs of labor and fuel were lower for processing carbon-treated syrup in spite of the additional manpower used and fuel required to operate the carbon treatment station. The increased materials cost for the carbon-treated juice was attributed to increased filter aid consumption. More recent increases in fuel costs have led to disuse of this system.

During the Holly test in 1965, carbon losses amounted to 6.3% of the carbon reactivated. The burn ratio, defined as lbs of carbon reactivated per 100 lbs of sugar treated, was 2.2 lbs. Sugar lost by retention on the carbon corresponded to 0.019% of the sugar processed. This amounts to 0.86% by weight of the carbon.

It is of interest to compare these results with those obtained earlier in other plant-scale evaluations of granular, activated-carbon systems. The results of a study of a very similar installation for thick juice treatment at the York factory of the British Sugar Corporation have been published (22). Sugar quality was compared before and after the start-up of the carbon system. After the start-up significant improvements in sugar quality were observed as measured by visual inspection, polarographic height, color, foaming index, and turbidity. It was found that the overall purity of the carbon-treated syrup increased by 0.1%, just statistically significant (3). At York, the amount of sugar retained on the carbon was 1.2% and the burn ratio 0.95%. The fact that significant improvements were achieved at a much lower burn ratio than that used at Carlton may be due to differences in syrup quality. The Carlton report mentions that highly colored syrups are processed at their test site.

The carbon losses at York were determined to be 7% of the carbon reactivated. However, the determination was made at a time when operating difficulties were encountered with the furnace. Average losses during normal operations were estimated to be in the 4 to 5% range.

In a third plant test the installation at the Woodland plant of the Spreckels Sugar Company, which incorporates the CAP columns described earlier, was evaluated (2). The test involved the comparison of two 17-week operating periods, one from the 1959 fall campaign and one from the 1956 fall campaign in which the quality of the beets was comparable. The granular carbon installation was in operation during the 1959 period.

The yield of white sugar was substantially higher and the molasses purity substantially lower during the 1959 campaign. A higher percentage of sugar was crystallized in each boiling and quality sugar could be prepared from lower purity liquors and massecuites. Because of these factors the capacity of the factory was increased by 236 tons of beets per day. The estimated savings effected by reduced sugar end recycling, reduction in molasses purity, and increased factory capacity are summarized in Table 10-6 with the additional costs incurred. Net savings of 7.3 cents per bag were obtained. While the cost figures in the table are out of date, comparable savings should be effected at the present time.

Extremely low floc content and marked reduction in the solution colors, turbidity and foaming characteristics in the finished sugar were reported.

The plant installations described above are concerned with thick juice treatment. Hiltgen and Matz (8) have summarized the results of a number of laboratory and pilot-scale tests on the carbon column treatment of juices ranging from 9 to 62 rds, from a wide variety of geographical sources. In these tests full-scale bed depths were employed, and the flow rates were adjusted so that the same conditions were used on the basis of pounds of sugar solids treated per unit of time. The burn ratio in most cases was 1.3 lbs carbon per 100 lbs sugar. Under these conditions apparent purity increases in the 0.6 to 1.0 unit range were noted and were essentially equal for thin and thick juice. Similarly, essentially equal degrees of decolorization were obtained for the two syrups. In one test, carbon treatment of thin juice eliminated the need for antifoaming agents in the evaporation step. Further, on the basis of one test, carbon used for thin juice treatment was more easily reactivated than that used

Table 10-6 Savings and Costs Summary (Woodland 1959)

	$ Per Bag of Sugar Produced		
	Saving		Cost
Reducing recycling in sugar end	$0.014		
Reduced purity of molasses produced	0.082		
Increased factory capacity	0.078		
Operating labor			$0.013
Operating materials and expense			0.071
Maintenance			0.016
Sugar loss			0.001
TOTALS	$0.174		$0.101
Savings before fixed charges and taxes with the CAP using CAL carbon		$0.073	

for thick juice treatment. In 1968 the Carlton installation was operated on thin juice with reportedly very satisfactory results.

Reactivation of Granular Carbon. The spent carbon withdrawn from the adsorption column, desweetened and dewatered, will contain the color bodies and other impurities adsorbed from the liquor treated as well as a small quantity of sucrose and 40-50 wt % water, not conveniently removed by conventional dewatering equipment. In the reactivation furnace the water is driven off and the adsorbed organics volatilized, charred, and oxidized primarily to volatile gases and vapors. Reactivation is usually performed in a direct-fired, multiple-hearth furnace at maximum temperatures in the 920 to 980 °C range. Steam and a small quantity of excess air are passed through the furnace.

Although the chemistry of the reactivation process is not well defined, it is likely that the principal reactions are those of the charred impurities with the steam and carbon dioxide present in the furnace atmosphere.

$$H_2O + C \rightarrow CO + H_2 \quad (3)$$

$$CO_2 + C \rightarrow 2\,CO \quad (4)$$

The principal function of the excess air is to convert the carbon monoxide and hydrogen back to carbon dioxide and water.

$$2\,CO + O_2 \rightarrow 2\,CO_2 \quad (5)$$

$$2\,H_2 + O_2 \rightarrow 2\,H_2O \quad (6)$$

The multiple-hearth furnace, Figure 10-23, consists of a vertical cylindrical shell lined with refractory which encloses a series of horizontal, circular hearths stacked one above the other.

A central shaft extends the full height of the furnace, supporting cantilevered rabble arms above each hearth with rabble blades attached to them. The rotation of the shaft and the attached arms spreads the carbon over the surface of the top hearth and takes it in a spiral path from the entry point near the center to drop holes at the outer edge. In the second hearth the carbon is raked from the periphery to a central drop hole. This alternate pattern of "out-hearths" and "in-hearths" is continued for the remaining compartments.

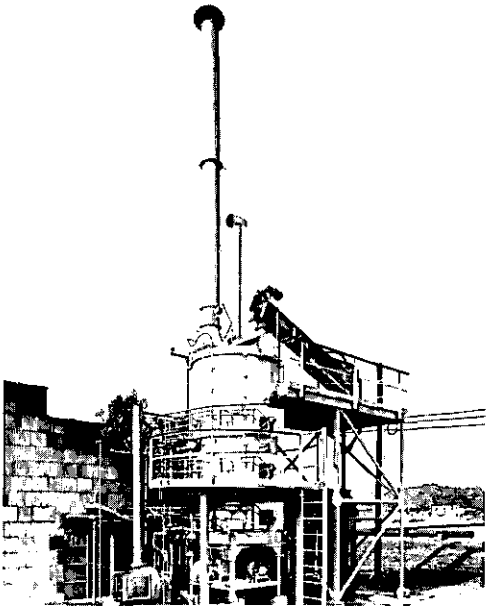

Figure 10-23. Multiple hearth furnace and dewatering screw.

Furnaces used in reactivation service generally range in diameter from 3 ft to 13.5 ft and may consist of from 6 to 10 hearths. One typical unit is 7 ft in diameter and has 8 hearths. The furnace is fired on hearths 4, 6, and 8, numbered from the top, with natural gas, and has an air blower that supplies supplementary air to the hearths as well as primary air to the gas burners. Hearths 4, 6, and 8 are also equipped with steam lines. Steam requirements range from 0.5 to 1.0 lb steam per lb dry carbon.

Care should be used in the selection of test methods to control furnace operation and to evaluate the quality of the reactivated carbon. Specifications for sugar-treatment carbons normally include such physical properties as hardness or abrasion resistance, apparent or packed bulk density, and particle size. Adsorptive properties are characterized by surface area measurements or by tests involving the adsorption of specific substances from solution, such as iodine, or the colored components of blackstrap molasses. Apparent density measurements as well as iodine and molasses adsorption tests have been used to determine the degree of reactivation of service carbon; however, the results should be interpreted with caution.

The apparent density of activated carbon may increase in service either by an accumulation of oxidation products of adsorbed organics in the fine pores or by a small but measurable accumulation of ash in these pores. For this reason, attempts to restore the virgin apparent density may result in over-reactivation and activity loss. Accordingly, apparent density measurements should be used as guide lines in conjunction with activity tests.

Similarly the use of iodine adsorption as a single test is not recommended. Iodine adsorption is indicative of total surface area, and the fine pore blockage will also reduce the total surface area. However, the relatively large molecules con-

stituting the impurities in sucrose solutions do not penetrate into the very fine pores of the carbon. Consequently, the blockage may not affect the capacity of the carbon for the components of interest. This is illustrated by an investigation (3) which reveals that although the iodine adsorption of a reactivated beet sugar carbon was markedly lower than that of the virgin adsorbent, the reactivated carbon had considerably better acid-floc and saponin capacity than the virgin material.

Molasses color adsorption is a better indicator than iodine adsorption of the surface area available to the larger molecules. Ideally, however, the performance of the reactivated granular carbon should be evaluated using syrup representative of that encountered in the plant. Isotherm-type tests are of value in this respect. A recently published (1) ASTM general isotherm procedure should be adaptable to reactivated beet sugar carbons.

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Chapter XI

EVAPORATORS AND FUEL ECONOMY*

Introduction. Evaporation of water from the beet juice is done in two stages, the first stage in the evaporators and the final stage in the vacuum pans. This chapter is concerned with the first stage.

Heat is required to evaporate water from the beet juices and sugar syrups encountered in beet-sugar factory processes. This heat is universally supplied to evaporators and vacuum pans in the form of steam. Water evaporated from the beet juices and sugar syrups leaves as steam. Thus to understand the evaporation process it is necessary for the reader to understand certain thermodynamic concepts.

Thermodynamic Definitions. Water exists in three states, solid, liquid, and gaseous or, in other terms, ice, water, and vapor or steam. Steam is a special term for water vapor. However, in the beet-sugar industry, steam and vapor are given special definitions to improve the facility and accuracy of communication. "Steam" refers to water vapor first formed in a steam generator, such as a boiler. "Vapor" refers to water vapor first formed in an evaporator or vacuum pan. Heat and temperature units are defined as functions of these states. The "ice point" is the temperature of pure melting ice under standard atmospheric pressure, in contact with pure water saturated with air, at the same temperature. The boiling point of water is that temperature at which the vapor pressure of the liquid water equals the pressure above the surface of the water. Evaporation of water is brought about by the addition of heat to the water in sufficient quantity to cause it to change from the liquid to the gaseous state.

Fahrenheit and Centigrade temperature scales are in common use in beet-sugar factories. The Fahrenheit scale divides the temperature interval between the ice point and the steam point into 180 equal parts, while the Centigrade scale divides it into 100 equal parts. On the Fahrenheit scale, the ice point is defined as 32 °F, and on the Centigrade scale as 0 °C. Then by the above definition, the steam point on the Fahrenheit scale is 212 ° F, and on the Centigrade scale 100 °C. To change Centigrade to Fahrenheit degrees use the following formula:

$$F^\circ = \frac{9}{5} C^\circ + 32 \qquad (1)$$

To change Fahrenheit to Centigrade (Celsius) degrees use the following formula:

$$C^\circ = \frac{5}{9}(F^\circ - 32) \qquad (2)$$

Thus, 100 Centigrade degrees are equivalent to 180 Fahrenheit degrees, or 1 C ° = 1.8 F °.

Pressure is defined as the force acting upon a unit area. The unit of pressure most commonly employed is *pounds per square inch* or *psi*. Standard atmospheric pressure is the pressure exerted by a column of 76 centimeters or 29.922 inches of

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mercury at sea level and 0 °C at standard acceleration of gravity, and is equal to 14.696 psi. Standard atmospheric pressure is considered equivalent to the normal atmospheric pressure at sea level. The common pressure gauge indicates gauge pressure, psig, which is the *absolute pressure* minus the actual atmospheric pressure. To obtain absolute pressure (psia), use the common pressure gauge reading and add to it the actual atmospheric pressure obtained from a barometer, or use a special absolute pressure gauge.

In the United States the common unit of heat is the British thermal unit or Btu. It can be approximately defined as the quantity of heat required to raise the temperature of a one pound mass of water through 1 °F. It is defined exactly as 778.26 foot-pounds.

The beet-sugar factory evaporator evaporates water from the beet juice, so the thermodynamic or heat properties of water are of concern. The heat content of water and water vapor, as well as other fluids, is separated into several classifications, depending on the physical state.

The heat content of water is called the enthalpy of water and it is the heat content of one pound of water above the heat content of water at some arbitrary temperature, generally 32 °F.

The *enthalpy of evaporation (latent heat)* of water is the heat required to evaporate one pound of water at constant temperature and pressure. The resultant pound of steam, termed *saturated steam,* will have been evaporated from water at its boiling temperature to steam at the same temperature. It is to be noted that the enthalpy of evaporation is not constant but varies with the boiling temperature, which, in turn, varies with the absolute pressure in which the liquid is boiling. For example, pure water boiling at 35 °F will be in an absolute pressure of 0.09991 psia and will have an enthalpy of evaporation of 1073.8 Btu per lb mass. At 366 °F and 164.968 psia the enthalpy of evaporation is 856.9 Btu per lb mass (2).

Superheat is additional heat added to the saturated steam. It raises the steam temperature above the saturated steam temperature. There are other classes of heat which will not be discussed here. Some of these are heat of crystallization and dilution, latent heat of fusion, heat of sublimation, etc. Further information is available in many standard texts on thermodynamics (9,11,15,17). Heat in these classifications is usually measured in terms of the Btu or its metric equivalent, the calorie.

Specific heat is not a quantity of heat, but rather the ratio of two particular quantities of heat. The specific heat of a substance is defined as the ratio of the heat required to raise the temperature of that substance one degree, to that required to raise the temperature of an identical weight of water, usually from 62 ° to 63 °. As it has no units it has the same value in the English and metric systems.

An essential tool for heat calculations are the steam tables (2). These tables list for water the enthalpy of the saturated liquid, the latent heat of evaporation and the sum of these two, or the enthalpy of the saturated vapor, for a wide range of temperatures and pressures. The enthalpy of superheated steam is also tabulated.

Description of Evaporators. Many years ago the horizontal-tube evaporator, patented by Wellner and Jelinek in 1879, was in almost universal use in the beet-sugar industry of the United States. This evaporator is usually rectangular in horizontal section and inverted U in vertical end section with the top being semicylindrical. A horizontal-tube bundle is located in the bottom portion of the evaporator body. At each end of the tube bundle there is a steam chest which pro-

trudes from the evaporator shell. Steam is on the inside of the tubes and juice is on the outside. The tubes are quite small in outside diameter—usually between 0.75 and 1 in. diameter.

For many years calandria-type evaporators have found favor. Also known as the Robert or standard type they have vertical cylindrical shells with dished bottoms and heads. There is usually an entrainment separator mounted in the head to remove liquid droplets resulting from the boiling action and carried up with the vapor. The tubes are vertical, from 5 to 10 ft in length, and are usually 1.5 to 2 in. outside diameter. The tube bundle is located in the bottom portion of the evaporator body, the bottom tube sheet being within a foot or two of the dished bottom. There is usually a cylindrical center well down through the center of the tube bundle, which is usually about one-fourth of the diameter of the evaporator shell. When operating, liquor circulates up through the tubes and down through the center well. Liquor is usually fed in under the bottom tube sheet and usually leaves at a point near the top of the center well. There are many designs for feeding and removing liquor from a calandria evaporator. Many of these attempt to reduce or eliminate the recirculation of liquor within the evaporator body, to increase capacity and to minimize destruction of sucrose by heat. Most of these attempts are only partly successful because instrument control of the evaporator function is also desired, and so far all the instrumentation systems require some recirculation of liquor within the calandria to maintain stability (1,18).

A number of long-tube, vertical-film type evaporators have been installed in recent years. These evaporators have vertical tubes 18 to 32 ft long and about 1.5 in. outside diameter. There is no center well. Two types are in use, the climbing film and the falling film. In the climbing film type the liquor enters below the bottom tube sheet and flows up inside the tubes. As the liquor flows up through the tubes, heat for evaporation is furnished by steam on the outside of the tubes so that a mixture of liquor and vapor leaves the upper end of the tubes with a high velocity. The top of the tube bundle extends one or two feet into a cylindrical chamber or dome. An umbrella-type baffle separates the liquor from the vapor in the dome so that the liquor can be withdrawn from the bottom of the dome and the vapor from the top.

In the falling film type the liquor is fed into the top of the tubes. The design is such that each tube is assured of its proportionate share of liquor. As the liquor flows down through the tubes, heat for evaporation is furnished by steam on the outside of the tubes so that a mixture of liquor and vapor leaves the bottom end of the tubes with a high velocity. Means are provided at the bottom of the tube bundle to separate the liquor and vapor.

In beet-sugar factories, the horizontal-tube type of evaporator has not been used with steam pressures over about 17 psi, the calandria type over about 40 psi, and the film type over about 45 psi.

Entrainment separators are normally installed with each evaporator body. These are located in the leaving vapor stream and can be either inside the body or in the vapor line external to the body.

Evaporator Operation

Thin juice, after sulfitation, is pumped through the thin-juice heaters and into the evaporators, where a large part of the water is removed from the beet juice. For

the most economical operation the thin-juice heaters should discharge juice at or above the saturated temperature of the first vapor. This increases the capacity of the evaporators and facilitates both heating and evaporation. The usual set consists of four or five bodies or effects in series, and are known as quadruple- or quintuple- effect evaporators. Figure 11-1 shows a quintuple-effect evaporator.

An evaporator body is a single evaporating unit. An evaporator effect can be a single evaporator body or several evaporator bodies connected in parallel. In the beet-sugar industry an evaporator effect generally consists of only one body, although there are many cases of two-body effects and some three-body effects.

It is common practice to feed the thin juice into the first effect, throttle juice from the first effect into the second effect, throttle juice from the second effect into the third effect, etc. This is known as forward feed of the evaporator. Water is evaporated in each effect and so as the juice progresses through the evaporators, its density increases. Juice from the last effect is called thick juice. Backward feed is occasionally used, wherein thin juice enters the last effect, and the juice is then pumped out of each effect into the preceding effect and finally out of the first effect.

Exhaust steam is piped into the first-effect steam chest. On condensing, this steam gives most of its heat to the juice in the first effect and the transfer of heat causes the juice to boil. Vapor from the boiling first-effect juice passes through the evaporator dome and is piped into the vapor or steam chest of the second effect. First-effect vapor in the second-effect vapor chest gives most of its heat to the juice in the second effect, and so produces second-effect vapor. This process continues down through the evaporators until the vapor from the last effect is led to a condenser. This condenser maintains the last vapor at a low pressure, usually well below atmospheric pressure and consequently at a low temperature. Thus, each evaporator effect acts as a condenser for the preceding effect. Hence, each suc-

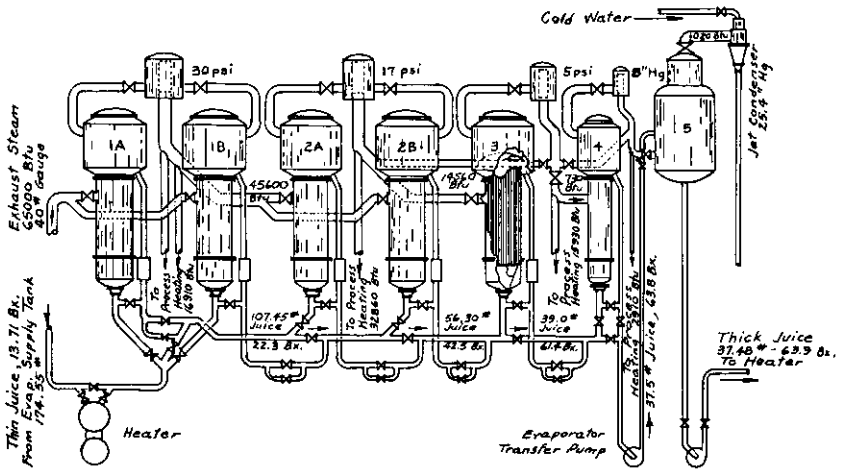

Figure 11-1. Typical modern long-tube vertical evaporators, with two first bodies, two second bodies, and calandria-type fifth effect. Heat and material quantities from Tables 11-1, 11-2, and 11-3. All pressures shown are gauge pressures.

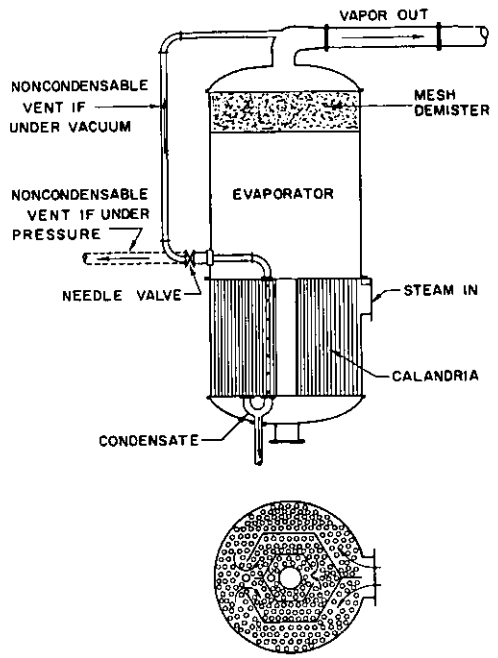

Figure 11-2. Sectional elevation of a short-tube vertical evaporator body.

ceeding vapor pressure, and, of course, temperature, is proportionately lower with the lowest vapor pressure in the last effect under control by the condenser.

One pound of steam admitted to the first-effect steam chest evaporates approximately one pound of water from the juice in the effect. One pound of the first vapor thus formed, when admitted to the second-effect vapor chest will evaporate approximately one pound of water from the juice in the second effect. This process continues through each succeeding effect. Consequently, one pound of steam admitted to the first-effect evaporator will evaporate approximately five pounds of water in a quintuple-effect evaporator, four pounds of water in a quadruple-effect evaporator, etc.

The accuracy of this approximation depends on the vapor-pressure and temperature levels on the steam and juice sides of the heating surface of the evaporator effect in question. The actual amount of water evaporated in an effect, per pound of steam or vapor condensed, can be calculated from the steam and juice temperatures by the use of the steam tables. For example, assume an evaporator effect has a steam chest temperature of 231.8 °F, and a juice temperature of 217.9 °F. Juice will be throttled into the evaporator at the temperature of the juice in the previous effect, which is the same as the steam-chest temperature in the evaporator under discussion of 231.8 °F and leaves at the juice side temperature of 217.9 °F. In this example we have assumed no boiling-point rise and a specific heat of 1.0. Evaporation comes from two sources: juice "flash," and condensation of steam or vapor in the steam chest.

Evaporation by juice flash occurs because the juice entering at 231.8 °F is above the saturation temperature of the effect in question, and so must cool to the saturation temperature of 217.9 °F by self-evaporation, which is termed flashing. Each pound of juice will give up the difference in enthalpy of a saturated liquid between 231.8 °F and 217.9 °F.

Enthalpy of saturated liquid at 231.8 °F = 200.18 Btu/lb.

Enthalpy of saturated liquid at 217.9 °F = 186.11 Btu/lb.

Difference given up by juice in flashing = 14.07 Btu/lb.

The heat required to evaporate one pound of water at 217.9 °F to form one pound of vapor at 217.9 °F is 966.6 Btu. Hence each pound of juice, when flashed, will evaporate from itself 14.07/966.6 or 0.015 pounds of water. Assuming that there are three pounds of juice flowing through this evaporator effect for each pound of steam condensed, the evaporation by flashing, per pound of steam condensed, will be 3×0.015, or 0.045 pounds.

The amount of evaporation obtained from one pound of condensing steam can be calculated using the enthalpy of evaporation, or latent heat of evaporation, obtained from the steam tables for the two temperatures.

Enthalpy of evaporation at 231.8 °F = 957.5 Btu/lb.

Enthalpy of evaporation at 217.9 °F = 966.6 Btu/lb, and so 957.5/966.6, or 0.991 pounds of water are evaporated from the juice by one pound of steam. Hence the total evaporation per pound of steam is 0.991 + 0.045, or 1.036 pounds.

Beet-sugar factories usually use exhaust steam in the first-effect evaporator. Exhaust steam, as the term implies, usually consists of the exhaust from the turbines, which use high-pressure steam directly from the boilers. It is possible to keep the demand for exhaust steam by the evaporators and other equipment exactly equal to the production by the turbines. If the production of exhaust steam is greater than the demand, the excess production must be relieved to the atmosphere or "blown off," and represents a total loss. For this reason, in a well-designed factory, the turbines never make enough exhaust steam to satisfy the demand. To make up this deficiency of exhaust steam a pressure controller, actuated by the exhaust steam pressure, bleeds high pressure boiler or "live" steam into the exhaust system. It should be pointed out that this exhaust-steam pressure controller should be so set that the live-to-exhaust steam make-up valve will close below the pressure setting on the exhaust steam relief valves to atmosphere.

Heat Transmission

In studying the transmission of heat from one substance to another in various types of apparatus, valuable use is made of the *overall coefficient of heat transfer,* which is defined as the heat transferred per unit of time, per unit area, per degree overall temperature difference. In engineering units it is the number of Btu which will pass in one hour from a warmer to a colder fluid through one square foot of surface, when the temperature difference between the warmer and colder fluids is one °F. The usual English units of the overall coefficient of heat transfer are Btu per hr per sq ft per °F. The overall temperature difference in the evaporator effect discussed above was 231.8 – 217.9 or 13.9 °F.

The area of heat transfer surface is rather difficult to determine precisely, when the surface consists of tubes. If the tube walls are relatively thin, the area of surface can be closely approximated by calculating the surface on the liquid side.

For the horizontal-tube evaporator this is the outside surface of the tubes; for the calandria evaporator and the long-tube vertical film-type evaporator this is the inside surface of the tubes.

The capacity of any given set of evaporators, or quantity of water that can be evaporated, depends almost entirely on how rapidly the heat can be transmitted through the tubes; or, in other words, depends upon the overall coefficient of heat transfer. Consequently, anything which affects this overall coefficient affects the capacity of the evaporators.

Factors affecting the overall coefficients of heat transfer and the capacity of the evaporators are: vapor velocity, air and noncondensable gas removal, steam or vapor superheat, condensed water removal, surface cleanliness, tube composition, juice height in body, juice viscosity, temperature difference, boiling-point temperature, surface tension of juice, and others.

The overall coefficient of heat transfer is composed of three or more individual coefficients which exist in every heat transfer surface. In a beet-sugar factory evaporator the first resistance met is that offered by the film of steam and condensate on the steam side of the tubes, involving the steam-film coefficient of heat transfer; the second resistance met is the metal tube wall, involving the coefficient of heat transfer for the metal tube wall; the third resistance met is the scale on the tube, involving a coefficient of heat transfer for the scale; and the fourth resistance met is the juice film adhering to the scale, involving the liquid film coefficient. Each of the factors listed above affects one of the individual coefficients and in so doing affects the overall coefficient (6,10,13,16).

Vapor Velocity

In theory, the transmission of heat will be better, the greater the velocity with which the steam passes over the heating surface. In practice, unless the overall coefficient is very high, the vapor velocity over the heating surface has but little effect on heat transmission. It has been shown that the vapor velocity against a heating surface caused by condensation alone is sufficient for most conditions met in practice (3,7). In general, there is little that can be done to improve conditions on the steam side except in two cases: 1) changes in the coefficient due to a change from film-type to dropwise condensation, and 2) changes due to a change in the amount of noncondensables.

In 1930 it was discovered that there are two types of condensation: film-type and dropwise. In film-type condensation the steam condenses in a continuous film over the heating surface, and in dropwise condensation the steam condenses in drops which grow rapidly and run down the heating surface. A change from film-type to dropwise will, in certain cases, double the overall coefficient of heat transfer. A very slight amount of oil or grease promotes dropwise condensation.

Air and Noncondensable Gas Removal

Beet juice contains certain compounds of nitrogen, and the carbonation process leaves some free and combined carbon dioxide in the juice. When the juice is boiled, some of the nitrogen compounds break down into ammonia and most of the carbon dioxide is driven off. In the evaporators these two gases, carbon dioxide and ammonia, as well as air and other gases, leave the beet juice and mingle with the vapor. These gases will not condense along with the steam or vapor and so they are

termed noncondensables. As such they interfere with and slow down the condensation of steam or vapor. Since very little of these noncondensables can leave the evaporator with the condensate, they tend to collect in pockets which will blanket off a portion of the heating surface and interfere with heat transfer through that surface. To prevent these pockets from forming, the noncondensables must be bled off to a lower pressure.

As a stream of steam or vapor passes over a heating surface, it tends to sweep the noncondensables along with it, and as the steam condenses, the mixture that is left becomes richer and richer in noncondensables. This means that in an evaporator or heater where the steam has a definite path, as in horizontal-tube evaporators or in long-tube film-type evaporators, the noncondensables will be swept to the far end of the path and can be removed there quite completely.

Noncondensables in a steam chest separate out as described above, rather than by gravity. A common example is the fact that water vapor in air does not rise to the top of a room but stays thoroughly mixed with the air even though it is lighter than air. In other words, a gas mixture consists of a mixture of molecules of each constituent, the molecules of any single constituent being distributed uniformly through the entire space and also acting or moving quite as if they occupied the space alone. This statement follows from Dalton's law, which states that every portion of a mass of gas enclosed in a vessel contributes to the pressure against the sides of the vessel the same amount that it would have exerted by itself had no other gas been present. Hence, these pockets of noncondensables have a continuous tendency to diffuse back into the vapor.

So, in the horizontal-tube evaporator, the vents can be located anywhere past the ends of the last tube pass. In the long tube vertical film-type the steam should be admitted at one end of the tube bundle and the noncondensables removed at the other end. In calandria-type evaporators the steam path is not well defined. Consequently, most calandria designs include vertical baffles for the purpose of creating a steam path, so that the noncondensables can be successfully vented from the end of the steam path. Unfortunately, the steam flow in many calandrias is not completely positive from the steam inlet to the vents, and so dead spaces or pockets tend to form where there is very little flow or sweeping action of the steam. In these dead pockets noncondensables will collect as the steam which brings them is condensed. Laboratory tests have shown that these pockets may be merely very thin layers of noncondensables on the heating surface, and yet these layers effectively prevent the transfer of all but small amounts of heat through the surfaces blanketed.

Usually, high concentrations of noncondensables against the heating surface produce accelerated corrosion at that point. In such a case, an extra vent can be installed.

Vent lines from the evaporator effects can be connected to discharge to various points. Noncondensables can be bled from a steam chest to the vapor dome of the same or any following effect; however, where the steam or vapor pressure is above atmospheric pressure it can be vented to the atmosphere. Venting can be to the evaporator condenser or even to a vacuum filter condenser. Steam in the first effect chest usually carries very few noncondensables and is frequently vented to the atmosphere. The vent from the second-effect steam chest receives all of the noncondensables boiled off in the first effect, which forms the largest portion of noncondensables to be vented. If these are bled to second vapor, they will materially impair the overall coefficient of heat transfer in the third effect, so the best practice is to vent them to the atmosphere. If the second-effect steam chest is below atmospheric pressure, it can be vented to the evaporator condenser, although this will

impose more load on the condenser. Remaining effects may be vented to any of the points mentioned; however, venting an effect to its own vapor should be done with discretion. At times it can be economical to vent to a surface or contact heat exchanger on another service. With a surface heat exchanger the coefficient of heat transfer is likely to be low and the unit can be subject to corrosion. With a contact heater the fluid absorbing the heat is likely to absorb considerable noncondensable gases as well. Similar principles for venting noncondensables may be applied to other heat exchangers in the beet-sugar factory.

Too much venting wastes steam. Too little venting reduces capacity. It is best to vent a little more than is absolutely necessary. An open bleed to atmosphere can be regulated by adjusting a throttle valve to give a small visible plume of condensed vapor at the outlet. A closed bleed can be adjusted to give optimum operation of the evaporator or heat exchanger as indicated by the instrumentation or as determined by a heat balance.

Steam and Vapor Superheat

Superheat in the steam or vapor may reduce the overall coefficient of heat transfer. The exact amount of superheat necessary to materially affect the overall coefficient of heat transfer is indefinite. A test of a horizontal-tube evaporator with saturated steam and steam superheated 25 °F, showed no change in the overall coefficient of heat transfer. A test on a vertical-tube evaporator with 40 °F superheated steam also showed no difference (4,8). Operation of a long-tube vertical evaporator using 180 °F superheated steam showed no appreciable difference at the Woodland, California factory. It is likely that superheat has but very little affect on the overall coefficient of heat transfer in beet-sugar apparatus.

Condensed Water Removal

Condensed water should be removed from the evaporator as it is formed so that it cannot cover the heating surface and impede heat transfer. In horizontal-tube evaporators the tubes should be slightly inclined, so that the water drains to the steam chests, from which it can be removed by pumps or through traps. In calandria-type evaporators, several condensate outlets from the bottom tube sheet are usually provided. In the long-tube film type one design allows the condensate to run over the edge of the bottom tube sheet into a condensate chamber from which it can be removed by traps or pumps. Condensate from the evaporators, vacuum pans, and heaters is quite valuable because it is hot and contains very few impurities. Its high temperature and purity enables some of it to be used as boiler feed water and press wash water. The remainder can be used in diffuser supply water and for general washing up. From the standpoint of heat and fuel economy, live and exhaust steam condensate are best for boiler feed water and first vapor condensate second best. Juice heater condensate is not normally used in boiler feed water since the juice pressure is usually higher than the vapor pressure, hence any leaks will allow juice to contaminate the condensate.

Gauge glasses should be provided on all steam chests to indicate the level of condensate and should be watched carefully to see that the condensate is being removed as rapidly as it is formed. In some types of apparatus, condensate accumulation will cause water hammer, which can produce dangerous stresses in the

apparatus. If a heater has ample capacity, a high condensate level does no particular harm as long as it does not cause water hammer. A properly designed heater and condensate system is not subject to water hammer.

There are many different arrangements for condensate removal. It is not within the scope of this chapter to discuss all the various condensate systems, excepting perhaps one type which is particularly economical of heat and fuel. This type successively bleeds the condensate from one effect to the next, and in so doing, recovers heat from the condensate. For example, if condensate comes from an effect at 220 °F and is bled into a chamber connected to saturated steam or vapor at 210 °F, the condensate will flash down to 210 °F. The 10 °F the condensate lost will be converted into saturated steam or vapor. Hence, we can successively flash first-effect condensate to first vapor, second vapor, third vapor and fourth vapor in a quintuple-effect evaporator. In a like manner, second-effect condensate can be flashed to second vapor, third vapor and fourth vapor. This system is usually only partially carried out because of boiler-feed water economies which were discussed above, and because a certain volume of high temperature water is required for use in the factory.

Surface Cleanliness

Scale on the liquid side of tubes is the largest and most important factor retarding the transfer of heat in beet-sugar factory evaporators. Even extremely thin scales can be very detrimental. One writer (5) reports that the removal of a scale so thin that it could be easily rubbed off leaving only a greyish stain on the finger raised the overall coefficient from 1100 to 1600 Btu/(hr) (sq ft) (°F) with a constant temperature drop of 40 °F. While commercial evaporators and heat exchangers seldom show heat transfer coefficients as high as these, the important effect of scale is illustrated.

If the tubes become covered with scale, their heat conductivity is greatly reduced and the capacity of the evaporators is lessened. There are certain salts which are forced out of solution as the juice is concentrated. A further requirement for a true scale-forming substance is that its solubility decreases with increasing temperature. As the juice thickens in the evaporators, some of the substances deposit on the tubes, producing a heat-insulating scale. The rate of scale formation can be minimized by proper treatment of the juice in carbonation and sulfitation before it enters the evaporators.

Scale is usually of such a nature that dilute hydrochloric acid alone will not dissolve it, and so the ordinary procedure is to boil first with a solution of soda ash and caustic soda, which changes the nature of the scale so that acid will attack it, and then to boil with the acid. In some cases ammonium bifluoride [NH_4HF_2] has been used. Boiling out is usually accomplished without shutting down the evaporators. The juice is bypassed around the body which is to be cleaned and the body emptied. The soda solution is introduced and boiled; the soda is drained out, and the acid introduced and boiled. In this procedure the boilout solution must be boiled at the same rate and at the same temperature as the juice it displaced. As high-pressure evaporators have come into use, such a procedure causes difficulty because of the high boilout temperatures required, which cause a portion of the acid to vaporize and enter the vapor system. To overcome this problem, vapor bypasses can be installed so that any body can be isolated. Arrangements can then be made to boil out at any desired temperature and rate. Much better boilouts result.

Deposits on the steam side of the tubes are not ordinarily important. However, any such deposits should not be overlooked if an evaporator is not performing up to capacity.

It is desirable to have a measurement of the capacity of each evaporator effect. This will indicate: the proper times to boil out; operations that cause rapid scale formation; the value of scale preventatives; and the thoroughness of each boilout. A good measure of capacity can be obtained by charting each day the difference in temperature between the steam side and the juice side of each effect.

Tube Composition

Different metals conduct heat at different rates. The rate of conduction is usually expressed in the units Btu/(ft) (sq ft) (°F) (hr). The rates of heat conduction or thermal conductivities for several metals follow: silver 243, copper 222, aluminum 118, red brass 89, Admiralty brass 65, yellow brass 63, mild steel 35, 302 stainless steel 17.

A metal tube wall normally offers only a small portion of the total resistance to the flow of heat from the steam side of an evaporator to the juice side. Consequently, the type of tube metal used is usually of minor consequence for the overall coefficient of heat transfer. For example, if an evaporator has 1.25 in. od steel tubes, 0.14 in. thick, obtaining an overall coefficient of heat transfer of 207 Btu/(sq ft) (°F) (hr), and a change is made to copper tubes only 0.067 in. thick, the overall coefficient will be increased to 221 Btu/(sq ft) (°F) (hr). The influence that a tube metal has on the overall coefficient increases as the overall coefficient increases, so with very high overall coefficients tube metals can be important.

Corrosion is another factor dependent in some cases on the type of tube metal used. Copper is attacked by ammonia, and steel will rust. Bimetallic tubes are sometimes used to counteract corrosion. The products of corrosion are often much like scale and similarly retard heat transfer.

In applications where it is necessary to clean tubes mechanically, the tube metals should be suitable for this operation.

The rate of heat transfer in evaporators of the horizontal tube and calandria type is a function of the celerity with which the steam bubbles leave the heating surfaces and are removed from the juice. The level of the juice in the evaporator is important in this regard, as the lower the level, the quicker the bubbles are removed. This explains the fact that it has been found advisable in these types of evaporators to keep the juice level inside a body just over the tops of the tubes for maximum capacity.

The gauge glass on the body does not show the true height of the boiling juice, which is expanded and raised by the steam bubbles. If the juice should cease boiling and the bubbles subside, the level inside would correspond approximately with the level shown on the gauge glass. It is advantageous to run a few tests to find the optimum juice level for maximum capacity because the general rule given here is modified by the specific design.

Long tube film-type evaporators are not subject to hydrostatic head except in the rising film version where juice enters a body below boiling temperature.

Juice Viscosity

The rate of heat transfer is dependent to a high degree on the viscosity of the juice, because a more viscous juice is less easily penetrated by steam bubbles.

Viscosity increases with increasing concentration and with decreasing temperature. Since both these changes occur with forward feed as the juice passes from one evaporator body to the next, the juice becomes more viscous, and the rate of heat transfer decreases in the later effects. Note that in a backward fed evaporator the effects of concentration and temperature on viscosity operate in opposite directions.

Temperature Difference

When the temperature difference increases between the steam or vapor used for heating and the juice to be heated, the amount of heat transmitted increases and consequently the amount of evaporation. This statement is only true, of course, when there is no change in other factors. Increased temperature difference means there will be more steam bubbles rising and consequently greater circulation in the evaporator effect. This will increase the overall coefficient of heat transfer.

It is well to point out that the overall coefficient of heat transfer is markedly influenced by circulation. Circulation, in turn, is almost entirely determined by whether or not forced circulation is used and many of the factors under discussion.

Boiling Point Temperature. The higher the boiling point temperature, the lower the viscosity and the better the circulation and the overall coefficient of heat transfer.

Surface Tension of Juice. Laboratory and pilot plant experiments have shown remarkable increases in the overall coefficient of heat transfer by the addition, to the evaporator feed, of very small amounts of wetting agents or materials which reduce the surface tension. The main difficulty in the beet-sugar industry is to find one that is nontoxic, that is not too costly, and that will not distill over into the condensate to cause trouble in the boilers.

Other Factors. Several other quite minor factors affect the overall coefficient of heat transfer. Some of these are pH of juice, density of steam or vapor, density of juice, condensate temperature, and thermal conductivity of the liquid.

Boiling Point Elevation. A water solution such as beet juice boils at a higher temperature than pure water. The increase in the boiling point over that of pure water is called *boiling-point elevation,* or *boiling point rise.* The boiling point elevation varies with the concentration of the juice or syrup, its purity and the pressure under which the juice is boiling (12,14).

It has been proved experimentally and in practice that vapor leaves the boiling juice at the temperature of the juice. This means that the vapor leaves the juice slightly superheated by the amount of boiling-point rise. This small amount of superheat is quickly eliminated by radiation or upon entering the next evaporator effect.

The total temperature drop across a set of evaporators is equal to the sum of the temperature drops across the heating surfaces, plus the sum of the boiling-point elevations. That boiling-point elevations absorb some of the total temperature drop, is demonstrated in the following example: A particular juice boiling at 193 °F, has a boiling-point elevation of 2.2 °F. Hence, the vapor from this juice will be superheated by the boiling-point elevation of 2.2 °F. This vapor at 193 °F upon entering the steam chest of the next effect, immediately loses its superheat and becomes saturated at 190.8 °F. Hence, this vapor chest has a temperature of 190.8 °F rather than the juice temperature of the preceding effect at 193 °F, and 2.2 °F is lost, in that it is not available as a driving force for evaporation, although the energy released contributes to the evaporation.

When dry, saturated steam is throttled it becomes superheated. If vapor is throttled between evaporator effects by a valve, by too small a pipeline, or any other restriction, it becomes superheated, and evaporator capacity is lost in a manner similar to that due to boiling-point elevation. It is desirable to have vapor lines of adequate size.

Total or Overall Temperature Difference. The difference in temperature between the condensing steam in the first effect and the vapor from the last effect is defined as the "total temperature difference." if this difference is increased in any way, as by increasing the first-effect steam pressure, or by reducing the pressure of the final effect vapor, the capacity of the evaporator will be increased.

All beet-sugar factories have a limit on the pressure of the steam entering the first effect, which is set by the physical limitations of the equipment installed and cannot be exceeded. Some of these physical limitations are: the maximum-allowable back pressure, or exhaust pressure of the turbines; the maximum-allowable pressure on the first-effect steam chest; and the maximum-allowable pressure in the first-effect vapor dome. If none of these physical limitations has been reached it still may not be economical to raise the exhaust steam pressure, if by so doing the steam (water) rate on the turbines is increased to such an extent that excess exhaust steam is produced which must be relieved to the atmosphere. Pressure and temperature of the vapor leaving the last effect are governed by the condenser and noncondensable-removing apparatus.

The best operation from the standpoint of fuel economy maintains the pressure in the first-effect steam chest at a maximum, and regulates the final vapor pressure in accordance with the vapor capacity desired. This method of operation has two objectives: it keeps the vapor pressures at a maximum, thus benefiting process heating; and it keeps the thick juice leaving the last effect at a maximum temperature, thus reducing the amount of thick juice heating necessary. It should not be forgotten that higher temperatures increase the rate of sucrose destruction. Increased detention time also will increase the amount of sucrose destruction. For these reasons many high temperature evaporators are designed to retain the juice at higher temperatures for only a very short period of time.

Another method of increasing the total temperature drop, and hence the capacity of a set of evaporators, is to superimpose a new high-pressure first effect on an existing set of evaporators. Sometimes, in so doing, it is necessary to rearrange the old bodies or perhaps to discard one of them.

Still another method of increasing evaporator capacity is to install a steam-jet thermocompressor. These machines take live steam that would otherwise be bled into the exhaust steam system by entrainment. This reduces some of the intermediate vapor pressures and thus increases evaporator capacity. They also increase evaporator economy and reduce one or more of the vapor pressures, unless they are installed to compress vapor going to the condenser. Thermocompressors can be in the form of steam jet pumps or centrifugal compressors driven by steam turbines or electric motors.

Sugar losses in the evaporators should be small. Loss by decomposition can be minimized by short retention time, low temperatures, and proper processing of thin juices. Entrainment loss results from particles of juice being carried over mechanically with the vapors; it is aided by high juice levels and foaming. To prevent this, baffles, catchalls, and demisters are installed to separate the juice particles from the vapor, and the juice should be kept at the correct level. Where foaming occurs, a nontoxic foam breaker can be added.

Fuel and Energy Economy

As with most commodities the cost of fuel and energy has been rising for many years. However, in the past several years fuel and energy costs have undergone major increases. Future prospects point to further major increases. Thus fuel and energy usage in the beet-sugar factory has become even more important than before.

Fuel economy in a sugar factory depends upon a great many factors, such as the efficiency of the boiler house; the proper balance of steam used in turbines with respect to power requirements and exhaust steam requirements; the amount of water used for sugar extraction in the diffuser; the amount of filter wash water; the dilution of massecuites and syrups and the amount of reboiling done in the sugar end; the manner and extent of vapor usage from the evaporators; the amount of molasses dilution in the Steffen process to produce molasses solution; and the inherent quality of the beets.

Fuel economy can be measured by several standards or units. None of these standards is a perfect criterion. Some of the standards in use are a) weight of fuel to boilers, percent on beets sliced; b) weight of fuel to boilers, percent on sugar produced; c) weight of steam from boilers, percent on beets sliced; d) weight of steam from boilers, percent on sugar produced; e) Btu in fuel to boilers, per ton of beets sliced; f) Btu in fuel to boilers, per bag of sugar produced.

The first two of these standards would be satisfactory if every beet-sugar factory used exactly the same fuel. As it is, beet-sugar factories use a variety of fuels, such as coal, oil and natural gas so the fuel weights are not at all comparable. Furthermore, the heating value of any one fuel such as coal will vary widely from factory to factory.

Standards c) and d) would be satisfactory if every beet-sugar factory boiler produced steam at identical pressures and temperatures and every boiler had the same efficiency.

The last two standards, e) and f) have none of the disadvantages outlined for the first four standards. These standards have been coming into more general use. One of the two, f) is the more valuable because it refers to the product, and because it reduces inconsistencies caused by changes in sugar content of the beets, Steffen or non-Steffen operations, etc. The efficiency of the boiler house is treated in Chapter XX.

It is important to maintain the proper balance between live steam usage in the prime movers, power needs, and steam requirements for the process. The turbines should produce sufficient power for the factory, but in so doing should not produce more exhaust steam than the factory demand. To achieve a satisfactory balance one or more of the following can be done: live steam pressure, or boiler pressure, can be increased; live steam users, such as coil vacuum pans, can be changed to exhaust steam users, such as calandria pans and exhaust steam pressure can be reduced. This will likely require more investment in larger evaporators and more efficient or lower water rate turbines. A corollary here is to use an electric motor rather than a small turbine having a very high water rate.

The inherent quality of the beets influences fuel economy, and the operator has no control over this factor. For example, an increase from 17 to 18% sugar in beets will reduce the fuel requirement about 25 000 Btu per bag of sugar produced.

The manner and extent of vapor usage from the evaporators is extremely important. The beet-sugar industry discovered and developed long ago the principles of multiple-effect evaporation and the economy resulting from vapor heating. Four general principles to be followed for maximum fuel economy may be stated:

1) Expand all high pressure steam from the boilers to exhaust steam pressure, either through turbines, which is preferable, or through reducing valves. All other uses of live steam, such as in vacuum pans, heaters, and steam injectors, should be eliminated. A number of factories have achieved almost complete conformance to this principle.

2) Use all exhaust steam in the first-effect evaporator. All other uses of exhaust steam such as in vacuum pans and heaters, except for the final thin-juice heater, should be eliminated. This has been practically realized by several factories.

3) Do all possible evaporation in the evaporators to reduce the load in vacuum pans, and in juice boilers to a minimum. Concentrators is an an expedient and a half measure because they are not as economical in fuel as the multiple-effect evaporator. In other words, control the thick juice densities so that the optimum standard liquor density will be obtained. Many factories achieve this.

4) For all factory heating, such as vacuum pans, heaters, and melters, with the exception discussed below, use the lowest pressure vapor possible. This is the vapor from the last effect. As yet no scheme has been devised whereby all heating or even a large portion of the heating can be done by the last vapor because it has a low temperature level. Also a theoretical consideration enters to modify this principle. The evaporator feed in a forward-fed evaporator, for maximum fuel economy, should theoretically be heated in a series of heaters; the first heater in the series being on the last vapor and the last heater in the series being on first vapor.

In practice this is adhered to rather well. For example, with a quintuple-effect evaporator, diffusion juice may be heated with fourth vapor and then third vapor, juice after carbonation with second vapor, and thin juice on its way to the first effect with second vapor and then with first vapor. It is evident that this exception to the general principle occurs when the evaporator feed temperature is lower than the first vapor temperature.

The above four principles have been evolved from the fact that one pound of steam from the boiler may be used as many as seven times; in the steam turbine, first effect, second effect, third effect, fourth effect, fifth effect, and a heat exchanger on the fifth effect vapor ahead of the condenser. Of course, it is not feasible to use all steam from the boiler seven times, but it follows that the more times it is used the more work will be obtained from each pound of boiler steam and hence the greater the fuel economy that will be achieved.

The extent to which these four principles can be applied by an operator is limited by the equipment he has at hand. It is the operator's job to use this equipment to the best possible advantage. In doing so it will probably be necessary for him to strike a balance between capacity and fuel economy, and to end up with neither maximum capacity nor maximum economy. To accurately calculate any fuel saving, complete heat balances should be made. In many cases it is possible to use approximate formulas and calculations.

The difference in fuel costs between using live steam and exhaust steam depends upon the respective properties of the two steams. This difference in cost is usually quite small.

When a process water heater is changed from exhaust steam to a vapor, the reduction in exhaust steam usage can be approximately calculated by the formula:

$$S = \frac{V}{N} Q \tag{3}$$

where S is the exhaust steam saving, V is the kind of vapor proposed (for third

vapor, V = 3, etc.), N is the number of evaporator effects in use, and Q is the quantity of exhaust steam or vapor usage in the heater. For example: If a heater condenses 1000 pounds of exhaust steam per hour, what would be the exhaust steam saving if the heater was placed on first vapor from a quintuple-effect evaporator? Third vapor? Exhaust steam saving with first vapor = 1/5 × 1000 = 200 pounds of exhaust steam per hour. Exhaust steam saving with third vapor = 3/5 × 1000 = 600 pounds of exhaust steam per hour.

This formula can be deduced as follows: If the heater in the above problem is changed from exhaust steam to first vapor, it will still take 1000 pounds of steam an hour, only it will be 1000 pounds of first vapor rather than 1000 pounds of exhaust steam. This 1000 pounds of first vapor will require 1000 pounds of additional exhaust steam to enter the first-effect evaporator and will there evaporate 1000 pounds of water from the first-effect juice. However, the overall amount of water to be evaporated from the juice will remain constant, the original exhaust steam to the first-effect will have to be reduced by the amount equivalent to 1000 pounds of evaporation, and since this original exhaust steam is giving five pounds of evaporation for each pound entering the first effect, the original exhaust steam will be reduced 1/5 of 1000 or 200 pounds per hour. So there is a saving of 1000 pounds of exhaust steam to the heater and 200 pounds of exhaust steam to the evaporators, and a further cost of 1000 pounds more exhaust steam to the evaporators; giving a net saving of 200 pounds of exhaust steam per hour. This exhaust steam saving can easily be converted into fuel saved and then evaluated as money saved.

Although the overall use of exhaust steam in the factory was decreased 200 pounds per hour in the above example, nevertheless the exhaust steam to the evaporators was increased 800 pounds per hour. This illustrates the greater load that vapor heating places on the first few evaporator effects and means that these must now be larger than the latter effects.

So that these four principles of fuel economy can be put into greater effect, a gradual change is taking place in the beet-sugar industry. Steam and vapor pressures are being increased, and apparatus design is changing to allow the use of evaporator vapors in place of exhaust or live steam in heaters and pans.

Calandria-type vacuum pans are replacing coil pans because calandria vacuum pans can operate on much lower steam pressures than coil pans. Closed heaters are being substituted for open injection heaters because a closed heater will operate on a lower vapor pressure and because a closed heater prevents the condensate from diluting the sugar liquor. Also the recovered condensate is valuable for its heat content and purity. Furthermore, some waste heat is being regained from Steffen waste and condensate. Thermocompressors are being used, diffuser draft is being reduced by better diffusers, the amount of filter wash water is being reduced, and modern centrifugals require less wash water.

In order to raise the vapor pressures so that equipment can be operated with vapor of a lower order and a fuel saving effected, either the exhaust steam pressure must be raised or the size of the evaporators be increased, or both. When the exhaust steam pressure is raised the live steam pressure must be raised, in turn. This will reduce the steam rate of the turbines so that the exhaust steam production can be maintained lower than the demand.

With vapor instead of exhaust steam heating on batch users such as vacuum pans, it is highly desirable to integrate the operation of the pans and evaporators because large changes in vapor withdrawals from the evaporator will change its

capacity. By installing two pans on each boiling, and with simple scheduling, relatively uniform vapor withdrawals can be obtained.

Bypassing of Steam or Vapor in Evaporators

In many evaporator installations it is necessary that provision be made to completely isolate any evaporator body so that repairs, such as retubing, may be done without shutting down all the evaporator bodies and consequently the factory. The vapor piping arrangement necessary to effect the isolation of an evaporator body from steam or vapor will very probably be equivalent to a bypass between the vapor chest and the vapor dome of the evaporator body. Consider a third effect evaporator having one body. The vapor chest contains second vapor from the second effect and the dome contains third vapor on its way to the fourth-effect vapor chest. To isolate this body second vapor must be sent to the fourth-effect vapor chest. In effect, this is bypassing second vapor to third vapor.

Table 11-1 Theoretical Material Balance

Steffen factory, excess molasses. Saccharate purity, 90.3; 1800 tons daily slice of beets; 8% sugar in beets; 40 tons excess molasses; 151 tons total molasses.

Item	Product	% on Beets	Composition			% on Beets	
			Rds	Purity	Sugar	Non-sugar	Total Solids
1	Beets	100.0		84.0	18.0		
2	Battery loss				.34		
3	Diffusion juice	142.00	14.30	87.0	17.66	2.64	20.30
4	Lime cake loss				.03		
5	Thin juice from beets			90.3	17.63	1.89	19.52
6	Sweetwater; H_2O entering	7.70					
7	Thin juice from saccharate	24.85	17.75	90.3	3.98	.43	4.41
8	Total thin juice to evaps.	174.55	13.71	90.3	21.61	2.32	23.93
9	Evap. thick juice	37.48	63.9	90.3	21.61	2.32	23.93
10	Evaporation in evaps.	137.07					
	White massecuite system						
9	*Evap. thick juice*	37.48	63.9	90.3	21.61	2.32	23.93
11	Dust collector returns	1.42	40.0	100.0	0.57		0.57
12	High remelt	6.49	100.0	98.5	6.39	.10	6.49
13	Steam to melters	1.06					
14	Low remelt	5.04	100.0	96.0	4.84	.20	5.04
15	Thin juice to low melters						
16	Total standard liquor	51.49	70.0	92.7	33.41	2.63	36.04
17	White massecuite	38.96	92.5	92.7	33.41	2.63	36.04
18	Evap'n in white pans	12.53					
19	White sugar to granulators	19.10	100.0	100.0	19.10		19.10
20	White sugar packed	18.53	100.0	100.0	18.53		18.53
21	High wash plus high green	22.57	75.0	84.5	14.31	2.62	16.93
22	Wash and dilution	2.71					
	Intermediate massecuite system						
21	High wash plus high green	22.57	75.0	84.5	14.31	2.62	16.93
23	Inter. footing for low pan	4.19	88.0	84.5	3.12	.57	3.69
24	Inter. massecuite	14.16	93.5	84.5	11.19	2.05	13.24
25	Total evap'n inter pan	4.22					
12	High remelt	6.49	100.0	98.5	6.39	.10	6.49
26	Intermediate syrup	9.25	73.0	71.1	4.80	1.95	6.75
27	Wash and dilution	11.58					
	Raw massecuite system						
23	Inter. footing for low pan	4.19	88.0	84.5	3.12	.57	3.69
26	Intermediate syrup	9.25	73.0	71.1	4.80	1.95	6.75
28	Total to raw pan	13.44	77.8	75.7	7.92	2.54	10.46
29	Raw massecuite	11.16	93.7	75.7	7.92	2.54	10.46
30	Evap'n in raw pan	2.28					
14	Low remelt sugar	5.04	100.0	96.0	4.84	.20	5.04
31	Final molasses (wash .31)	6.43	84.0	57.0	3.08	2.32	5.40

Table 11-2 Theoretical Heat Balance

Steffen factory, excess molasses. Saccharate purity, 90.3; 1800 tons beets daily slice; 18% sugar in beets; 40 tons excess molasses; 151 tons total molasses

Item	Station	Specific Heat	Temp °C. Enter	Temp °C. Leave	Temp. °C.	Diff. °F.	% on Beets	Beets, Btu 100 lb.	Exh. Steam	1st Vapor	2nd Vapor	3rd Vapor	4th Vapor
	Steam pressure								40	30	17	5	8" Hg
	Steam temperature, °F								287	274	254	227	197
	Steam temperature, °C								141.3	134.3	123.3	108.3	917
	LHV (Exhaust, 60° superheat)								952	931	945	962	981
1	Calorizators—cossettes entering	1.00	30			54	100.0	5400					
	—water leaving	1.00	50			90	242.0	21800					
	Total heat							27200					
	Total heat input												
	Diffusion juice leaving	.95		60		108	142.0	14600					
	Wet pulp and water	1.00		60		108	200.0	21600					
	Total heat							36200					
	Total heat input							9000				9.35	
2	Raw juice heater	.95	60	72	12	21.6	142.0	2910					2.97
	Raw juice heater	.95	72	85	13	23.4	142.0	3160				3.28	
3	Second carbonation heater	.95	80	92	12	21.6	174.55	3580			3.79		
4	Second filter heater	.95	89	92	3	5.4	174.55	890				.93	
5	Thin juice boiler	.95	98	101	13	23.4	174.55	3880			4.11		
6	Thin juice heater	.95	96	111	15	27	174.55	4480			4.75		
	Thin juice heater	.95	111	126	15	27	174.55	4480		4.82			
7	High and low melter (open inj.)	.60	35	85	50	90	51.49	2780			2.78		
8	Standard liquor heater	.60	85	95	10	18	51.49	560				.58	
9	White pan evaporation	LHV = 992					12.53	12430		13.38			
10	Intermediate pan evaporation	LHV = 992					4.22	4190			4.44		
11	Raw pan evaporation	LHV = 1018					2.28	2320				2.41	
12	Receiving tanks*							600	.65				
13	Cold waste heating (open inj.)	1.00	18	90	72	129.6	79.8	10360			10.36		
14	Sacch. milk heater (open inj.)	1.00	40	85	45	81	72.0	3590			3.59		
	Total vapors, % on beets								.65	18.20	33.82	16.55	2.97
	Total Btu									16910	32860	15930	2910

Table 11-2 (Continued)

Water evaporated in evaporators	157.07	68.30	
Steam to granulators		.50	
Steam to beet cutters		1.25	
Steam to turbines (Elect. and Mech. power)		6.60	
Total steam % on beets, at 254°F., = 17 psig, LHV = 945		77.30	
Total steam, % on beets, from and at 212°F., LHV = 970			75.30
Pounds of steam per ton of beets, from and at 212°F.			1506
Pounds of steam per bag of sugar, from and at 212°F.			406
Gas fuel, at 1040 Btu, 82% boiler eff. (cu feet per ton beets)			1710
Gas per bag of sugar, 96.8% produced of total entered			461
Btu per bag of sugar			480,000
Oil, % on beets (18,500 Btu oil, 86% boiler eff.)			3.86

* 300 feet 1.5 inch od copper tubing.

With the third body in operation what happens when second vapor is bled into third vapor through the bypass valve or through leaky evaporator tubes? It has previously been pointed out that with a quintuple-effect evaporator, any steam bypassed does only about 4/5 the normal amount of work. Bypassing steam does not alter the amount of work to be done, so for every five pounds of steam bypassed the evaporators must be given an extra pound of exhaust steam, and, in turn, the boilers must generate an extra pound of steam, which takes more fuel.

With these considerations in mind it can be seen that if second vapor is bypassed into third vapor the load on the third effect will be reduced while the load on all other effects will be increased. This means that the first and second vapor temperatures and pressures will be reduced and that the third and fourth vapor temperatures and pressures will be increased. The evaporator capacity change is a complicated and particular problem for each case in each factory. Usually, but not always, the evaporator capacity will be reduced.

Bypassing in any form is expensive practice, although at times it may be the lesser of two evils.

Evaporator Calculations

It is fortunate that the practical theory of evaporators is rather completely developed, and can be used accurately as a practical basis of operation. Evaporators can be subjected to almost exact calculations which can be used to locate the weak points in any installation, point out the improvements that can be made, and show how much these improvements are worth in dollars and cents.

A theoretical material balance, a theoretical overall heat balance, and a theoretical evaporator heat balance for a modern beet-sugar factory are given in Tables 11-1, 11-2, and 11-3. These balances illustrate in detail the manner in which economy can be obtained.

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Capter XII

CRYSTALLIZATION*

A Driving Force

An earthen kettle with a wood fire below, a long handled, wooden ladle, and great patience have been the tools of some boilers of sugar (191a, 303a).

A controlled population, salt-out seed continuously flowing into a multi-compartment, forced circulation, continuous crystallizing pan, heated with an economical third vapor, and fed with a regulated purity and constant high density syrup, all at steady rates and steady equilibrium states, are the tools of other sugar boilers.

This range of crystallizing capability is indeed the result of dissatisfaction and ingenuity of skilled sugar boilers, engineers, and researchers in all parts of the world.

Bit by bit, batch-boiling systems have been perfected to yield more uniform, reproducible crystal crops. A basic technology has evolved. A better understanding of the fundamental chemistry of condensed systems is, however, essential to the continued improvement of batch crystallization and more particularly of continuous crystallization.

The impact of fuel costs has imposed a new economic philosophy in all industries. Much progress has been made (75a), but still more is needed. Fortunately, a new technology in sugar crystallization has been slowly evolving—continuous sugar boiling. With a steady process flow, the heat flow is smoothed. With impending equipment design, continuous crystallization becomes a means for significant fuel savings.

A treatment of continuous crystallization theory and practice has been blended into the following descriptions of batch technology in generous measure.

Energetics-Macro and Micro

Chemical change in a condensed system is possible *if* it results in a new structure of lower energy than the original structure.

Our boiler of sugar with the open pot and wood fire knew that if he boiled and evaporated long enough, crystals would suddenly appear; and with a little more slow boiling, these crystals would grow to a useful size.

Without realizing it, our sugar boiler had surmounted an energy barrier in the syrup, which led to the formation of crystal structure having a lower energy level, Figure 12-1 and Figure 12-25.

In many research laboratories, skilled investigators have been able to determine these respective energy levels under a variety of macro conditions. In these and other laboratories, the nature of the chemical bond, along with the rapidly fluctuating thermal energy conditions, and the wide variance in degree of energy fluctuations evident in each of the molecular and ionic species present, has been explored in the micro sense and, in part, has been defined.

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* by G.W. Cossairt

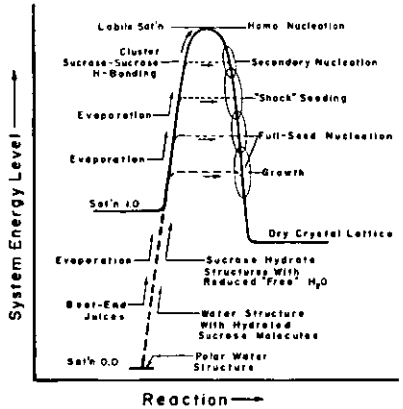

Figure 12-1. Energy barrier surmounted in boiling-to-grain. The energy level in the final crystals is at a significantly lower level than in the saturated solution.

While our sugar boiler sees a gently boiling pot of syrup, our inquisitive, probing, crystal chemistry investigators open the door for all to see an amazingly active, dynamic equilibrium in this molecular soup: the genesis of nucleation and growth of crystals.

To better appreciate the contributions of the molecular activity of the solute, the solvent, and of the interfering impurities, they will now be examined individually and then collectively.

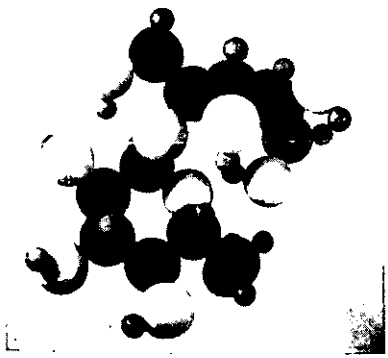

Figure 12-2. Space model of sucrose molecule.

Properties of Solute and Solvent

Crystal Chemistry. Initial chemical studies define sucrose as $C_{12}H_{22}O_{11}$. Structural chemists resolve the molecule into two-dimensional structural groupings, as noted in Figure 2-9 (left), to help identify locations of functional groups. The energetics of reactions require the introduction of the third dimension [Figure 2-9 (right)].

Many of the properties of solids and solutions are dependent on the individual molecular configurations. A space model (316) has been beneficial in giving a clearer visualization of the molecule of sucrose (Figure 12-2). This style of molecular representation stresses the space occupied by each atom.

The "fit" of molecules, one beside another, establishes many of the properties and the shape of the crystalline solid. The determination of inorganic molecule fit

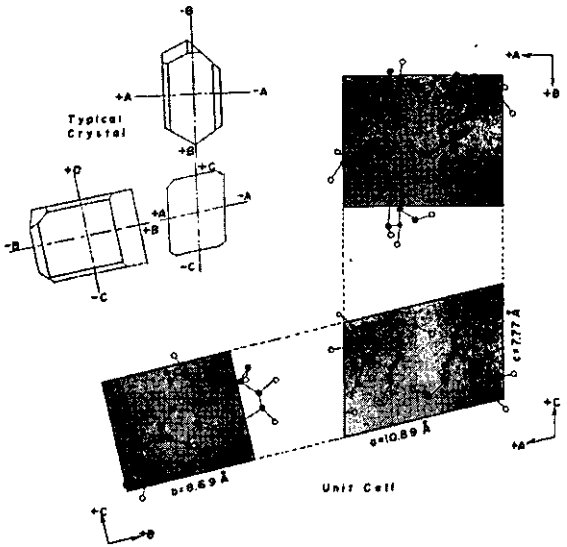

Figure 12-3. Relative orientations of a crystal, its crystal lattice cell, and the sucrose molecules within the unit cell. ***[after Mantovani, et al. (210). By permission CITS]***

was among the earliest of the many successful X-ray diffraction studies. Organic molecular crystal studies were much more difficult. Neutron diffraction studies (30,43) have since provided a rather precise location of sucrose molecules in the crystal structure (Figure 12-3). In this instance, the ball-and-stick method of illustration permits stress of bonding angles and interatomic distances.

Crystal Systems. As an aid to the understanding of crystallization, a brief review of crystal systems is given. There are many excellent references in the literature.[1]

Crystals occupy space which can be identified by the plane surfaces enclosing them. A simple three-axis system is useful to identify each plane. Seven basic crystal systems have been established.

Cubic and monoclinic systems and a typical sucrose crystal are illustrated in Figure 12-4. While the right-hand perspective is normally used, some authors prefer the left-hand perspective when considering sucrose, as the —B end usually exhibits the greater number of faces (406).

The shape of the geometrical unit representing the crystal system is a reflection of the shape of the ionic or molecular "building block." For sodium chloride this unit is cubic and is ionic in nature. The unit for sucrose is monoclinic and molecular in nature. For simplicity of illustration, cubic "units" will be used in development of most of the concepts in this chapter.

The crystal lattice can be considered as an orderly grouping of the building blocks into a three-dimensional array. As an example, Figure 12-4(b) suggests a simple cubic arrangement. Modification of the basic cubic stacking of lattice units will yield new planes of crystal surface, as shown by the heavy dotted lines of the other arrays in Figure 12-23.

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[1] See refs (249, 276, 383, 405)

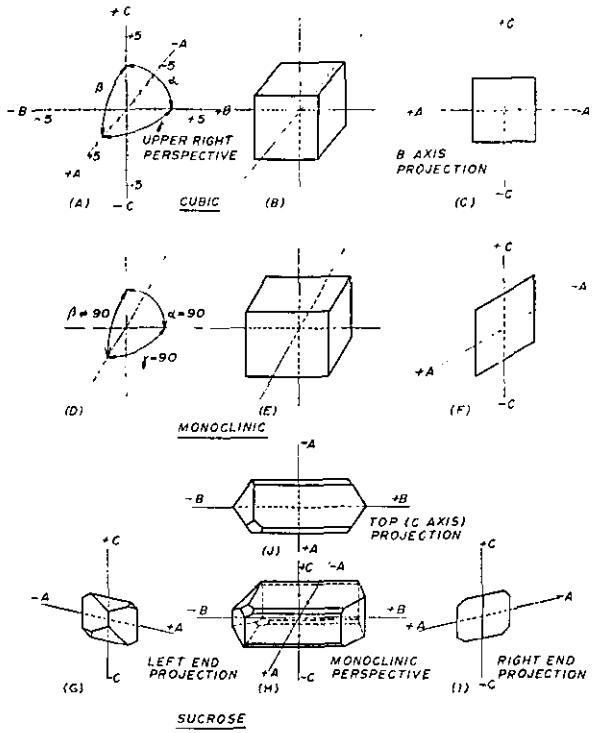

Figure 12-4. Monoclinic system of sucrose.

If it is kept in mind that these building blocks are in the one-to-ten Å range, the molecular roughness will have no significance in the *macroscopic* consideration of the surface, and it will appear as smooth as any of the prime cubic faces.

In the study of the influence of various impurities upon the shape or morphology of product crystals and upon the nature of the inhibition of growth rates on the various crystal faces (213a, 343a, 188a), a positive identification of those faces is by Miller indices.

A tabulation of probable faces is contained in "Atlas of Sugar Crystals," (404), Bureau of Standards Circular C-440 (18), the text of Honig (284), and a private publication of Powers (288). Bloss (35a) provides a detailed description of crystal geometry and of Miller indices.

Crystal Studies. For those interested in the artistry of crystals, crystallography has provided an organized basis for better undertstanding (91,283). Recently visual observations have been enhanced by still and lapsed-time photography, through the microscope (285). For an introduction to this avenue of study, the suggestions of Powers are recommended (279,288).

X-ray diffraction provided the initial tool for crystal structure determination.[1] X-ray studies have been limited, however, as only carbon and oxygen atoms of sucrose could be identified spacewise, with certainty. The newer tools, including

[1] See refs (29, 30, 94, 249, 276, 367, 383, 384, 405)

neutron diffraction (31), have permitted more refined structure studies including quite precise location of the hydrogen atoms. Electronic images with a resolution of 3.4Å have been achieved with improved scanning electron microscope techniques (179, 258, 271). These "micro-micro" studies have been quite productive.

Water, the Solvent. The behavior of water has been noted earlier with various impurities and their removal. In the beet end of the factory, there are relatively dilute solutions in which chemical rejection is the major mechanism of impurity removal. In this environment, the ratio of water molecules to sucrose molecules is quite high.

In sugar crystallization, the same properties of water are again important in the final "squeezing out" and rejection of impurities. The dipole and hydrogen bonding attractive and orientative forces present between water and sucrose, and between sucrose and sucrose molecules, make water a particularly fitting solvent in sucrose purification. The solvent capabilities of water and its thermodynamic properties are defined by its molecular structure (216) (see pp. 159, 160). Its unsymmetrical shape is illustrated in Figure 12-5.

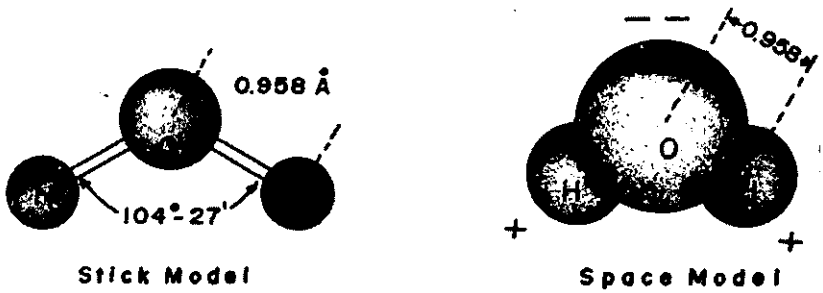

Figure 12-5. Dipole structure of water.

An overlap of atoms is noted in the space-occupying model, typical of covalent bonding. However, as with an ionic bonding, the electrons are shared unequally. This unequal distribution of charges produces a dipole or polar molecule.

Part of water's unusual success as a solvent lies in association and attraction properties through the –O– –H–O– hydrogen bonding (217,262). Just as two water molecules, if properly oriented, will bond together, a third, fourth, and more molecules can bond together as a chain, a branched chain, a looped chain, and as a three-dimensional cluster (1c, 81).

The bonding of the first pair is more difficult as compared to the second and additional hydrogen bonds. As a consequence, once a few molecules have bonded, the formation of large clusters becomes easy, Fig. 12-6. A subsequent energy pulsation in that micro volume of water probably will cause the clusters to break up. As these energy pulsations are localized and random, while one cluster is breaking apart, other clusters are forming. Dielectric studies suggest a short life span for such clusters -10^{-10} second – thus the term "flickering clusters." These clusters may be 50 to 100 molecules in size and may endure as many as 1,000 fluctuations before breaking up.

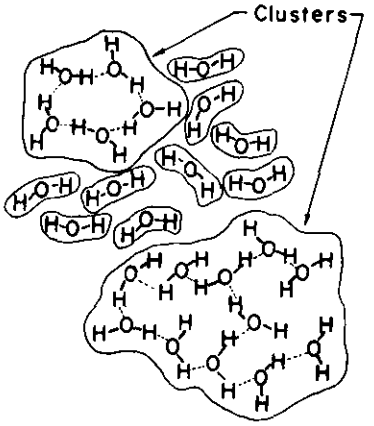

Figure 12-6. Water structure — "flickering clusters" model of water structure. Dielectric studies show short life, hydrogen bonded and dipole orientations. Cluster sizes range up to 50-100 molecules for a life of about 10^{-11} sec. (1c). *(Reproduced by permission Academic Press.)*

Although the micro structure is one of fluctuations, the macro properties reflect stability and average values of energy, consistent with bond-making and bond-breaking.

Molecules in a liquid are separated about 10% as compared to the locations of molecules in a crystal. This minor amount of looseness imposes a constraint on molecular movement. Surrounding molecules create a restraint or a *solvent cage effect*, Fig. 12-7a.

The vibrating molecules bounce against one another (statistical probability of about 150 times during that 10^{-10} sec interval) until there is an instantaneous energy sufficient to break through and escape into another cage. The activation energy of diffusion has been exceeded and an escape or molecular jump occurs. Fig. 12-7b becomes a theoretical model for viscous flow (1c).

The frequency of jumps is related to temperature through the Boltzman function. In a solution at rest, the jump will be random in direction, Fig. 12-7c. With liquid in mechanical or chemical stress, the jumps will be more frequent in the direction of stress for a net directional diffusion.

The mechanisms of both diffusion and viscosity are directly attributable to the liquid structure. One also can sense the fluctuating nature of bonding with solute molecules.

Impurities. The influences of nonsucroses in the sugar-end syrups become evident all through the boiling and crystallization systems. Fluid properties, crystallization kinetics, and final product quality are all affected.

Various investigators[1] have demonstrated the variance in factory juice quality as related to growing area, cultural practices, and climatic conditions.

Beet-end processes eliminate many nonsucroses, but they have their physicochemical limitations. Fortunately, crystallization is also an impurity-rejection mechanism. Unfortunately, those same soluble impurities affect the ease or difficulty of crystallization of an acceptable product (213b).

Resonant destruction moieties, alkali ions, neutral amino bases, spoilage substances characterized by dextran and weak acid anions are among the non-

[1](See refs. (51, 224a, 254a, 328, 409a, 411c)

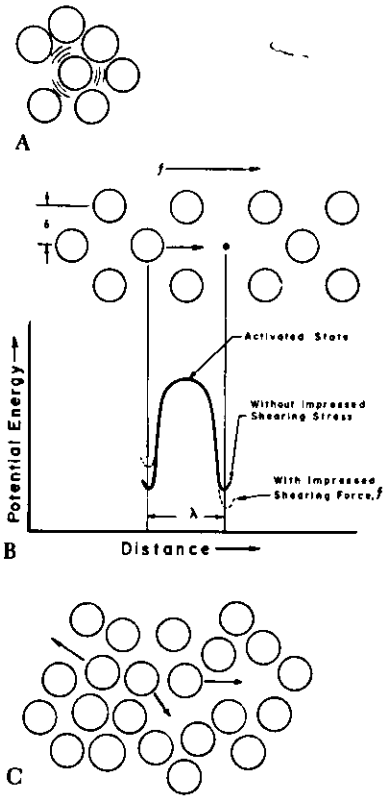

Figure 12-7. Molecular Neighbors.

A) Each molecule in the solution vibrates against its immediate neighbors which, in turn, form a somewhat confining "solvent cage."

B) Molecules with sufficient bursts of energy may escape from the immediate surrounding neighbors and escape or "jump" through the restricted space between vibrating neighbors and enter a new cage of neighbors. The potential energy curve identifies that energy level needed to make the jump.

C) In a liquid at rest, the direction of jump is random. In a liquid in motion, in "B" above, the line of force "f" identifies the shear stress (and viscosity) and the more probable direction of jump. ***[Viscosity model 12-7b, reproduced by permission of Academic Press (1c.)]***

sucroses affecting such solution properties as viscosity, solubility, conductance, nucleation kinetics, growth inhibition, crystal habit, crystal color, and others.

Solution Properties

Solution Structure. The structural formula of sucrose, Fig. 12-8, shows the eight cationic sites for hydrogen-bond attachment. Allen (8a) reports that three of the sites become internal or intramolecular and five are exposed sites for attachment to other molecules.

Surrounding the solute molecules in solution, the polar water molecules will associate or hydrogen bond to the exposed sites of these sucrose molecules. A typical site and two bonding possibilities are shown in Fig. 12-8 a); chains of successive water molecules and b) a nonrepeating bonding.

This water sheath contributes to the structure of the solvent-solute system. Just as in the water system, localized energy fluctuations will affect the extent of the chain formation, its breakup, and re-exposure of the primary cationic sites.

Below 30% sucrose concentration, the system is reported[1] to be a water structure, re-enforced by the presence of hydrated sucrose molecules.

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[1]See refs. (215b,321,322,323,410)

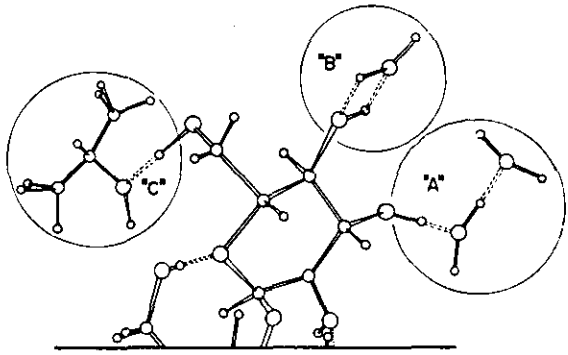

Figure 12-8. Typical bonding sites exposed to solution for hydrogen bonding:

A) Water-alcoholic hydroxyl single hydrogen bonding, having probability of extended water chain or sheath.

B) Water-alcoholic hydroxyl double bonded, having low probability of extension.

C) Isopropyl alcohol—alcoholic hydroxyl to alcoholic-hydroxyl hydrogen bonding forming a "waterproof" sheath. ***[After Allen (8a), Brown and Levy (43), and Mak and Kelly (208a).]***

If the sites are filled with the nonrepeating, double-bonded configurations, Fig. 12-8b, the sucrose molecule and its associated water molecules become a more defined structure within the solvent.

As the ratio of solvent to solute is modified, the number of "free" water molecules decreases until still further evaporation leads to a reduction of the number of water molecules "bonded" to each sucrose molecule. The macro properties of the fluid change. The fluid structure is more resistant to shear, thus viscosity increases. The solution vapor pressure is lowered, thus the solution boiling point is raised (boiling point rise, bpr). The refractive index of the solution increases. These fluid properties are all useful for understanding and manipulating the crystallization process.

Solubility. Solubility is an equilibrium mechanism. Molecules must be held in rather rigid locations in the lattice. Freedom to wander would destroy the lattice structure. Two opposing forces are in operation: a) the attractive forces between the molecules and b) the thermal energy of the molecules themselves.

In a solution, the water molecules surround the crystal lattice. The crystal lattice bonds are the result of sucrose-sucrose dipole and hydrogen-bond attractions. Water is also a dipole and has hydrogen-bond attraction. The relative values of these two forces determine crystal growth or crystal dissolution.

Not all hydrogen-bond attractions are of the same magnitude. There is evidence (8a) that hydrogen bonds between alcohol hydroxyl groups and water are stronger than between alcohol-alcohol and between water-water complexes.

Obviously, a multitude of interactions are at work. However, some conclusions can be drawn to bring a macro sense of order. In the solution, the relative concentration of solvent will identify the relative sucrose bonding potential of the liquid phase as compared to the sucrose bonding potential of the lattice. Localized system fluctuations shift the magnitude of the two potentials such that some sucrose molecules find a more energetically favorable "home" in the solution, while in other micro locations, the more energetically favorable "home" is in the lattice. When the "escape" and "capture" rates in the micro sense are in equilibrium, the solution is defined as saturated.

This is an equilibrium obviously in the presence of an excess of solute. As the system temperature directly influences the vibrational energies of the molecules, the solubility equilibrium changes for each temperature change of the system. Tables of solubility at various temperatures are a basic tool for crystallization studies.

Agitation of a surplus of crystals in water solvent at a controlled temperature for a fixed period of time will establish equilibrium of the various force energies and will thus permit the determination of sucrose solubility in water (188).

A similar procedure using molasses (224a) or a process syrup is the basis for determining the sucrose solubility in an impure solution.

The Herzfeld (1892) values of solubility of pure sucrose in water were generally accepted for many years (21). Grut's tables of solubilities (65, 138a) at various purities for beet syrups have been convenient and representative of plant operations in many locations. Refer to Appendix for the Grut tables.

Through statistical analysis of the available data of many investigators, Vavrinecz (408) prepared new tables of solubility ICUMSA (1974) tentatively

Table 12-1a Sucrose Solubility in Water

Values of Vavrinecz for the solubility of sucrose in pure water. Tentatively approved by ICUMSA (177a).

t °C	Z	t °C	Z	t °C	Z
−4	178.89	31	217.07	66	309.66
−3	179.44	32	218.82	67	313.43
−2	180.03	33	220.62	68	317.27
−1	180.64	34	222.46	69	321.17
0	181.27	35	224.36	70	325.15
1	181.93	36	226.29	71	329.20
2	182.62	37	228.27	72	333.33
3	183.35	38	230.28	73	337.52
4	184.11	39	232.37	74	341.79
5	184.89	40	234.50	75	346.15
6	185.70	41	236.68	76	350.58
7	186.54	42	238.90	77	355.08
8	187.41	43	241.18	78	359.64
9	188.32	44	243.50	79	364.27
10	189.26	45	245.89	80	368.99
11	190.23	46	248.32	81	373.78
12	191.23	47	250.82	82	378.65
13	192.27	48	253.37	83	383.58
14	193.33	49	255.97	84	388.59
15	194.44	50	258.63	85	393.68
16	195.58	51	261.34	86	398.82
17	196.75	52	264.11	87	404.01
18	197.95	53	266.96	88	409.28
19	199.19	54	269.86	89	414.64
20	200.47	55	272.81	90	420.04
21	201.79	56	275.84	91	425.49
22	203.13	57	278.92	92	430.96
23	204.52	58	282.07	93	436.51
24	205.94	59	285.28	94	442.12
25	207.43	60	288.56	95	447.75
26	208.93	61	291.90	96	453.40
27	210.47	62	295.32	97	459.09
28	212.06	63	298.82	98	464.84
29	213.68	64	302.36	99	470.59
30	215.35	65	305.96	100	467.34

Z = grams sucrose per 100 grams water, at saturation.

recommended adoption of these values for pure sucrose-water systems, Table 12-1a.

The inhibiting effects of impurities become particularly important in the lower purity crystallization processes discussed later in this chapter. Through the combined efforts of Wiklund (425a), Wagnerowski (411d,411e), and Vavrinecz (409a), a correlation was developed between the saturation coefficient and the nonsucroses present, Fig 12-9 and Table 12-1

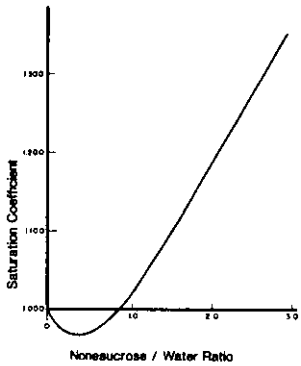

Figure 12-9. Dependence of solubility upon the nonsucrose-water ratio. Data from Grut's table as plotted by Vavrinecz (409a). *[Courtesy of C.D. Rhoten (249a).]*

The inhibiting effects of impurities become particularly important in the lower purity crystallization processes discussed later in this chapter. Through the combined efforts of Wiklund (425a), Wagnerowski (411d, 411e), and Vavrinecz (409a), a correlation was developed between the saturation coefficient and the nonsucroses present, Fig. 12-9 and Table 12-1.

The influence of impurities upon sucrose solubility can be expressed as:

$$Z' = C \cdot Z \tag{1}$$

$$\text{or } C = \frac{Z'}{Z} = \text{saturation coefficient} \tag{2}$$

where Z = solubility of sucrose in *pure* solution, grams sucrose/100 g water

Z' = solubility of sucrose in *impure* solution, grams sucrose/100 g water

A valid and useful relationship has been carefully and extensively investigated for the saturation coefficient and the nonsucroses concentration, (N/W):

$$C = m \cdot N/W + b + (1-b) \cdot e^{-c\,(N/W)} \tag{3}$$

For a given beet quality and plant processing capability, a relationship between C and N/W can be plotted as in Fig. 12-9. From such a plot, the constants m, b, and c can be determined.

McGinnis (224a) reports that the Grut data of impure solution solubilities are satisfactory for many beet growing areas and normal process molasses. Equation 3 then becomes:

$$C = 0.178(N/W) + 0.820 + 0.180e^{-2.1(N/W)} \tag{4}$$

Wiklund recommended that this solubility equation be used only in the range of N/W = 1.7 to 3.5 on the linear portion of the plotted curve. Vavrinecz, however, demonstrated the excellent fit of Grut's data in the] to 1.7 N/W range. The careful investigator will redetermine the constants of the Wiklund relationship for his own

beet growing area and for his own plant's process, i.e., straight house, Steffen, Quentin, etc.

However, for those who find solubility tables other than Grut's better fit the nonsucrose compositions of their molasses, Vavrinecz has furnished values of the constants of his equation, as follows:

Table 12-1. Coefficients for solubility equations for sucrose in beet molasses, for data of various investigators, as determined by Vavrinecz (409a).

Investigator	m	b	c
Schokow	0.234	0.752	2.85
Grut	0.178	0.820	2.1
Nees, Hungerford	p.322	0.563	1.6
D'Orzi	0.271	0.767	1.5
Schwerbljanski	0.287	0.797	2.1
Silin	0.354	0.516	2.0
Wagnerowski, et al	0.200	0.750	1.69

Supersaturation. Crystallization is a two-step process: Nucleation and then growth. In the previous section it was noted that the hydrogen bonds between alcoholic hydroxyl groups (−OH group attached to a ring or methyl carbon of the sucrose molecule) and water, are stronger than alcohol-alcohol (sucrose OH to another sucrose OH). This would suggest that by evaporation removal of water, the solution energy level more and more represents that of sucrose-sucrose groups, a prerequisite to nucleation.

Supersaturation can be used as a measure of that solution energy level and of the concentration necessary to exceed the energy barrier for nucleation.

Crystal growth is a mass transfer operation, i.e., a transfer of sucrose molecules from the solution phase to the solid phase. A concentration difference is the driving force that causes the transfer of mass to take place. A negative concentration difference would imply that the solid will dissolve in the surrounding solution. A positive difference — supersaturation — identifies crystal growth.

Ostwald (259b) is generally credited with the concept of supersaturation and its distinctive regions of "metastable" and "labile." Miers (240b) formulated a con-

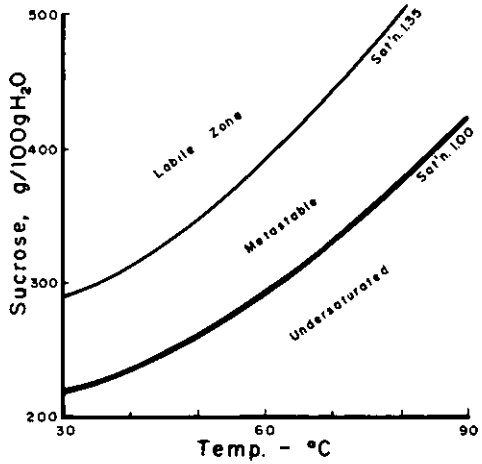

Figure 12-10. Miers' classical diagram of the regions of saturation for pure sucrose.

cept diagram, which is very useful in visual understanding of various pan boiling and crystallizer processes.

The identified regions of Fig. 12-10 have the following significance:

Labile	nucleation can take place in the *absence* of seed.
Metastable	nucleation can take place *only* in the presence of seed. Growth can take place in both the metastable and labile regions.
Unsaturated	neither nucleation nor growth can occur in the unsaturated region.

Studies of crystallization of many substances in a wide range of environments and equipment have demonstrated that the true picture is much more complex. The regions are indeed present, but the separations between regions are diffused, depending upon the substance being crystallized, the population or density of seed present, the size of the seed, the rate of saturation change, agitation, and, of course, the nature and amount of soluble impurities.[1]

The diffused nature of the metastable region, as it blends into the labile region, has led some investigators to expand the Miers' chart with an "intermediate" region, Fig. 12-11.

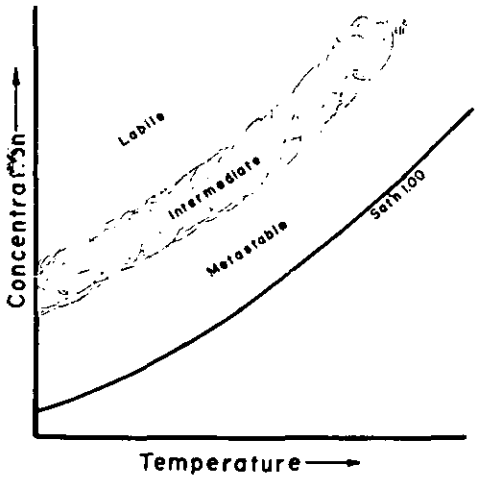

Figure 12-11. Intermediate supersaturation region for the study of secondary nucleation or "false grain" formation. *[After Webre (422a).]*

Modern day batch sugar boiling (if well done with full seeding) operates in a limited region: totally metastable.

Some continuous boiling procedures may operate entirely in the intermediate region. Other continuous sugar boiling procedures are designed for totally metastable conditions. Still other systems make use of the mechanisms of two regions.

The very nature of the micro conditions of nucleation and growth demands a probability perspective in visualizing these regions of supersaturation. The macro properties are time averages.

A deliberate state of disequilibrium is induced in the syrups on pan floor by the sugar boiler to bring about crystal formation and growth. The measure of this non-equilibrium is generally termed, "supersaturation," a condition which cannot be measured

[1] See refs. (292a, 369a, 392a, 403, 422a).

directly. However, a number of solution physical properties can be measured and the relative amount of disequilibrium can be calculated.

Supersaturation is defined in many ways, depending upon the needs of the investigator—differences, ratios, log functions, etc.[1] These "tools" and many tables of physical data are available to the researcher.

For the sugar boiler and his supervisor, two expressions suffice—first, as a difference for basic mass transfer and reaction rate studies, and second, as a simple ratio to quickly assess the ongoing, dynamic production nucleation and growth environments.

Symbols: (Based upon the raw data of the factory control laboratory to the sugar boiler).

rds = refractive dry substance or "Brix" determination of % total soluble solids in the solution

W = 100 − rds = water content, %

P = purity = % ratio of sucrose solids in the rds total solids

$S = \frac{rds \times P}{100}$ = sucrose content, %

N = rds − S = nonsucrose content, %

S/W = sugar-water ratio, grams sucrose/gram solvent water

N/W = nonsucrose-water ratio

O = oversaturation

SS = supersaturation

For clarification of the conditions of the saturation comparisons, the following subscripts are definitive:

E = equilibrium saturation (given temp)

B = instantaneous saturation (same temp)

O = saturation comparison of pure solution

P = saturation comparison at identical purity

N/W = saturation comparison at identical N/W

For mass transfer, growth rate and nucleation rate-order studies:

$$O = C_B - C_E \tag{5}$$

$$SS_O = \frac{C_{B,O}}{C_{E,O}} \tag{6}$$

For pan boiling, the values of S, W, and N are all rapidly changing. Comparison of saturations of an instantaneous condition of the syrup is adequately expressed by the rds, purity data:

$$SS_P = \frac{C_{B,P}}{C_{E,P}} = \frac{(S/W)_{B,P}}{(S/W)_{E,P}} \tag{7}$$

[1]See refs: (58, 182, 191b, 224b, 343a, 394a)

In this instance, the saturation data of the Grut's table are totally satisfactory. Most BPR instruments have been calibrated to this relation.

For control of crystallizer operation (where there is a rather considerable dynamic time lag) and for the precision studies of investigators of growth rates, etc., the constant N/W basis is officially preferred. The equations of Vavrinecz are applicable.

$$SS_{N/W} = \frac{C_{B,N/W}}{C_{E,N/W}} = \frac{(S/W)_{B,N/W}}{(S/W)_{E,N/W}} \tag{8}$$

McGinnis has illustrated the wide variety of numerical values of supersaturation that are possible, using the same basic data in the several equations for expressing supersaturation (224b). Before published saturation data are compared, the author's data should be examined as to the methods of expressing supersaturation.

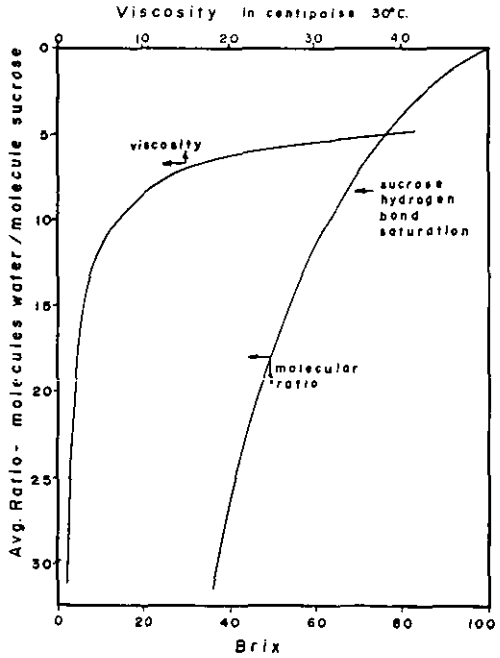

Figure 12-12. Molecular composition and solution structure of the sucrose-water system.

Viscosity. The structural nature of the sucrose-water solution has been shown on previous pages to range from that of pure water to that of sucrose aggregations having only a part of the normal water of hydration, Fig. 12-12.

Fig. 12-7b was referred to as the model for viscous flow. From this is derived the relation

$$\eta = n \cdot h \cdot e^{\epsilon^*/kt} \tag{9}$$

in which viscosity η is proportional to the molecular concentration n, and is exponentially related to temperature. ϵ^* is the activation energy required for a jump.

The structure of the fluid (polar and hydrogen bonding) influences the probability of a jump. With added solution structure (sucrose-water and sucrose-sucrose bonding into larger structures), viscosity increases.

This structural fluidity or rigidity is reflected in the degree of shear stress or resistance to fluid flow. This shear stress is measurable and is expressed as viscosity. It becomes important that this solution property be fully appreciated, as it is the basis of management and control of several aspects of the total crystallization and separation system.

As shown in Fig. 12-20, viscosity is related to sugar concentration and temperature. The influence of impurities (additional solute having varying solution structural properties) is also evident, Fig. 12-13.

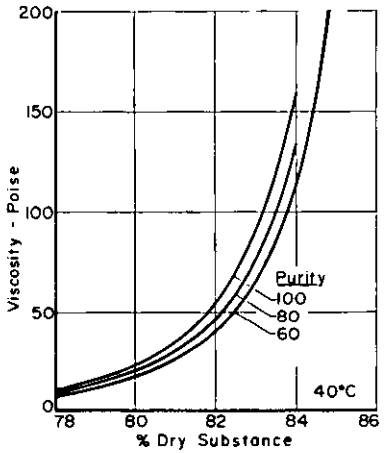

Figure 12-13. Reduction of molasses viscosity due to presence of normal beet impurities. Data of Bennett and Nees. (See Chapt. 19.)

Tables of viscosity of pure sucrose solution are available in Circular C-440 (20). The 14th ICUMSA adopted the data of Schneider in the range of 75-86% concentration as supplementary to the NBS tables. Typical viscosity values for beet sugar syrups were measured and reported by Bennett and Nees (31a), Table 19-1.

Boiling Point Rise. Certain properties of solutions relate to the *concentrations* of the solute molecules rather than to the nature of the molecules. Among these colligative properties is boiling point rise.

A solution of a nonvolatile solute always boils at a higher temperature than that of the pure solvent. In dilute, ideal solutions, the difference in temperatures (the boiling point rise) is proportional to the concentration of the solute.

Adamson (1c) reports the relation to be:

$$\Delta T_b = \frac{R(T^\circ_{b\bullet 1})^2 M_1}{1000 \bullet \Delta H^\circ_{v,1}} \bullet m \qquad (10)$$

Where: ΔT_b = normal boiling point elevation of the solution, $T_b - T^\circ_{b\bullet 1}$

T_b = normal boiling point of a solution of solvent activity a_1.

$T^\circ_{b\bullet 1}$ = normal boiling point of the pure solvent

R = gas constant

M_1 = molecular weight of the solvent

m = molality of the solute

ΔH°_{v+l} = standard heat of vaporization of pure solvent.

With ideal solutions, equation 10 reduces to

$$\Delta T_b = K_b m \tag{11}$$

Where K_b = boiling point constant

and K_b depends only upon properties of the pure solvent. The value for water as solvent is 0.514.

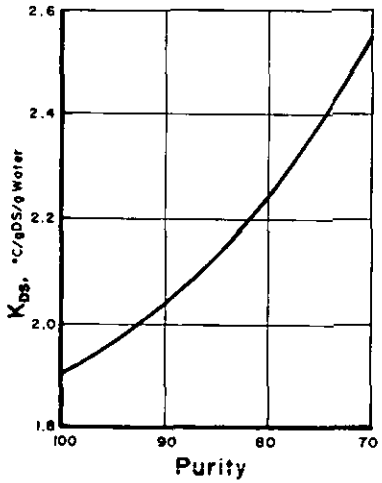

Figure 12-14. Boiling point elevation constant as modified by beet nonsucroses. *[After Ziegler (453a).]*

For the practical conditions of sucrose crystallization, the solutions deviate considerably from ideality. Sucrose tightly binds about six molecules of sugar — a compound formation between solvent and solute.

For impure solutions, the ionization of the solute salts present, binds an additional number of solvent water molecules for still more departure from ideality.

As several of the saturation—supersaturation expressions are in terms of grams solute per gram water, it is convenient to express "m" in grams dry substance per gram water solvent. The boiling-point elevation constant is then not only modified to accommodate the concentration conversion but also to accommodate the shift in average molecular weight of the solute - in this case dry-substance, K_b becomes K_{ds} (Table 12-2).

Table 12-2. Boiling Point Elevation Constants for Beet Syrups at 75°C.

Purity	K_{ds}
100	1.90
95	1.95
90	2.03
85	2.13
80	2.25
75	2.37
70	2.52

Refractive Index. Light refraction in sugar solutions has a direct relation to the sucrose solids present. In 1966 ICUMSA adopted an "International Scale of Refractometer Indices for Sucrose Solutions."

For factory juice and syrups, refractometers are used in most U.S. beet-sugar laboratories and attest to the usefulness of this property of sugar solutions. While laboratory measurements are related to 20° C, special refractometers are available for on-line measurements at normal pan-boiling temperatures.

Conductivity. Solutes may be classified as to their ability to conduct electricity when dissolved in water. Some substances, such as sucrose, dissolve as electrically neutral molecules, and form a nonconducting solution. Other substances dissolve as ions in the water and can conduct electricity. Each solute has its own degree of conductance, thus the measure of the solution conductance becomes a measure of the concentration of that substance.

In those solutions where one of the solutes present contributes to the solution viscosity, the mobility of the ions in the solution becomes related to the solution viscosity. For sugar solutions of a given purity, a conductance measurement can be related to the solution viscosity.

For a given pan feed syrup, a typical pattern of conductance will be established from which concentration or supersaturation can be estimated (162, 207a). With proper circuit design, the measurement can be "tuned" to the feed syrup at hand.

Crystal Slurry Properties

Crystal Population. For batch pans the number of crystals in the batch as dropped is equal to the number of crystals in the seed slurry or to the number generated by nucleation. In only a few seconds of the total boiling time, the crystal crop is established. The need is then to maintain and protect that initial population, a critical boiling syrup's specification.

For nonseeded, single-tank, back-mixed crystallizers, Fig. 12-73, the crystal nucleation rate of generation must equal the rate of product population removal. As will be developed in a later section, the nucleation rate is dependent upon supersaturation. Growth is area-dependent and saturation-dependent. In this type crystallizer, operating in the intermediate saturation zone, crystal slurry density must be stabilized.

In a batch boiling, the number of seed initiated will determine the final crystal size as the crop is completed and dropped to the centrifugals. In Table 12-3, Gillett illustrates the effect of MA—crystal size—with respect to relative surface area for growth and exhaustion and also the effect with respect to free drainage of the mother liquor from the crystals in the batch centrifugals.

Table 12-3 Effects of Crystal Size

Mesh Size	Size Microns	% Relative Surface Area	Relative Pore Cross-section Area
65	208	200	100
60	246	170	140
48	295	142	200
42	351	119	280
35	417	100	400

After Gillett (124a)

Crystal Size Distribution (CSD). Particulate processes have been intently studied and described with suitable mathematical models. Among these processes, crystallization, and more specifically sucrose crystallization, the characteristic sizes and shapes may have an influence on the processes from which they were produced. The average size, and the variance in sizes can be modeled for one dimensional and multi-dimensional crystal size distributions. These distributions may be better known as MA-CV and CSD. McCabe's ΔL law states that all crystals in a given environment will individually grow in equal amounts within the same time interval (215d,292b).

A carefully performed, uniform-size seeding of the syrup at the first of the boiling will yield a reasonably uniformly-sized final crystal crop, but, of course, of a much larger size. The CSD or CV at the end will be the same as the seed or slightly greater. As growth is the objective, the MA will be much larger, Fig. 12-15.

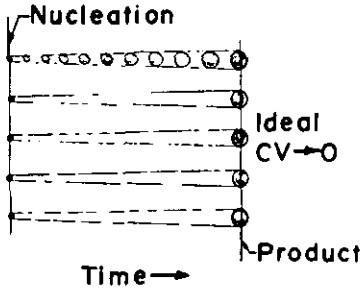

Figure 12-15. Ideal Batch Crystal Growth. For nuclei of uniform size and under conditions that McCabe's ΔL Law prevails, then the final product crystals will all be of uniform size. ***[After Saeman (314).]***

If, however, the initial seed is widely variant in size (high CV), then one can expect an equally high CV in the product, Fig. 12-16.

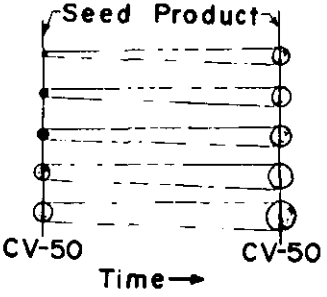

Figure 12-16. Product CV as influenced by seed CV. McCabe's ΔL Law prevails.

In continuous, single-stage pans, an equilibrium condition is established with respect to saturation and the crystal slurry properties. Ideally, a slurry of uniform population and of similar size distribution is generated and maintained in all parts of the pan volume. The product crystals leaving the pan are of the same size and size distribution as in the slurry in the pan. Such crystallizers of the continuous, mixed-suspension, mixed-product-removal type (MSMPR) are widely used in continuous chemical and continuous sucrose crystallizing systems.

In the higher saturation environment for secondary seeding, as found in a continuous, single-stage pan, the CV would normally be 50%. As discussed later, this can be modified with product slurry classification and other techniques.

In a stabilized, MSMPR crystallizer, the crystal surface area for growth is:

$$A = N \cdot f(S) \quad (12)$$

when A = total area

N = total crystal population

$f(S)$ = characteristic surface area of a crystal.

The factor $f(S)$ is a function of the variance in sizes of the crystals (CV) and of the average size (MA).

The total crystal area, in part, identifies the rate of supersaturation decay. As nucleation rate is highly dependent upon supersaturation, slurry population is a function of supersaturation.

In batch systems, the *probable* final product MA-CV will be established within a few seconds or minutes of the pan seeding.

In continuous MSMPR systems, a long-term balance among several factors in the crystal slurry influence the final product MA-CV. Both nucleation and growth can be shown to be dependent upon supersaturation, but each responds in a different manner (292, 292a, 292b):

$$\text{Growth rate} = \text{length/time} = (\text{supersaturation})^x \quad (13)$$

$$\text{Nucleation Rate} = \text{number/time} = (\text{supersaturation})^y \quad (14)$$

$$\frac{\text{Growth Rate}}{\text{Nucleation Rate}} = \frac{\text{length}}{\text{number}} = \text{an index of crystal size} \quad (15)$$

Since: $y > x$

Then crystal size index decreases with increasing supersaturation.

Crystal Slurry Bulk Viscosity. The viscosity of the syrup in a pan helps to identify the concentration of that syrup. Upon seeding, a crystalline slurry is established. Initially, the suspended, tiny crystals offer little interference to the normal viscous flow of the syrup. As these crystals grow and evaporation brings the crystals closer together, a structural interference to liquid flow becomes evident. The apparent viscosity of the slurry becomes the sum of the normal syrup viscosity plus the hindrance to the normal flow character of the syrup, due to the added crystalline particles.

As crystallization proceeds, the bulk viscosity or fluidity changes to one of stiffness, and a limiting condition develops. In a thin slurry there is a large excess of fluid between the crystals. As sucrose is taken up by the crystals and as evaporation removes the water, the volume of liquor between crystals is reduced to the extent that only enough remains to "lubricate" the crystals as they are discharged from the pan or crystallizer.

As in Fig. 12-17, Gillett (124) noted the % true crystal volume and % apparent crystal volume for low raw crystallizers. The difference between the % apparent volume and 100 is the volume of lubrication syrup. For best process yields and economy, this must be low *at pan drop.*

Practical system operation dictates that the final crystal slurry must be able to flow from the boiling pan and must not be so stiff that the crystallizers and mixers are mechanically damaged.

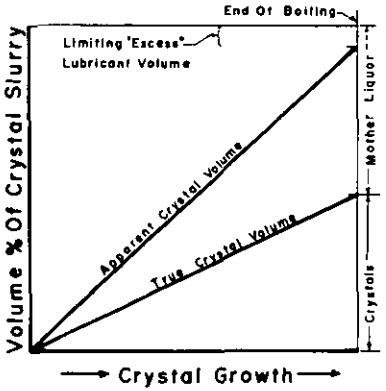

Figure 12-17. Percent crystal volume in crystal slurry. *[After Gillett (124).]*

Silina (340a) has shown the relation between slurry viscosity and syrup viscosity, Fig. 12-18. A practical limit to crystal growth in any given slurry is suggested. As will be developed later, this limit can be circumvented to a degree.

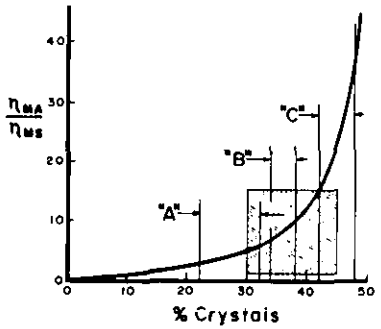

Figure 12-18. Silina ratio of viscosities. Viscosity of crystal slurry as compared with viscosity of the mother liquor:

A) range of % crystals during pan boiling.

B) range of % crystals at pan drop.

C) range of % crystals in final massecuite to the centrifugals. For detail in shaded area, see Figure 12-19 *[After Silina (340a).]*

Silina also developed the relationships between final slurry ds and % crystals and limits of % crystals for various temperature and purity conditions.

The small crystal sizes and low purity syrups of low-raw massecuites relate to a crystal limit of 40-45%. The larger crystals of high purity slurries, along with the lower syrup viscosities, permit crystal yields up to 55-60%.

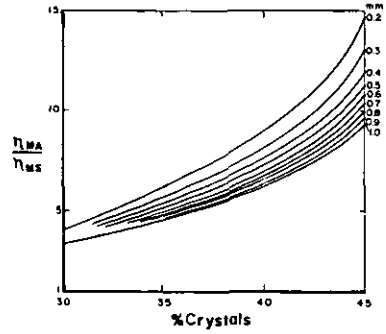

Figure 12-19. Effect of crystal size on massecuite fluidity as represented by Silina ratio. After Silina (340a), as expanded by Austmeyer and Kipke (11b), using the empirical expression of Gromkovskij and Fursov (137a).

Supersaturation Estimation by Viscosity. For a given syrup temperature, the viscosity of the syrup can identify the degree of supersaturation. The initiation of a crystal crop can be reproducibly performed during each batch.

The usual laboratory viscosimeter depends upon a small rotating cylindrical surface immersed in the fluid under study. The viscosity or liquid shear stress is measured by the torque required to rotate the surface.

In a similar manner, the Siemens viscosity measuring device (330a) has a cylindrical element which is mounted through the pan wall. The torque necessary to maintain a constant rpm is the measured value and relates to viscosity and indirectly to supersaturation of the syrup (131, 307).

Measurement of fluid shear by a wobbling element in the fluid is the basis of the P & L Rheometer (370a).

Following seeding, the presence of a fine, well-dispersed seed crystal has little influence on the viscosity measurement. As the crystals grow and the slurry density increases, the bulk viscosity properties gradually dominate. The viscosity measurement degrades and is no longer a measure of supersaturation.

The Ultra-viscoson-Bendix viscosity sensor consists of a vibrating element in the fluid, the means of inducing the element vibration ($28KH_z$), and a transducer for measuring the degree of vibration dampening due to the viscous structure of the surrounding fluid. The device has been used for very accurate investigation of viscosity, temperature effects, impurity effects, crystal-slurry effects, and the indirect measurement of supersaturation,[1] Fig. 12-20.

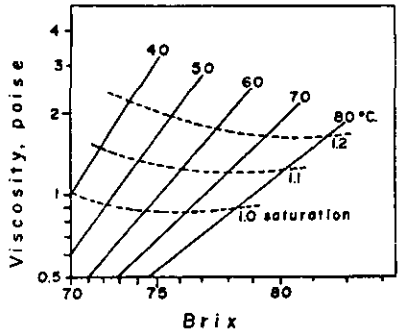

Figure 12-20. Effect of temperature on viscosity in pure sugar solution. ***(Courtesy of CITS.)***

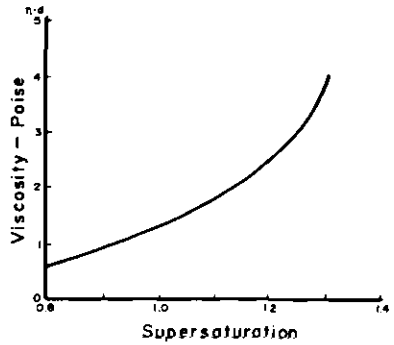

Figure 12-21. Viscosity and supersaturation. For temperature range 70-90°C and for pure sucrose solution. ***[After Ahari (1).]***

Supersaturation Estimation By Boiling Point Rise. Holven employed two resistance thermal elements in a characterized bridge circuit to measure "supersaturation." One element was in a pot of boiling water arranged to yield a saturated

[1]Ref. (1,10,12,295).

vapor at the pan absolute pressure. The second element was located in the center well of the pan. The system provided a reproducible temperature differential, useful for seeding reproducible crystal crops.[1]

The original thermal elements unfortunately exhibited an undesirable time lag. The hydrostatic head and the partial short-circuit flow introduced an increasing error as the massecuite level rose.

A later system (452) involved the measurement of the temperature of superheated vapors leaving the massecuite and fixing the boiling at a constant absolute pressure. Unusual disturbances in the absolute pressure regulation bias the supersaturation, even with excellent regulation of feed by the single measurement of vapor temperature.

A more recent unit (453a,453b) uses two low-mass thermisters to reduce measurement lag. One element is exposed to the superheated pan vapor, while the other is immersed in a tiny chamber of hot water continuously flashing down to saturation temperature at the existing vapor absolute pressure. The elements will respond well when exposed to a good vapor velocity and when the hot water feed is carefully installed.

Solubilities of sucrose solutions, Table 12-1a, are determined under nonboiling conditions (409a). Several investigators (22b, 32) have studied the effect of syrup superheat resulting from higher than equilibrium syrup temperatures. Such superheat is a normal action in creating a heat flux adequate for economical evaporation (ebullition) rates. Other factors of superheat are syrup level, crystal content, circulation rate, and syrup purity.

The lowest temperature in the syrup will be found at the top of the syrup, i.e., at the syrup-vapor interface. Fig. 12-22 illustrates this temperature-saturation relation. The highest saturation condition relates to the temperature of the interface.

The fixed thermal element in the well is subject to the syrup superheating error. Measurement of the superheated vapor temperature minimizes this error.

As evident in Fig. 12-14, the nonsucroses present influence the bpr. With high purity boilings, the change in purity is small. With low purity feed syrups, the purity

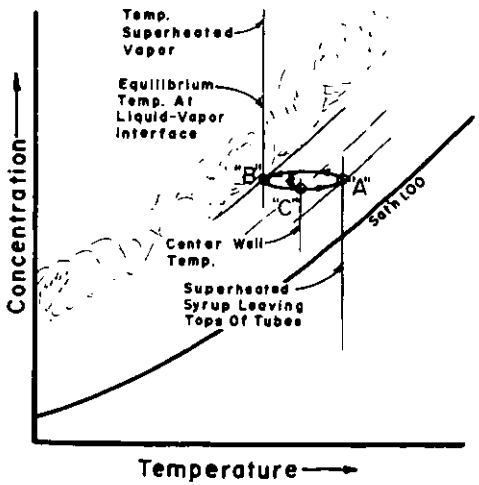

Figure 12-22. Effect of massecuite superheat on bpr measurement in center well:

A) temperature of syrup just above calandria.

B) temperature of syrup at vapor-liquid interface at top of massecuite.

C) temperature of syrup in center well with partial short-circuiting.

[1]Ref. (122,130,160,161,233,306,414,422)

change during boiling is significant. If necessary, a purity change compensation can be a function of level.

Temperature measurement of the superheated vapor can be erroneous if there are significant water leaks or poorly draining catchall systems, due to their desuperheating effect.

As with other systems, bpr can yield a useable, relative measurement. Each instrument must be "tuned in" to its particular boiling system. As it is measuring the properties of the mother liquor, it is a useful indicator of saturation conditions during the final minutes of the boiling.

Supersaturation Estimation by Refractive Index. The optical properties of solutions have long been used to measure the concentration of soluble solids. A typical, very successful application of refractive index (RI) for measurement and control of supersaturation has been reported by Neilson and Blankenbach (59, 254b). The usual requirement for rapid temperature compensation was avoided by designing the pan-boiling scheme for a constant 80.0 °C temperature during the critical graining and initial crystal growth period.

The RI measured is that of the mother liquor film on the prism surface. Thus we have a direct measurement of the solution properties. As crystals grow and the syrup film on the prism face becomes less homogeneous, the usual sharp refraction shadow becomes diffused and finally becomes unsatisfactory for control.

The nature of the total batch-boiling control scheme would indicate if this is a deficiency. As the boiling proceeds, the supersaturation may "sag" below the target (limiting) value and thus eliminate itself as an operating criterion. Consistency becomes the regulation-of-feed control parameter for the balance of the boiling cycle.

The boiling pattern of Neilson and Blankenbach is typical of a process-engineered balance of evaporation rate and evaporation need — a programmed pattern of Δt for ability to evaporate and a fixed syrup feed rds for a fixed need for evaporation. As crystals begin to grow, supersaturation gradually slips below the limiting value, thus refractive index measurement of supersaturation fits the needs ideally.

Other calandria pan systems with a generous ratio of heating surface per cubic foot of massecuite per strike, pans with a steam (vapor) supply at a pressure higher than needed, a steam valve which by habit is "full open," a condenser system and water temperature permitting a high boiling vacuum (a large Δt and high heat transfer rate), and pans fed with very high density feed syrups, all tend to have evaporation capabilities that overrun the crystal growth rate. Supersaturation *can* become excessive all through the crystal growth period, thus there is the need in such boiling patterns to be able to measure and control supersaturation for graining and continuously on through the brixing-to-drop period.

For the pan floor that needs to *push* supersaturation to reduce boiling time as much as posible, refractive index would become inadequate as the crystal content in the massecuite increases. Many pans have been equipped with simple optical refractometers to guide the sugar boiler through seeding and a few minutes into the crystal growth period.

For assurance that the syrup film on the prism will be constantly displaced by fresh syrup, the refractometer must be mounted so that the prism face projects three or four inches into the pan massecuite belt.

Preventive maintenance of the RI sensor includes a weekly (usually much less frequent) cleaning of the prism.

Supersaturation Estimation by Conductance. As has been shown earlier, a conductance measurement can be related to solution concentration of nonconducting sucrose. A factory syrup will normally have a typical impurity composition and thus will have a typical conductance for a given syrup purity. Changes in conductance for that typical syrup can then be related to the syrup viscosity or concentration or supersaturation (72, 162).

Typical conductance-purity determinations on local syrups become the foundation for designing the conductance bridge circuitry for the necessary compensation adjustments to "tune" the instrument to the local syrup properties. An ac bridge permits measurement of conductance without polarization of the electrodes in the massecuite.

Supersaturation Estimation by Dielectric Constant. With high purity syrups, the nonsucroses are present at relatively low levels and the changes in nonsucrose concentration are also relatively small. Conductivity is not satisfactory as a control. However, the S/W ratio changes are rather large.

The use of a high-frequency (150 MHz) transmitter, with its antenna in the massecuite, permits measurement of its dielectric constant. The measured value is the sum of the individual dielectric constants for water (constant = 80), sugar (3.85), and impurities (most organics in range of 1 – 10). Due to the large constant for water, the measured value is essentially a moisture measurement.

In the 90-100 purity range, Madsen (207a) reports only minor changes in the dielectric measurement due to impurities. The unit does not discriminate between sucrose in solution or as crystals. A level function compensation is needed.

While process stream measurement of dielectric constant has long been used in chemical process streams (1c, 330b), its application to crystallization is relatively recent.

Comment. To achieve reliable nucleation or seeding and to continue growth of the crystals at an economical level, an accurate measure of the solution supersaturation is needed from *seeding to pan drop*. Regardless of the crystal slurry consistency, secondary grain can be induced by neglect of the supersaturation of the surrounding syrup.

Consistency Measurement

Viscous Drag. As noted in the section on crystal slurry bulk viscosity, there is a decrease in fluidity of a slurry as water is evaporated. The syrup volume decreases and the crystals crowd together. The density of the crystal population per unit volume of slurry is of importance in avoiding or inducing secondary nucleation. In batch boiling and some forms of continuous crystallization, a high slurry density is desired. In other continuous crystallizing systems, the slurry density is maintained exceptionally low to induce secondary grain formation.

In a prior section, the Siemens viscosity unit, the Rheometer, and the Ultraviscoson were noted as being crystal dependent. The measurement was, in fact, bulk viscosity – the measure of the fluidity or the stiffness of the slurry.

In some series-continuous crystallizers, the mixing-cooling elements rotating through the slurry transmit a load to the crystallizer shaft. Motor load becomes a measurement of stiffness. In some mechanically agitated pans, the horsepower requirement of the impeller becomes the indicator of consistency.

Gamma Ray Densitometer. Many chemical process streams are being monitored for slurry density by the absorption of gamma rays passing through the slurry. For some continuous crystallizing systems, the final crystal slurry density is measured by this absorption principle. The measurement is total solids concentration (rds). One principal precaution is to sample the stream without incorporation of air bubbles.

Crystal Formation.

In the first paragraph of this chapter, our sugar boiler knew that if the syrup were boiled and evaporated long enough, it would suddenly be full of crystals. He had passed over the energy barrier. He knew it only as "boiling to grain."

The process technician (sugar boiler) of the second paragraph, carefully avoids the unstable boiling-to-grain condition. Nevertheless, he must scale a modified energy barrier where a more controllable crystal formation is performed.

Six general systems for initiating crystals are recognized (193, 292a, 392, 403a):

1. Homogeneous (Classical) — new crystal particle formation as the *result of supersaturation only* - a specific solution energy level is reached and exceeded.
2. Heterogeneous (Classical) — new crystal particle formation as the result of supersaturation *and* the presence of foreign insoluble material which reduces the net energy requirement
3. Secondary — new crystal particle formation as the result of supersaturation *and* in the presence of an ongoing, growing crystal crop
4. Attrition Collision — crystal population increase due to the fracture or impact chipping or breaking of existing, growing crystals into smaller particles, each capable of growth
5. Full Seeding — preparation of seed crystals by salting-out, grinding, screening, or centrifugal separation, mixing as a slurry or magma; adding the mixture as a controlled population into a supersaturated liquor *for growth only*
6. Ultrasonic — liquid conditions similar to homogeneous except a much lower supersaturation. Ultrasonic irradiation creates mechanical perturbations adequate to exceed the energy barrier of homogeneous nucleation (385b).

The Crystal Lattice. As molecules move close together, there is a tendency to seek the lowest level of energy. In organic compounds, covalent bonds are the predominant interatomic forces, establishing a relatively stable molecular architecture. The inter-molecular "glue" or forces holding carbohydrate molecules in their proper places in the solid structure are the intermediate strength hydrogen bonds and the much weaker van der Waals' forces.

The shape and dimensions of the unit building blocks are resultants of the various forces at work in achieving the lowest system energy level. This shape and its dimensions define a unit lattice (398, 407). The stacked array of units is known as the crystal space lattice.

In comparing a sugar crystal and its faces in the perspective view of Figure 12-23 with the conceptual stacking of sucrose building blocks in the projection

views, a logical development of faces is noted within the fundamental monoclinic system.

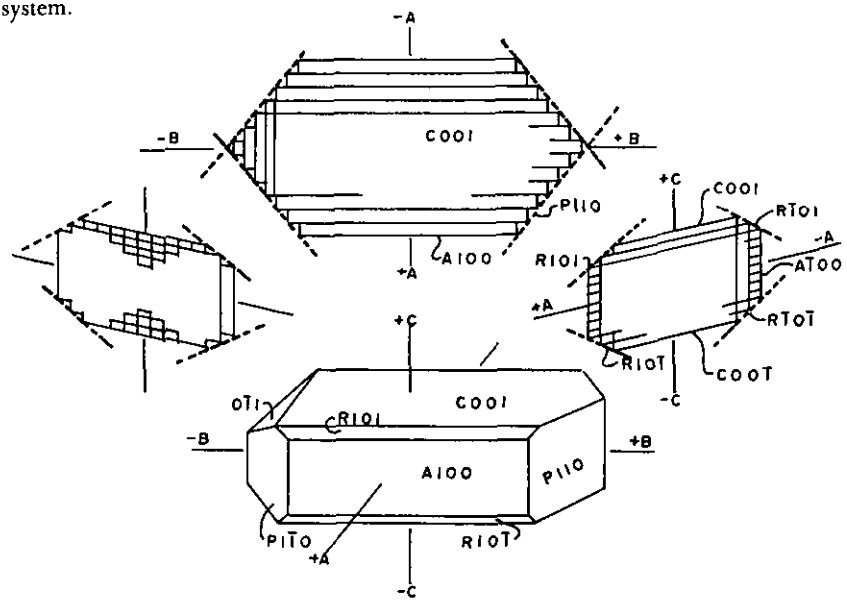

Figure 12-23. Stacking arrays and development of sucrose crystal faces.

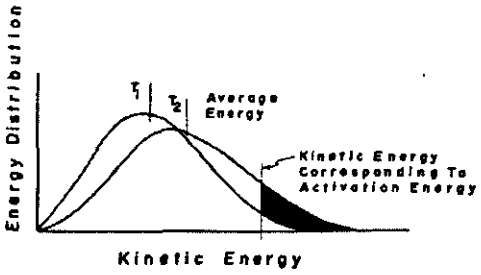

Figure 12-24. Energy distribution among molecules.

Energy Barriers and Fluctuations. Kinetic molecular theory recognizes the variance of energy by each molecule and utilizes probability to express the average status as a Maxwell-Boltzman distribution, Figure 12-24. A few of the molecules will possess high kinetic energy. Temperature increases the energy of all molecules present, and thus the average kinetic energy is increased. There is also a considerable increase in the number of molecules having the high level activation.

Reactions, whether physical or chemical, have energy barriers or levels which must be achieved before the reaction can proceed. Figure 12-25 is typical of many exothermic processes, of which crystallization is one. For the sucrose-water system, the energy level of the solution is increased by removal of water by evaporation.

For equivalent degrees of supersaturation, the temperature will enhance the probability of a molecule having sufficient energy to reach the system-activation energy level, and to proceed with the reaction, — in this case a phase change.

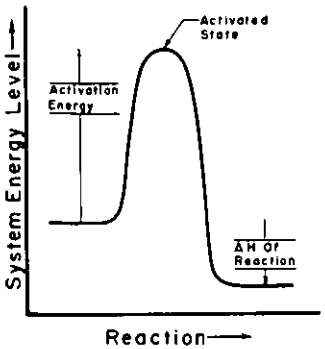

Figure 12-25. Energy barrier to change in state.

Wave mechanics provide another conceptual tool — energy concentration probability. In the reported procedure for determination of solubility, equilibrium establishment was stressed. While a macroscopic equilibrium may be established, the molecular equilibrium is still very active, with some under-saturated spaces and some over-saturated spaces in the system.

Using a three-dimensional probability representation (92, 157), Figure 12-26, varying degrees of energy activation may be expected throughout the space of the system. If the sphere sizes are considered to represent the probability of energy levels, energy concentrations sufficient to achieve the activation energy hump of Figure 12-25 are found. Increased supersaturation increases this probability.

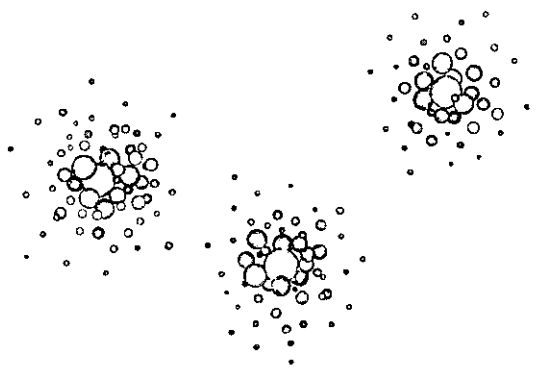

Figure 12-26. Three-dimensional probability of energy clusters. *[After Holden (157). By permission of Columbia University Press.]*

Homogeneous Nucleation. As a result of these fluctuations, the individual hydrated sucrose molecules frequently enter the area of attraction and rejection of one another and usually join momentarily. Most of these kinetic units quickly separate. Others cling together and are joined by still other molecules to become clusters — typical of reversible chain reactions (86,193,385):

$$A_1 + A_1 \rightleftharpoons A_2$$
$$A_2 + A_1 \rightleftharpoons A_3$$
$$A_{m-1} + A_1 \rightleftharpoons Am$$

where A_1 = an individual kinetic unit
m = the number of A_1 units in a cluster.

When "m" is small, the cluster still has no defined boundaries and is still a part of the solution phase. With an increase in "m," a new solid phase assumes an identity with boundaries, and is called an embryo. Being a reversible reaction, most embryos revert to clusters and even to individual units.

Some embryos in the supersaturated solution increase in size sufficiently to be in thermodynamic equilibrium with the solution. As an embryo reaches this critical size, it is called a nucleus. For sucrose, this critical size has been calculated to contain about 80-100 molecules (193, 292a, 403b).

Embryo Critical Size. Following the Gibbs deduction[1], creation of a new surface area requires work, ΔG_s, which is proportional to the extent of the new surface formed between the new solid phase and the parent solution phase. The volume of the new phase is at a lower energy level than the parent phase; thus there is a negative volume energy or work factor ΔG_v. Initially the surface work exceeds the volume work term, algebraically yielding a net positive system energy requirement ΔG as in Figure 12-27. As the size of the particle increases, the volume energy term becomes the larger factor and a system net energy maximum $\Delta G_{critical}$ on the main line is passed. Any array of less than the critical size will probably dissolve. Any array of greater than critical size will continue to grow (28, 86).

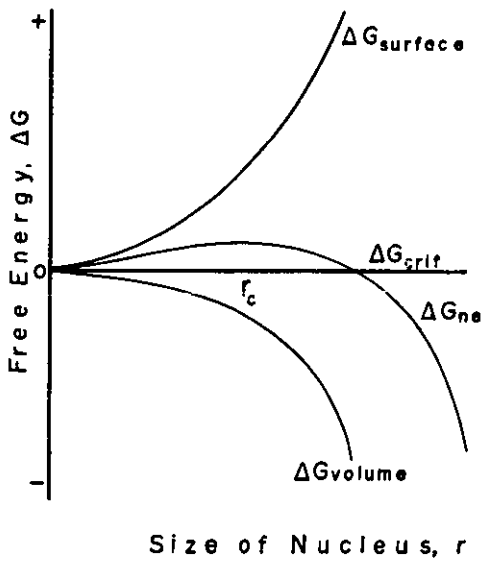

Figure 12-27. Embryo size, energy status, and stability for growth. *(After Mullen (251). By courtesy of Butterworth Inc.)*

This homogeneous nucleation model is a three-dimensional thermodynamic treatment of the molecular activity in the creation of a totally new solid surface, necessary for crystal growth.

Nucleation by "Boiling-to-Grain." Upon evaporation of a clean syrup, its viscosity increases rapidly. If a sample of the syrup is accumulated between the thumb and forefinger, then by spreading the fingers, the length of the string of syrup can be tested before it breaks. Spontaneous grain will not appear until a "long-string" condition is reached. This condition relates to being well into the labile zone, area A in Fig. 12-28a.

[1]See references (251, 359, 379, 385, 392)

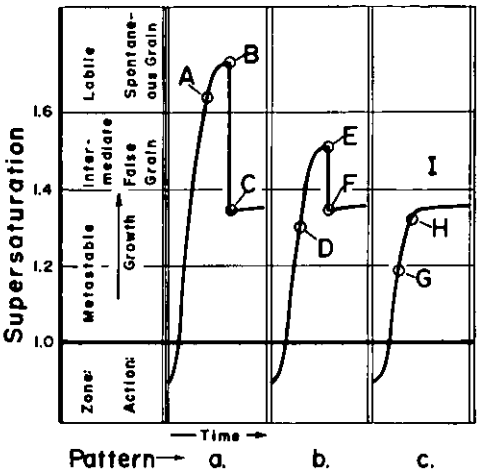

Figure 12-28. Typical supersaturation pattern during nucleation: a)boil to grain, b) shock seeding, c) full seeding.

Once started, fine grain will appear and will rapidly increase in quantity. The proper ending of the nucleation period point B, Fig. 12-28a, is a matter of experience and skill of the sugar boiler and his observations of syrup samples on the proof glass, of the splash on the sight glass, or better yet, by observations through the pan microscope.

A rapid return to the metastable zone is effected by a change in temperature and a reduction in concentration. Vacuum is decreased and a heavy, short syrup feed is introduced into the boiling syrup. Further nucleation is ended, but the just-formed crystals will continue to grow, point C, Fig. 12-28a.

Heterogeneous Nucleation. Heterogeneous nucleation occurs on flat surfaces or in cavities, on foreign particles, and on crystal defects. Part of the surface of the newly forming embryo is not exposed to the solution phase. Thus a lower energy level is required to get "over the hump" (380). In practice, this is the mechanism normally found due to the localized concentrations of:

1) The surface energy at rough or pitted surfaces of the equipment in use; 2) the surface energy of sucrose crystals not removed by proper steam-out techniques; 3) the surface energy of centrifugal grain in the pan feed syrups; 4) the surface energy of foreign particles in the feed syrup due to poor filtration; 5) the induced energy through mechanical perturbations, such as cavitation at impeller tips, or from sonic irradiation; 6) the surface energy by deliberately added "seed" crystals at the desired moment of nucleation; 7) or the solute in the solution due to nonuniformity of massecuite circulation or of temperature variances.

The Volmer expression (403a,411b) connects the two-dimensional heterogeneous mechanisms with three-dimensional homogeneous nucleation through surface tension and the angle of wetting:

$$\Delta G^*_{het} = \Delta G^*_{homo} \frac{(2+\cos\Theta)(1-\cos\Theta)^2}{4} \qquad (16)$$

Θ = angle of wetting

ΔG = activation energies of the processes.

If sucrose seed crystals, having identical lattice parameters as the solute, are the foreign surfaces introduced, and if metastable supersaturation exists, growth will

begin immediately on the lattice fragments introduced. The foreign particle will be considered completely "wetted" ($\Theta = 0$). ΔG^*_{homo} reduces to 0, a seeded nucleation.

For complete repellency ($\Theta = 180$) — lattice misfit, smooth surfaces, low energy particles, etc., — this condition would amount to an ineffective nucleus.

For partial wetting, the free energy of the critical nucleus is reduced, and two-dimensional nucleation and growth is initiated.

Nucleation By Shock Seeding. In the search for a more reliable technique of initiating a crystal crop, a less drastic method of nucleation was introduced in the beet industry by Zitkowski (420a). A clean syrup is concentrated and string-proof tested until a short string (about an inch) is attained. This corresponds to the mid-to-upper metastable zone. Approximately a pound of powdered sugar is sucked into the pan and into the syrup, point D, Fig. 12-28b.

Concentration continues until a crystal crop becomes visually evident. Again, the sugar boiler must judge the proper time to end the nucleation, point E, Fig. 12-28b. A short, heavy drink of syrup feed is usually enough to reestablish the metastable condition, point F, Fig. 12-28 b. Some boilers also feel the need for a change in vacuum.

In formation of the new solid phase, the Gibbs relation is dependent upon overcoming the surface tension component of the energy requirement. Volmer (403a) defined the change in surface tension due to a "wetted surface" and thus explained the lower energy level required for critical nucleus formation. The surface of the suspended particulate matter reduces the energy needed for two-dimensional nucleation and growth.

VanHook (385a) and Dunning (86a) demonstrated the time lag in cluster formation, particularly in viscous solutions such as sucrose syrups. The supersaturation level for "shock" seeding is still high enough to create 3-dimensional clusters. The introduction of particulate surfaces not only contributes to two-dimensional nucleation, but in some catalytic manner, reduces the time lag in 3-D cluster formation.

The time-lag factor is important in the actual "shock-seeding" procedure. The number of crystal fragments in the powdered sugar introduced is only a small fraction of the total seed population needed. The powdered material should be introduced as soon as the upper metastable range is reached, to have time for the nuclei to become visible and adequate before continued evaporation carries the supersaturation too high. An early seeding and a rapid return to metestable (less viscous) conditions, after grain establishment, are reported to yield fewer conglomerates.

The term "shock seeding" is generally misleading and creates confusion. Careful examination suggests that this procedure deliberately generates the conditions leading to "false grain" or secondary nucleation (215a,292a,292b,453c).

Secondary Nucleation. Shock seeding and slurry seeding have often been performed with totally inadequate seed populations, but the final massecuite population has been near normal. To achieve this result, the sugar boilers often resorted to a short term, higher than normal, seeding saturation, Fig. 12-28b.

In 1911, Miers (240c) observed that when a seed crystal is introduced into a solution having a supersaturation not high enough for homogeneous nucleation, that seed crystal itself begins to grow, and it also induces other crystals to form and to grow some distance from itself. The primary seed crystals have been observed

(224f) to "give birth" to large numbers of new, secondary nuclei.

Almost immediately upon being immersed in the super-saturated syrup, the surface of a primary seed crystal becomes wetted with the hydrated sucrose aggregates of the solution. This surface layer (1,000 Å units or more, depending upon the degree of supersaturation) becomes even more highly organized as growth begins. Powers (288) suggested that some of the clusters in the surface film could be loosely held and sheared away by agitation, and thus could become a stable cluster for new particle growth. The second alternative is that at high saturations, dentritic-growth structures on the primary seed crystals are sufficiently fragile to be sheared off by the solution agitation and become secondary growth centers. These new nuclei can, in turn, become the initiators of further secondary nucleation. At high supersaturation, the secondary effect can proceed at catastrophic rates.

Such nucleation is the source of "false grain" in the usual boiling procedures for sucrose. Secondary nucleation is also the mechanism used in many continuous evaporative crystallizers for continuous seed generation within the crystallizer.[1] Its use and regulation identifies system productivity (population) and product quality (MA-CV).

Attrition and Collision Nucleation. Very common in chemical industry crystallizations, the crystal structure is quite fragile and the solution viscosity is low. Agitation is necessary for uniformity of nucleation and growth conditions. Impact of crystals by the agitator impeller and impact of crystals against the crystallizer walls, in part, describe attrition — the mechanical degradation of crystals in the slurry. The fragments become growing crystals and become additions to the population.

With sucrose solutions, the viscosity is high and impact is less severe. Also, the crystal sizes are relatively small and the inertial impact is less. With batch boiling, the agitator systems are designed with ample clearances and relatively streamlined flows.

With continuous systems depending upon secondary nucleation, the greater the attempt at capacity-forcing by higher supersaturations, the greater will be the possibility of dentritic growth which is more fragile and *might* be a source of attrition and collision fragments. However, at the higher saturations, it becomes more difficult to distinguish between secondary "breeding" and attrition effects.

Nucleation By Full Seeding. With the successful development of boiling point rise and other systems for estimating supersaturation, reproducible crystal crops are initiated at much lower levels of supersaturation.

Large size specialty sugars have been produced by introducing several thousands of pounds of carefully screened and sized crystals into a vacuum pan containing a moderately supersaturated syrup, point G, Fig. 12-28c. Each individual crystal introduced was a "seed" which immediately began to grow to become a larger, more desirable size.

With the usual readily available powdered sugar as the seed, it was found that if several pounds were used at saturations less than previously used for "shocking," point H, Fig. 12-28c, the total of the individual particles of powdered crystals approximated the population needed in the final massecuite. To reduce the volume of seed introduced, trials with the much smaller crystal fragments of "fondant" powdered sugar were successful. However, continued variances in populations were experienced.

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[1]See refs.(215a,239b,292a,292b,293a)

The investigations of Mak and Kelly (208a) have identified the unusual surface activity of dry ground and pulverized sugars. Surface charges induced an agglomeration of the dry fragments, leading to population uncertainty.

The continued investigations of Gillett (123) and others (164a,343d) with wet ball-mill grinding of a suspension of crystals led to a much more stabilized and reproducible seed. Also, with extended grinding times, smaller volumes of seed slurry were possible. With the smaller size seed, the slurry is introduced at a somewhat higher saturation, point I, Fig. 12-28c.

Several published boiling patterns are for "full seeding," however, they resemble the Fig. 12-28b pattern. The initial seed growth is "forced" by the higher saturation until a visual size of 40-50 μ is attained. Such forcing reintroduces a degree of uncertainty in the final population.

Some continuous, multi-compartment pans are full seeded, in fact, by continuously pumping a prepared magma into the first compartment.

Seed Slurry Preparation. Using a standard laboratory ball mill with porcelain jar and flint pebbles, a mixture of one lb of powdered sugar per one quart of isopropyl alcohol, ground for 24 hours, will yield a practical slurry for seeding.[1] The Ditmar slurry mill is being used by some boilers. It is reported to have a greater milling capacity. Seed size above 15 to 20 μ requires large amounts of seed materials. Seed size below 5 μ involves such small amounts of material that slurry measurement and handling errors become significant in establishing an assured population. Typical slurry volumes are (182):

White pans use 130-150 ml slurry/1000 cu ft massecuite
Intermediate pans use 160-220 ml slurry/1000 cu ft massecuite
Raw pans use 450-600 ml slurry/1000 cu ft massecuite.

These seed volumes must be adjusted to accommodate the variances of individual pan circulation, heat flux, vacuum uniformity, and saturation measurement errors. MA-CV (see pp. 547-548) analysis of the individual strikes will permit a rapid ad-

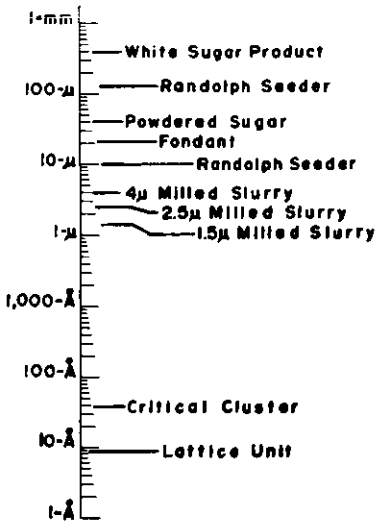

Figure 12-29. Relative size of seed.

[1]See refs. (119,123,127,141,165,181,197,234,302,337,393)

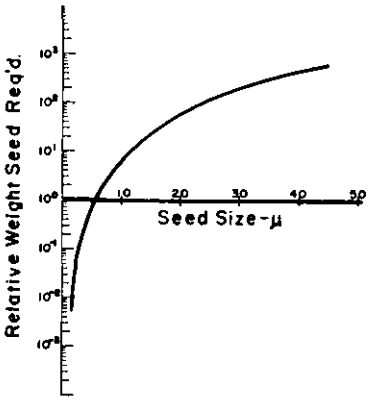

Figure 12-30. Relative weights of seed to achieve similar final product populations.

justment of slurry volume to yield a more reliable graining technique.

Seed slurry injection into the pan should be made without a simultaneous air introduction. To insure generation of the full population, an alcohol flushing of the seeding apparatus is common procedure.

The usual isopropyl alcohol seed slurry tends to settle easily. To automate batch pans more reliably and completely, Madsen and co-workers (207b,207c) suggest grinding sugar in a polyethyleneglycol solvent to obtain a slurry having a viscosity matching that of the pan syrup. Settling is less rapid.

Early descriptions of seed preparation for continuous boiling included a proportional, vibrating feeder for dispensing screened, dry crystals into a presupersaturated feed syrup, flowing into the first compartment of the pan. This system can be elegant in simplicity, but massive in logistics (88,99,100).

A continuous grinding mill, fed with just-completed magma, is an integral part of a closed loop seeder for some continuous, multi-compartment pan systems (15b), Fig. 12-31.

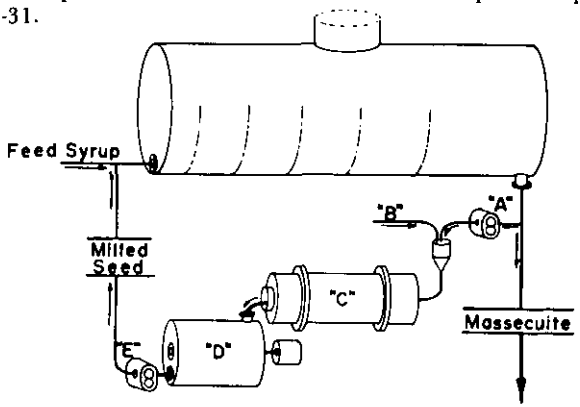

Figure 12-31. Continuous crystal grinding mill using massecuite to form seed slurry:
a) variable speed massecuite transfer pump.
b) dilution.
c) horizontal grinding mill.
d) agitated, milled-seed slurry receiver.
e) variable speed pump for seed transfer to crystallizers. *[After Barkow and Witte (15b).]*

Several continuous boiling systems are, in essence, full seeding. The elements of "einwurf" boiling are noted in some magma systems. The crystals separated from a low-grade boiling are mingled with a higher purity syrup to form a normal, pumpable magma. Upon entering the first stage of the crystallizer, the crystals simply begin to grow, thus the term "crystal enlarger" typifies the nature of these systems (273a).

If a reduced CV in the final product is of significance, the above magma method can be modified (71b,240a) by classification of the seed prior to use as the continuous magma footing, Fig. 12-32. Oversize is recycled through a grinding mill. The intermediate, selected fraction is mingled as above as the continuous seed source. The undersize with its "affination" syrup can be used as the footing for the initial boiling.

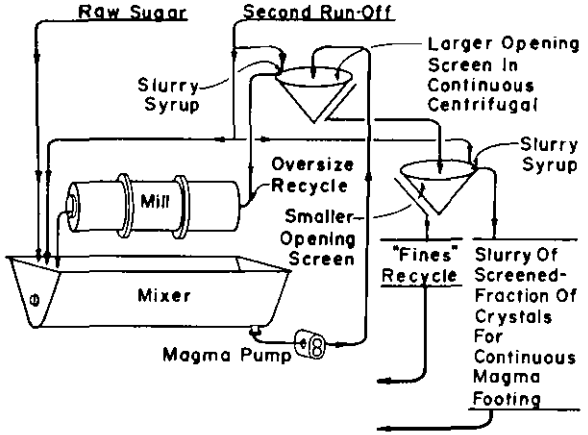

Figure 12-32. Classified size seed for magma seeded continuous crystallization, a dual classification.

The continuous, single tank crystallizer, Fig. 12-73, depends upon initiating secondary nucleation, first with a batch of seed slurry, and second, establishing a self-sustaining nucleation, part by continuing secondary action and part by an external classification and recycling of undersize product.

The nucleation rate in such a system is shown as curve "N" in Fig. 12-33. The linear growth rate is curve "G." A carefully controlled and sustained excursion into the intermediate saturation range can continuously induce secondary seeding and simultaneous growth.

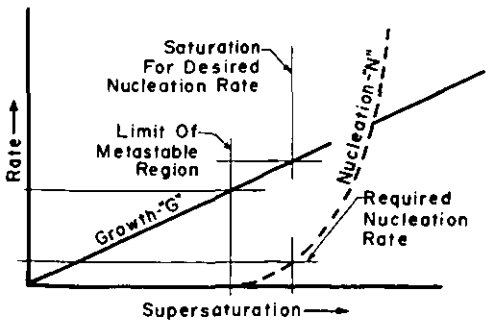

Figure 12-33. Continuous secondary nucleation and growth.

Kelly and Mak (188b) explored salt-out systems for homogeneous precipitation. An average particle size of 4.6μ was obtained with methyl cellosolve as the third component. With ethanol, particles with an MA of 60μ were formed. The particles were examined under an electron microscope and were generally observed to be porous structures.

Battelle (22a) developed a similar salt-out system using polar, organic solvents as the third component.

Randolph and co-workers (292c) have successfully developed a continuous salt-out system, using a mixture of organic solvents in a MSMPR-type crystallizer. This patented unit is an integral part of the continuous boiling system of one U.S. refinery (270a,270b).

Supersaturation During Nucleation. The Kelvin equation identifies the theory of enhanced solubilities of very small crystals (1c). This greater solubility has influenced part of the nucleation procedures developed through the years. In "boiling-to-grain," the sugar boiler evaporates and concentrates until the first "sparklers" appear. He continues to wait for particle growth, and then saturation is reduced, Fig. 12-28. The larger "sparklers" will continue to grow. Relieving of the supersaturation reduces the solution energy level, and some very small "sparklers" may be dissolved.

Achieving size of normal stability is estimated in Fig. 12-34. A reaction energy curve has been modified with particle size as the ordinate axis. It is probable that the higher saturation level is often used longer than necessary, as process insurance.

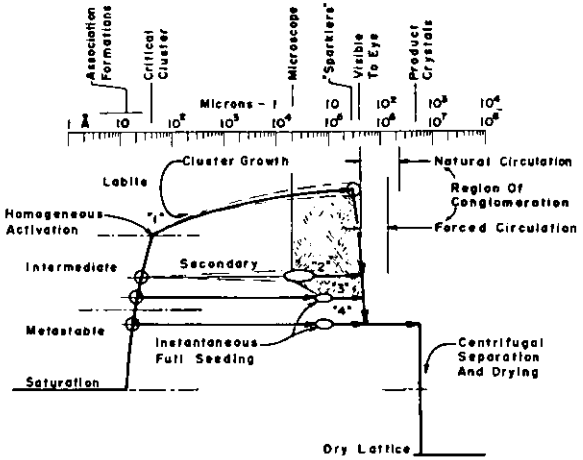

Figure 12-34. Practical approximation of saturations for assured nucleation and seeding:

a) homogeneous, critical cluster, saturation maintained until visible "sparklers" $\approx 25\mu$.

b) heterogeneous nucleation with 1-4 μ finely ground seed slurry, saturation maintained until visible "sparklers" $\approx 25\mu$.

c) "full seeded" with natural circulation.

d) full seeded with forced circulation.

e) shaded area is extra "insurance," as built into some batch and continuous systems. Compare this "reaction curve" with its equivalent in Fig. 12-1 and 12-25.

The size scale above the reaction curve helps us realize that the homogeneous nucleus increases in size very rapidly at first. This leads to the speculation that the first structure formation must be by agglomeration of many clusters into a mosaic structure which soon reaches the "sparkler" size.

In very pure solutions, this may be a matter of one or two minutes. With the elevated viscosities of impure syrups, assembly of the structure may require 15 to 20 minutes.

With such speculation as a background, we can better appreciate the magnitude of the activity that has taken place before we can ever see the new crystals. The simplicity of "full seeding" with larger-size milled seed is apparent.

Crystal Growth

Mass Transfer. Crystallization is the transfer of sucrose from the solution phase to the solid phase. Supersaturation is the measure of the "pressure" or driving force to cause this transfer to take place. Resistance to the transfer of sucrose molecules from the liquid phase to the solid phase is of two forms: 1) resistance to diffusion or movement of molecules from the bulk of the solution to the surface of the solid; 2) overcoming those energy requirements necessary to shed the water sheath, to orient into the lattice "fit" position, and to jump, or otherwise move into the lattice vacancy.

In the comparative evaluation of various mathematical models of growth, Ohara and Reid (258a) compared rates for various inorganic, ionized salts as well as sucrose, Fig. 12-36. The general power function of supersaturation was used for curve fitting:

$$R = f(S)^x \qquad (17)$$

where: R = growth rate

S = supersaturation

Based upon Smythe's data (27a,343b,343c), the family of growth curves indicates x = 1 (a linearproportionality) at higher saturations, and x→2 at lower saturations (a second order rate limits the net growth realized). Two interdependent, successive reaction steps are needed to define the crystallization-from-solution unit process.

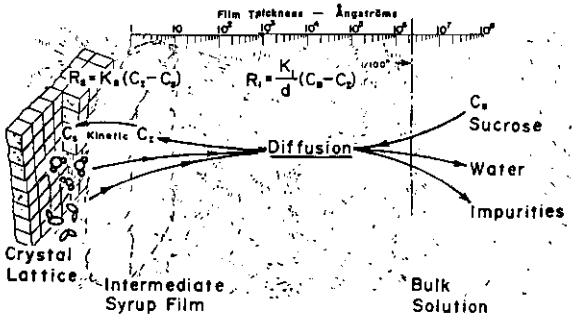

Figure 12-35. Molecular activity at crystal surface and in laminar surface film. Two reaction equations are needed to describe the separate activities and as the sugar molecules move through the two conditions in series, the slower rate sets the overall rate for growth.

Fig. 12-35 represents recent growth rate data for sucrose. At higher saturation levels, a first-order rate prevails, while at lower saturations, a second-order rate is dominant. Silin (340b) identified a two-step mechanism for crystal growth: a) diffusion of molecular sucrose through the surrounding syrup, and b) the surface kinetics or the incorporation of these molecules into the crystal lattice, Fig. 12-35. In the first step:

$$R_1 = \frac{k_1}{d} \cdot (C_B - C_I)^x \quad (18)$$

where: R_1 = rate of diffusion
k_1 = diffusion constant
d = thickness of the nonagitated syrup film (up to 250μ)
C_B = concentration in the solution bulk
C_I = concentration in the intermediate layer next to the crystal surface
x = reaction order (usually $x = 1$)

For the second step:

$$R_2 = k_2 \cdot (C_I - C_s)^y \quad (19)$$

where: R_2 = rate of surface incorporation
k_2 = surface kinetics constant
C_s = concentration at the crystal surface (252)
y = reaction order (y varies from 1⟶2)

Arrhenius (1c) observed that rate constants showed a temperature dependence expressed as:

$$k = Ae^{-E^*/RT} \quad (20)$$

Where k = reaction rate constant
A = preexponential or frequency factor
E^* = activation energy, a characteristic energy that must be added to the reactants for the reaction to occur
R = gas constant
T = temperature in °K.

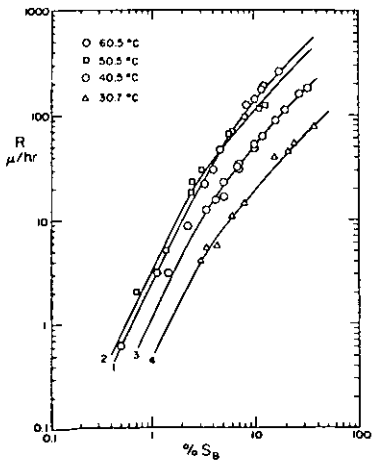

Figure 12-36. Growth rates for pure sucrose. The BCF Surface Diffusion Model appears applicable (258a). Smythe used high stirring rates to reduce effects of fluid velocity (343a). *(Reproduced by courtesy of Prentice-Hall, Inc.)*

The activation energies for diffusion, for viscosity, for nucleation, and for crystal growth all have different values, thus we can expect different reaction rates at differing temperatures and concentrations.

Both the transport and surface reactions are influenced exponentially by temperature, but other factors alter the net relationship of both. In diffusion, there is a structure-breaking influence upon molecular flow. In surface activities, there are comparatively low energy van der Waals attractions, hydrate-bond breaking, and impurity-desorption bond breaking, all of which slow down the rate of sucrose incorporation. The dipole and molecular lattice structure demands that the oncoming molecule lose its three freedoms of rotation and become precisely oriented and positioned, to be accepted into the lattice.

There are many aspects of sucrose crystallization rates that still need to be explored and defined. However, the work of many investigators provides a practical consensus of understanding:

1) The diffusion and surface reaction rates both contribute to the net growth rate, observed and measured.

2) At low temperatures, the rate of surface incorporation is at a rate far below the diffusion rate. The net rate approaches second order — surface kinetics control.

3) At intermediate temperatures (about 50 °C), there is a transition from second-order to first-order rate.

4) At higher temperatures, the surface kinetics far exceed the diffusion rate, which becomes the limiting first-order rate.

For crystallization-by-boiling procedures, practically all of the activity is above 50 ° and is at moderate-to-elevated supersaturation. The simplified growth rate equation becomes:

$$\text{Unit growth rate} = \frac{dw}{dt} = K \cdot (C - C_0) \tag{21}$$

On the basis of growth on all the surface area of the crystals of the crystal slurry system:

$$\text{Growth} = A \frac{dw}{dt} = K \cdot A \cdot (C - C_0) \tag{22}$$

In equations (21) and (22), and in the following original graphs of Smythe's data:

C = gm sucrose/100g H_2O in the solution being studied
C_0 = gm sucrose/100g H_2O at equilibrium solubility at the same temperature
S = supersaturation = $(C - C_0)$

In the original graph of Ohara and Reid (Fig. 12-36), the concentration driving force is expressed as % S_B where:

$$S_B = \frac{C_B - C_{BE}}{C_{BE}} = \frac{C - C_0}{C_0} \tag{23}$$

In Fig. 12-37, a crystal surface is surrounded by a supersaturated, intermediate film of syrup only a few Å thick. Sucrose molecules in this layer are the source of sucrose for the surface kinetics of crystal growth. From this layer, the adsorption on the surface, the orientation, the bonding into the lattice, the desorption of displaced hydration water, the desorption of displaced impurities, and the diffu-

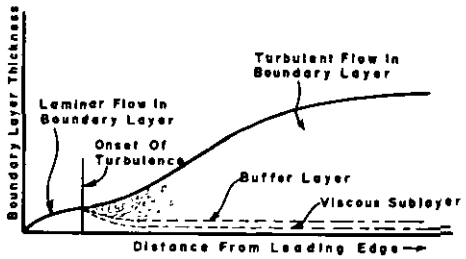

Figure 12-37. Viscous sublayer at surface of solid is always laminar and relates to viscous shear flow of Figure 12-76. First transition to turbulent flow is in the buffer layer. The asymptotic rate-limiting effect of Figure 12-38 relates to the nonturbulence in the viscous sublayer. *(Reproduced by permission McGraw Hill) (215d).*

sion-migration of these desorbed products into the outer mass of mother liquor will materialize.

For continuation of the surface incorporation activity, the concentration of sucrose molecules in the intermediate film must be sustained by constant diffusion of other sugar molecules through the surrounding laminar film and from the bulk syrup.

If the crystal slurry is only gently agitated, the diffusion path for sucrose replenishment of the intermediate layer is the more difficult, longer path and the diffusion rate may (will, for sucrose) limit the growth process.

If the crystal slurry is strongly agitated, the turbulence in the bulk syrup may disrupt the surrounding film to as little as 1/100 inch final film thickness. (Estimated from the theoretical model of "boundary layer" (215d), Fig. 12-37).

In the theoretical development of the "viscous flow" model, Fig. 12-7b, Adamson (1c) and Hughes (173a) identify that by definition, zero-flow must prevail at the solid-liquid interface. If each successive micro "plane of fluid shear" in the liquid is assumed to be 10Å and that the 1/100 inch thickness of the macro "viscous sub-layer" is realistic, there should be some 250 000 planes of shear in that 1/100 inch. Fluid flow rate in those first several hundred shear layers, immediately adjacent to the lattice surface, will be negligible to zero. Diffusion through these remaining 2 500 000 Å is a residual, rate-liming factor as evidenced by various investigators[1] and Smythe, Fig. 12-37.

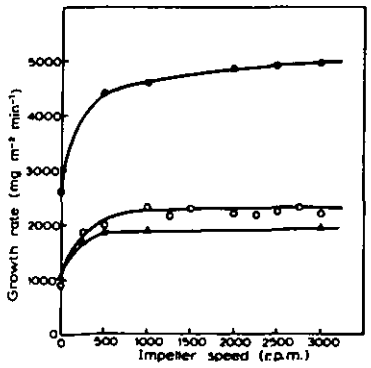

Figure 12-38. Effect of stirring on crystal growth rates. Laboratory test data: limited improvement with increase in rpm (343a).
● 40°C ($C - C_s^\circ$) = 70g/100g H_2O
○ 50°C ($C - C_s^\circ$) = 17g/100g H_2O
▲ 40°C ($C - C_s^\circ$) = 27g/100g H_2O
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[1]See refs.(89,173a,190c,215c,343a,397)

Smythe's data not only illustrate the benefit of fluid movement upon reduction of the diffusion rate resistance, but even more particularly identify the asymptotically limiting aspect of this benefit. These data suggest a doubling of growth rate for only nominal agitation.

In general, at high saturation and low turbulence, the system tends towards mass-transfer or diffusion control, while at low saturation and high turbulence the control becomes that of surface reaction (358). Many authors (27,395) report favorable results with agitation. Various stirrer speeds are given, but a quantitative hydraulic environment is seldom defined.

The Rodgers and Lewis data (298) on the effect of circulation rate on bound moisture and ash content of the final product can be interpreted, in part, by the surface mechanism of crystallization as follows:

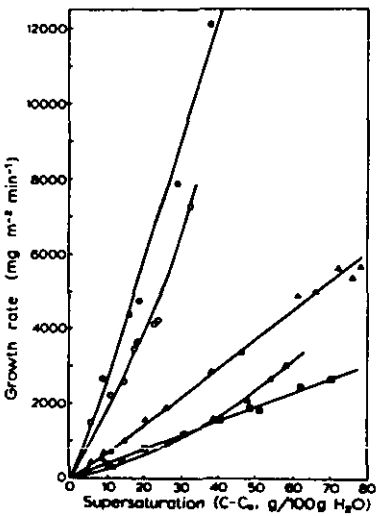

Figure 12-39. Effect of stirring on crystal growth rates at different temperatures (343a).
- ● 70.5 °C 3,000 rpm
- ○ 70.C free fall
- ▲ 41 °C 3,000 rpm
- △ 40 °C free fall
- ■ 41 °C crystal stationary

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1. Sucrose molecules must shed their sheaths of hydrated water molecules to be accepted into the lattice. The water molecule concentration rises near the crystal surface and increases the probability of the relatively small water molecule being occasionally incorporated as an interstitial impurity. Powers' exhaustion sleeve experiments demonstrate the presence of the dilute layer (278).

2. Impurities rejected by the lattice tend to accumulate, just as water, and become considerably more concentrated at the lattice surface. Wilcox (427) has pointed out that occlusions may contain liquor even more concentrated, with respect to impurities, than the mother liquor from which the crystals were grown.

A reasonable circulation rate would reduce the surface film thickness and lessen the probability of impurity incorporation.

Growth Mechanisms. The macroscopic observations of crystals and their growth provide an insight to the molecular aspects of crystal growth. Plane faces establish the geometrical boundary of the crystal. Growth mechanisms appear to have some restraints which promote the layer growth on those plane faces. Lapsed-time photographs of growing sugar crystals have confirmed the assumptions of growth by layers moving across the crystal surface (279,282). The step height is quite low as compared to the distance between steps. This can be deduced from the

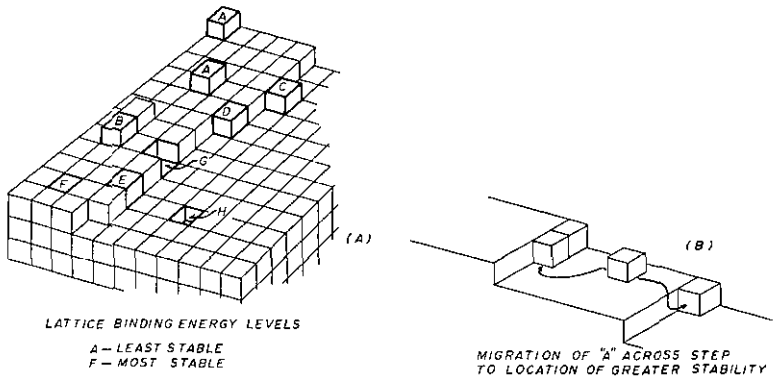

Figure 12-40. Repeatable-step layer growth.

relative constancy of the interfacial angles. Powers' cinemicrophotos and the spiral growth reported by Albon and Dunning (2,3,83) have opened new vistas in the understanding of crystal growth, not only for sucrose but in many new fields.

Of the various sites for a molecular "fit" into a lattice, Figure 12-40, each has an energy status or binding energy. These energy levels suggest a mechanism of growth. As the model is examined, the environment should be kept in mind:

1) The lattice model is surrounded on all sides by a multitude of sucrose molecules in various stages of hydration; 2) the lattice surface retains bound hydration water; 3) the intermolecular collision forces; 4) unassociated water molecules tending to separate the sucrose molecule from its position on the face of the lattice; 5) and the variances in all the above due to concentration and temperature fluctuations.

The total binding energy for a single sucrose unit would be the sum of energy between the single unit and each adjacent neighbor in the lattice. In Figure 12-40a, the "A" position is the least stable and could be swept away again into the solution by molecular impact, thermal fluctuation, or saturation fluctuations.

Position "D" yields a third-level stability, and, in turn, provides a third-level, adjacent site for another oncoming molecule. The probability of a vacancy at "E" is low, and the probability of an "H" vacancy is even more rare. Thus it must be concluded that the "D" position has the greatest probability of existence and provides a reasonable level of stability. This "repeatable step" structural configuration provides the basis for layer growth, Figure 12-40b.

The separation between steps can be related to the lower probability of an "A" position building block remaining long enough for an even lower probability second building block forming the "B" condition. The concept of "A" and "B" type units, migrating across the face of the plane surface to the repeatable step, accelerates the rate at which units are deposited and bonded at the step (361). The further development of an edge row or a corner will provide the initial "D" condition for the next repeatable step.

The formation of new steps has a low probability, and thus growth rate has been calculated as being near zero, or at best, very slow (48). Actual growth rates suggest that an additional factor is involved. Frank's dislocation screws (106) pro-

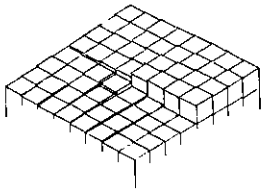

Figure 12-41. Frank's dislocation screw [After Frank (106). *By courtesy of The Faraday Society.*]

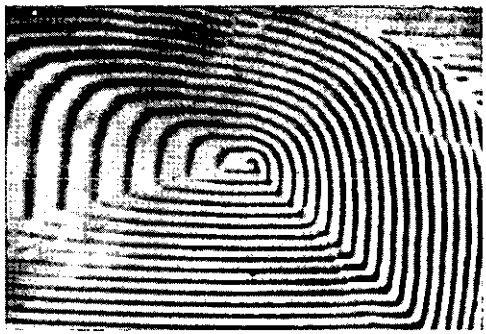

Figure 12-42. Growth spiral observed on growing sucrose crystals. (Photo by courtesy of CITS.)

vide the need for a continuously forming repeatable step, Figure 12-41. These dislocation screws have been observed on sugar crystals as moving spirals (83). Such screw steps must be multi-molecular in height, to be visible with optical microscopes, Figure 12-42.

Deposition rates in the magnitude of 50 layers per second are necessary to account for a crystal 0.5 mm in length within the usual high grade strike boiling time.

The association mechanism in the liquid phase leads to a speculation as to how the clusters enter into the lattice building activities. Bonding energies are reached which permit association, but which are insufficient to nucleate homogeneously. The clusters are of sufficient energy to deposit in a kink or step of macroscopic dimensions in a manner resembling molecular, single-layer step growth.

It is suggested (288) that in saturated or slightly unsaturated solutions, such clusters continue to exist and alter the subsequent crystallizing characteristics of that solution or syrup.

Grisdale (136) postulates that these clusters or aggregates may be of sufficient size to constitute a colloidal system with its electrokinetic potential barriers. Long range van der Waals-London forces which may be effective over several hundreds of Ångstroms distance, may be the driving force for the surface mechanism growth.

While the step theory of growth is in a single molecular layer scale, the cluster effect and the visual observation of step growth suggests a zone of considerable thickness. Garner (113) refers to "polymolecular sheets" and a visibly detectable series of steps.

The lack of sharp resolution of X-ray diffraction studies of some crystals, suggests a mosaic structure (93, 411). A subassembly of blocks of relatively perfect unit assemblies can be visualized. The resulting block wall is ill-fitted, having frequent misorientations of several seconds of arc. Such mosaic structures would be more probable in elevated supersaturation environments.

Imperfections and Occlusions. Dunning's (84) graphic confirmation of the range of variance of growth rates of individual crystals in identical environment sug-

gests such variance is related to a structure-sensitive growth process.

Surface imperfections on sucrose crystals are essential to acceptable crystal growth rates. It is probable that future investigators will demonstrate the need of occasionally introducing a new set of surface imperfections by short intervals of an etching environment, to reestablish an acceptable crystal growth rate particularly if large crystals are to be formed. The technologist must strive for imperfections which enhance the growth rate and the system economics, but he must also avoid those imperfections that are degrading to the product quality. The influence and importance of imperfections is common to crystal growth of nearly all chemicals, minerals, and alloys. A brief, partial list of kinds of imperfections follows (112,360,426):

A. *Point Defects.*

1. Vacancy—a lattice site which is unoccupied.

2. Interstitial—an extra molecule in a normal, filled lattice, and usually causing lattice strains. Single interstitials are additional molecules found in a single lattice unit. The extra molecule may be sucrose or of foreign nature. If the foreign molecule is relatively small, the lattice will not distort, such as water molecules in sucrose lattice. Powers (280, 281) reports up to 0.4% "bound water" in large crystals, a portion of which may be interstitial.

3. Substitutional—a foreign molecule occupying the space normally filled by a sucrose molecule. Substitutional impurities require a reasonable lattice fit and energy fit. Raffinose is the most prominent of the substitutional impurities. It is believed that some ash and colored degradation products may be substitutional.

B. *Line Defects.*

1. Edge Dislocations—a wedged-in, extra "slice" of lattice units, terminating externally. Line defects may be formed by internal thermal stresses. However, the crystal mass and the temperature environment normally found in practice suggest this is a minor source of defect. Examination of a nucleation event suggests that the clustering or agglomeration of molecules to form the nucleus yields a denteric or mosaic, ill-fitted structure. As the embryo grows, the spaces between the denteric branches begin to fill in with a very high probability of mismatch and dislocation formation.

2. Screw Dislocations—an imperfection equivalent to a partially sheared and slipped lattice, yielding a helical ramp, upon which spiral step growth becomes established. Frank's screw dislocations result from the more complex of the structured mismatches noted above in which a ramp forms. An unending step growth mechanism facilitates rapid growth.

C. *Plane Defects.*

1. Grain Boundaries—the interface between two continuous crystals of dissimilar orientation, yielding poor molecular fit. Grain boundaries are common in all clusters and also in a single crystal which has overgrown a small crystal adhering to its surface.

2. Coherent Boundaries—the interface between two crystals with a relatively good molecular fit, for example, twin crystals.

D. *Inclusions.*

1. Bubbles—cavities filled with gas or mother liquor. Gaseous bubbles from the mother liquor adhere to the crystal surface and become overgrown. As the practical environment at growth is in a vacuum, it would not seem that many air or vapor inclusions would form.

Liquid-filled bubbles occur frequently. Pits or corners provide the sites for overgrowth of a portion of mother liquor into a bubble-like cavity.

Delavier and Hirschmüller (74) report inclusions of solution and air in crystals growing from pure sucrose solutions. The numbers of inclusions were in accordance with the rate of crystallization and the solution conditions. It may be reasonably concluded that the bubble inclusions are not caused by impurities, but are the result of the cyclic effects of high heat transfer rates, irregular circulation, feed variances, and other boiling mechanics.

2. Fjords—deep canyon-like fissures. Repeated dissolution and regrowth have been demonstrated as the function of deep etches in the form of channels or grooves (203, 428) or fjords (280). These fissures seal over as the crystal reforms its former sharp outline.

Mackintosh and White have demonstrated considerable variance in etching and regrowth among crystals treated simultaneously in the same environment. It is suggested that the number of etchings is related to lattice defects. The defects appear to predominate at the edge of a crystal plane. The number of inclusions decreases with an increase in temperature.

3. Veils—thin layers of small occlusions. Veils of irregular orientation and shape have been observed to be the result of thermal stress cracks that form, fill with mother liquor, and seal over (281). Other veils are the result of area overgrowth. It has been suggested that a localized surface area becomes blocked for further growth. Growth rate decreases, and surrounding areas bridge over to seal off the "poisoned" area. Then growth rate is resumed.

4. Planar envelope—oriented veils parallel to a crystal face. The planar envelope is a series of veils formed by changes in growth environment. The outer surface of the crystal experiences a greater than normal occlusion situation on a cyclic basis. The veils form a series of envelopes representing the previous crystal surfaces, and thus form the oriented, parallel planes.

5. Clouds—clusters of fine, spherical droplets, probably the result of saturation variances.

6. Solids—small crystals or fragments, foreign particles. Nearly all samples of massecuite will have some large crystals with an occasional adhering small crystal. Depending upon the combination of the faces exposed to growth, the small crystal may become overgrown or may continue to grow rapidly enough to retain its individuality.

Turbid pan feed syrups invariably yield crystal crops with occluded insoluble matter. Screw dislocations can be formed by growth around an adsorbed foreign particle (374), Figure 12-43.

Again referring to the growth equation (1), the area factor can now be recognized as not just a simple macroscopic surface factor. Spirals, pits, and the specific face being considered are among the several complexing factors.

For practical purposes, line defects are the keys to crystal growth rate. Their presence greatly enhances growth but does not necessarily increase the population of inclusions. Further process technique improvements will undoubtedly reduce point, plane, and inclusion defects, and thus will reduce the impurity content. Growth rate could then be optimized at a higher level.

Impurity Effects. Coloring matter and other degradation products, nitrogenous substances, salts, turbidity, and colloidal material vary in their growth retardation effects. The molecuar fit into the lattice, similarity of portions of the molecular structure, the hydrogen bonding capability of some of the degradation fragments, the shifting of solubilities through phase relationships, and concentration

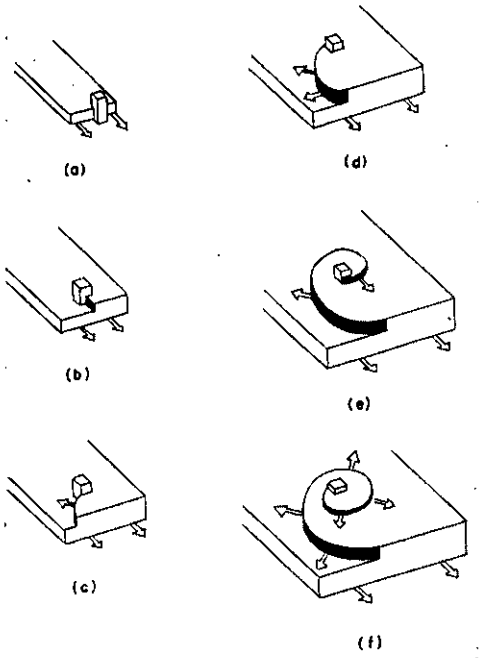

Figure 12-43. Inclusion of foreign particle in crystal growth layer and formation of a screw dislocation growth center. [After Tiller (374). *By courtesy of John Wiley and Sons.*]

effects are some of the retardation factors (46).

An approximation tool for closer correlation of impurity concentration and retardation has been provided by VanHook through application of the activity concept.[1]

Mak (208b, 208c) indicated the behavior of ionic impurities upon growth rates is currently related to the adsorption of those ions at the polarized hydroxyls exposed at the crystal face and on the surrounding sucrose molecules. Adsorption of the ions reduces the effectiveness of these growth sites for hydrogen bonding between sucrose molecules.

Present studies suggest that some of the differences in growth rates can be related to the ionic radius and the electro-negativity of the various ions.

Many impurities are occluded in the lattice structure by the overgrowth of material trapped in a pit or etching in the crystal's surface. Mackintosh and White (203) have studied the numbers of occlusions as related to alternate periods of dissolution and regrowth. As will be detailed later, such saturation changes are realized in some poorly circulating pans. Increasing temperatures yield comparatively fewer occlusions. Schliephake and co-workers (323) report that coloring matter and higher molecular substances are adsorbed to a lesser degree at the higher temperatures.

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[1]See refs. (250,389,391,396,399)

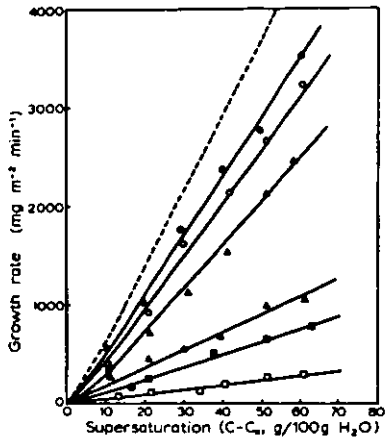

Figure 12-44. Effect of invert sugar on crystal growth rate. Circulation rate constant at 3,000 rpm (343a).

Pure sucrose
- ● 17 I/W
- ○ 34 I/W
- ▲ 47 I/W
- △ 101 I/W
- ■ 139 I/W
- □ 220 I/W

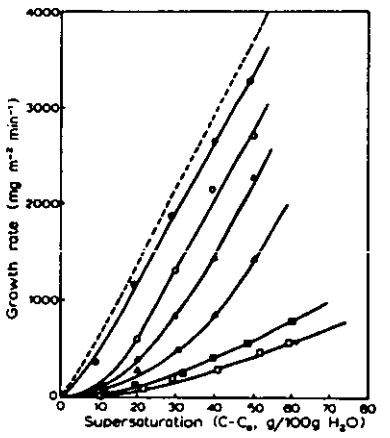

Figure 12-45. Effect of raffinose on crystal growth rate. Circulation constant at 3,000 rpm (343a).
- ● 0.5 I/W g/100g H_2O
- ○ 1.4 I/W g/100g H_2O
- ▲ 2.8 I/W g/100g H_2O
- △ 5.6 I/W g/100g H_2O
- ■ 23.0 I/W g/100g H_2O
- □ 34.3 I/W g/100g H_2O

Crystallization provides an elimination factor of 99.5 to 99.7% for most impurities (259). As impurities accumulate at the crystal surface, polar sucrose molecules tend to reject the nonpolar species. The higher attraction between polar molecules tends to squeeze out nonpolar intruders. Hydrogen bonding has a similar effect (145).

Ionized impurities can, to a degree, be considered as "swept away" from the surface by the clustering of water dipoles around the ions (253).

Verhaart and co-workers (411) demonstrated, Figure 12-46, that sodium, potassium, and lithium salts are excluded from the lattice and as being adsorbed.

The results of one series of tests are related to the amount of one ion (B) added at the start of the boiling of a crop of crystals while the sister ion (A) was added after crystallization and just prior to purging.

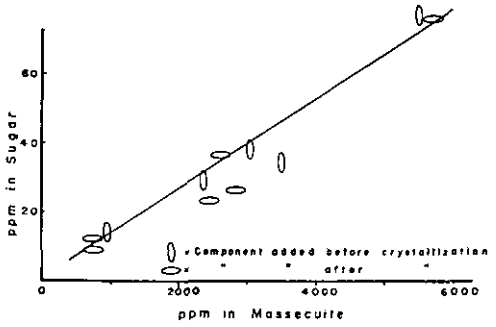

Figure 12-46. Impurity adsorption and rejection. *(Courtesy of CITS).*

Table 12-4

ppm in Mass.		ppm in Sugar		ppm in Sugar / ppm in Mass.	
K	Na	K	Na	K	Na
5710A	5530B	76	77	1.3%	1.3%
3500B	2420A	36	23	1.0	1.0
3030B	2610A	38	36	1.3	1.4
2840A	2395B	26	29	0.9	1.2
988B	685A	14	9	1.4	1.3
794A	689B	12	11	1.5	1.6

The residual impurity is a function of the amount of wash water used in the centrifugal and to an incomplete washing of the "back side" of the crystals, as related to the location of wash water sprays.

Delavier and Hirschmüller (74) have been unable to confirm a relationship between crystal size and ash content. They reemphasize that uneven solution environment yields uneven crystal composition.

As syrups increase in impurity, the solubility of sucrose is altered (Figure 12-9) and the family of curves of Figure 12-10 will shift up or down scale, depending upon the specific impurity. In low-grade boiling, the general influence of purity on the metastable zone is shown in Figure 12-47.

Some impurities are physically adsorbed on the different faces of the crystals in varying and selective amounts (116, 253, 400). A portion of the lattice sites becomes blocked, thereby reducing or retarding growth. Crystal habit modification becomes evident.

Frank's spiral growth centers show the effect of impurities through the observed shape of the spirals. Dunning (85), Figure 12-42, reports sucrose spirals of a "D" shape on the 100 face with the flat part of the "D" perpendicular to the 010. Square and triangular shapes have been observed as well.

Addition of 1.5% raffinose changed the D-shaped spirals to irregular shapes. He reports a restoration of the D shape after replacement of the raffinose solution with pure sucrose solution. A two-dimensional habit change is indicated. Dunning suggests that the impurities are relatively strongly adsorbed and trapped by the growth of the step. Glucose, fructose, galactose, maltose, lactose, and melibiose did not cause modification.

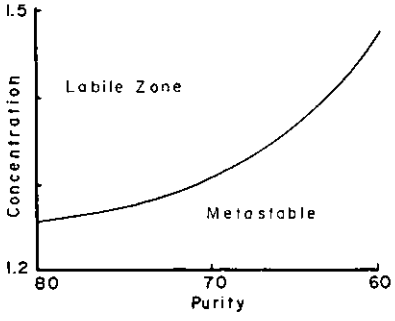

Figure 12-47. Influence of purity on limits of metastable zone.

Mantovani (210) and Smythe (343a) have illustrated sucrose molecular orientations in the crystal, the hydrogen bonds between molecules, the dipole nature of the molecules, and the variance in the exposed bonding sites for each face of the crystal. The varying steric fit necessary at the different surfaces becomes evident.

The dipole nature of the sucrose molecule is essential to the molecular orientation at the surface (340). The hydrogen bonds are the "glue."

Ion-exchange is most helpful in reducing the tonnage of impurities in the syrup and correspondingly reduces the sucrose loss in molasses. Ion-exchange will reduce the effect of ionized salts and coloring matter and organics carrying a charge, and thus will partially restore the crystallization rate. Those syrups containing raffinose and other selected sugar impurities will continue to be characterized with retardation and habit modification.

Dextran products of various molecular weights result from infections of *Leuconostoc*. Sutherland and Paton (366) have demonstrated a remarkable, needle-

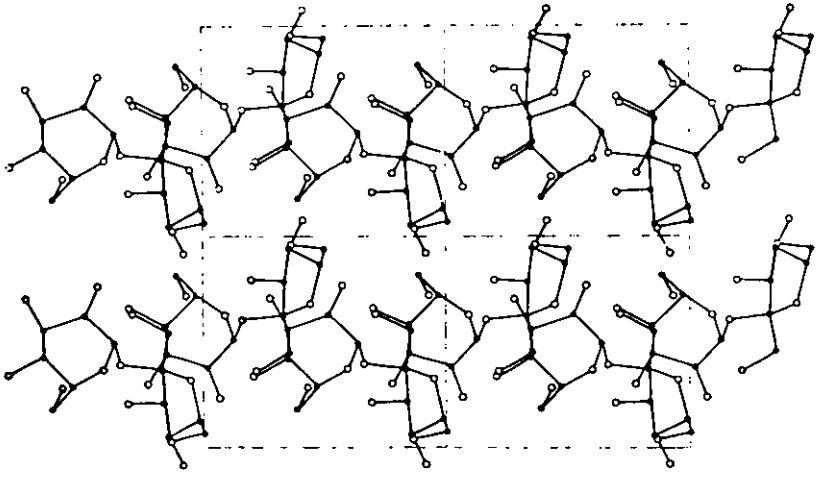

Figure 12-48. Variance in exposed sites of impurity adsorption or of sucrose-water bonding on the various surfaces of the crystal lattice. For clarity, only the carbon-oxygen molecular framework has been shown. The steric fit and probable variance in electronegativity of exposed sites becomes evident (210). *(Reproduced by permission CITS.)*

like elongation of the C axis of the crystal by the highly 1-6 linked dextran (see Figure 16-2, p. 546). The lower 1-4 and 1-3 linkages also yield refractory and slow-boiling syrups, but show little C axis elongation.

Vavrinecz (404) correlates a large number of mother liquor environments and illustrations of habit modification.

Variance in Individual Crystal Growth. Studies of particle size distribution of sugars crystallized under various conditions indicate conclusively that McCabe's ΔL law (13) has degrading interferences. Wright and White (439) and Schliephake and co-workers (323) have used distribution curves to illustrate such interferences, Figure 12-49.

Among the factors involved in the degradation of the particle-size distribution the following must be considered:

1. Variance of supersaturation across the face of any given face of a crystal. This will be dependent upon the embryo initiation and the population of structural defects originating at that time (47, 440).

2. Variance in the inhibiting effect of the several kinds of nonsucroses and the boiling temperature (323).

3. The concentration of impurities in the mother liquor and the delaying of their effect during the strike or crop growth. The use of activated carbon or macroreticular ion-exchangers will reduce some of the concentration and inhibiting effects. The feeding of impure syrup should be delayed through the use of segregated purities, if storage facilities permit, i.e., charge and feed as long as possible on a higher purity syrup and finish up with the lower purity syrup. Consistent with other crystallization factors, a loose boiling will further delay the impurity effect.

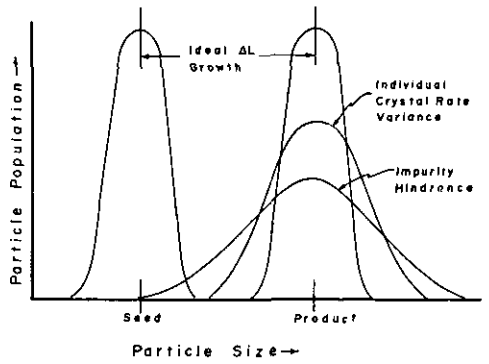

Figure 12-49. Degradation of particle size distributions. ***(Courtesy of CITS).***

4. The geometry of the massecuite space in the pan and the elimination of stagnant or nondirected flow conditions (286, 438).

5. Variance in supersaturation as massecuite flows along the pan's heating surfaces. Those crystals close to the heating surface become elevated in temperature and depressed in saturation until heat can be released (412). With plug-flow as an extreme condition, those crystals only a fraction of an inch away from the heating surface may experience little if any change in their saturation environment. Turbulent flow will lessen the saturation depression effects, Figure 12-50.

A high density feed syrup, a low temperature vapor boiling, and a heat input just sufficient to evaporate water in the feed only, will reduce the saturation depression effect at the transfer surface.

Forced agitation will reduce the hydrostatic effect on the boiling temperature at the submerged heating surfaces.[1]

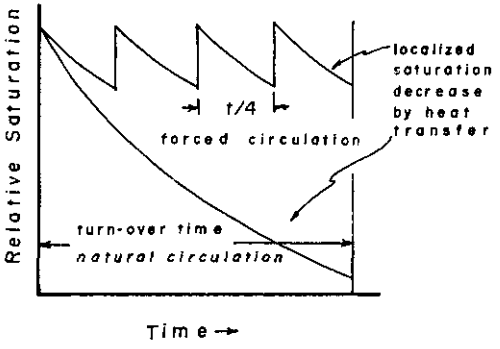

Figure 12-50. Saturation environment for crystal growth, natural circulation versus forced circulation.

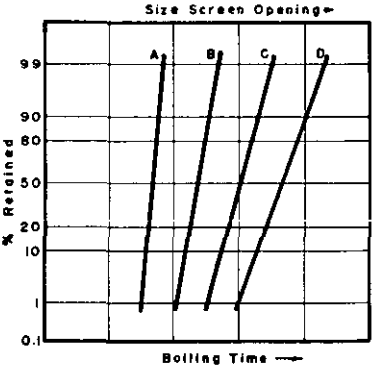

Figure 12-51. Gradual change in CSD with boiling time. Degree of change dependent upon: a) boiling time, b) quality of massecuite circulation past superheated heat transfer surfaces, and c) impurity inhibiting effect. *[After Orlowski and Schlophake (259a).]*

Batch Sugar Boiling

The Batch Process. Broken down into its elements, the batch sugar boiling process is relatively simple. Manipulation of just three valves is needed to achieve repeatable environments in each of the four phases of each strike. Consideration of the process in these simplest elements permits the trainee sugar boiler to accelerate considerably his learning and mastery of the technology. It also permits the logical application of measuring devices to sense and control the values existing in each of the four steps, thereby reducing art to basic physical chemistry and applied chemical engineering:

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[1]See Refs. (7,299,309,312)

1) *Introduce sufficient syrup to cover the heating element and concentrate to a* supersaturation suitable for a controlled nucleation; 2) initiate an embryo crystal population which will yield the desired final product size; 3) maintain a saturation environment which will yield an optimum crystal growth rate; 4) and, as the batch size is reached, continue to maintain a stable saturation and concentrate or "Brix-up" the strike to an optimum crystal yield.

Operation of auxiliary on-off valves in a relatively fixed sequence permits discharge of the batch of crystals, a cleaning of the pan, and a reestablishment of vacuum for the next cycle.

Absolute pressure. The uniformity of the vacuum in the pan is critical for uniform grain establishment and to avoid conglomeration in the early portions of the growth period. A sharp reduction in absolute pressure during the Brixing period can induce a secondary fine grain. Individual condensers and noncondensable pumps yield individual flexibility and reliable control.[1] A step change of set point of an absolute pressure control should yield the step response illustrated in Figure 12-52.

Although variance of the absolute pressure is one technique to control saturation, a uniform and constant absolute pressure is essential and is recommended for simplicity of saturation control, see Fig. 12-55$_1$.

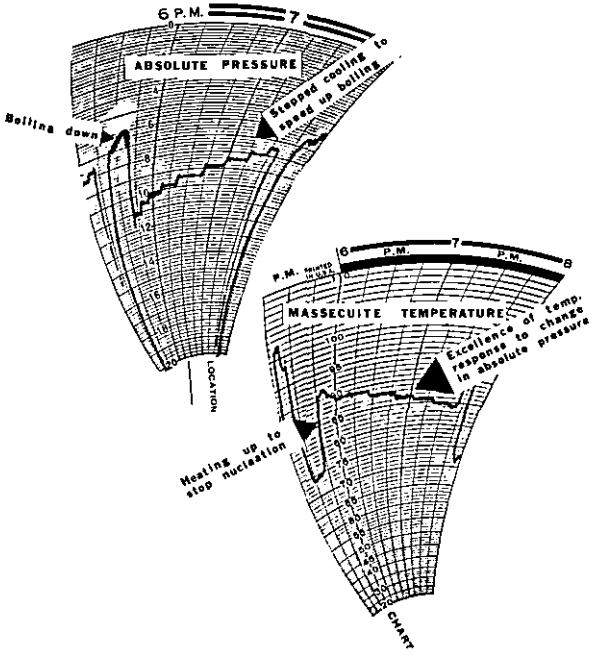

Figure 12-52. Excellence of temperature response to absolute pressure regulation in a circulated pan.

[1]See Refs. (69,70,176,225,246,308,344,357,424)

Graining Charge. A graining charge volume of 25% or less of the final strike volume is considered desirable for uniformity of crystal crop establishment. As the crystals at inception are of negligible volume as compared to the mother liquor, the space between the nuclei is great. In these spaces, fluctuations may continue to start additional embryos on their way as a second or even continuing crop. With larger initial volumes, the probability of stagnation volumes is greater and the parallel probability of conglomeration is also greater. The equipment design, of course, fixes the charge-to-strike volume ratio (71,124,231).

The elimination of centrifugal grain or foreign suspended matter is an obvious prerequisite to controllable nucleation and growth.

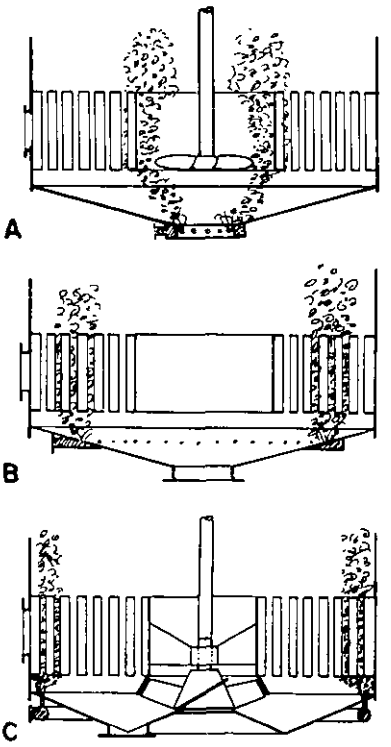

Figure 12-53. Effect of location of feed syrups into massecuite. To assure no residual centrifugal grain in feed, the temperature in the storage tanks *may be* higher than pan boiling temp.

A) any flash in this pan will be violently upward into center well and counter productive to agitation and may add to conglomeration.

B) flash under the calandria adds to the natural convective flow.

C) flash near outside of calandria provides extra circulation where most needed in space above calandria along side walls of massecuite belt.

Miers' Chart and Boiling Scheme. The Miers chart, Fig. 12-54, is useful in illustrating the temperature-supersaturation relationships during a boiling cycle. The change in absolute pressure in scheme A-B-C-D introduces saturation "bounce" if not performed well. Fig. 12-52 introduces a fluctuation with each step change in absolute pressure. C & H (129a) and others (11a,60) have used a characterized cam to effect a *smooth* temperature lowering, for example, cooling a low-raw boiling prior to dropping the massecuite into the cooling crystallizer system. This boiling pattern was typical of most "boil-to-grain" procedures in earlier years.

A constant pressure, full-seeded, forced circulation boiling would probably resemble the EFGH scheme. The excursion into the intermediate zone, point G, is typical of many full-seeding schemes (207a,370a), Fig. 12-54.

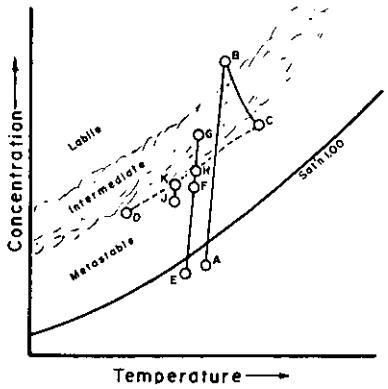

Figure 12-54. Batch boiling patterns.

1) *Boiling to Grain:* A) unsaturated feed concentrated to B), homogeneous nucleation, then vacuum reduction and large drink to reduce supersaturation to C), growth in metastable saturation with the optional increase in vacuum for "cool" pan drop at D.

2) *Shock Grain:* E) unsaturated feed evaporated to G, with seedling at F enroute to intermediate saturation, as sparklers show, return to metastable range at H by feed drink. A gradual small increase in bpr acceptable, due to impurity change. A fixed absolute pressure pattern.

3) *Forced Circulation Full Seeding:* Supersaturated charge J, full seeded at K, constant absolute pressure and minor increase in bpr due to purity lowering as in (2) above.

For a minimum heat requirement, the JK scheme would represent a presupersaturated feed and charge syrup, constant absolute pressure, and full-seeded, forced-circulation boiling. The system of Neilson and Chapman (254c) indeed starts with a preconcentrated charge at J in the metastable area, as noted. No steam is applied until after the seed starts to grow.

While there are many options in boiling patterns (221), the essence is: 1) a repeatable, controlled condition for graining each successive batch, 2) constant absolute pressure, and 3) a minimum fuel use: a high density feed syrup.

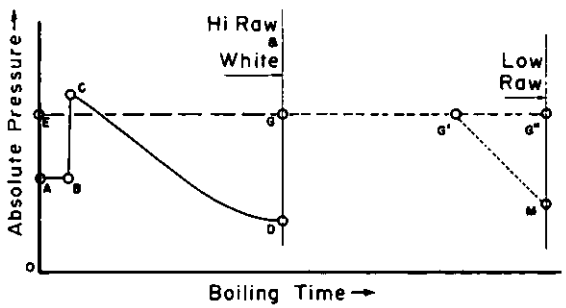

Figure 12-55. Absolute pressure patterns (correlate with Figure 12-54).

A) ABCD-typical of "boiling to grain" and some "shock grain" patterns. Pressure increase, B to C, heats the just-grained syrup, reduces supersaturation, and slows evaporation rate.

B) EG and EG^{II} constant absolute pressure, boiling temperature changes only as bpr changes. Typical of many "shock grain" and "full seeding" boilings.

C) EG^{I}M- early constant Absolute Pressure (82 - 87°C) for more rapid crystal growth in lower purity, followed by cam-generated AP reduction to reduce massecuite temperature before transfer to cooling crystallizers. This pattern suitable for reducing thermal load input to crystallizers having a temporary cooling inadequacy.

Seeding Environment. As charged liquors are normally unsaturated, evaporation must be initiated by steam admission to the heating surface. Each plant's steam system dynamics will determine the rate of steam or vapor turn-on. A fast start-up often causes entrainment in the system. Where steam economy is carefully practiced, a steam flow limiting device is often used. Calandria pressure can be used as a control value, but it is relatively insensitive to improper calandria venting and trap action. Steam flow is the more fundamental measurement.

Supersaturation must be established before seeding can be effective. In the absence of mechanical sensing devices, the sugar boiler watches the bubbles and splash on the sight glasses at the syrup level. At first, the bubbles rise rapidly to the surface and the liquor splash runs down rapidly and drains freely. With increasing viscosity (and indirectly, concentration) the bubbles will be elongated and will rise more slowly, the light reflections will become more brilliant, and the syrup splash will run down the glass more slowly with slow-draining tails.

Pan microscopes are of particular help in following the crystal initiation and growth sequences. Visual observation of particle density, the presence of conglomerates, the presence of abnormal habit and relative size at any instant is a positive aid to prompt corrective measures (191,452).

Crystal Growth. Seeding initiates an irrevocable growth period. Any irregularities developed at seeding remain throughout the rest of the growth cycle, unless detected quickly, washed out, and a new cycle is started. As this is time-consuming and destroys capacity, the conditions at seeding must all be correct.

The embryo crop is of very small surface area. The growth in pounds per minute is negligible. The crystals cannot accept sugar except as the surface area develops. Any attempt to push the process by means of a high evaporation rate will yield a mixed crystal crop (182,362,394).

"Setting the grain," "maturing," "hardening the grain," "breaking-in of grain," are all phrases descriptive of the art but are without foundation from a physical-chemical point of view. Single sucrose crystals are all of similar hardness. Clusters, when rubbed between the fingers, will shear at the planes of contact and will give a sensation of having been easily "crushed." In our economic haste, conglomerates may be formed. The artisan's descriptive phrases have been useful to give emphasis to the value of this time period for initial growth in which patience is mandatory (107,413).

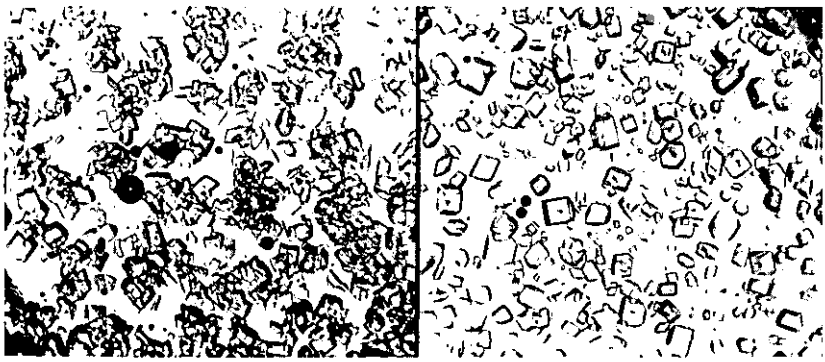

Figure 12-56. Conglomerate and individual grain.

Conglomeration. Rolled grain, spherulitic grain, ball grain, agglomerated grain, and conglomerated grain are terms describing an undesirable crystal formation, Figure 12-56 (left). In contrast, Figure 12-56 (right) illustrates individual, separate crystals. Both massecuites, however, suffer from a mixed grain problem.

Powers (286) draws attention to the "fluidized layer" of molecules on the crystal surface which may have a capability of cementing crystals together. The surface tension differences of the dilute "sleeve" may also have some initial holding or retaining effect, once collision has been made. Möller (244) notes a peak period of conglomeration. Initially, Brownian movement from molecular collision becomes less a factor and agglomeration probability increases. With still more growth, the crystal mass becomes more sensitive to mother liquor movement and the agglomeration probability decreases.

These theories suggest mechanisms. The common, practical conclusion is, however, that positive circulation will minimize the conglomeration effect.[1] Rodgers and Lewis (297) reported a CV improvement in strikes, i.e., 38 for noncirculated vs. 31 for circulated. Additional quantitative data of CV and percent conglomerates suggest that pan geometry has a decided influence on stagnation space. Other data (60) indicate the yield improvement of high-raw and low-raw boilings through circulation. Twin calandria pans were used. One was fitted with a circulator. Comparison of a drop in purity between the massecuite and the green syrups yielded the following averages:

Table 12-5

Massecuite	Purity Drop	
	Circulated	Noncirculated
Intermediate	11.5 (8)*	10.9 (11)
Raw	16.8 (3)	16.0 (8)

*No. strikes in test series.

The generalization is that conglomeration is inversely proportional to crystal velocity in the mother liquor, and is directly proportional to excessive supersaturation.

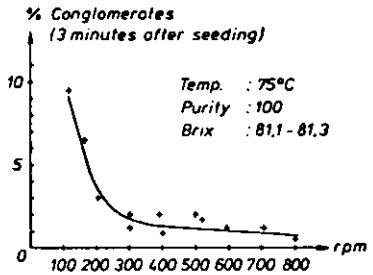

Figure 12-57. Conglomeration reduction by fluid movement. Laboratory tests of dispersion of seed crystals and relative fluid movement (190c). ***(Reproduced by permission Elsevier Scientific Publishing Co.)***

Knøvl and Møller (190c) recorded quantitative reductions in conglomeration at various impeller rpm. Other tests identified a critical time period (244) (crystal growth through the 50μ-120μ range) for conglomeration, Fig. 12-58. Abbott and Rich (1b) have shown significantly improved yields in white massecuites following installation of circulators and control systems.

Product Quality and Boiling Program. Some batch-boiling patterns of commercially available, automated and programmed systems are identified as mill-

[1]See refs. (60,127,198,287,353,386,388)

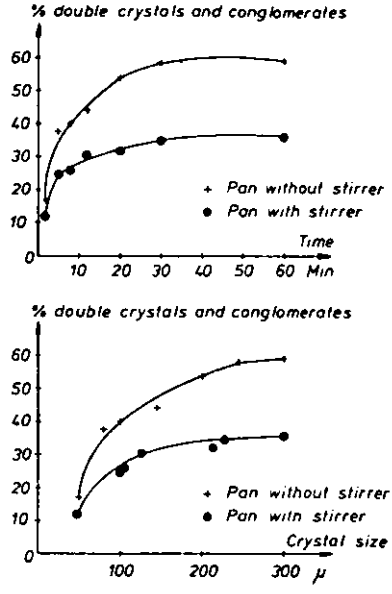

Figure 12-58. Conglomeration and intensity of circulation. Clustering and growing together starts almost immediately after milled seeding in syrups that are: a) high purity, b) high supersaturation (60). At lower purities, growing together starts at about 40-50μ. Growing together ends: a) about 120μ for *well*-circulated pans, and b) up to 250μ for some natural circulation pans (190c). *(Reproduced by permission Elsevier Scientific Pub. Co.)*

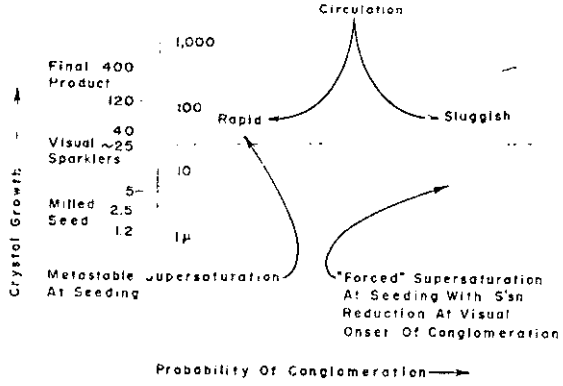

Figure 12-59. Critical crystal size range of conglomeration formation.

ed seeding procedures. The several methods of supersaturation estimation yield patterns similar to Fig. 12-60. Some possible conclusions might be: 1) natural circulation, high heat flux, high evaporation rate, high liquid superheat; thus an elevated saturation is needed to offset the localized undersaturation and seed melting, 2) super-fine grind of milled seed, requiring an elevated supersaturation to "baby-sit" the tiny fragments through their first, super-solubility period of growth, and 3) a deficient seed population introduced, requiring supplementary "shock seeding" (secondary nucleation) for final population adjustment.

The early-high supersaturation creates a condition of high viscosity and saturation during that short interval leading into conglomeration and twinning. Some systems use a "heating" or "thinning" approach for a return to metastable conditions. With higher purity massecuites, the conglomeration may have started

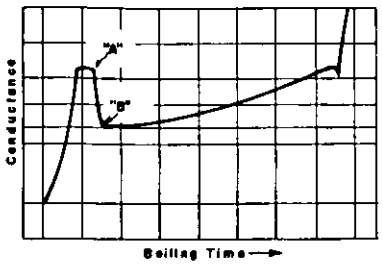

Figure 12-60. Typical boiling pattern for milled-seeding. Saturation "A" elevated for seeding followed by reduction at "B" to metastable range just prior to onset of accelerated conglomeration rate period. ***(After Madsen, et al., 207a.)***

even during the few seconds following seeding. A storage-and-color sensitive final product is created in such high purity seeding situations.

If the pans are so tightly scheduled to productive capacity that one cannot *take the time* for milled seeding (as defined on page 402), should we then consider positive circulation and metastable, coarse-milled seeding in a time-saving *supersaturated feed syrup?*

Mixed Grain. Figure 12-56 illustrates the effect of a continuous, heterogeneous seeding through neglect of elimination of centrifugal grain in the pan feed syrups. Excessive supersaturation, excessive circulator rpm, and vacuum fluctuations are also causative factors.

Refractory Syrups

With pans having a history of falling-off to a near zero circulating rate, injection of some low pressure vapor into the pan feed ring or into a perforated coil under the calandria will often restart the circulation and sustain it for the rest of the boiling. Injection should be near the outer periphery of the calandria. Any injection into or near the center well will be counterproductive.

The basic cause of the poor circulation should be determined and corrected: high lime salts, other syrup qualities, sealed heat transfer surfaces, poor venting, location of feed ring, etc.

Crystal-Slurry Fluidity and Mother-Liquor Viscosity. At seeding, the mother liquor structure is a guide to the supersaturation, Fig. 12-21. As the crystals increase in size and the space between crystals decreases, their influence upon fluid flow becomes measurable and controllable (124,425), Fig. 12-18.

The quality of fluid dynamics is of importance all through a boiling: 1) velocity and turbulence for heat transfer and diffusion rates, 2) product quality; conglomeration and occluded moisture or other impurities, and 3) stiffness as related to % crystal yield.

The terms "loose boiling" and "tight boiling" beg for a more quantitative definition. Varying degrees of confusion exist in the literature in the descriptions of boiling. The degree of agitation, the area/volume ratio of heat transfer surface, the pressure of the vapor used as heat source, the Brix of the feed syrup, and mother liquor purity are among the items in the compromise reached for the preferred fluidity-stiffness pattern for a given pan.

With respect to fuel economy, there is a middle path between heat transfer reduction due to early increased hydrostatic head and heat transfer reduction due to reduced fluid velocity.

With respect to crystal growth rate, a more fluid slurry delays the mother liquor purity drop. Also, a more fluid slurry enhances turbulence and a reduction of diffusion resistance.

With respect to yield, a final tightness must be achieved. A gradual transition appears preferable.[1] A function generator related to level, can program the optimum consistency pattern for any given pan (60).

An occasional sampling of massecuite during the boiling, for "% crystal" analysis will help quantify this parameter.

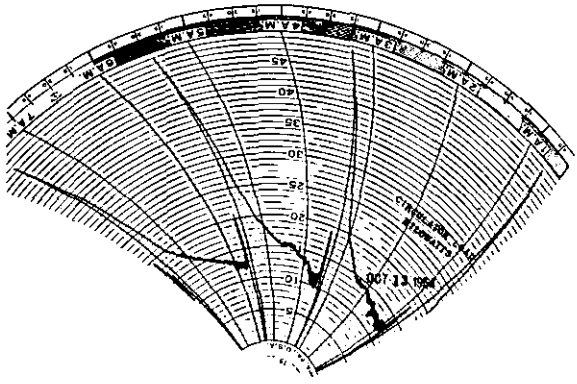

Figure 12-61. High-raw massecuite fluidity as measured by kw load of circulator: a) left, moderate fluidity during boiling, with level-actuated cam to program fluidity controller, b) center, hand-fed pan by a skilled sugar boiler, c) right, cam-controlled boiling with a deliberate tightness. All boiling with moderately high density pan feed and low, "starved" impeller suction at seeding. Test boilings 1954, Alvarado, California (60).

Massecuite Height at Pan Drop. Tippens (369b) compared massecuite height and daily pan productivity and found an optimum height to be 5½ to 6¾ ft. above the top tube sheet. Others (120,426) have found similar optimum heights when evaluated from a CSD spread (see p. 388 and Fig. 12-49) or bound-moisture, sugar quality approach.

The ebullition activity at the top of the massecuite is very intense in various areas. This rapid, everchanging rate of vapor release entrains considerable particulate lumps of massecuite. Much of this ejected and entrained material will fall back into the boiling massecuite if its velocity is reduced. That which does not lose velocity and is carried forward, has been an amazing B.O.D. source and a yield and extraction loss.

Most fabricated calandria pans have been provided with a generous massecuite belt height. As noted above, a pan has its time-average optimum-boiling height. The extra height is designed for entrainment disengagement. Unfortunately, it has been used to set super records of bags-per-strike. Also, unfortunately, such practice has its inevitable, but less visible, heavy economic penalties.

Highly visible markings on the face of the pan are recommended for "seeding" and for "end of strike."

Pan Discharge Steam-Out. Pan dischage simply involves shutting off steam,

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[1]See refs. (109,155,201,235,298,301,413).

breaking vacuum, and opening the massecuite discharge valve. However, seemingly minor technique often spells the difference between poor and excellent product quality.

Incomplete steam valve shut-off will caramelize massecuite. Delayed vacuum reduction permits additional flash evaporation and uncontrolled stiffness. Opening of the foot valve too early permits air slugs to entrain massecuite which is lost.

Pan cleanliness is essential to avoid uncontrolled nucleation at the start of the next strike and to avoid caramelized or burned deposits on the heat transfer surfaces. The foot valve must be well cleaned to avoid vacuum leaks during the next strike. The steam-out may be strictly a steam injection or it may include a water or liquor spray-down. The spray shortens the high temperature environment and reduces color formation. Such fluid should be returned to the melter or centrifugal receiving tank to avoid crystal solution in the mixer or mingler.

Vacuum break by injection of a low pressure vapor is becoming a common practice. Air vacuum break involves massive noncondensable removal to reestablish vacuum.

The Artisan Sugar Boiler. With only their powers of observation and their memories of the events of prior boilings, sugar boilers crystallized thousands of tons of sugar with only the open kettle. With the introduction of steam-heated vessels came the more sophisticated tools: a pressure gauge, a vacuum gauge, and a thermometer.

Our sugar boiler continued to rely upon his powers of observation of the string-proof, the splash-on-the-glass, and the appearance and feel of the crystal slurry on the proof glass. While there have been several generations of equipment improvements, several pan floors continue to depend totally upon the sugar boiler's power of observation and his sense of timing.

Hibbert and co-workers (143b) reported extensive data on sugar quality and the various factors affecting that quality. While many pan design features have a positive contribution on improved quality, these are reported as "marginal" as compared to the sugar boiler's influence. Process and instrument engineers have been simplifying the sugar boiler's duties to the extent that some are without the basic skills to keep the process moving if the little black box blows a transistor or suddenly is afflicted with a case of dirty air "influenza." Both take time for diagnosis and recovery.

Whether he be called sugar boiler, instrument man, process technician, system monitor, or shift supervisor, someone must have the basic skills of the artisan boiler, a) to fill in for an ailing system, b) to be aware when a system is ill, and c) to continuously perform the boiling skillfully in the absence of any control system.

Natural-Circulation, Batch Boiling. Good quality crystal crops have always been dependent upon a rapid circulation of the crystal slurry. Ebullition or the rapid formation and rise of vapor bubbles from the heat transfer surface stimulates the movement of the syrup upward. Upon release of the vapor bubbles at the syrup surface, the cooler syrup then flows downward through the center well and again starts an upward flow past the heating surfaces.

To establish a reasonable, natural flow, the boiling rate must be assured by a relatively low-Brix-syrup feed and a fairly high steam pressure (temperature).

To initiate a boiling cycle, the pan is steamed-out, closed-up, and the vacuum pump and condenser are started. As the vacuum begins to rise, the syrup charge valve is opened. The vacuum continues to rise. As the heating surface becomes bare-

ly covered, the charge valve is closed and the steam valve to the heating surface is opened in small, spaced increments until boiling has begun. The smaller feed valve is opened enough to keep the heating surface barely covered. The steam valve is left throttled or is gradually opened, according to the available steam, the process rate that is required, and the vacuum that has been established. This early, boil-down-to-grain period can be extended considerably if a smoothing of vapor draw from the evaporators is necessary.

For a given vacuum level, there will be a gradual rise in temperature of the syrup as it continues to concentrate. The syrup splash-on-the-bull's-eye glass becomes more brilliant in its light reflection and it begins to run down the glass more slowly. A sample of syrup pulled out with the proof stick and caught between the fingers will exhibit an increasing consistency or viscosity as the fingers, covered with syrup, are spread apart.

As the syrup becomes more concentrated, the evaporation rate will tend to slow down. The condenser water valve must be frequently adjusted to maintain the desired vacuum level, and the steam valve must be adjusted to assure full boiling rate as the graining time approaches.

With the constant vacuum condition, the boiling-syrup temperature, the splash-on-the-glass, and the string-proof length all become the signals to the sugar boiler to "full seed" the pan. The seed slurry (as described earlier) is introduced and is quickly followed by an isopropyl alcohol or other flushing agent, to assure a "full population" seeding.

Being aware of the superheated boiling temperature near the heating surfaces, due to ebullition, the seeding pattern of Fig. 12-28b is probably used by the sugar boiler to assure no loss or melting of the tiny seed fragments during the first short interval of growth.

Careful observation of the slowly rising syrup temperature, the change of the splash from a great brilliance to a faint but increasing opalescence or dullness, and the first evidence of tiny sparklers of reflected light on the proof-glass, all provide the several indications that a crystal population is indeed being established. In this "forced" condition, it is imperative that the sugar boiler respond uniformly to each successive boiling to assure a "full" but not excessive population.

A retreat to a metastable supersaturation can be achieved by a short, heavy drink of syrup. The temperature will fall a small amount, but the high natural circulation rate will not be slowed. The saturation condition should now compare with point F, Fig. 12-28b . Maintenance of full circulation at this time is of particular importance to minimize conglomeration.

By continued observation of the splash-on-the-glass and the rise of bubbles across the glass, the fluidity of the young massecuite can be followed. If the condenser water valve is adjusted to maintain a constant vacuum, the boiling temperature can continue to be a guide to avoid straying into the intermediate saturation region. It will, however, contain an increasing error due to hydrostatic head, as the massecuite level rises.

Shortly after introducing the large drink to reduce supersaturation, a slow feed is applied to maintain the metastable condition. The viscosity or consistency will slowly increase as the crystals grow in size. With natural circulation, the tendency will be to stay somewhat low in consistency to maintain rapid boiling. There is, however, a counterproductive aspect, a more rapid increase in hydrostatic head and a loss in effective temperature differential for heat transfer. That delayed loss in

evaporation rate fortunately comes later in the growth period when the probability of conglomeration is much less.

As the normal pan volume is approached, the feed rate can be reduced and finally shut off. The crystal slurry will become more viscous and stiff at an increasing rate. Increasing stiffness of proof-stick samples, the slow movement of bubbles across the glass, and finally, the appearance and then the increasing intensity of "flash" in the upper sight glasses, provide the guideline for the termination of the boiling.

Assuming a proper timing and scheduling of an empty receiver, the steam is quickly turned off, and the vacuum breaker valve is opened. As the vacuum drops to three or four inches, the foot valve can be opened and the massecuite can be discharged. After a short drain, steam-out of the foot valve is started. As soon as a clean, tight closure can be made, the foot valve is closed, and an adjacent, smaller valve to the corresponding melter or centrifugal syrup receiving tank is opened. A short, pan-feed syrup spray-down flushes the pan walls and heating surface rapidly. This is followed by a shortened steam-out and displacement of air in the pan. The steam-out valve is closed, the vacuum breaker valve is closed, the drain valve is closed, and the vacuum system is turned on, in preparation for the next boiling.

In this description of a natural circulation boiling procedure, specific values of temperature, pressure, and vacuum have been avoided. By repeated, hands-on boiling cycles, a sugar boiler is taught, and quickly learns, the more satisfactory pattern for *his particular set of pans.* He also becomes aware of the emergency responses necessary for steam, feed, or vacuum failure.

Forced Circulation, Batch Boiling. The pan, in this instance, is fitted with a circulator, absolute pressure controller, a supersaturation by boiling-point-rise indicator, a center well mounted, resistance thermometer, and a pan microscope. The circulator impeller is of large diameter, of good hydraulic design, and is responsive to major changes in mother liquor viscosity as well as to % crystals.

As the pan is charged with syrup, the circulator is turned on just prior to its covering the calandria. As the circulated juice begins to spill over the calandria and into the center well, the steam can be slowly turned on.

Where a 20 to 25 psi first evaporator vapor may have been necessary with the natural circulation boiling, a third or even fourth vapor (3 to 5 psig) can be used for the circulated pan boil-down with a high density pan charge and feed (68 to 72° rds).

With the reduced evaporation rate resulting from the high density feed syrup, the last "window" of the entrainment separator is reduced in open area to create a higher vapor velocity past the measuring elements of the boiling-point-rise thermisters.

The absolute pressure controller is set for a fixed value to yield the desired syrup boiling temperature at the time of seeding, and is simultaneously low enough to set up a temperature difference for an adequate evaporation rate with the lower steam vapor pressure.

As the boiling point rise indicates an approach to point H, Fig. 12-28, the steam flow rate is reduced to zero[1], the center well temperature is noted, and the pan is full seeded. Very shortly, observation through the pan microscope assures one that crystals are growing. The excellent forced circulation keeps the crystals in a

[1]See refs. (182,295,305,421).

very turbulent movement, thus excluding nearly all conglomeration.[1] With the steam shut off, the possibility of drifting into the intermediate supersaturation zone is circumvented, and the absence of ebullition superheating reduces any seed dissolution by localized momentary undersaturation.

As the center well thermometer indicates the first fall-off of supersaturation, the steam is slowly turned on over a period of 20 to 25 minutes. At first the circulating charge level is maintained and then slowly increased. As the evaporation rate resumes, the bpr measurement will again be effective. With the rise in crystal slurry level, the center well thermometer slowly begins to exhibit the normal hydrostatic-head measurement error. This measurement has fulfilled its critical period need.

It takes time for crystals to grow! If quality crystal crops are the objective, forcing with a borderline, secondary supersaturation is not the answer. A steady, assured metastable supersaturation (bpr) is the guide for increasing the steam rate. As bpr tends to fall, the evaporation rate can be increased. The pan feed rate can be increased as the crystal surface area increases, and as the consistency or stiffness of the crystal slurry tends to rise too rapidly.

After a few cycles of experience, the sugar boiler neatly balances steam flow rate with time needed to achieve the produce crystal size, and with a "full" pan. Feed is shut off, and the bpr is carefully watched. If it tends to rise, the steam rate *must* be throttled accordingly.

The circulator motor load becomes the indicator of final massecuite consistency for good crystal yield and for *proper* centrifugal loading and separation. Steam is turned off, the vacuum breaker is opened, and the strike is dropped in the usual manner.

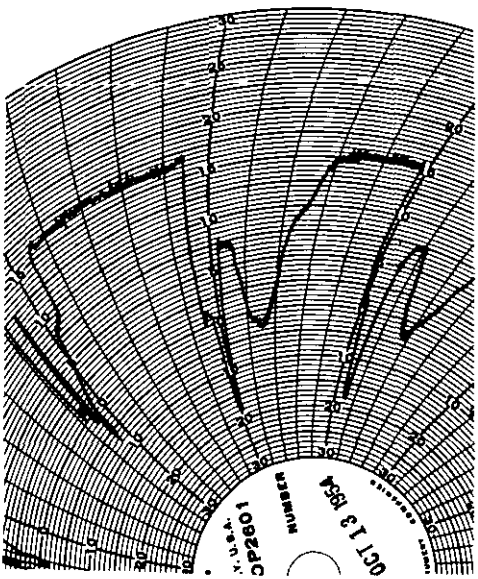

Figure 12-62. Calandria steam pressure: a) left, full pressure for total boiling, b) right, steam only as needed. Difference in area under the two pressure curves suggests unnecessary steam use. Test data, Alvarado, California (1954) (60).

[1]See refs. (60,198,353,386,388).

With these several support features, our sugar boiler produces a more uniform quality crystal crop with a remarkably lower heat requirement and without pulling a "proof," except to maintain his "touch."

Low-Brix pan feed and high-Brix pan feed require two different boiling philosophies. Both have their place, but only one leads to reduced fuel costs.

Batch Crystallizing Equipment

Heat Transfer. Fabricated steel construction and its greater design flexibility has fostered many heating element arrangements, Figure 12-63. In contrast to the coil design, the calandria heating surface is all available at the start of the cycle. The ratio of surface area to final massecuite volume varies from 1.2 to 2.0 with most designs in the 1.6 to 1.8 range. The higher ratios permit use of lower vapor pressures and increasing economy.

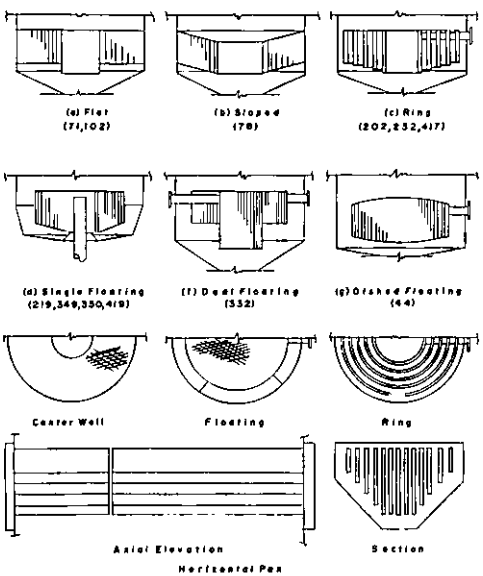

Figure 12-63. Calandria design modules.

Sight glasses installed in the top of various pans permit observation of boiling patterns at various massecuite levels. With the calandria just covered, heat release patterns from the calandria may be studied. The regions of lower activity can often be related to the accumulation of noncondensables and steam velocities past the tubes.

Circumferential distribution belts admitting vapor uniformly around the calandria are typical of modern pans (71,78,102). Various baffling and tube arrangement schemes are in use to reduce noncondensable blanketing.[1]

Massecuite Belt. The height of massecuite above the calandria is generally limited to 4.5 to 5 ft to assure reasonably good circulation (120). To further aid pan capacity, the massecuite belt diameter (Figure 12-64) has been enlarged by 2 to 5 ft.

[1]See refs. (78,118,226,420)

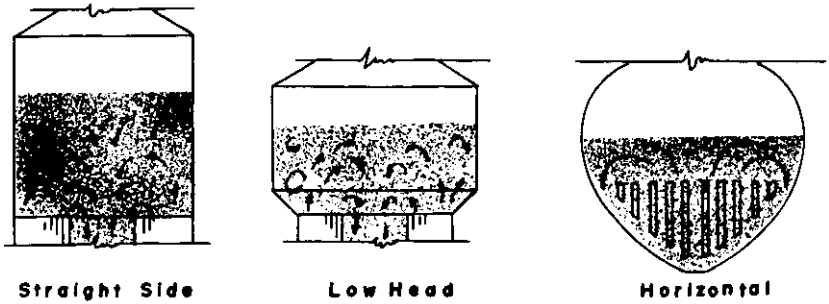

Figure 12-64. Massecuite belt designs.

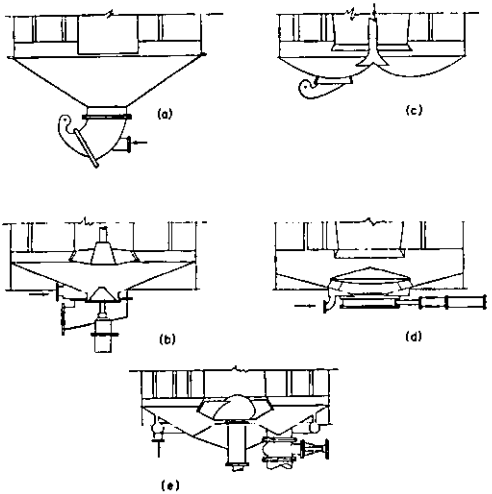

Figure 12-65. Pan bottom, feed and foot valve design modules: a) Steep cone bottom, single central feed entry, flapper-type foot valve, b) Low angle cone bottom, hydraulically operated cone foot valve, circular feed ring at valve, c) "Saucer bottom," contoured plug-type swing-out valve, center feed distributor, d) Low angle cone bottom, piston-operated thin wafer valve, annular ring feed under deflector, e) Fabricated double cone bottom, standard gate valve foot valve, pipe ring feed distributor.

Several studies have demonstrated partial stagnation by means of an isotope tracer, internal moisture, and other techniques (436). Forced circulation reduces the degree of stagnation and reduces the dependence upon a low strike height (120,230). Internal baffling helps in reducing stagnation in some circulation patterns (152). In others, it detracts from the product quality (437).

Pan Bottom. As the influence of circulation, conglomeration, and crystal size became known, pan designs were improved to achieve a saucer or similarly streamlined bottom (229,417). Hydraulics were given consideration to have enough cross-sectional area for unrestricted massecuite flow to the underside of the calandria. All excess space was eliminated to obtain a minimum ratio of seeding volume to final strike volume.

Foot valves of the flapper design are giving way to hydraulically operated vertical disc-and-seat assemblies, or to thin gate-type valves. The valve operators are more compatible with automatic cycle controls (45,232,315). (See Figure 12-65.)

Feed to the pan is now introduced in multiple small nozzles in the pan bottom, well under the calandria. Another feed arrangement includes a single baffled inlet nozzle in the center of the pan well (5). In circulated pans, feed is often introduced into the feed well on the impeller suction side, and in others it feeds into the lower

side of the hollow impeller hub. In both instances, a well-mixed injection is sought to minimize variances.

Impellers. Pumping of high volumes of viscous crystalline slurries at a minimum energy expenditure has undergone intensive study.[1] Among the various high-volume impellers are axial flow and mixed flow. The number of vanes, vane solidity, vane angle and curvature, the configuration of the impeller hub and the casing or shroud all have a large influence on the impeller performance.

At the normal saturation levels in batch pans, the impeller rpm and tip speeds do not appear to contribute to collision or attrition nucleation. In one instance, the rpm was increased with improved pan performance.

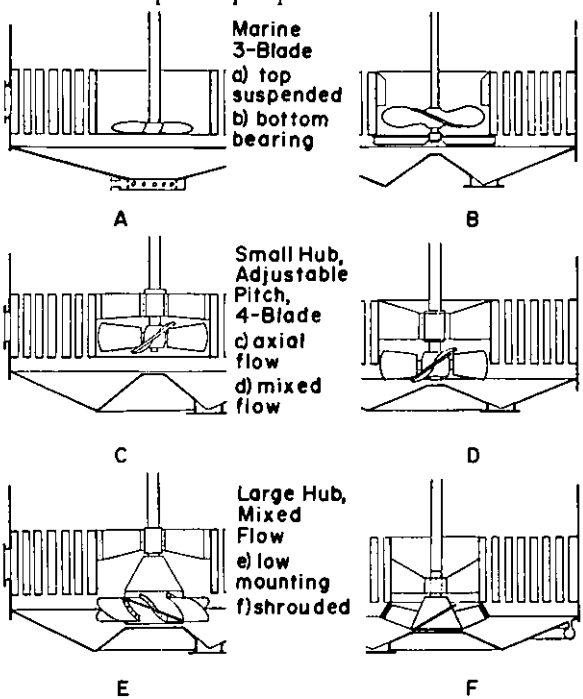

Figure 12-66. Impeller Systems. *[After Abbott and Rich (1b).]*

Abbott and Rich (1b) compared the performance of several circulator system designs, first on the basis of water circulation patterns at the graining charge level, and second, by crystallization yield improvements during successive years. Lower calandria steam pressures were realized with the forced circulation heat transfer improvement. The configuration of the pan bottom and impeller location was found to be of particular importance in crystal yield improvement in some installations. Similar improvements in heat transfer and product quality were reported (11b) for a characterized ring-calandria circulator system.

A number of circulator systems are top-of-pan supported only, i.e., no lower steady bearing. To accommodate the long overhung shaft and its inherent flexure, the ratio of impeller outside diameter to shroud inside diameter may be as low as

[1]See refs. (120,151,152,254,298,354,415).

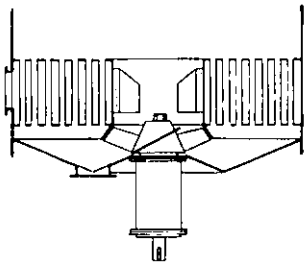

Figure 12-67. Bottom drive agitator. Shaft-mounted speed reducer (not shown) would be mounted on lower shaft extension.

.75. Circulators with a lower bearing are designed for a ratio of .95-.98, and reflect a considerably improved hydraulic efficiency (354). Petri (270a,270c) has reported the need of high fluid velocity past the heat exchange surfaces if minimum spread of CSD and minimum conglomeration are to be achieved. A low ratio impeller may provide excellent mixing within the center well, but may not necessarily provide good massecuite movement, heat transfer, and conglomerate reduction.

Bottom-drive agitators are of simple design and construction. A good hydraulic efficiency can be achieved and shaft-mounted reducers permit drive flexibility. Although a shaft seal for syrup is required, the design avoids the top shaft seal with its air and seal-water leakage. Both top seal deficiencies can upset the quality of bpr measurements.

An external axial-flow pump is suitable for lower viscosity systems, Fig. 12-73. A 5 to 7 ft/sec flow rate at the heating surfaces of the standard shell-and-tube heat exchanger provides improved heat exchange rates and minimum liquid superheating.

Some of the present, transition, batch-converted-to-continuous systems utilize the batch circulator systems (270a,270b). As new systems are designed, and if product quality and fuel economics are indeed of concern, each of the multistages will be fitted for positive, forced circulation.

Where superior sugar quality, optimum yields, and the ultimate in steam economy are desired, a calandria pan designed to accommodate forced circulation offers the more satisfactory arrangement (155). Of the two calandrias suitable for forced circulation, the flow through calandria tubes is more uniform than past the various elements of a ring or ribbon calandria (150).

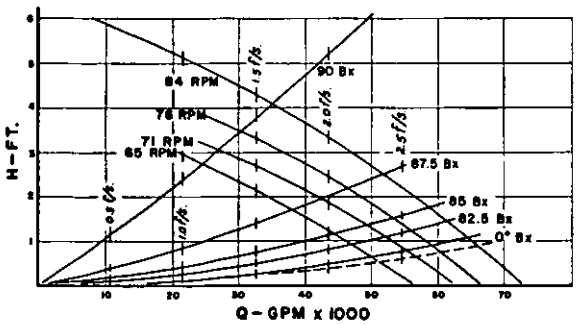

Figure 12-68. Q-H System head curve for pan-impeller system design. H vs Q for 2¼" tubes and H vs Q for impeller speeds in the design of one alternative system, as reported by Cole (59a). *(Reproduced by permission ASSBT.)*

Other Circulation Aids. Vapor injection into the massecuite below the calandria has been used to a limited extent for many years (120,416). Recent new equipment uses the venting from the calandria steam chest into a distribution ring under the calandria (71). The horizontal pan uses the same vent vapors to augment natural circulation of the plate heating elements (300). Massecuite circulation rate measurements have included the benefit of such vapor injection (71).

Continuous Sugar Boiling

With but two exceptions, sugar processing is a completely continuous process; the white centrifugals and sugar boiling. Both are under increasingly intensive study. Continuous boiling systems have been repeatedly studied in all parts of the world.[1] The highly mixed crystal size has been the major retarding factor.[2] The problem is not unique to sugar but has been solved in an acceptable manner in the general chemical industries.[3]

Continuous crystallization has several aspects of interest and economic advantage:

1. Pan storage liquor feed tanks need not be large. Storage time at elevated temperatures and the accompanying losses from sucrose destruction are reduced.

2. Second or third vapor requirements can be expected to be as uniform and continuous as the rate of transfer of thick juice into the sugar end.

3. Surge-type mixer-mingler equipment is unnecessary. The uniform consistency effluent from the continuous equipment feeds directly to the centrifugals. Mixer wash-up and steam-out losses are eliminated.

4. Centrifugal operation is on a continuous, never-ending mass flow status. Sugar drying, sugar reheating and blending, molasses disposition, and all other sugar end activities enjoy the benefits of smooth flow rates and energy demands.

5. The system is amenable to unattended, centralized, and computerized on-line control.

6. A sugar end utilizing continuous boiling for all grades can use "plug-in units" to match the physical equipment to the impurity load of the season.

Single-Tank, Mixed-Slurry, Mixed-Product-Removal Pan (MSMPR). Due to continuous seeding or nucleation, a single vessel, continuous crystallizing pan is characterized by a wide variance of crystal sizes, Fig. 12-69. Randolph (292a) indicates that from a plot of population density and crystal size, Fig. 12-70, the nucleation and growth rate in such a system can be identified. The crystal size distribution data can be related to particle numbers, length, area, and weight, Fig. 12-71. The influence of retention time on crystal size is illustrated in Fig. 12-72. Many models have been proposed and tested in pursuit of a better understanding of the continuous systems.

Earlier trials of a Swenson FC crystallizer and an external centrifugal classifier were reported by Petri and Bennett (270). The massecuite was held to 10-30% crystals and at a supersaturation level of 1.65 to 1.85 (prob. Classen relation). Secondary nucleation initiated the seed. Although the crystal-growth rate was higher than usual, the entire crystal growth was at the final, reduced purity and at a considerably higher viscosity.

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[1]See refs. (11,63,76,77,87,117,190,274,364,381)
[2]See refs. (41,50,61,97,148,177,204,205,209,214,215,270,296,313,363,390,440)
[3]See refs. (64,159,194,241,311,325,346,390,439)

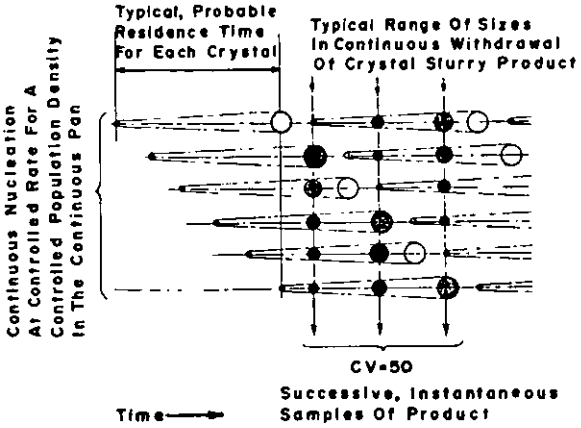


Figure 12-69. Idealized product size from a continuously seeding, MSMPR crystallizing pan. McCabe's ΔL Law is assumed.

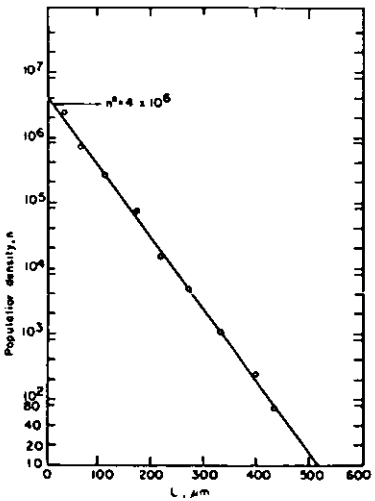


Figure 12-70. Population density from MSMPR crystallizer. From this type plot, both nucleation and growth rates can be determined (292a). *(Reproduced by permission Academic Press.)*

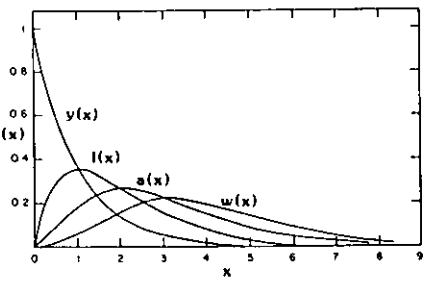


Figure 12-71. Typical distribution functions of an MSMPR crystallizer, in terms of number (y), length (l), area (a), and mass (w) (292a). *(Reproduced by permission Academic Press.)*

A continuous centrifugal, with a 400-micron opening screen, was used for product separation — a coarse sugar, i.e., 80-85% on 16-, 20-, and 30-mesh screens. The mother liquor was partly recycled to the pan.

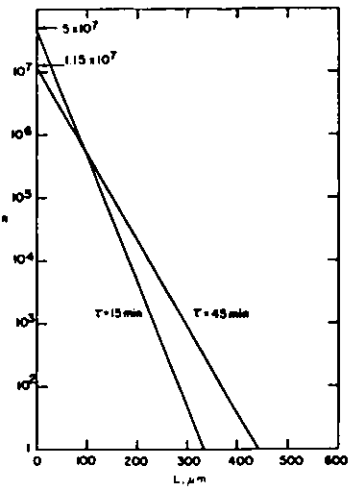


Figure 12-72. Effect of holding time (T) on crystal size distribution (292a). *(Reproduced by permission Academic Press.)*

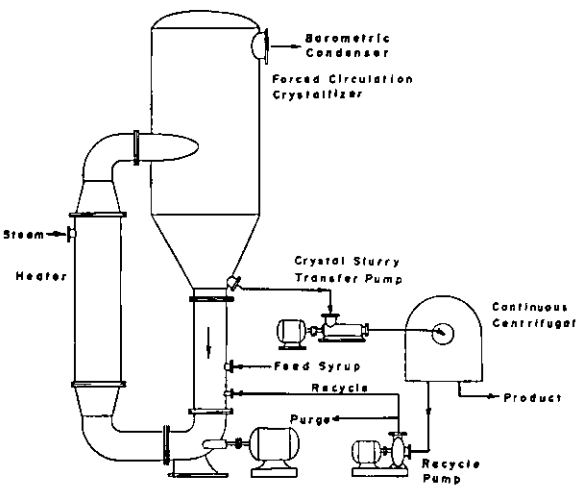


Figure 12-73. Single-tank, continuous, forced-flow crystallizer with solids classification. *[After Petri and Bennett (270). By courtesy of Sugar Industry Technologies, Inc.]*

Randolph (292a) illustrates product-size classification in Fig. 12-74. The entire mixture of sizes enters the centrifugal classifier with selected sizes (in shaded area right of L_p) leaving the system, with the mother liquor and smaller crystals being recycled. A portion of the mother liquor must be removed for purity control.

Petri (270a) describes a three-stage white sugar crystallization system in which: a) a continuous Randolph salt-out seed generator produces a seed of 60-100μ, b) a Swenson FC crystallizer that enlarges that seed to about 200μ in a pulsed bed manner, c) a seed mixer-receiver, and d) a set of batch, forced circulation calandria pans, converted to continuous operation. This system yields a white massecuite purgeable in Krauss-Maffei pusher-type centrifugals.

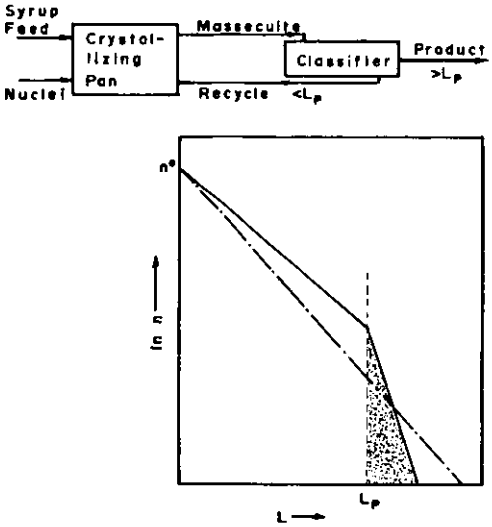


Figure 12-74. Classified product removal with undersize recycle in MSMPR crystallizer.[1] Idealized population distribution plot, a) normal unclassified and b) classified product removal. Product size is L_p and larger. ***[After Randolph and Larson (292a).]***

Of the recognized methods of crystallization, De Vries (76,77) employed the self-evaporation system for continuous flow (71a). A high purity syrup is concentrated to saturation at 105 °C. The hot syrup is seeded and introduced into the bottom of an inclined crystallizer, the upper end being under high vacuum. Crystals are scrolled to the upper end and removed through a barometric leg, Fig. 12-75. As the mother liquor flows up the inclined crystallizer, the hydrostatic head decreases and the equilibrium vapor pressure (temperature) decreases. The solubility decreases and crystal growth is maintained.

The improved system utilizes a dual, inclined cascade for improved product size. The initial 105 °C helps to "pack" a relatively large amount of sucrose into solution at the start of the crystallization. This is known as the "SWS" or "Dinteloord" system.

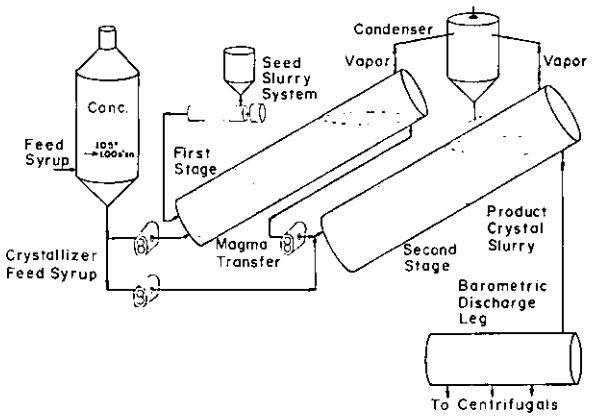


Figure 12-75. Self-evaporation crystallization, the SWS (Dinteloord) system. ***[After De Bruyn (71a).]*** [1]

[1] See refs. (148,154,158,194,314,325)

Multiple-Tank, Back-Mixed, Continuous-Flow Evaporative Crystallizing Pans

In the 1936-1957 period, De Vries (76) developed a 10-compartment, horizontal system for his self-evaporation system. Each compartment was under a successively lower vacuum.

Fives-Lille has successfully divided a horizontal pan into 4 to 7 compartments to improve the crystal size distribution of the crystals. A number of units are in use on different beet and cane products. For simplicity of control, these systems operate at constant and uniform vacuum in each section.

The 4-compartment, magma-seed enlarger is the simplest system, Fig. 12-76. A low-grade boiling is centrifuged with the crystals being mixed with a higher purity syrup and is continuously fed to the continuous pan. Crystal size is enlarged as an intermediate grade boiling. Smooth material flows and steam use are inherent. Crystal size (MA) is set by the rate of magma-seed addition. Fewer seed will relate to a longer residence time.

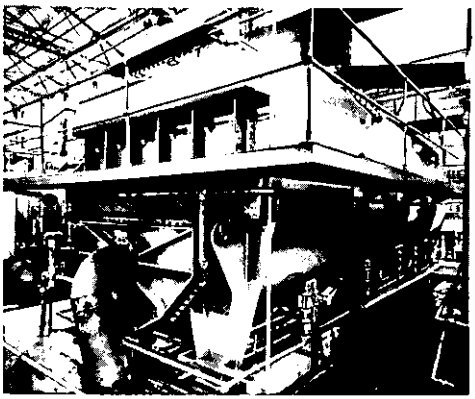


Figure 12-76. Magma-type continuous pan, typical for low grade beet and cane massecuites. *(Reproduced courtesy Fives-Cail Babcock.)*

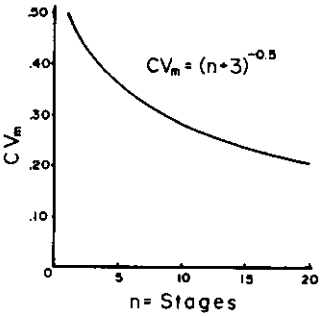


Figure 12-77. CV in multi-compartment crystallizer. Refinement of CV with added stages in series. Nucleation in first stage only (292b).

Where the CV quality factor is of greater importance, the 7-compartment pans are being used (267). Fig. 12-77 illustrates the value of additional compartments on the refinement of the CV, based on mass.

In contrast to the dynamic, ever-changing conditions of the batch pan, the continuous pans are static, or nearly so, in each of the control parameters: level, supersaturation in selected compartments, final consistency, feed syrup saturation, syrup and steam flows, etc. (15c,449b). Feed syrup is continuously proportioned to each of the several compartments according to steam flow. Bpr (Holven method)

regulates feed to the first one or two compartments. Gamma ray adsorption regulates last compartment consistency in several installations.

The constantly present driving force for crystal growth tends to generate a fouling condition on the pan walls and other surfaces (116a,132a,140a,430a). Various wall-construction materials have been tested: ordinary steel with well-ground welded joints, polished stainless, brass, Teflon, etc.

Some units have been jacketed around the boiling liquid for heating of these surfaces by the noncondensables being vented. Upper, vapor-space surfaces have been periodically cooled to induce a flushing, dissolving condensation. Other surface cleaning has been related to the inorganic scaling character of the feed syrups. Other pans are designed with rotary sprays for feed syrup introduction.

Crystallization by Cooling

Sucrose molecules, as molecules, possess dipole and hydrogen-bonding fields of force that are very selective in a mixture of sucrose-solvent-impurity molecules. Sucrose molecules associate easily with other sucrose molecules. As noted earlier, the alcoholic hydroxyl-alcoholic hydroxyl is reported to be a stronger hydrogen bonding than adsorbed ionic attachments or the poor steric-fitted bonding of other impurities. One might anticipate that as sucrose molecules orient and move together, the lesser bonds are broken. Associating sucrose molecules become sucrose crystals and leave behind the impurities — the nonsucroses and the water. Sucrose crystallization is an impurity-rejection mechanism.

As successive crystal crops are grown, the rejected impurities begin to dictate the chemical and structural nature of the residual syrups. For example, the machine syrup from the high-raw boiling is of higher viscosity. Nucleation in this less-pure "molecular soup," is somewhat inhibited. Crystal growth, which is simply a two-dimensional extension of heterogeneous nucleation, is also restrained. The important fact is that while the rate is slowed, it has not stopped.

At one time it was believed that in a practical sense, syrups with purities less than 76% could not be nucleated. Now, with full seeding, nucleation is being performed in the 60's. Some operators shorten their boiling time by seeding with a magma. Growth starts almost immediately. Other operators "cut" strikes, i.e., drop only half the strike, then resume boiling with the remaining massecuite as seed. Each technique has its advantage, and each has its *set of problems.*

As sugar recovery is the essence of good raw-side performance, the formation of surface area for growth is basic, followed by the effective use of that same surface area, and finally, a careful separation of that surface area from the exhausted mother liquor.

As was developed earlier in the section on properties of crystal slurries, the major portion of recoverable sugar in high-raw-machine syrups can be crystallized in three-to-five hours. The % crystals, plus the somewhat elevated viscosity of the mother liquor, generate a fluidity or stiffness limitation to further crystallization-by-evaporation. As this boiling reaches this temporary limit, its temperature is in the 70 to 85 °C range. This is the temperature of the saturated mother liquor. The usual end-product temperature for storage or shipping is 35 to 50 °C. Reference to Table 1a suggests a rather remarkable amount of sucrose, that is recoverable, simply by temperature reduction.

A controlled rate of cooling generates a degree of supersaturation which will continue to induce crystal growth. The % crystals developed in crystallization-by-evaporation can thus be extended beyond the circulation limits, characteristic of the boiling vessel.

The boiling time for a low-raw massecuite may be 1.5 to 2 times that of a high raw and will depend upon the feed syrup purity, the kinds of impurities present, the size of the seed crystals, supersaturation, and upon excellence of circulation.

At one time, long-boiling times were believed to assure good crystal growth. Often the boiling time was considerably extended by "boiling on water." In view of today's fuel costs, this technique no longer seems valid.

With the tools available for today's boiler, the plan of "forced-circulation, batch boiling," described earlier, is modified for low raw, only by having even greater patience, by using different calibrations on the supersaturation instrument, and for the consistency measurement.

Supersaturation maintained just below the intermediate zone will establish the concentration driving force.

With a high rds pan feed, there will be a reduction in the evaporation required, a reduction in the syrup superheating at the heat transfer surface, and a reduction in the spread in CSD. Fewer small crystals will be passing through the centrifugal screen to reduce % crystal yield and to increase molasses purity.

The assured, forced circulation circumvents the need for water feed. Steam is applied only to sustain the bpr or supersaturation. Feed is added as needed to hold the crystal slurry viscosity in a reasonable range through the first tightening and on up to the final tightening, for a positive high N/W ratio.

A narrow CSD crystal crop leaving the vacuum pan becomes a narrow CSD crystal seed for the cooling crystallizers. A high N/W reduces the "W" in the final mother liquor—the principal carrier of sucrose out of the system.

The high Brix feed and "steam-only-as-needed-to-maintain-bpr," are parts of reducing total pan steam requirement to the 14.5% steam on beets noted by Zagrodski (449a) and toward the tantalizing 11.0% of Christodoulou (57a).

The Cooling Cycle. The fast-cool cycle of Fig. 12-78 has been estimated on a Miers' chart, Fig. 12-79. In the preceding pan boiling, the supersaturation of the mother liquor was carefully controlled in a well-circulated boiling environment.

The supersaturation was maintained in the upper metastable range, just short of secondary seeding in the intermediate zone. With excellence of circulation and

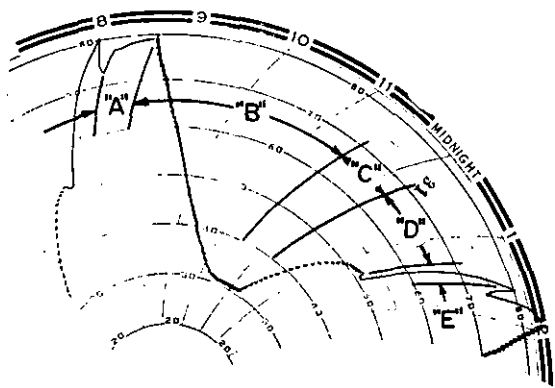


Figure 12-78. Typical fast-cooling cycle in Lafeuille crystallizer: a) crystal crop transfer from boiling pan to crystallizer, b) massecuite cooling, 17.8 °C/hr, c) cold dwell period, in this instance stretched to 50 min., d) reheat, 19.9 °C/hr, e) drain to centrifugal mixer and steam-out of Lafeuille crystallizer.

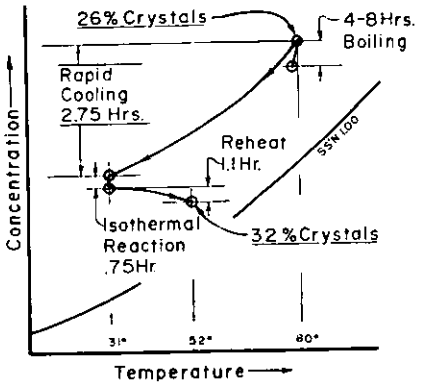


Figure 12-79. Miers' diagram of rapid cooling as in Figure 12-78. An early, high saturation is generated for rapid growth. With an extended isothermal crystallizing time (Period "C," Figure 12-78), the final saturation after reheating would be more nearly 1.00 with an improved % crystal yield.

with watchful persistence in maintaining an aggressive supersaturation, and an 80° boiling temperature for accelerated growth surface reaction, the growth rate was optimized.

Within a relatively short period, the fixed crystal population assumes a volume equal to the working capacity of the boiling vessel. Various crystallization formulas identify the fact that a high crystal yield is the result of a high pan-drop rds (264). In pursuit of this objective, further water was evaporated and eliminated until the crystals became deficient in lubrication. Only enough fluidity remained to permit the crystal slurry to drain from the boiling vessel in a 15 to 20 minute period.

The temperature chart shows a rapid cooling of the massecuite for a temperature shift in solubility and hopefully a significant increase in crystal growth (263). As the temperature was decreased, the mother liquor supersaturation was sustained and crystal growth continued[1]. As long as the temperature change showed a smooth transition, the massecuite continued to be fluid.

The Lafeuille temperature chart exhibits an excellent cooling pattern. The chart *does not* show a simultaneous, continuous addition of lubrication fluid: water. Without the lubrication of the mass, it would show temperature aberrations from nonflow of the massecuite.

Once water is started, crystallization is "capped" at that "just fluid," % crystal level. Any crystallization effort by additional cooling or by extended time is mostly wasted effort.

The Silina relation suggests that for a limiting consistency, there will be a corresponding limit to the % crystals tolerable by the equipment (Fig. 12-80). In a growth environment, (supersaturated), the % crystals will tend to increase. To prevent mechanical destruction, the % crystals can be held constant by providing more mother liquor. Water added will dissolve sugar until the new solution is of the same saturation as the other mother liquor[2]. The new volume of massecuite is restricted to its tolerable % crystals — all at the expense of much of the added crystal yield (as % on original massecuite).

It becomes evident that it matters little whether the crystallizer is in a fast-cool mode or is of the 1 ° C/hr configuration. The potential crystal yield in both systems is grossly inhibited once water is added as lubrication.

[1]See refs. (126,265,351,352,409)

[2]See refs. (36,109,191,208,239,268)

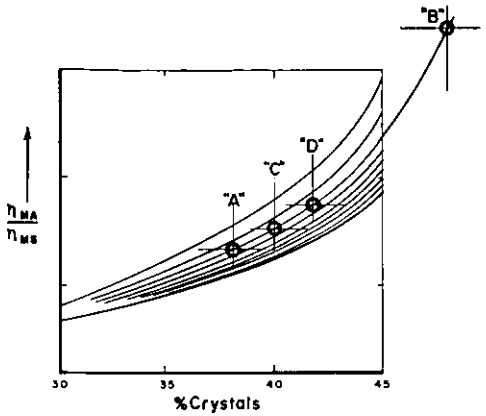


Figure 12-80. Massecuite consistency and relative % crystals yield:
A) Massecuite from evaporation pan dropped at low rds (low N/W).
B) Massecuite dropped at high rds (high N/W)—potential yield if no physical constraints are imposed.
C) % crystal yield limited by mechanical deficiencies of crystallizer.
D) Improved % crystal yield by ruggedness of crystallizer equipment.

Where the economic values of sucrose in the bag and as sucrose in molasses are not too dissimilar, molasses exhaustion is of little consequence and water dilution becomes the inexpensive insurance for equipment protection. Where there is a significant difference between dry sugar and wet sugar values, the economics quickly justify a correspondingly significant effort in molasses exhaustion.

Analysis of good crystal yield based only upon temperature and solubility, suggests the need for a high rds at pan drop which, in turn, is a high N/W ratio. A comparison of massecuite N/W with molasses N/W (should theoretically be the same) would suggest a molasses rds in the 87 to +89 range. The usual molasses rds is significantly lower than this expected value.

Two plants in close proximity and with similar purification systems are shown in Fig. 12-81. One plant with a 5-hour pan boiling plus a 7-hour Lafeuille cycle, achieved a 36% crystal yield. The second plant with a 14-hour campaign average pan boiling and a 47-hour slow cooling crystallization, also yielded a 36% crystal massecuite.

Assuming a positive, economic desire for molasses exhaustion, a conclusion drawn from this plant performance example is that it matters less about how much crystallizing time is recorded, but it matters greatly about how the first few hours are utilized. A properly operated fast-early-cool system will provide good exhaustion and a considerably reduced volume in process. Control of the % crystals becomes the key problem.

Critical inspection of various sets of plant operating data, yields similar patterns of exhaustion compromise due to less-than-rugged equipment and to crystal "capping."

Water Dilution. Using the 36% crystal yield of Fig. 12-82 for illustration, the use of dilution water might be represented by Fig. 12-83. An equipment or procedural limitation thwarts the attainment of the potential crystal yield. Depending upon the mother liquor temperature, rds and purity, 3.5 to 5 parts sucrose are dissolved per part dilution water added Table 12-1; Fig. 12-9.

Additional lubricating mother liquor is created and thus restricts the % crystals to 36%. Of the potentially recoverable crystals through temperature (saturation) reduction, only 17 to 22% is recovered using water dilution procedures. The rest is used to saturate the added water to form lubricant.[1]

[1]See refs. (135,289,327,339,423,429)

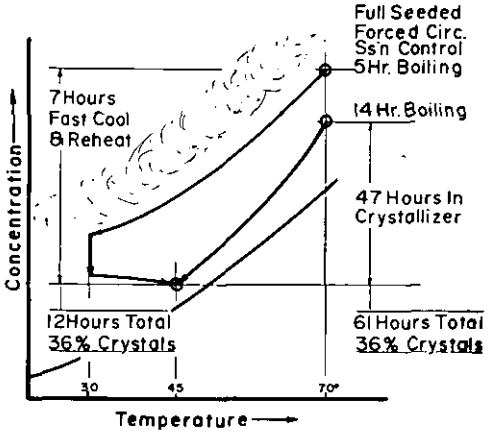


Figure 12-81. Mother liquor exhaustion is not dependent upon *long* hours of boiling and cooling. Exhaustion is highly dependent upon effectiveness of use of each hour that is available. A 12-hour system and a 61-hour system both yielded 36% crystal crops.

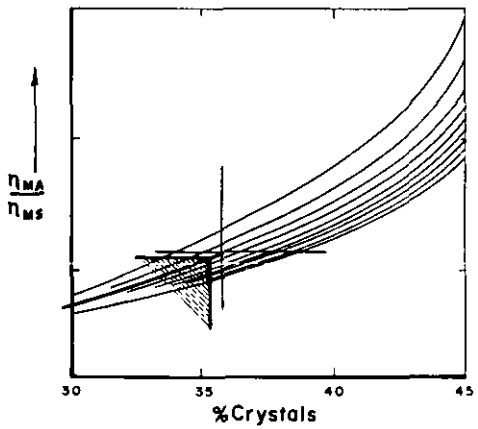


Figure 12-82. Limited cooling cycle. The nature of the impurities in the massecuite mother liquor unfortunately may create a higher than normal mother liquor viscosity. For an equal, final massecuite consistency, the % crystals must be reduced. For an equal flowability through the centrifugal screen, the N/W of the massecuite (and consequently the N/W of the mother liquor) must be restricted. A foreshortened cooling cycle to 55-50°C and immediate centrifugal separation may accommodate the high mother liquor viscosity limit.

Mother Liquor Viscosity. Viscosity is one of the two factors in the Silina ratio: a massecuite consistency expression. In addition, mother liquor viscosity, all too often, is the ultimate limitation of the low-raw process (264a). The usual Poiseuille laws of the flow of fluids through porous media apply to centrifugal screens as well as to vacuum and pressure filtration.

Very viscous mother liquors, typical of low purity molasses systems (45-50 purity), may drain from the crystal surfaces and flow through the screen openings at uneconomically slow rates. Equation 22 identifies the desirability of a large surface area for sucrose exhaustion. As per Table 12-3, this is best achieved with small crystals.

This table also identifies the value of larger crystal sizes for improved film drainage from the crystal surface and the attainment of a more satisfactory crystal

[1]See refs. (135,289,327,339,423,429)

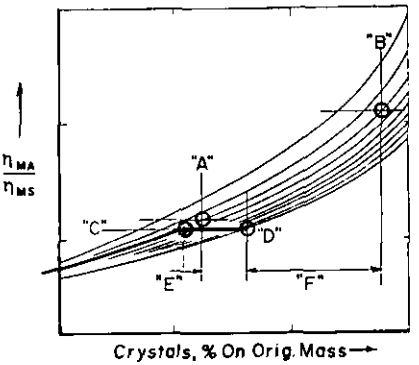


Figure 12-83. Water dilution limit of yield.
A) physical limits of equipment.
B) potential yield if no physical limits were imposed.
C) water added to prevent exceeding equipment and consistency limits.
D) % crystals at end of cooling period.
E) probable yield increase.
F) potential yield used to provide lubrication.

purity after separation. Conversely, a higher viscosity can be processed for an equivalent crystal purity at a larger crystal size.

The Poiseuille equation for definition of viscosity and fluid flow is:

$$\eta = \frac{\pi (P_1 - P_2)\, r^4 t}{8 V \ell}$$

η	=	viscosity in poises
r	=	radius of opening through which the fluid must flow
ℓ	=	length of tube or hole through which the fluid must flow
$P_1 - P_2$	=	pressure drop across the opening or capillary tube
V	=	volume of fluid passing through the opening or capillary
t	=	time required for flow of volume V through the opening through which the liquid must flow

For relatively small changes in "r," the increase in viscosity that can be handled at equivalent flow, pressure drop, etc., is of interest for improved exhaustion (Table 12-6).

Where flow is through an opening other than a round orifice, the irregular orifice can be related to the conventional opening through the *"hydraulic radius"* R_H (267a). Tapered holes have been incorporated into good screen design. This has the effect of changing the length of the flow path -"ℓ" in the denominator of the flow equation. Both factors significantly aid the flow of molasses through the screen openings.

Possibly, for an overall improvement in exhaustion, a small compromise is desirable — a moderate increase in crystal MA, a determined effort to achieve a small CV, and a corresponding increase in screen perforation, all to accomodate a higher mother liquor viscosity and a much improved massecuite N/W.

With whichever screen perforation selected, it is essential that the crystal size corresponding to the lower edge of the CSD does not extend down into the size of the perforation. The ideal molasses does not contain fine crystals, Fig. 12-84.

Precentrifuging. Another of the means of modifying the Silina ratio to avoid excessive bulk viscosity of the massecuite is precentrifuging. While the mass is still hot, a portion (about 25%) is diverted to continuous centrifugals. Crystals are removed and the mother liquor is returned to the crystallizer. By selecting the

Table 12-6. Incremental change in capillary radius, r, and relative, permissable change in viscosity at constant flow rate.

r	r^4
1.0	1.0
1.1	1.46
1.2	2.07
1.3	2.86
1.4	3.84
1.5	5.06
1.6	6.55
1.7	8.35
1.8	10.5
1.9	13.0
2.0	16.0

Permissible viscosity, relative to screen hole radius = 1.0, is proportional to r^4.

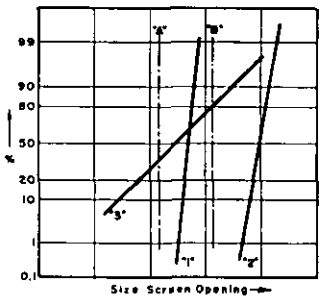


Figure 12-84. Centrifugal screen perforation and size of crystal crop to be separated: a) CSD (crystal size distribution) for normal screen perforation, "A;" b) CSD required for larger perforation, "B;" c) CSD with high CV and increased probability of a high purity molasses.

amount of crystal removal[1], the recombined slurry will be suitably lubricated to allow continued crystal growth without mechanical damage.

Some sugar-end systems must handle elevated low-raw pan feed syrup purities. The crystal volume developed becomes excessive for even the boiling pan and seriously excessive for the crystallizers. By precentrifuging, an adjustment of the % crystals (by their partial removal) permits continued room for crystal growth and recovery as the mother liquor temperature is reduced to induce further growth.

Fig. 12-85 illustrates precentrifuging with respect to the crystal slurry. Genotelle (117c) and McGinnis (224c) report an expression suitable for computer modeling for optimizing the amount of massecuite withdrawal. Several schemes for mass withdrawal and for mother liquor return are discussed by McGinnis.

Due to the economics of an added process stage and centrifugal equipment, use of the system (224d) is associated with high purity sugar ends and with Quentin syrups. In most instances, some other means of % crystal control is utilized.

Massecuite Lubrication. Genotelle suggests that a 38-40% crystal massecuite is optimum. A well-boiled massecuite, handled properly in the cooling crystallizers,

[1]See refs. (117a,117b,224c,248a)

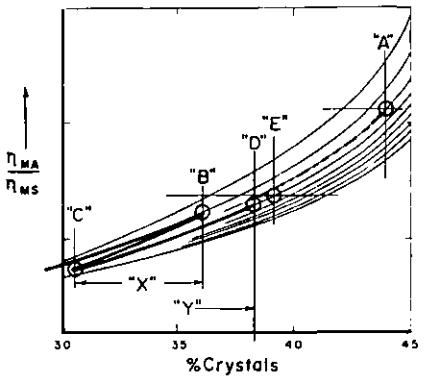


Figure 12-85. Improved % crystal yield by precentrifuging:

A) potential % crystal yield created.

B) crystal content early in crystallizer while warm and suitably fluid for centrifugal separation—part of mass diverted to precentrifugals to remove "X" component of crystals.

C) mother liquor returned and mixed with rest of mass for a net crystal content identified by "C".

D) remaining crystals continue to grow to condition "D"—just short of crystallizer structural limits at "E." Total crystals recovered = x + y which approximates that of "A," but which was not directly attainable.

can yield 48% crystals and more. The mechanical aspects of such a slurry become limiting, Fig. 12-80, and illustrate an exponential increase in bulk viscosity.

If a saturated mother liquor corresponding to that already in the massecuite is added, the % crystals can be controlled at a level that is not mechanically limiting. The crystals, % on original pan-drop-massecuite volume, will continue to become larger. By continuous lubricant addition, the massecuite consistency is stabilized at a value for safe equipment operation and for sustained "high level" exhaustion growth.[1]

One crystallizer set, in particular (224e), measures the motor load on each crystallizer in the series, Fig. 12-86. Repeated small doses of moderately heated, saturated molasses are added *only as needed* to hold the consistency at the acceptable but high levels. Straight house (non-Quentin) molasses purities have been gradually worked down from 62 to 53 averages. Concurrent with this has been the reduction of low-raw-massecuite purities from 78 to 68 averages. It must be noted that natural alkalinity of the diffusion juice is relatively low and no process soda ash is used. On-going testing suggests 46 as a viable interim purity objective.

With the same set of crystallizers, the weekly averages on molasses product purity have followed, to a remarkable degree, the changes in maturity and the tissue quality of the beets sliced. An observation of limited scope suggests that in the quest for lower purities, mother liquor viscosities may become more sensitive to the kind of impurities present, than previously. Impurities become an increasing, solution-structure determining factor.

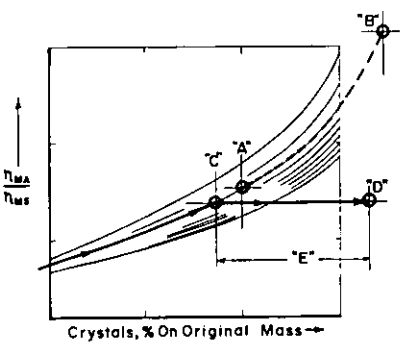


Figure 12-86. Improved exhaustion of massecuite by molasses lubrication:

A) physical limits of crystallizer.

B) potential % crystal yield developed in massecuite prior to pan drop.

C) consistency level for controlled molasses dilution at optimum high crystal surface area for mother liquor exhaustion.

D) probably actual crystal yield without mechanical overloading of crystallizer.

E) % crystals remains constant in the present, modified massecuite but % crystals, *based upon original massecuite from the pan,* increases a remarkable amount.

[1]See refs. (137,197,284,429).

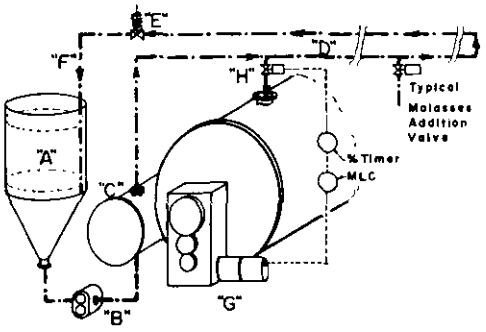


Figure 12-87. Molasses lubrication system.

A) molasses, nondiluted, tank filled on daily basis.

B) low volume gear pump.

C) shell and tube heat exchanger with vapor heating. Temp. control not shown.

D) heated molasses header.

E) 15 psi back pressure valve.

F) submerged return.

G) individual motor drive on each crystallizer.

H) snap-action, full-diameter molasses dosing valve operated by motor load limit relay and series-connected %-timer.

Cooling crystallizers have been constructed with varying degrees of mechanical ruggedness. Those designed for deliberate structural strength have paved the way for more complete exhaustion of the residual, impure mother liquor.[1]

Many crystallizer stations which were previously adequate may have been overextended and need supplemental equipment. Wear and obsolescence or structurally lightweight design may dictate a new system. Horizontal crystallizers have been and will continue to be used. Excellent exhaustion can be attained.

With a premium on the square-foot cost of floor space for enclosed equipment, the trend toward vertical crystallizers is to be expected. Some of these systems have been installed outside the factory building, Figures 12-93 and 12-95. Excellence of thermal insulation becomes an obvious requirement. While the basic objective is to cool the massecuite, uneven one-sided atmospheric cooling may easily overwhelm the mixer shaft and arms, induce a secondary graining, plug a massecuite transfer line, or rupture a water line.

Some vertical designs include a cone bottom with a lower skirt. The skirt is extended enough to provide a protected space for pumps and for some piping and valves within its confines.

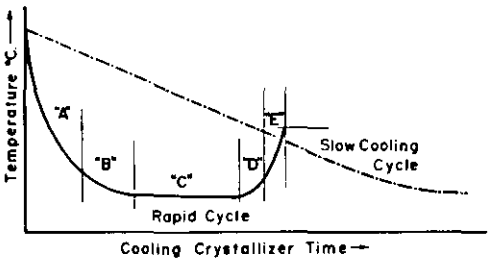


Figure 12-88. Cooling and reheat equipment design for rapid cooling cycle and reasonable exhaustion.

A) Rapid, high intensity mixing while surface reaction rate is at moderate rate. Rapid cooling, short of secondary nucleation. Molasses dilution as needed to avoid excess mechanical stress. (Small diameter mixer-cooler, high surface area per unit volume mass.)

B) Intermediate rpm, intermediate intensity mixing as surface reaction slows and starts domination of growth rate. With molasses addition.

C) Slow rpm, conventional size and rpm, crystallizer units (low surface area per unit volume mass). Molasses addition to each unit as needed. Economical retention time 6-12 hours.

D) First stage of reheat, intermediate reheat Δ t, intermediate rpm (viscosity high) small diameter, high surface area per unit volume massecuite. Molasses addition only as needed.

E) Last stage of reheat, high intensity mixing, rapid rpm to safely use higher reheat Δ t, small diameter unit.

[1]See refs. (25,68,90,126,237,327).

A number of vertical installations consist of a group of four units: three for cooling and one for reheating. Through the years, several horizontal, batch units have been joined together to form a series of 6 or more units.

The observation has been made that the larger number of smaller units permit a *closer tuning* of the equipment *to the process dynamics* — the rate of cooling, the more frequent adjustment of limiting % crystals, the more selective use of elevated slurry mixing rates before surface kinetics become dominant, a shorter, more intense reheating, and, in general, a more effective exhaustion potential.

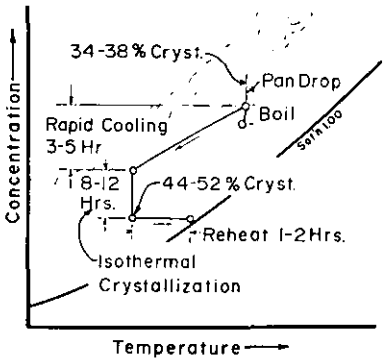


Figure 12-89. Rapid cool cycle with cold dwell and molasses lubrication. Correlated with Fig. 12-88.

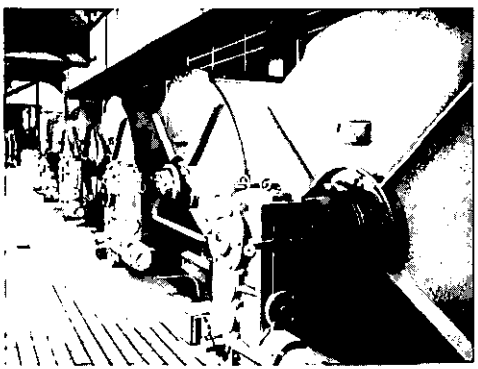


Figure 12-90. Battery of horizontal, cooling crystallizers. Motor loads of drives can be used for consistency measurement and regulation. *(Courtesy, BMA.)*

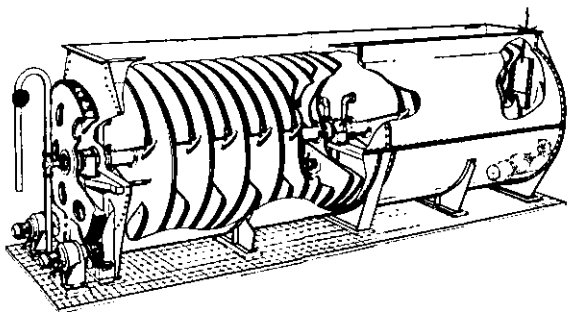


Figure 12-91. Rotating cooling elements in horizontal crystallizers, typical of many units in service. It is to be noted that some more recent designs use stationary cooling elements along with rotating, non-cooling elements for massecuite mixing. *(Reproduced with permission of Stork-Werkspoor Sugar (138a).)*

In close examination of crystal growth-reaction kinetics with actual mother-liquor temperature, one becomes aware that some rules-of-thumb may be conservatively safe. With the introduction of Lafeuille crystallizers, it became evident that rapid, intensive cooling could be achieved without secondary grain formation. Un-

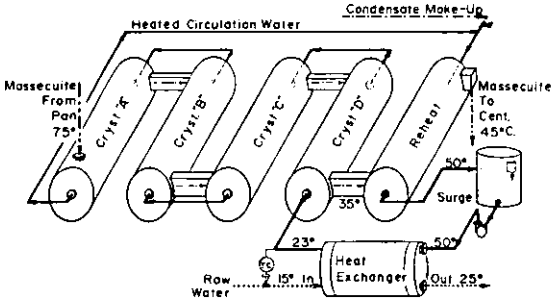


Figure 12-92. Circulating water cooling and reheating system. Closed system for corrosion protection of heat exchange surfaces.

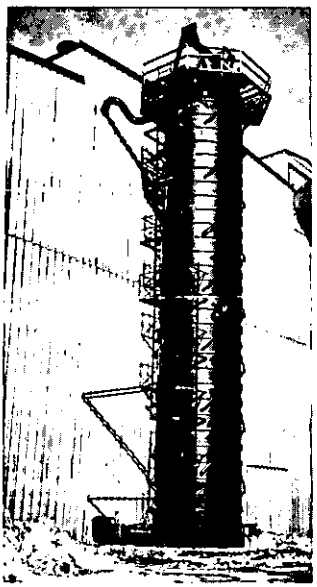


Figure 12-93. Vertical crystallizer in outside installation prior to insulation of shell. Interconnection of water boxes at successive levels. *(Courtesy, Silver Engineering Works.)*

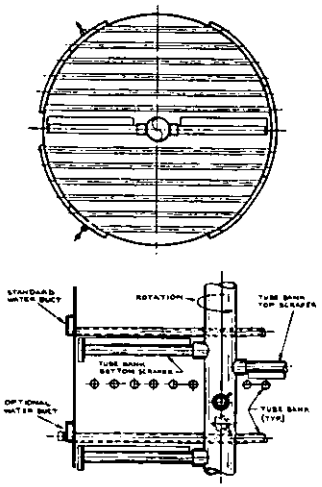


Figure 12-94. Typical cooling elements. Welded construction of stationary cooling pipes. Center shaft carries only agitation arms with scraper blades above and below cooling pipes. *(Courtesy, Silver Engineering Works.)*

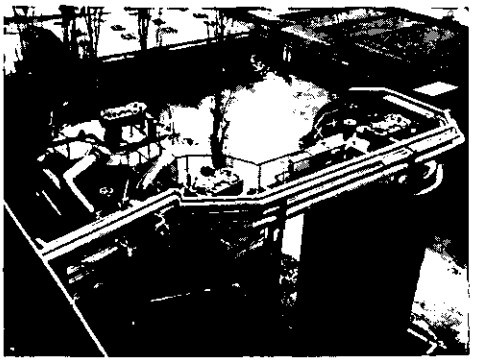
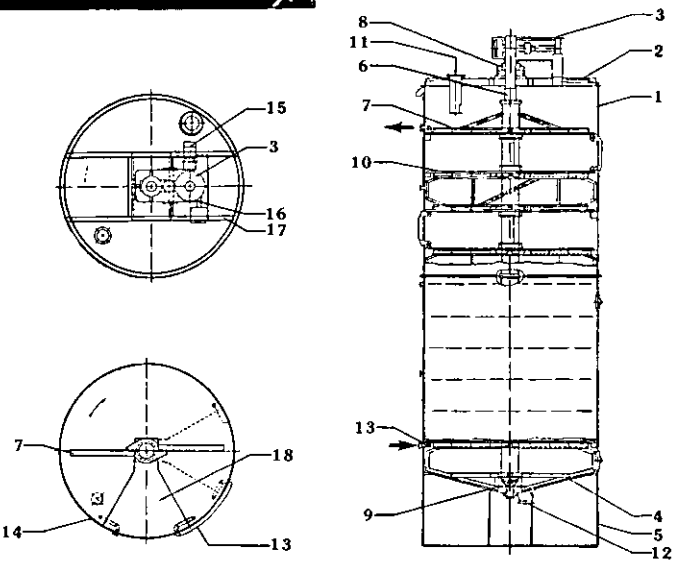


Figure 12-95. Vertical, series-connected crystallizers. Insulated covering on tanks and piping for outdoor operation. *(Courtesy, BMA).*



Sectional view of vertical crystallizer

1 Rolled shell
2 Reinforced cover
3 Drive with spur-wheel reduction gear
4 Conical bottom
5 Supporting frame
6 Split tubular shaft
7 Stirring arm
8 Upper shaft bearing
9 Lower shaft bearing
10 Cooling element
11 Magma in
12 Magma out
13 Water in
14 Water out
15 Three-phase motor
16 Torque arm
17 Cover reinforcement
18 Segment cut

Figure 12-96. Major design elements of vertical, cooling crystallizer. *(Courtesy, BMA.)*

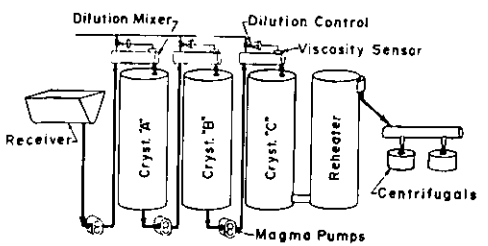


Figure 12-97. Vertical crystallizers with dilution system for each cooling unit. Rotating disk sensor for slurry viscosity measurement and control by water or molasses dilution. *(After Genotelle.)*

fortunately only one crystallizer was assigned per boiling pan. An equally rapid reheat was invoked simply to clear the equipment for the next batch. The much-needed additional 6 to 8 hours of "cold dwell" were not made available, thus there was the condemnation of Lafeuille crystallizers as non-efficient exhausters.

Several investigators have been reassessing the crystallizer cooling curve[1]. The slow, linear rate of prior practice is giving way to an initially very rapid but supersaturation-limited rate of cooling. This is followed by an asymptotic leveling-off at the lowest temperature for an extended surface (kinetic) reaction time.

To achieve such a rapid initial cooling without excess supersaturation at the cooling surfaces, the usual crystallizer agitator rpm needs adjustment upward. It has been suggested that agitation intensity should parallel or exceed the desired exponential cooling curve.

An analysis of the "agitation" in any given crystallizer should be evaluated *not* in terms of a mass crystal slurry moving past the inspection manhole – rotating

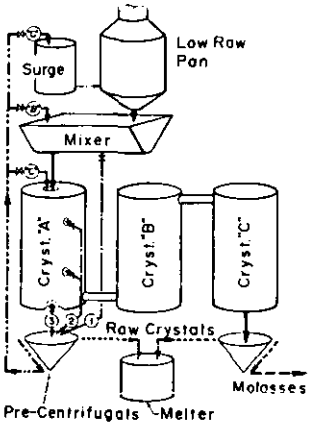


Figure 12-98. Precentrifuging flow schemes for % crystal redution. Optional points of massecuite withdrawal and optional points of mother liquor return are illustrated. After McGinnis (224d).

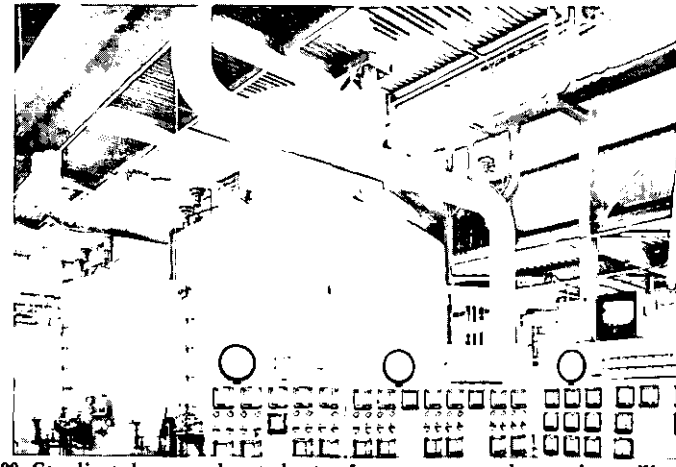


Figure 12-99. Coordinated sugar-end control system for pans, syrups, melters, and crystallizers. Direct accessibility of instruments to sugar boilers, enhances the the operator-equipment interface.

[1]See refs. (6,67,126,223,265,351,352,409).

with the rotary cooling elements or mixer arms. The agitation should be examined more properly with respect to crystals moving past crystals: a scrubbing, a "massaging," a kneading of the liquid film surrounding each crystal.

Horizontal crystallizers with inefficient mixing may be improved with stationary breaker bars. Vertical crystallizers have been designed with periodic penetrations of the mass flow by the stationary cooling elements.

Upon review of operating results of horizontal and vertical crystallizers, the conclusion of the author is that both styles are potentially excellent exhausters and both are capable of limited exhaustion.

Sugar End Control Management

Control Philosophy. Irrespective of the control method, whether natural, semi-automatic, or fully automatic, the fundamental requirement for uniform product is a consistently uniform crystallization technique. A sugar boiler with such a technique seldom pulls a proof and seldom has a quality problem.

As a first step, the preparation of the pan feed syrups is essential. Assuming excellence of beet-end purification work and a uniform density evaporator effluent, additional regulation of syrup flows, of syrup blending, and of temperature and density must be provided. The pan feed continues to be a dominant variable.

Quantitative data are rapidly being gathered on the influence of color, conductivity, pH, Brix, lime salts, and of other selected impurities. Their influence on crystallization rates and the preferable crystallization environment is being defined. It will soon be possible to "dial" these values into a composite, empirical crystallization model. Control with superior repeatability will be obtained.[1]

Application. As a second step, the boiling vessel's deficiencies must be identified and corrected. The addition of controls to a faulty pan is an invitation to process and control failure. Such an unsatisfactory performance would become an improper condemnation of automation.

Throughout the sugar-producing world,[2], intensive study of the control of batch sugar boiling has yielded improved product quality and extraction. Continuous boiling control is being implemented in a similar manner (100). Common to all schemes is the requirement for measurement reliability, simplicity, and relative freedom from measurement lag (306,310). Equally important is the representativeness of the measurements; in particular, the saturation temperatures and the massecuite stiffness (219,299,309).

Pans have been fully automated and have performed reliably (see p 718). Further reliability insurance is an integrated monitoring of each of the process variables for "off limits" conditions. Peripheral services can adversely influence the uniformity of the crystallization environment. The condenser water temperature and steam trap performance are typical. Off-limits annunciator systems call for immediate attention and correction action.

Process Management

As with any process supervision responsibility, awareness of the system status is mandatory for proper management and performance. The communication tools are the laboratory analysis sheets, the station logs, the repair supervisor's log, the process supervisor's own log, and the factory manager's coordination log of instruction.

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[1]See refs. (56,182,197,246,317,318,334).

[2]See refs. (78,79,104,128,183,185,191,206,227,269,290,302,320,345,442, 443,444,452)

Laboratory Data. The promptness of availability of laboratory data on what has just happened is a key to the supervisor's ability to anticipate the process status for the next several hours. The sugar-end supervisor's knowledge of the beet end and of the significance of certain beet-end data is helpful to reinforce his own status forecast. With improved, rapid analytical techniques and with closed circuit TV, analytical data are available within a few minutes of sampling and are simultaneously available with entry on the laboratory control sheets.

Data Logging. The trends of the process become obvious through data logs. The maintenance of such a log on the pan floor or in a central control provides coordination for the sugar boiler and for the process supervisor. The data logged would include: influent juices, syrup blends, each strike as completed, the inventory of syrups and massecuites, and of effluent products.

The sugar end equipment has its respective capacity limitations. Uniform operation permits the most efficient use of the equipment (111). Measurement of material flow rates provides the basis for operating uniformity. A data log permits additional supervisory assurance of uniformity.

Process Flow Schemes. The two-boiling system is no longer used in the beet sugar industry. It does, however, provide a simplified scheme for study of the manipulation of syrups and sugars according to their purities (224). The object is to achieve the proper raw massecuite purity which, in turn, permits a normal sugar loss in molasses, Figure 12-100.

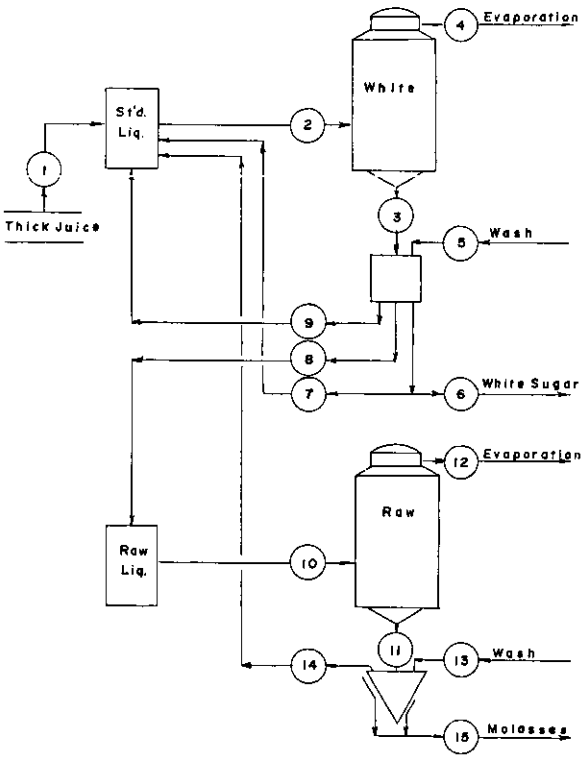

Figure 12-100. Two-boiling, materials flow (244).

Wash syrup of the white massecuite centrifugal is recycled to avoid raising the raw massecuite purity. The wash syrup purity is low and tends to hold the white massecuite purity down. So that the green syrup purity is not too low for the raw pan, some white product sugar is recycled and thereby tends to increase the white pan purity.

TABLE 12-7
TYPICAL MATERIAL BALANCE—TWO BOILING

Item	Identification	Brix	Purity	% on Beets			
				Total	D.S.	Sug.	N.S.
1.	Thick Juice	56.3	89.1	34.4	19.3	17.2	2.1
2.	Standard Liquor	62.7	89.6	57.3	35.9	32.2	3.7
3.	White Massecuite	92.4	89.6	38.9	35.9	32.2	3.7
4.	White Evaporation			18.4			
5.	White Wash Water			3.6			
6.	White Sugar Product			14.0		14.0	
7.	White Sugar Remelt			3.1		3.1	
8.	Green Syrup	74.6	78.6	13.7	10.2	8.0	2.2
9.	Wash Syrup	74.3	81.9	11.6	8.6	7.1	1.5
10.	Raw Feed Liquor	74.6	78.6	13.7	10.2	8.0	2.2
11.	Raw Massecuite	94.3	78.6	10.8	10.2	8.0	2.2
12.	Raw Evaporation			2.9			
13.	Raw Wash Water			0.6			
14.	Raw Sugar		98.0	5.0	4.9	4.8	0.1
15.	Molasses	82.7	60.4	6.4	5.2	3.2	2.0

TABLE 12-8
TYPICAL MATERIAL BALANCE—THREE BOILING

Item	Identification	Brix	Purity	% on Beets			
				Total	D.S.	Sug.	N.S.
1.	Thick Juice	59.2	89.2	32.5	19.3	17.3	2.0
2.	Standard Liquor	67.6	92.7	45.0	30.4	28.2	2.2
3.	White Massecuite	91.6	92.7	33.2	30.4	28.2	2.2
4.	White Evaporation			11.8			
5.	White Wash Water			2.1			
6.	White Sugar Product			14.4		14.4	
7.	White Machine Syrup	76.3	85.8	20.5	15.6	13.4	2.2
8.	Intermediate Liquor	76.3	85.8	15.4	11.7	10.1	1.6
9.	Raw Liquor	76.8	72.0	0.4	0.3	0.2	0.1
10.	Intermediate Massecuite	92.6	85.4	13.0	12.1	10.3	1.7
11.	Intermediate Evaporation			2.8			
12.	Intermediate Wash Water			1.8			
13.	Intermediate Sugar		98.7	7.1	6.1	6.0	0.1
14.	Intermediate Machine Syrup	76.8	72.0	7.8	6.0	4.3	1.7
15.	Intermediate Liquor	76.3	85.8	5.1	3.9	3.3	0.6
16.	Raw Liquor	76.8	72.0	7.3	5.6	4.1	1.5
17.	Raw Massecuite	93.4	77.5	10.2	9.5	7.4	2.1
18.	Raw Evaporation			2.2			
19.	Raw Wash Water			0.4			
20.	Raw Sugar		98.5	4.8	4.6	4.5	0.1
21.	Molasses	86.6	58.3	5.7	5.0	2.9	2.1

The white pan purity is too low to yield a high quality white sugar product which will meet today's competitive market requirements.

Three-boiling schemes are the most common in American practice for production of a competitive product quality and for a reasonably efficient sugar recovery. With first thoughts toward the proper raw massecuite purity for optimum molasses purity, it will be noted in Figure 12-101 that the intermediate machine syrup is 72.0 purity. This is too low to achieve a workable 58 molasses purity and too low for rapid crystal growth. This is offset by using intermediate liquor as the charge for a most favorable nucleation environment (124), and then by using raw liquor for pan completion. The final pan purity is proper to achieve the 58 molasses purity.

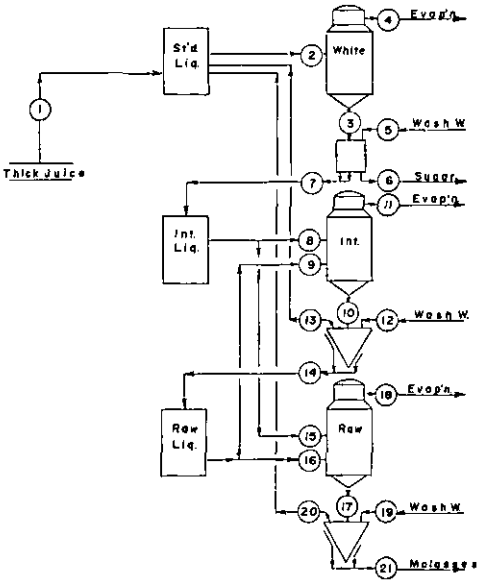

Figure 12-101. Three-boiling, materials flow (244).

It is noted throughout this scheme (224) that there is no separation of wash and green syrups. A minor amount of back-boiling is shown for the intermediate pan (365).

With full-seeding techniques, an excellent crystal crop can be established and grown without a high purity "footing" or initial charge. The intermediate pan purity and the separation at the intermediate centifugal establish the raw pan feed purity. Only one pan feed liquor is used for each grade of strike. The crystal growth rate may be comparatively slower, but, process control is simplified.

Where an exceptional purity white sugar product is required, or where the thick juice contains a high color content, a four-boiling system can be used, Figure 12-102. Such systems obviously require an extra set of centrifugals, syrup tanks, and a pan. Steam requirements are relatively high. As the need for such a superior product is rare, or the duration of high color thick juice is usually short, a modified three-boiling scheme is normally used for such conditions.

Process Flexibility. Beet quality determines the sugar-end process to be used. In those agricultural areas experiencing a large variance in beet quality during the campaign, the sugar end process must be flexible.

TABLE 12-9
TYPICAL MATERIAL BALANCE—FOUR BOILING

Item	Identification	Brix	Purity	% on Beets			
				Total	D.S.	Sug.	N.S.
1.	Thick Juice	60.0	90.3	32.2	19.3	17.5	1.8
2.	White Machine Syrup	76.0	96.2	9.3	7.1	6.8	0.3
3.	#1 Raw Liquor	63.6	91.9	41.5	26.4	24.3	2.1
4.	#1 Intermediate Massecuite	91.6	91.9	28.8	26.4	24.3	2.1
5.	#1 Intermediate Evaporation			12.7			
6.	#1 Intermediate Wash Water			3.9			
7.	#1 Intermediate Sugar		99.0	15.2	13.1	13.0	0.1
8.	#1 Intermediate Mach. Syrup	76.0	85.0	17.5	13.3	11.3	2.0
9.	#2 Intermediate Feed Liquor	76.0	85.0	12.8	9.7	8.3	1.4
10.	#2 Intermediate Massecuite	92.6	85.0	10.5	9.7	8.3	1.4
11.	#2 Intermediate Evaporation			2.3			
12.	#2 Intermediate Wash Water			1.4			
13.	#2 Intermediate Sugar		98.7	5.7	4.9	4.8	0.1
14.	#2 Intermediate Mach. Syrup	77.0	71.1	6.3	4.8	3.4	1.4
15.	#2 Intermediate Liquor	76.0	85.0	4.7	3.6	3.0	0.6
16.	Raw Feed Liquor	77.0	71.1	6.3	4.8	3.4	1.4
17.	Raw Massecuite	93.4	77.0	9.0	8.4	6.5	1.9
18.	Raw Evaporation			2.0			
19.	Raw Wash Water			0.3			
20.	Raw Sugar		98.5	4.2	4.0	3.9	0.1
21.	Molasses	86.6	57.8	5.1	4.4	2.6	1.8
22.	White Machine Syrup	76.0	96.2	13.2	10.0	9.6	0.4
23.	Dilution Water			7.6			
24.	Standard Liquor	70.0	98.0	46.2	32.3	31.7	0.6
25.	White Massecuite	91.0	98.0	35.5	32.3	31.7	0.6
26.	White Evaporation			10.7			
27.	White Wash Water			2.2			
28.	White Sugar Product			14.9		14.9	
29.	White Machine Syrup	76.0	96.2	22.5	17.1	16.4	0.6

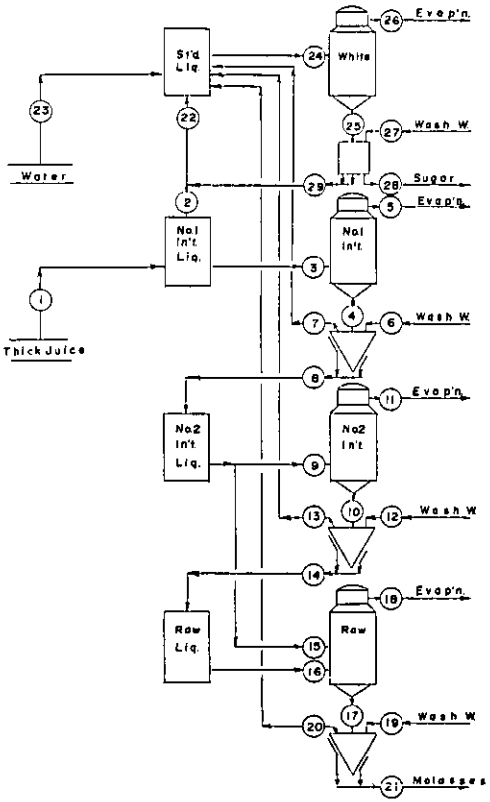

Figure 12-102. Four-boiling, materials flow (244).

Early season beets normally permit all or nearly all the thick juice to be processed directly in standard liquor: point "A" on Figure 12-103. This, of course, is the simplest and most economical production scheme.

With climatic extremes during harvest or extended pile storage, color and lime-salts type impurities become significant and tend to affect the sugar product quality (319,368,445). The ratio of remelted sugars to thick juice must be increased to dilute color solids sufficiently to permit continued production of a superior product quality.

Dilution of the color impurities is accomplished by splitting the flow of thick juice between the white and intermediate side, points "A" and "B," Figure 12-103. Such a split is determined and monitored by analysis of the following materials on a "color units" per 100° Brix basis:

Anticipation	quality	control	—thin juice
"	"	"	—thick juice
In-process	"	"	—standard liquor
"	"	"	—wet sugar from centrifugals
After-the-fact	"	"	—dry product sugar.

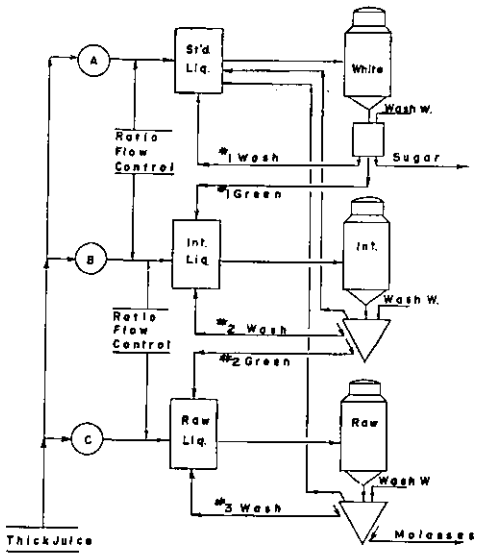

Figure 12-103. Thick juice quality and process acceptance.

Thick Juice		Flow Ratio			Criteria for Flow Ratio
Color	Impurity	A	B	C	
low	low	1.0	0	0	No Split
medium	—	Split	Split	0	Std. Liq. color "purity"
high	—	0	1.0	0	Std. Liq. color "purity"
—	high	0	Split	Split	Intermediate Mass. impurity

Flow ratio control of the thick juice entering the sugar end can be set to maintain a purity balance. The standard liquor color, as related to wet sugar color, will fix the ratio with the least time lag. Where beet quality fluctuates rapidly, analytical determinations must, of course, be made frequently. Where beet quality is poor and colors are intense, the quality of the product is very sensitive to even small amounts of occlusion and conglomeration. In this instance, all thick juice must be processed through the intermediate, point "B."

Figure 12-103 assumes some type of "laundry" or upgrading of the raw sugar. In this instance, raw wash separation is illustrated for simplicity. Wash separation is shown for all three boilings and is helpful in stabilizing impurity balances as various thick juice ratios are required (111,293,382).

Separation, early or late, is of course variable. With high colors, an occasional "color purity" analysis of the wash syrups will dictate the separation. With high lime-salt type impurities and introduction of the thick juice at "B" and "C," the white and raw purities will escalate, requiring some back-boiling to reduce the purities. An early separation is helpful.

The slicing or thick juice flow rate must be restricted to the maintenance of a controlled purity balance throughout the sugar end (110). The syrup inventory will tend to accumulate at the system bottleneck. Again, the thick juice influent rate must be restricted to avoid excessive in-process inventory.

Flexibility in Equipment Use. When the processing of all thick juice is required through the intermediate pan, the material balance for white and in-

termediate work is reversed. When the pan and centrifugal equipment for white and intermediate use are sized for normal purities, the material flows can be interchanged in the two sets of equipment and thus some of the total system capacity can be recovered. This assumes that both sets of centrifugals are compatible with white product operation. In other instances, part of the equipment, such as pans and centrifugals, can be shifted in "swing" fashion to better match the boiling and centrifugal loads.

The sugar-end supervisor should be constantly alert to any equipment limitation and record his observations in his log.

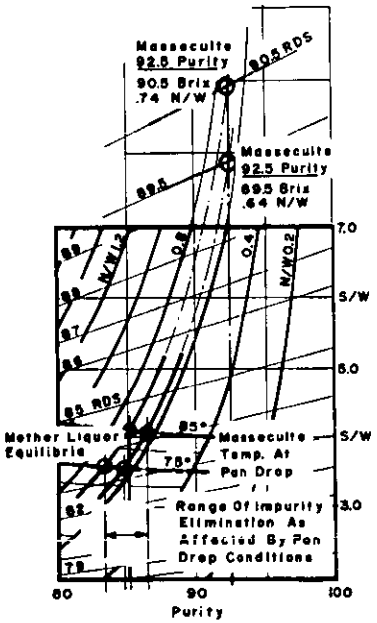

Figure 12-104. Influence of pan-drop Brix and of pan-drop temperatures on potential crystal yield of first-boiling massecuites. The chart has been expanded to show attainment of the "N/W track" at pan drop. For the 89.5 Brix massecuites, yields at 80°C and 75°C are 51.0 and 56.9 respectively. For the 90.5 Brix massecuites, yields at 85°C and 75°C are 55.9 and 61.2.

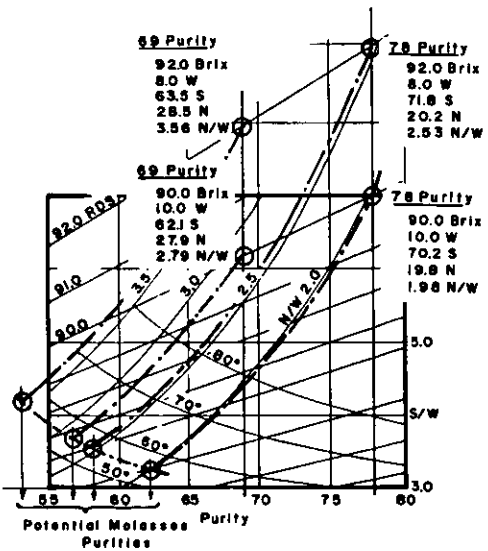

Figure 12-105. Influence of pan-drop Brix and of pan-drop purity on potential crystal yield of low raw massecuite at end of *evaporative and cooling* crystallization at a final 1.00 SS, 55°C. At the higher "N/W track," the mechanical limitations of % crystals in the massecuite may become the limit to further exhaustion. Potential molasses purities are significantly modified by the conditions at pan drop (453c).

Pan Feed Syrup Economics. High Brix (72 °) thick juice from storage can be blended to yield a high density standard liquor. A circulated pan can function very satisfactorily with a high Brix feed,[1] or even on a somewhat supersaturated feed syrup (100). The pan vapor requirement can be reduced by 30-35% by simply raising the feed Brix from 67 to 74.

Yields and Losses. In brief review, lack of crystal yield is related to: conglomeration, fine grain, low Brix as dropped from the pan, or irregular centrifugal work.

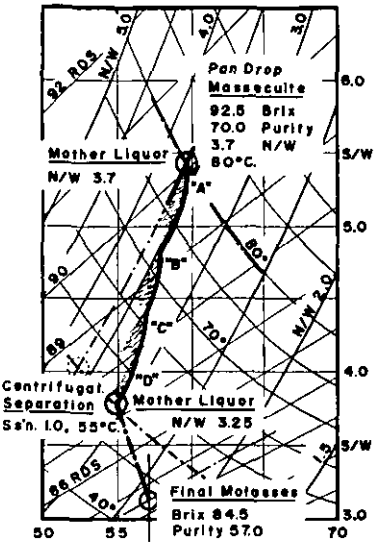

Figure 12-106. Influence of water dilution during cooling-crystallizer and centrifugal operation. Point A is the result of excessive pan steamout into the receiver (327,339,423). Points B, C, and D are dilutions to restrict bulk viscosity build-up (See Figure 12-83). Point E is the result of excess wash water to achieve an "acceptable" sugar for remelt.

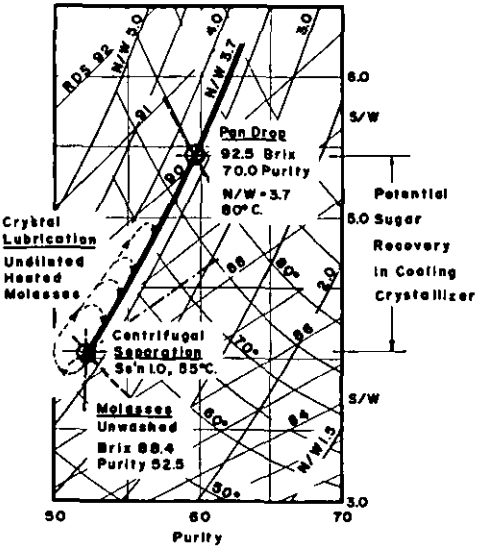

Figure 12-107. Influence of undiluted molasses recycle to provide crystal lubrication and to mechanically permit continued crystal yield development (See figure 12-87). In the shaded area, the use of a no-wash separation, followed by an affination for the necessary "crystal laundry" effect, is assumed, thus no purity rise during the *last few critical seconds* of impurity separation.

[1]See refs. (49,121,227,303,333).

To grasp visually the influence of various factors in the sugar-end processes, recent charts (294a,453d,453e) (See Figs. 104-107) of saturation equilibrium conditions, based on Grut's data, are useful. Several sugar-end situations have been charted. Other conditions of wash-volume and separator settings, of high raw purity balance, etc., can be easily illustrated also.

In using such a chart, it must be constructed to fit the solubility data of the local area (Equation 3) and it must be remembered that the massecuites (mother liquors) are usually still supersaturated, thus there is a "lag" in reaching the ultimate purity represented by the "N/W—temperature" intercept on the chart.

For any given equipment scheme, a rather narrow range of purities will be found by experience, which will yield a workable final massecuite and a low molasses purity. There is a corresponding intermediate massecuite purity which will, with good pan and centrifugal work, yield the intermediate green purity required for the raw pan feed. Excellence of sugar crystallization and subsequent centrifugal operations and the correct intermediate pan feed liquor purity are the keys to good loss management.

On the white side, nonsugar purity, color purity, and the absence of turbidity of the standard liquor are the initial product quality control factors. In addition to the items above, there is the requirement for a well-circulated pan to achieve a low moisture sugar suitable for bulk storage and shipping.

Upgrading

Surface Impurities. Low-raw massecuite treatment is a key factor in attaining the sugar-end objectives of product quality, maximum extraction, and optimum capacity. The beet quality, the beet-end purification effectiveness, and boiling procedures fix the mother liquor viscosity and the ease with which it can be spun off the crystals.

With a normal viscosity of mother liquor and free-draining crystals, a simple centrifuging can yield a low-raw sugar suitable for direct remelting in standard liquor. A viscous mother liquor will leave a greater amount of impurities on the crystal surfaces. The sugar is then unsuitable for entering the standard liquor. Remelting for intermediate crystallization obviously adds a large load to the intermediate system and effectively reduces the sugar-end capacity (111). The impurities are predominantly on the crystal surface. Several "laundry" techniques are used to avoid unnecessary recirculation.

In-place Washing. The simplest situation is that of a relatively free-draining crystal crop in viscous mother liquor. Application of a small amount of wash water may effectively raise the sugar purity three to five units without abnormal increase in the molasses purity.

Massecuite with good crystal drainage but with a tenacious mother liquor will often accept wash water in greater amounts than usual to yield a satisfactory raw sugar for standard liquor (331,433,434). The centrifugals must be fitted for wash separation, Figure 12-108. The raw wash is normally used to finish up or "top off" the next raw pan. This is a relatively simple solution but does require extra raw pan and crystallizer capacity. This is a requirement common to each of the upgrading operations.

Affination. Massecuites of mixed grain wash poorly. The affination technique provides an excellent laundry effect.[1] Raw massecuite treated without wash, in

[1]See refs. (53,184,195,218,336).

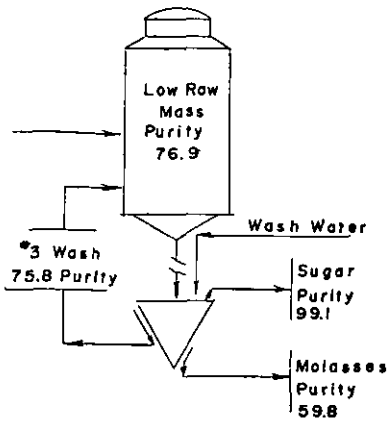

Figure 12-108. Low-raw wash separation.

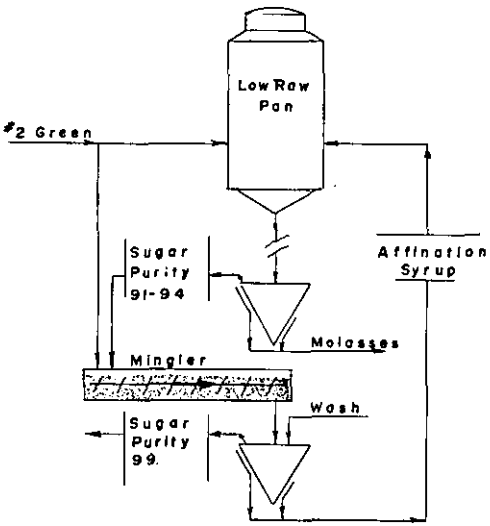

Figure 12-109. Affination of low-raw sugar.

batch or continuous centrifugals, will yield a minimum purity molasses and a sugar of 91 to 94 purity. Mixing this sugar with a saturated intermediate green or intermediate wash to form a moderately stiff magma, causes a dilution of the molasses film and reduction of the film viscosity. A second centrifugal treatment then yields a considerably upgraded sugar of about 99 purity, Figure 12-109. The affination syrup is used in the next raw pan.

With this system, the process is not particularly sensitive to mixed grain if continuous centrifugals are used. The impurity recycle is confined to the raw system. The standard liquor is of improved quality. This is of particular importance with syrups of high color. Lott (196) has reported an 18 to 20% reduction in intermediate massecuite volume by the use of affination.

Separation of Crystals and Syrup*

Massecuite Treatment Prior to Centrifuging. The quality of the massecuite fed to the centrifugals should be uniform throughout the strike in grain size, density, fluidity and temperature. White and intermediate massecuites are usually purged at pan temperature. While awaiting purging, this temperature should be maintained. Many beet factories use rotating heating coils in the lower portion of the modified U-shaped mixers with a stirrer in the wide upper portion. In addition covers are used, to help maintain the original temperature.

Cylindrical mixer tanks have come into wide use for high purity massecuites, varying in diameter from 8 to 13 ft. A temperature drop in the massecuite of the order of 0.5 °C per hour takes place. Some tanks are insulated. The size must be sufficient to hold a strike plus a portion of the preceding one, so that the centrifugals can be kept in continuous operation. The circular cross section is advantageous since the volume can diminish as the massecuite is purged, with a minimal change in head on the massecuite in the centrifugal charging gate. Almost perfect homogeneity can also be maintained with a single, center-shaft stirrer.

In raw crystallization, the crystallization is started in the vacuum pan and completed in the crystallizer with cooling and stirring. The final product from the crystallizer is at a temperature of about 40 °C. This material is too viscous for efficient separation by centrifugation, and heating simply reduces the viscosity. Fortunately in cold massecuite the syrup surrounding the crystals is supersaturated. The massecuite can, therefore, be heated to the saturation temperature of the mother liquor without melting grain. Generally the saturation temperature is between 55 and 60 °C.

Stevens Coil

The Stevens coil is almost universally used in the American beet industry for this purpose. It is a coil of steel pipe mounted on a hollow center pipe which is rotated in the lower section of a U-shaped mixer tank. Hot water is circulated through the rotating coil.

The object of the device is to raise gently the temperature without localized overheating. The coil rotates at 9 rpm with the 32 in. od coil, and at lower speed for larger diameters. A large volume of water, 275-300 gpm, is circulated at a temperature of about 8 °C higher than the saturation temperature.

The sizing of the coil is dependent on the volume of massecuite to be heated, the length of the mixer tank feeding the centrifugals, and the desired temperature increase. The heat requirement for a temperature rise of 20 °F (11 °C) for one cubic foot of low massecuite is:

$$H_1 = 93.5 \times 0.44 \times 20 \times 1.33 = 1080 \text{ Btu} \quad (1)$$

where 93.5 is the weight of the massecuite in lbs/cu ft (6), 0.44 the specific heat of the massecuite in Btu/°F/lb (4), 20 the temperature rise in °F, and 1.33 the multiplier covering losses in the system of 25%.

The heating surface of some typical coils follows: 3 in. pipe, 32 in. od coil on 16 in. diam. center pipe, 7 in. pitch = 15.3 sq ft/ft; 4 in. pipe, 48 in. od coil on 16 in. diam. center pipe, 8 in. pitch = 24.4 sq ft/ft.

Assuming: a mean water temperature of 68 °C (155 °F), and a mean massecuite temperature of 46 °C (115 °F) the available difference for heat transfer,

*by A.H. Stuhlreyer

Δt, is 40 °F. The heat transfer factor for low raw massecuite is in the range of 17.5 Btu/ °F/sq ft/hr, and thus the thermal capacity is:

$$Tc - 32 = 15.3 \times 17.5 \times 40 = 10710 \text{ Btu/hr/ft.}$$
$$Tc - 48 = 24.4 \times 17.5 \times 40 = 17080 \text{ Btu/hr/ft.}$$

Since 1080 Btu under the stated conditions are required to raise one cu ft of massecuite 20 ° F, these capacities result:

32 in. coil—10710/1080 = 10.0 cu ft heated per hr, per ft of coil.
48 in. coil—17080/1080 = 15.8 cu ft heated per hr, per ft of coil.

It follows that coils can be fitted to any practical requirements by changing such factors as spacing, and coil diameter.

Compartment Heating

A properly functioning reheater must deliver massecuite to each of the centrifugals at the same temperature. Numerous ways to do this are used, but a description of one will suffice.

Stevens promoted the compartment type mixer tank, see Figure 12-110. The U-type tank has a sloping horizontal deck plate located just above the coil. Underneath the deck are vertical partitions which create equally sized compartments, one for each centrifugal. A slotted opening in the deck plate allows cold massecuite to enter the compartment. The gate spout opening is in the tank bottom. The massecuite, from its entrance in the upper corner of the compartment to its outlet in the diagonally opposite lower corner, travels in and around the coil and is thus uniformly heated. Both the heating surface and the volume are the same for all compartments so that the temperature of the heated massecuite will be the same.

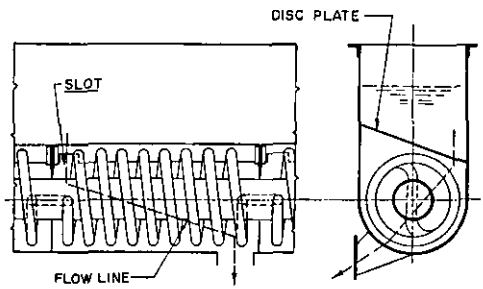

Figure 12-110. Compartmented Stevens coil, for low-raw massecuite.

The circulating water enters and leaves the coil at the drive end. It passes through the coil to the opposite end and returns through the center pipe. The temperature of the inlet water is constant. The circulating water pump is fitted with an on-off control which reacts to the massecuite temperature.

The use of continuous centrifugals for raw massecuite invites a different approach to the matter of heating before centrifuging. Since the massecuite flow through the heater is not intermittent, the compartments are no longer desirable. The ideal is that all massecuite should flow through the full length of the heater coil and then be fed to the machine feed valves. This can be accomplished in a variety of ways depending on the individual station layout.

The value of heating as a means of reducing viscosity is well stated by Dr. George Meade—"Shaw's curves show that the viscosity of a molasses approximately doubles for each 10 ° drop in temperature from 100 ° to 70 °F" (348).

The Centrifugal Station
The Batch Separator

Historical

The centrifugal machine is the device used to separate the crystalline sugar from the mother liquor. The difference in weight between the liquid and the solids, and the viscosity, prevent a fast gradation by sedimentation. Originally an incomplete separation was obtained by filling barrels or inverted cone-shaped vessels with massecuite. Holes in the bottoms of these vessels were fitted with plugs which were subsequently removed and the uncrystallized syrup drained off. On occasion water was filtered through to improve the quality of the sugar (73).

In the latter half of the 19th century, the Weston-type centrifugal, employing a single point suspension, came into general use, and this same suspension principle is used in the present-day batch centrifugals in the sugar industry. The cylindrical drum or basket is fixed to its shaft by means of a spider which forms the central discharge opening. The mode of suspension at this single point is usually a ball and socket joint, but in some makes a conoidal-shaped rubber buffer is used into which the bearing housing is inserted. In the ball and socket type suspension a ring-shaped rubber buffer is arranged outside and around the bearing housing so that it distorts as the basket swings out. The internal friction in the buffer acts as a retardant to this swing-out. The centrifugal, with its free spindle and its capability of oscillating, is self-balancing. The basket with its loads finds its own center of rotation which corresponds quite closely to the center of gravity. The ball joint suspension is shown in Figure 12-111 (9).

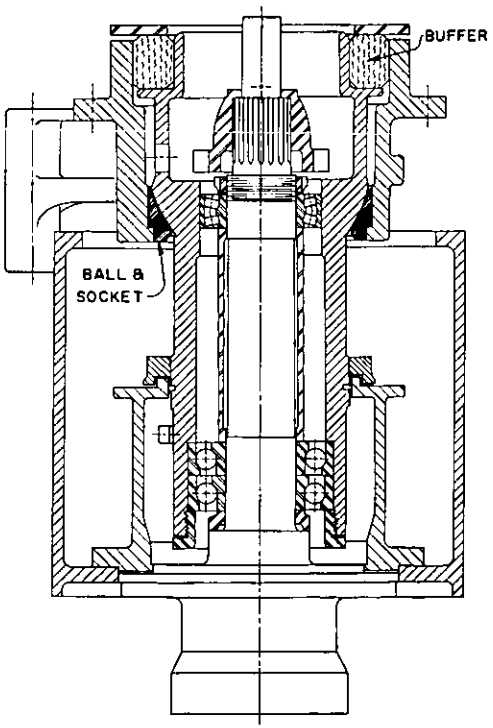

Figure 12-111. Centrifugal head showing ball and socket suspension.

The substitution of centrifugal force for gravity alone, both shortened the separation process and vastly improved its efficiency. The utilization of Weston's principle made possible a mechanically practical machine.

Theoretical

Centrifugal force is defined as that force which tends to impel a thing outward from a center of rotation. The action follows well-known physical laws. The faster a body is moving around a circle the greater is the centrifugal force, which is proportional to the square of the speed.

Also for the same peripheral speed, the centrifugal force will be proportional to the radius of the circle, or F is inversely proportional to the radius or rotation.

Centrifugal force then is directly proportional to the mass of the rotating body and to the square of the peripheral speed and is inversely proportional to the radius of rotation:

$$cf = \frac{mV^2}{R} \tag{2}$$

mass m = w/g, where w = weight and g = falling acceleration due to gravity,

$$cf = \frac{wV^2}{Rg} \tag{3}$$

In place of V, the peripheral velocity in feet per second, it is desired to use rpm as a practical measure:

$$V = 2\,R \times \pi \times rpm/60 = RN/9.55 \tag{4}$$

where N = rpm.

$$\text{so,} \qquad cf = \frac{wR^2\,N^2}{R \times 32.2 \times 9.55^2} = \frac{wRN^2}{2935} = wRN^2 \times 0.00341 \tag{5}$$

For force per unit pound,

$$w = 1, \text{ and } cf = RN^2 \times 0.00341\ (200). \tag{6}$$

The term "gravity factor," or "G" is the centrifugal force multiple which may apply to any given weight. G varies directly with the basket diameter when the speed is constant. Examples:

rpm	Basket Diam.	Gravity Factor
1200	20 in.	410
1200	40 in.	820
1200	50 in.	1025

G varies as the square of the speed at a given diameter. Examples:

rpm	Basket Diam.	Gravity Factor
200	48 in.	27.3
400	48 in.	109.8
800	48 in.	436.8
1200	48 in.	982.8
1500	48 in.	1534.6

Basket Design

Sugar centrifugals are usually top-driven. The basket is made up of a perforated-plate steel cylinder, having a partial top with a center opening. The basket is loaded through the top, the diameter of the opening of which establishes the volumetric capacity. The bottom is composed of an annular plate and a central hub connected to each other by spokes. The vertical driving spindle is fixed to this hub. The top, bottom, and cylindrical side sheet are welded into a unit, and usually the side sheet is fitted with shrunk-on, high tensile strength, reinforcing rings. The rings afford a higher safety margin and make the use of a lighter side sheet of stainless steel both structurally and economically feasible. The basket is lined on the inside with a woven backing screen which supports the perforated filtering screen. The liquids are expelled through the holes and the crystals are retained in the basket. After the major portion of the molasses has been eliminated, the sugar can be washed with a fine spray of water dispersed over the sugar wall. If low moisture is required, the spinning operation continues after the wash. The purged sugar is discharged by means of a mechanical plow through the openings between the spokes of the basket bottom. Please see Figure 12-112.

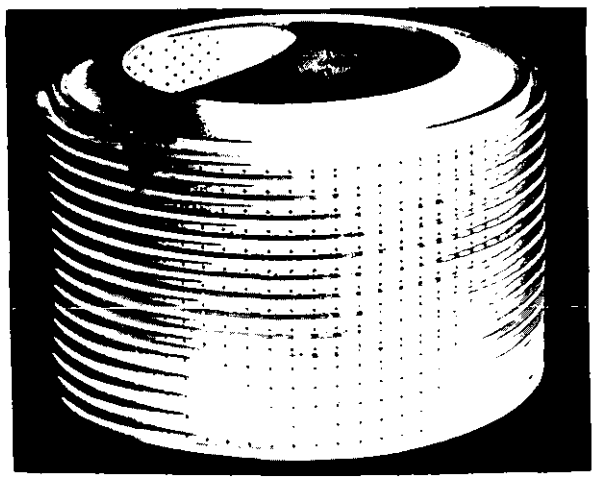

Figure 12-112. Centrifugal basket.

Drive Design

Practically every type of prime mover has at one time or another been used to drive centrifugals. In modern plants individual electric motors, both a-c and d-c, are most widely used, although there are numerous belt-driven, gear-driven, and water-turbine driven machines still in service.

The belt- and gear-driven machines are gradually being phased out. They are driven in multiple from a common line shaft which rotates continuously with the individual centrifugals connectable to the line shaft by friction clutches. As a result, any maintenance work on a single machine may require stopping the entire battery, thus causing an excessive loss of production. These centrifugals also are not easily made fully automatic.

Many types of individual electric motor drives are presently in use. All have at least two steps of acceleration, and some types using variable d-c voltage (Ward

Leonard or Thyristor-controlled) not only have multiple acceleration steps but also adjustable spinning speeds (174).

An equally divided two-speed motor, such as 600/1200 rpm, theoretically halves the acceleration heat loss as compared to a single-speed motor. A four-speed motor is even more effective since the power loss in heat is inversely proportional to the number of speeds. Increasing the number of accelerating steps, however, complicates the motor and the motor starting equipment. In North America, single-winding, consequent-pole, two-speed a-c, squirrel-cage induction motors are used for the most part because of their relative simplicity, ruggedness, lower first cost and available 1200 rpm speed.

Thyristor controlled dc drives are used to a greater degree in countries where the standard ac frequency is 50 hertz and the 6-pole ac pole-change motor speed is therefore 1000 rpm.

The two-speed motor makes electric regenerative braking possible. With the motor running at top speed, it can be de-energized and then re-energized on the low speed connection which changes the motor into an induction generator. The motor decelerates rapidly using no power and actually returns power to the system. As the speed approaches the half-speed synchronous speed, the decelerating torque nears zero, and the motor is disconnected from the power source. With about two-thirds of the stored energy removed electrically, the mechanical brake takes over and brings the basket to rest. The driving motor is, in fact, an electrical accelerator-decelerator. It must be especially designed to repeatedly start and stop the high inertia load involved, and to dissipate the heat resulting from the electrical losses.

The motor must be designed for the specific service, taking into account the diameter of the basket, its loaded weight, the top speed, and the length of the cycle. Experience has indicated that a gravity factor or G of 920; i.e., 48 in. at 1160 rpm, is a good one for high purity massecuite and an 1800 G factor; i.e., 40 in. at 1760 rpm, for raw massecuite. These two figures are the basic gravity factor criteria which are used when fixing the combination of basket size and speed for a particular application. Examples of basket diameters and speeds with resultant gravity factors are shown in Table 12-20.

The trend is to ever larger machines and thus fewer units for a given production. Baskets 54 in. in diameter by 40 in. deep are in service in many beet-sugar factories. In all probability, this size does not represent the ultimate.

Process Influence on Design

The varying qualities of different grades of massecuite influence the centrifugal force requirements, including the size and uniformity, or screen analysis, of the sugar crystals, and the viscosity of the mother liquor. As the purity of the massecuite decreases, the nonsucroses present in the mother liquor increase its viscosity and greater centrifugal force is required to effect separation. The required purity of the product influences either the centrifugal-force requirement, or the high-speed spinning time.

Increasing the basket depth is the most economical way to increase capacity. This increase is limited because problems of stability are created. The thickness of the charge is influenced by the uniformity of the crystals, the viscosity of the mother liquor, and the required sugar purity. The basket sizes, too, are influenced by the available electric current, and the physical and structural limitations of the basket materials. For practical reasons three basket diameters have come into common use

with varying depths and speeds to suit the application. These are shown in Table 12-21, while Table 12-22 lists the capacity of various basket sizes.

Table 12-20. Relative Centrifugal Force at Various Speeds

	Centrifugal Basket Diameters			
rpm	40 in.	42 in.	48 in.	54 in.
850				554
900	460	483	552	622
950	513	539	616	692
1000	568	597	682	767
1050	627	658	752	848
1100	688	722	875	929
1150	752	789	902	1015
1200	819	859	982	1105
1250	888	933	1066	
1300	961	1001	1153	
1350	1036	1088	1243	
1400	1114	1169	1337	
1450	1195	1255	1434	
1500	1279	1343	1535	
1550	1366	1434	1639	
1600	1455	1528	1746	
1650	1547	1625	1857	
1700	1643	1725	1971	
1750	1741	1828	2089	
1800	1842	1934	2210	

Table 12-21. Commonly Available Baskets

White & Intermediate		Low Raw	
Size	Max. Speed (Syn.)	Size	Max. Speed (Syn.)
48x30x7	1200	40x30x6	1800
48x36x7	1200	48x30x6	1800
54x40x7	1125	54x40x6	1500

Table 12-22. Volumetric Capacities of Conventional Sugar Centrifugal Baskets in Cubic Feet

Basket Dimensions	Capacity of Baskets with various width caps	
	6 in. cap	7 in. cap
40x24	8.90	
40x30	11.12	
42x20	7.85	8.91
42x24	9.42	10.69
42x30	11.78	13.36
48x24	11.00	12.63
48x30	13.74	15.79
48x36	16.49	18.78
54x40	20.95	23.92

Automatic Operation

The utilization of fully-automatic, batch-type centrifugals has become widespread. For intermediate and low purity massecuite continuous centrifugals are becoming ever more widely used. The improvement in performance is so marked as to almost preclude the use of batch machines for raw and intermediate massecuite in any new installations. These will be described in a later section.

Fundamentally the design of the batch centrifugals is the same for all duties. The speed, the basket size and the cycle components are changed to suit.

The two-speed a-c drive motors used have only a single winding, with the two speeds obtained by a pole-changing control. The low speed is, therefore, one-half the high speed, and the motors are designed for the same torque on either connection. In the automatic machines the motor is started by energizing the low-speed connection. After a preset time the motor is de-energized and immediately re-energized on the high-speed connection. The acceleration then proceeds until top speed is reached. High-speed rotation continues until the desired sugar dryness is attained. The proper process timer then initiates regenerative braking until almost half speed is reached. Then the mechanical brake brings the basket to rest and the separate discharge-drive motor is energized. Figure 12-113 is a speed-power-time curve for a 48 in. × 30 in., 600/1200 rpm, fully-automatic centrifugal for a white sugar or intermediate sugar service.

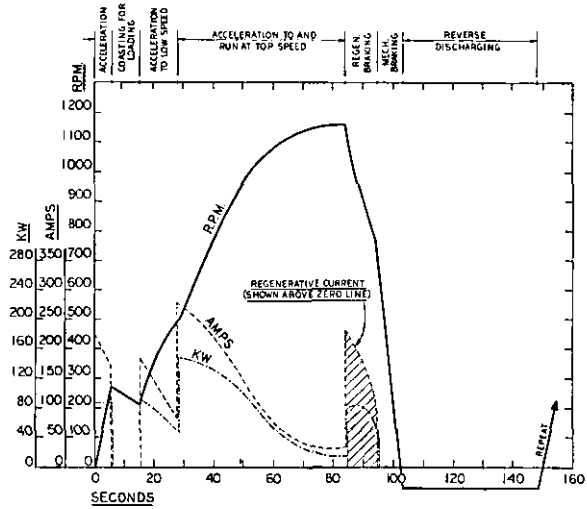

Figure 12-113. Typical performance curve of a fully-automatic centrifugal.

The change-over from low to high speed is performed automatically on the basis of a timed measurement. There is a precise best speed at which this change should take place, so the timer must be properly set. This is at the point in the low-speed accelerating cycle when the rate of rise begins to taper off. After change-over to the high-speed connection, the rapid rate of acceleration is restored. The net effect is that the basket is accelerated in the minimum possible time and with a relatively low current peak at the change-over. If the change-over is delayed, the time to reach top speed is extended. If the change-over is too soon, a greater current peak results and more rotor heating occurs.

Centrifugal drive motors must dissipate the heat generated by the electrical slip-loss during acceleration and during regenerative braking. Time is required to accomplish this, as is indicated by the fact that the motors are specified as suitable for a certain minimum load-to-load duty cycle.

There is a further limitation than simply time in establishing the minimum duty cycle of the motor. This is the high-speed running time from a point above 5/6 of the low-speed synchronous speed to the point where regenerative braking starts. In this range the motor is running at its highest speeds, and the rotor is being most effectively cooled. Therefore, the time for the curve segment "Acc. & Run at Top Speed" shown on Figure 12-113 may not be diminished for the motor in question. This limitation should be known and strictly enforced.

In the completely automatic batch-type centrifugals, all functions are performed without the intervention of an operator. The timed sequence for a typical 48 in. × 30 in. white-sugar centrifugal follows in Table 12-23: the timed intervals listed will vary quite widely for different quality feed material and with different technologists' judgments.

Table 12-23. Time Sequence of Operations in a Completely Automated, 48 in. × 30 in. White Sugar Centrifugal

	Time in Seconds from Start	rpm	
Start rotation	0	0	
Preflush of basket	1-3		
Open charging gate	5	275	
Close charging gate	15	210	
First wash on (6)	25	450	
First wash off	30	530	
Second wash on	65	1110	
Trip syrup separator to wash	69	1130	
Second wash off	75	1150	
Start regenerative brake	123	1160	
Start mechanical brake	132	760	
Start plowing speed	140	40	
End plowing, recycle	187	0	(10)

When these recycling machines are operated in a battery, an automatic control is used to sequence the starts of the machine. This control establishes the interval between the start of one centrifugal and the start of the next succeeding one. It keeps power demand peaks at a minimum and provides an even flow of sugar from the battery.

Figure 12-114 shows a section through a typical fully-automatic batch centrifugal. The control is accomplished by means of a combination of electrical relays, timers, limit switches and pressure switches. The use of solid state electric devices is a widely accepted innovation. They control the logic functions and replace the electro-mechanical relays and timers. The operations are mechanically and electrically interlocked to insure the proper sequence. Various steps in the process are monitored by an alarm system which calls attention to and approximately locates a malfunction.

Operational Guides

Fast acceleration within the limits of the available power system is desirable for white and intermediate centrifugation, but the drainage characteristics of the massecuite should be taken into account when establishing the acceleration time.

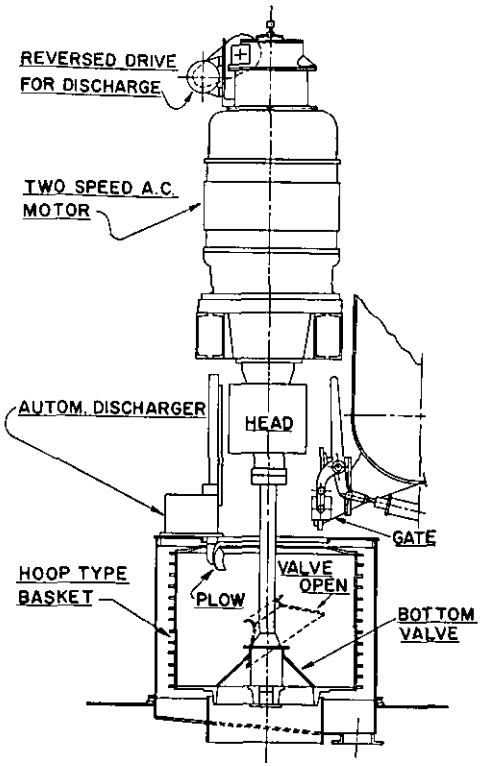

Figure 12-114. Section. Fully-automatic centrifugal, 48x36x7 (Western States Machine Company).

Conversely a slower rate of acceleration for the raw massecuite is suggested when batch machines are used.

The double application of wash water has been found to be effective when the sugars are to be washed to high purity. In the case of final crystallizer sugars, a single wash only is recommended, or possibly no wash.

The first of two washes should come on just as the syrup film leaves the face of the sugar wall. It should endure for not more than five seconds and a quantity of water equal to about 0.9 lbs per cu ft of massecuite should be applied. Sufficient time should elapse to allow most of the syrup to be filtered off before the second wash is applied. If the effluents are separated, the wash effluent will thus approximate the massecuite impurity. The separator should be tripped a few seconds after the start of the second wash. With intermediate, timing of the separator trip may be altered to control purity of the wash.

These observations are only guidelines. The amounts of water and the times of application will vary with local factors and objectives.

Washing

The wash water should be of the best quality, at a temperature of 82° to 110°C. Many factories use superheated water at 110°. This vaporizes as it leaves the spray nozzle, and, for the most part, condenses before reaching the sugar. A more even dispersion of the water over the sugar wall is accomplished.

Constant pressure wash water is vital if efficient washing is to be accomplished. Pressure regulators are available, but a more fool-proof system can be built around a properly selected centrifugal pump. A pump with a flat head capacity characteristic and with a rated point not more than 3 psi below the shut-off pressure will ensure a minimum variation with no control devices. Please see curve Figure 12-115. The piping must be sized for the maximum flow, with a small line from the end of the header returning to the supply tank. A continuous flow through the header will ensure uniform temperature and pressure.

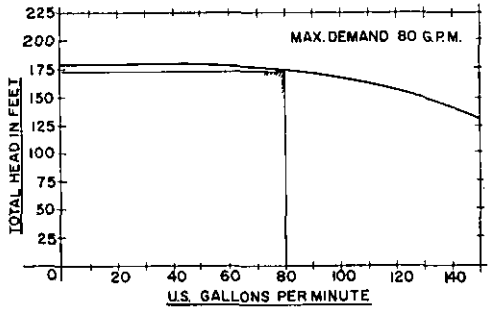

Figure 12-115. Performance curve for typical wash water pump, 2.5x2 in., single-stage, 7.5 hp, 3500 rpm.

Nozzles

There are diverse opinions about wash water nozzles. Single nozzles, two nozzles or even eight or nine nozzles are commonly used. The problem is to provide a relatively uniform application of water with a slightly higher concentration at the top and the bottom of the sugar wall. The real test is that the final wall of washed sugar in the basket be straight with slight undercutting at the top and bottom. A multiplicity of nozzles makes possible the best positioning of the nozzles but also creates the requirement for more frequent adjustment.

Modern centrifugals are equipped to automatically preflush the basket screen after the sugar has been plowed out and before the admission of the next charge. This operation properly performed is essential to efficient centrifugation.

Syrup Separation

The basket is surrounded by a stationary cylindrical casing which catches the molasses as it is expelled through the holes in the baskets. This casing is called the *curb*.

The molasses is collected in an annular compartment in the bottom of the curb from which it flows to the appropriate tank. The sugar is discharged from the basket through the openings between the spokes of a corresponding central opening in the curb bottom.

If the sugar is washed in the basket, the purity of the molasses is increased because of partial solution of the crystals. Normally wash is applied after the majority of the molasses has been expelled. This means that the low purity molasses can be separated from the high purity wash liquor by sending the first effluent to one tank and diverting the latter to another. If the separation can be made automatically, the value is further enhanced.

On some units the curb side sheet is flushed with water just before tripping the separator from molasses to wash.

A simple and effective effluent separation can be achieved by means of the "swing spout" device shown in Fig. 12-116. The spout is enclosed in a two-compartment box bolted to the molasses outlet in the curb bottom. It is air cylinder operated and moved from "green" to "wash" by the automatic control.

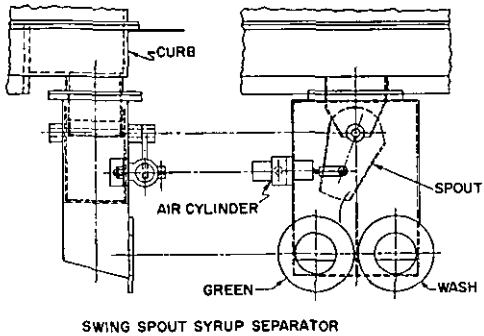

Figure 12-116. Type of syrup separators for centrifugal curbs.

Basket Bottom Closure

It is necessary to prevent as completely as possible the leakage of unpurged massecuite from the basket into the sugar conveyor. In a manually operated centrifugal, a flat steel plate usually covers the sugar discharge opening during charging and spinning. This is lifted during discharging.

This same function can be accomplished in automatic machines. Most machines are equipped with a conical basket valve and an air-operated lifting mechanism actuated by the automatic control. In some cases the lifted valve is cocked with respect to the spindle in order to provide room for the passage of the discharger.

A notably different device is the Poppleton valve developed by Broadbent. In this design the conical discharge valve is held flush with the basket bottom plate when closed by means of a compression spring. To open, the conical valve is pushed down below the level of the annular opening in the basket by a pneumatic thrustor. The lowered valve then creates a sump with a curved rotating bottom off which the sugar is spun.

Another simple device is a loading cone mounted on the spindle. This device is generally not used with white sugar because it does not provide as good assurance against massecuite leakage into the sugar scroll. If an occasional leakage can be tolerated, the cone is good since there are no moving parts to require repair.

Continuous Centrifugals

Historical

A centrifugal into which the massecuite could be continuously fed and from which the purged sugar and molasses could be separately discharged was an obvious and urgent need long unfilled. Sometime prior to 1939 a Cuban engineer, Frederico Sanchez, offered a continuous centrifugal to the sugar industry. It comprised a conical perforated basket mounted on a horizontal shaft. Movement of the sugar over the cone was induced by the rotational speed and the angle of the cone. The principle was proven but the success was limited, due in a great measure to the quality of the available screens.

In the early 60's screens with the required ultra-fine openings became available. These were made of acid-etched stainless steel or electrolytically deposited nickel. Screen openings could be made as small as 0.005 in. diameter or with slots 0.0024 × 0.065 in. and their use made the continuous centrifugal a practical machine. The screens were fragile, but experience and improvements, both in manufacture and utilization, extended the life to a reasonable degree. Chrome plating the screens extended their life, and a machine with the cone mounted on a vertical shaft also proved to be superior.

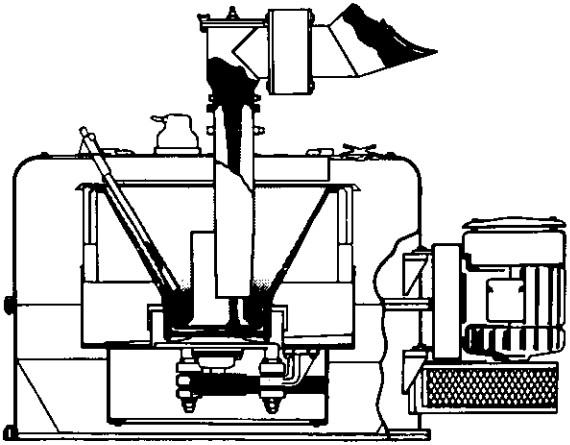

Figure 12-117. Vertical continuous centrifugal *(Western States Machine Company).*

A typical vertical spindle continuous centrifugal is shown in Figure 12-117. The machine is arranged with the large opening of the conical basket upwards. Thus the unit is fed by gravity into the loading bowl (accelerating pot) at the smaller end.

The massecuite is accelerated to the peripheral speed of the small end and moves up in response to the reaction forces generated. The material will move from bottom to top in accordance with the rate of feed. Generally the film of massecuite should be maintained at sufficient thickness to keep the "color line" (showing molasses elimination) approximately two-thirds up the screen wall. The purpose is to achieve maximum throughput commensurate with acceptable levels of molasses purity versus sugar purity.

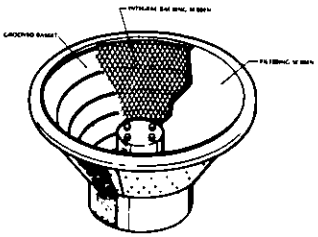

Figure 12-118. Typical cast conical basket for continuous centrifugal.

The conical basket is the heart of the machine. The one shown (Figure 12-118) is of a special alloy heat-treated stainless steel cast in a single piece and machined all over. For molasses elimination, grooves are provided on the inner surface and holes are drilled through the wall at the bottom of these grooves. A stainless steel backing screen supports the filtering screen which is held in place by the lip of the top-bolted loading bowl.

The monitor casing (curb) forms the housing and support structure for the rotating element. It contains internal annular compartments for sugar and molasses. A slinger seal prevents leakage between the compartments.

A three-point rubber buffered support system is employed for the center shaft. The top bearing is inside the basket and the bottom bearing is just above the drive pulley. The center line of this pulley coincides with the plane through the center of these support assemblies.

The bearings are forced oil lubricated and the drive is by means of a standard squirrel-cage motor mounted on a spring-loaded, self-adjusting tension base.

A feed pipe or spout connects the supply mixer to the feed control valve. A grating of steel bars is often employed in the feed system to prevent lumps from fouling the valve and possibly damaging the screen. The feed valve is usually air operated. The flow of massecuite from the valve to the loading bowl is normally enclosed to protect the flow from windage effects and to contain the steam which may be used for pre-treatment. In the center of the feed enclosure pipe is a hollow rod with facilities for the admission of water to the center of the stream of feed massecuite. Steam is added to the periphery of the massecuite stream as required.

An automatic feed control system is available which regulates the air-operated valve to maintain a preset load on the motor.

Utilization

The angle of the cone varies with different manufacturers and also with the quality of massecuite to be processed. Variations from 26° to 34° are common. The diameter across the top of the cone varies in the range of 34 in. to 40 in. Larger diameters may well be in the making. Various speeds are easily available and these may go as high as 2200 rpm.

In the beet-sugar industry in North America continuous centrifugals were first used on intermediate massecuite. Originally the purity of the effluent molasses from low grade continuous machines was too high in comparison with the batch machine.

Subsequent experience and improvement in operating procedures have shown results in capacity and quality which in many cases exceed the batch machines. The original objections were low capacity, crystal breakage, high purity molasses and low purity sugar. One single element (increased capacity) favorably affected all of these objections.

Increase in capacity means increase in feed rate without reducing the quality of sugar and molasses produced. Basic to this is a high static head on the feed valve, ample feed-pipe size and pre-treatment (fluidizing) of the massecuite below the valve. The enclosed feed pipe and central hollow rod previously described (see Figure 12-117) is one manufacturer's means of accomplishment. The careful and proper use of water and steam is critical.

The high throughput thus accomplished raises the color line, the position of which is an observable criterion of the results to be expected.

Continuous centrifugals on final crystallizer massecuites have almost completely displaced the batch machines for this service. The effectiveness of the separation is superior, the power requirement is lower, the capacity per machine is greater, and the labor is reduced. Supervision, however, is critical and cannot be ignored.

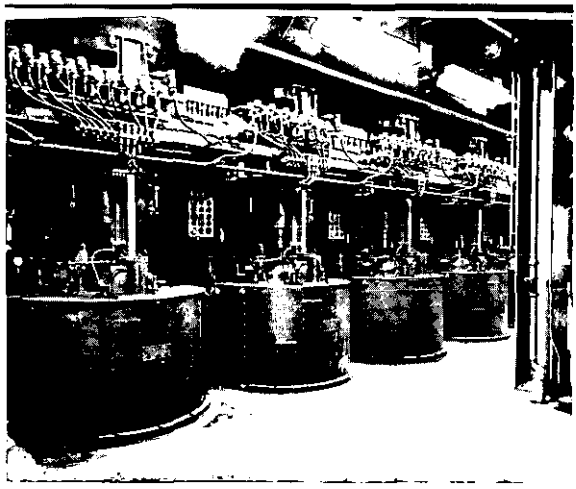

Figure 12-119. Battery of fully-automatic white centrifugals, 48 × 30, 600/1200 rpm, Shoup factory, Holly Sugar Corporation, Hereford, Texas.

Some users have increased exhaustion by boiling lower purity pans and others have, in addition, double-purged the final sugar. The fact that the continuous machines can process massecuites of smaller or mixed grain and lower purities, which batch machines cannot handle, has influenced favorably the crystallization and separation process on the low raw end.

Many attempts have been made to apply continuous machines to the finished product, white granulated and raw sugars. Some limited success has been achieved on raw sugar, but little or none on white.

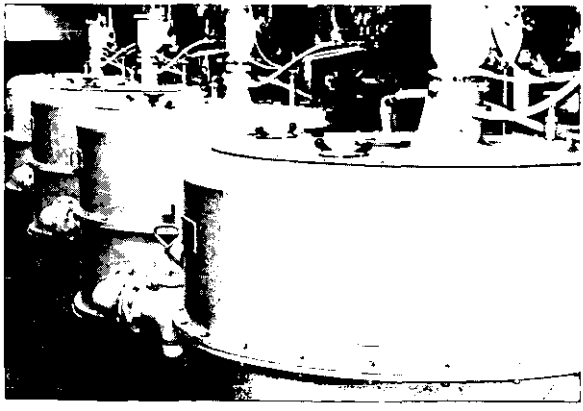

Figure 12-120. Battery of 37 × 30 continuous centrifugals for low raw massecuite, American Crystal Sugar Company, Crookston, Minnesota.

Centrifugals, both continuous and batch, should be treated as pressure vessels. The stresses to which the baskets can be exposed are very high and failures can be catastrophic. Regular inspection schedules must be established and followed. Inspection must not be superficial and at certain intervals the baskets must be removed for intensive examination.

There is much excellent literature available on centrifuges. If greater detail on the basic subject is desired, for batch centrifugals, the references appended are recommended: (9, 61a, 73, 167, 194, 199, 372, 411a). For continuous centrifugals: (1a, 51a, 61b, 15a, 143a, 175a, 175b, 185a, 190a, 190b, 194a, 219a, 239a).

Supplementary References on Crystallization For Further Reading

affination	102a, 335
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ultrasonics	448
viscosity	1c, 11b, 20, 117b, 156, 171, 173b, 187a, 190c, 272a, 370a

Abbreviations

Frequently cited reference sources for this chapter have been abbreviated as follows:

CITS (1963):	Comptes Rendus de la 12 Assemblée Generale de la Commission Internationale Technique de Sucrerie, 1963; CITS, Secretariat General, Tirlemont, Belgium
CITS (1967):	Comptes Rendus de la 13 Assemblée Generale de la Commission Internationale Technique de Sucrerie, 1967; CITS. Secretariat General, Tirlemont, Belgium.
Crystal Growth: (1949)	"Crystal growth. Discussions of the Faraday Society," No. 5, 1949, Butterworths, London, 1959.
ISJ:	*International Sugar Journal*
ISSCT: (1962)	Proceedings 11th Congress, 1962, International Society of Sugar Cane Technologists.
ISSCT: (1968)	Proceedings 13th Congress, 1968, International Society of Sugar Cane Technologists.
JASSBT:	Journal of American Society of Sugar Beet Technologists.
Princ. S. Tech. I:	"Principles of Sugar Technology" Honig, P., Ed. (1969), Vol. 1.
Princ. S. Tech. II:	*Ibid,* Vol. II
Princ. S. Tech. III:	*Ibid,* Vol. III.
QSSCT: (1962)	Proceedings 29th Conference, 1962, Queensland Society of Sugar Cane Technologists.
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Chapter XIII

GRANULATED SUGAR

Granulators*

The white sugar from the centrifugals contains from 1% to 2% moisture. This is removed in drying equipment called "granulators." Heated air is used to remove moisture and unheated air to cool the sugar. Most of the moisture in wet sugar is free moisture around the crystals, and is readily removed in the granulators down to a moisture content of 0.02% to 0.03%. Most of the moisture remaining is within the crystal and will change only by diffusion and time. The granulators also cool the sugar for storage.

The most common type of granulator is the Standard-Hersey granulator which consists of two rotary drums, one for drying and one for cooling (Figure 13-1). The wet sugar is fed into the upper end of an inclined drum. As the drum rotates, sawtooth flights pick up the sugar and discharge it as a curtain across the drum. Heated air is pulled through the drum countercurrent to the sugar. After the drying drum, the sugar is passed through a similar cooling drum with unheated air. Air from the drums contains sugar dust and must be put through a dust collector; a wet type scrubber is generally used.

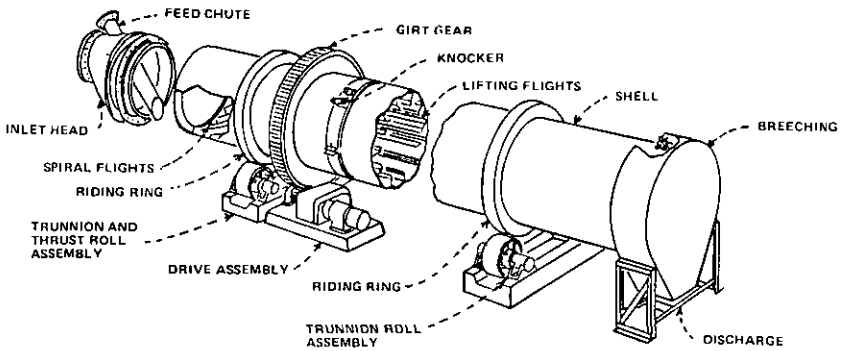

Figure 13-1. Direct-Heat Rotary Drier assembly for counterflow operation. Note difference in flights at feed end and at center of drier; spiral flights at feed end prevent leakage by accelerating forward flow. ***(Reprinted by special permission from Chemical Engineering, June 19, 1967, copyright 1967 by McGraw-Hill, Inc., New York, N.Y. 10036.) (1).***

Air for the drying drum is passed over steam coils and heated to a temperature of 100 °C to 130 °C. It is very important that the air entering the drums be free of dirt particles. This air should be filtered and sometimes an electrostatic precipitator is used. The sugar leaving the cooler drum should be cooled to 50 °C or less, and the moisture content should be .040% or less, depending on atmospheric conditions.

To provide sugar at lower temperatures for better bulk storage, refrigerator units have been used to chill water for circulating in the cooling coils of Hersey-type coolers. Care must be taken, however, to prevent water that may condense on the coils, when humidities are high, from being carried into the sugar by the cooling air. Sugar at a temperature of 35 ° C can be obtained in this manner.

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*by J.R. Corsberg

In recent years several other types of granulators have come into use. One of these is the Roto-Louvre granulator (Figure 13-2). It is a single drum with a moving bed of sugar that is dried and cooled by air entering louvres and passing up through the sugar bed. The sugar moves at the angle of repose with the sugar bed being thin at the feed end and deep at the discharge end. Therefore more hot air passes through the thin bed of wet sugar. As the bed becomes thick, less air passes through and there is less chance of overheating the sugar. The bed depth is critical, to maintain the proper air resistance.

BMA and Buckau-Wolf manufacture granulators designed to dry and cool in a single drum. The drying air and the cooling air are withdrawn together at a mid point of the drum. The hot, drying air moves concurrently with the sugar to avoid mixing dry sugar dust with wet crystals. The Buttner Turbo Tray Drier (Figure

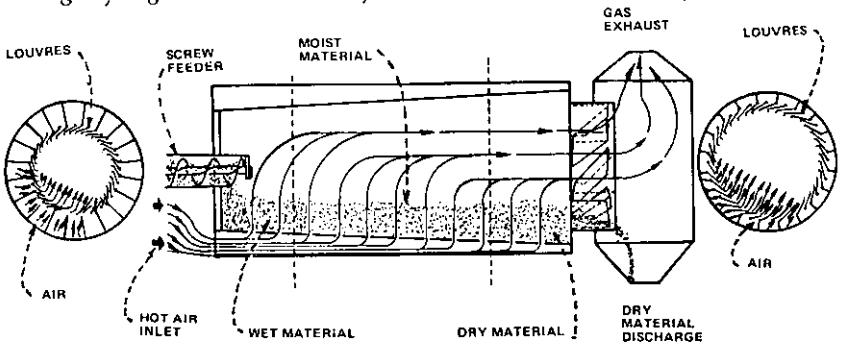

Figure 13-2. Roto-Louvre Drier has large volume of hot air going through thin bed of material near feed end, where moisture content is greatest. As bed thickens, less air passes through, thus preventing overheating. ***(Reprinted by special permission from Chemical Engineering, June 19, 1967, copyright 1967 by McGraw-Hill, Inc., New York, N.Y. 10036. (1)***

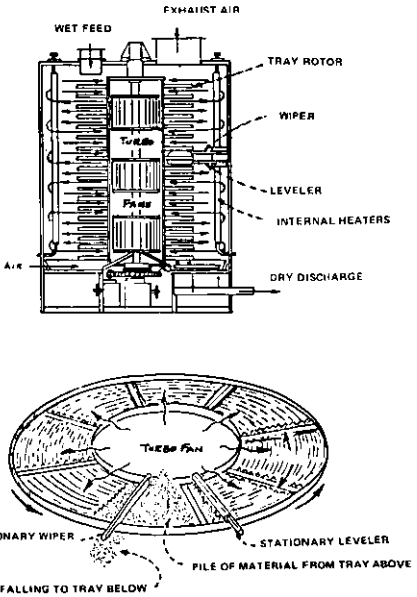

Figure 13-3. (a) Turbo Tray Drier has fans in center that circulate air over annular shelves, which contain material to be dried. Stock flows downward from shelf to shelf and exits at the bottom. (b) Shelf of Turbo Tray Drier is divided into trays. As shelves rotate, stock is wiped off onto trays below by stationary wipers. ***(Reprinted by special permission from Chemical Engineering, June 19, 1967, copyright 1967 by McGraw-Hill, Inc., New York, N.Y. 10036) (1)***

13-3) has a small rotary predrier and a large vertical cylindrical shell that contains rotating trays. The constant movement in the rotary predrier prevents agglomeration of the crystals. The predrier leaves 0.2% moisture in the sugar, which drops onto a rotating horizontal tray in the tray drier. At each revolution a scraper transfers the sugar to a lower tray. Cooling air enters the bottom of the tray system, and heated air passes through the predrier into the upper tray section. There is internal air circulation by turbine type blowers. A horizontal shelf separates the drying and cooling air. The exhaust air is removed by a fan and is nearly dust free.

These newer types of granulators have been developed to make sugar with a better appearance. They feature gentle handling of the crystal to reduce dust formation, and they try to prevent contact between wet sugar and the dust. The granulated sugar without this dust has an improved appearance with more sparkle and luster.

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Sugar Screening*

Screening, or sifting, is a process of separating particles of various sizes by means of passing the material over and through a screen cloth. The screen cloth is usually a woven fabric whose filament size and space between filaments is closely controlled. The size separation is accomplished by the particles smaller than the openings between filaments passing through the holes, while the coarser particles remain on the surface.

Screening Equipment

In the beet-sugar industry today, there are two general types of screening equipment used—gyratory and vibratory.

Vibratory. The two most-used vibratory screens are the Hum-Mer (3) by the W.S. Tyler Co., and the Rhewum by the National Screening Co. Both of these units agitate a steeply inclined screen surface by electromagnetic vibration. The Hum-Mer screen, Figure 13-4, agitates the screen 6) by means of an electromagnet 2) through rod 5). The amplitude of vibration is controlled by handwheel 1). Figure

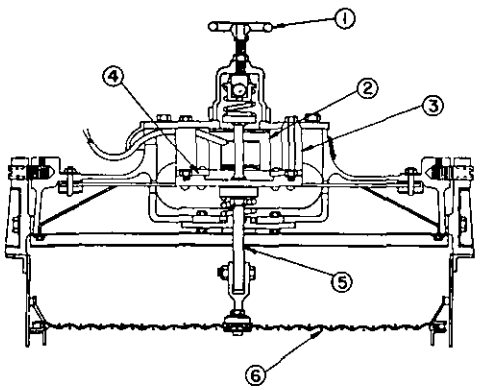

Figure 13-4. Cross-section elevation of Hum-Mer screen. *(Courtesy, W. S. Tyler Co.)*

*by C.H. Iverson and R.E. Munroe

13-5 shows a typical Hum-Mer installation. This type of screen has the advantage of low power cost, a minimum of moving parts, no lubrication, and easily varied action. The vibrator not only keeps the screen clean but classifies the feed, moving the finer sized particles to the screen surface. These types can also be obtained in multiple-screen surface units as shown in Figure 13-6.

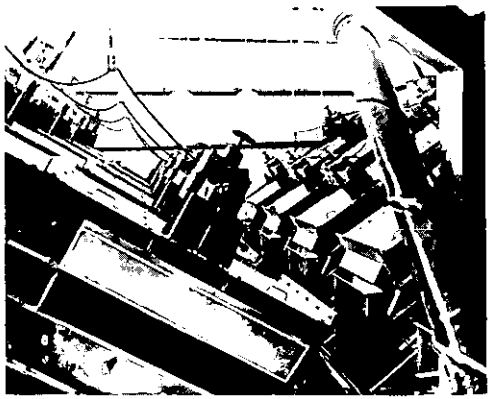

Figure 13-5. An installation of Hum-Mer screens. *(Courtesy, W. S. Tyler Co.)*

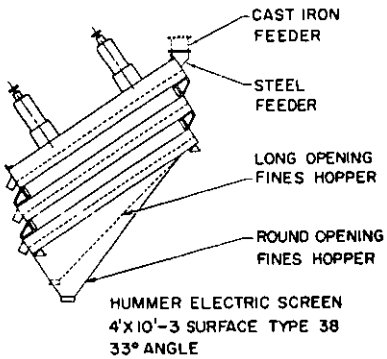

Figure 13-6. A multiple-surface Hum-Mer screen unit, for 3x10 ft surface, type 38, 33° angle.

Gyratory. The most typical gyratory types are the Rotex, manufactured by the Orville Simpson Co., and the Ro-ball (1) by the J.H. Day Co. Figure 13-7A shows a schematic drawing of a Roball single-surface screen assembly. In this unit the screen cloth presents a slightly inclined flat surface, with the sugar being fed at the upper end of the top surface of the screen cloth. A mechanical eccentric drive 1) beneath the upper end of the screen frame 2) creates a rotating action at the upper end of the screen, while at the lower end, mechanical restraints cause a reciprocating action.

This gyratory action spreads the sugar from the feed inlet 4) over the entire screen surface. The incline of the screen surface and the rate of gyration determines the rate at which the sugar moves down the slope to the discharge. A gyratory action also causes the resilient balls 13), of rubber or plastic, beneath the screen surface, to bounce against the screen surface to dislodge those crystals which are slightly oversized and tend to wedge in the openings. A coarse retaining screen below the balls prevents the loss of balls into the product.

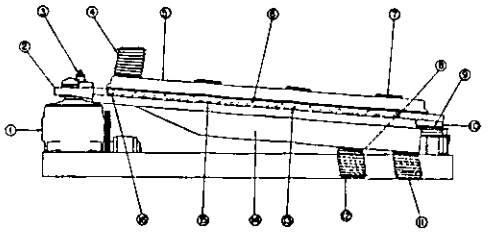

Figure 13-7A. Sketch of Roball single-surface screen assembly. *(Courtesy, J.H. Day Co.)*

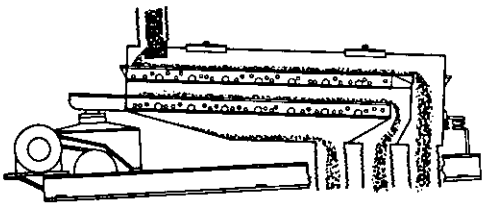

Figure 13-7B. Sketch of Roball two-deck screen assembly. *(Courtesy, J.H. Day Co.)*

In this type of screening device, the feed enters through a flexible sleeve 4). The oversized crystals move across the top of the screen surface and finally discharge over the end of the screen into the discharge sleeve 11); the undersized pass through the screen, and are carried by the bottom housing pan 14) to the other outlet 12). Figure 13-7B shows a two-deck screen which is capable of producing three products separated by two different mesh screens, the coarser mesh screen on top.

Screen Cloth

The screen cloth used in all of the above units is usually a woven metal wire mesh, although some companies find better results with silk.

Mesh sizes vary considerably between companies. *Scalping* screens vary from 12 mesh, often used on the end of the final granulator drum, to 26 mesh, used on sifting units. For specific sizes of grain, such as "Industrial fine" and "Baker's special," much finer meshes are used.

The word "mesh" denotes the number of filaments per inch. This term in itself does not specify the opening dimension unless the filament (wire) diameter is also given. For instance, a typical Baker's special screen for a gyratory sifter would be specified as follows: 64 mesh (filaments per inch), 0.0045 in. diameter wire, 0.0111 in. opening, 50.7% open area. A larger diameter wire would result in a smaller opening. The mesh size of a commercial screen cloth does not relate directly to U.S. Standard sieve specifications, because the screen filaments can be of any of many diameters, each diameter affecting the opening size, and hence the U.S. Standard sieve equivalent.

Laboratory Sieve Testing

Samples of sugar are tested in the laboratory by weighing the sample, and placing it on the top screen tray of a series of standard sieve trays. These trays may consist of the following U.S. Standard sieve sizes: 25, 30, 35, 40, 45, 50, 60, 70, 80, and 100. The trays are stacked one atop the other with the coarsest at the top and

the finest at the bottom. This assembly is agitated for a specified length of time, and the sugar remaining in each screen tray is then weighed. The analysis is usually presented as the percentage of the total remaining on a given screen surface.

The term MA, mean aperture, is the screen mesh opening that will allow exactly half of the total sample to pass through, and is a measure of the relative average size of the crystals in the sample. CV, or coefficient of variance, is a numerical evaluation of the size uniformity of the sample. A smaller CV would indicate a more uniform grain size, while a larger CV would indicate a larger percentage of crystal sizes at either or both ends of the distribution. A low CV, 28 or lower, is the more desirable product. Please see pp 511,512 for a detailed discussion of the MA-CV test procedure.

The graph plotted for any given screen test is valuable also for predicting and evaluating the performance of various industrial screens. The industrial screen may not have the same opening as the test sieve, but by reading the opening in inches instead of meshes per inch, the performance can be predicted.

Most companies collect samples of their final products, identified by each pan or strike, and screen analyses are made for the purpose of quality control and to identify errors in sugar boiling.

Screening Efficiency

The efficiency of the screening process depends on presenting the particles to the screen cloth openings in such a direction and at such a velocity that the undersized particles will readily pass through. Some of the factors affecting efficiency follow: a) The thickness of the bed of material passing over the screen cloth. Too thick a bed may prevent many of the undersized particles from ever reaching the screen surface and being thereby separated. b) Improper agitation of the material and screen cloth. Too weak an action will result in screen blinding caused by slightly oversized particles plugging the openings. Too violent an action bounces the undersized particles so that fewer pass through. c) Blinding of the screen openings. This effect can be the result of humidity or particle moisture content causing the filaments to accumulate material and the holes to grow smaller. Static electricity is another phenomenon contributing to blinding. d) The rate at which material passes over the screen. The longer the residence time on the screen, the more opportunities each particle will have to pass through an opening. e) Degree of screen inclination. This affects the effective size of the screen opening presented to the particle, and the rate at which the material passes over the screen. f) The open area of the screen. The larger the percentage of open area of a screen surface, the greater the efficiency of separation per square foot of screen surface. g) Screen openings. Efficiency is also affected by the size of the openings. The larger the openings, relative to the material particle sizes, the more efficient will be the screening action.

Screening efficiency can be determined by making a laboratory screen analysis of the feed and of the oversized product. This analysis will yield the percentage of undersized particles in the feed (Uf) and in the oversized product (Uo). These percentages may then be used in the following formula (2) for efficiency (E):

$$E = \frac{100\,(Uf - Uo)}{Uf\,(100 - Uo)} \qquad (1)$$

Sugar Screening

Sugar, as it is produced and being handled, is subject to various actions which may result in some off-standard product. These anomalies take the form of conglomerates, over- and undersized crystals usually resulting from boiling procedures,

dust from mechanical abrasion, and accretions from infrequent wash down of processing equipment. Screening, or scalping, is the accepted method of removing these accidentally produced particles from the bulk of the product.

Customer demand results in screening to meet crystal-size specifications. In this situation, the sugar is screened to produce the proper percentages of crystal sizes.

Most companies will scalp their total white-sugar production as produced. The oversized fraction is reprocessed through the melter, and the screened product either bagged or stored in bulk. If special screen sizes are required, these separations are normally made by sifting on the way to the packaging station or to bulk storage. In some instances, these specialty sugars are screened out when sugar is being moved from bulk storage for loading or bagging, or when being circulated. Nearly all bulk sugar being moved from storage is again scalped to remove any lumps which may have formed during the storage.

Sugar screening is a vital step in the production of quality sugar, and to satisfy customer demands for specially-sized sugars.

Screened Products

The Table 13-1 shows the sieve analyses of three sizes of screened products made by most companies, together with the particular screens used on gyratory sifters to produce them. Detailed data on screen specifications for various granulated sugars will be found on p 490.

Table 13-I

U. S. Standard	% Retained on Sieve		
	Scalped Production	Industrial Fine	Baker's Special
25	0.00	0.00	0.00
30	0.63	0.01	0.00
35	5.09	0.11	0.02
40	12.60	0.84	0.09
45	20.55	3.48	0.18
50	24.66	11.97	0.30
60	14.66	22.96	4.31
70	10.01	18.25	23.01
80	4.74	16.74	23.42
100	3.40	9.38	22.42
through 100	3.57	8.08	26.16
MA, in.	0.0128	0.0105	0.0072
CV	30	35	24

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Bulk Storage and Handling of Granulated Sugar*

The beet-sugar industry in the United States has been developing and improving facilities for the storage, handling, and shipping of granulated sugar in bulk form for about 40 years. The Great Western Sugar Company built the first concrete bulk bins at its Wheatland, Wyoming plant in 1930. The SSA-Weibull steel bin was

*by J.B. Powell, revised by Daniel Muller and W.D. Watson

developed in Sweden with the first being erected in 1933 for raw sugar storage. Since that time ten of the twelve beet-processing companies in this continent have installed facilities for storing from 200 000 to 1 million bags of bulk sugar at most of their presently operating factories. There are 43 beet sugar factories with bulk sugar storage, handling, and shipping facilities in North America today.

The reduction in labor cost for loading and unloading sugar in bulk as opposed to 100 lb bags, the reduction in freight costs and the flexibility of sacking the sugar produced throughout the year rather than during the campaign season only, are the chief reasons that make bulk storage and handling attractive both to the processor and to the customer.

Bulk Storage Facilities

The most common types of bulk storage bins used in this country are the SSA-Weibull steel bin and the concrete bin. The Weibull bin has the lowest initial cost for capacities above 400 000 bags. The concrete bin when constructed in units of more than one cylinder, offers flexibility for separation of several products; i.e., different screen sizes and sugars for special classes of customers.

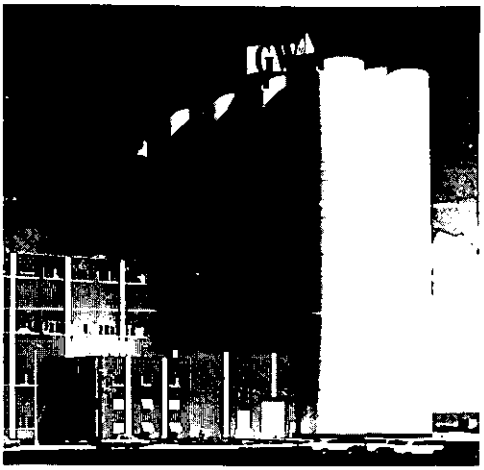

Figure 13-8. Concrete bulk storage bins, Kemp factory, Goodland, Kansas.

The Concrete Bulk Bin. Please see Figure 13-8. This type of storage bin is cylindrical in shape and has three basic parts. The bottom part or basement, which is sometimes below ground level, contains the conveyors bringing the refined sugar from the factory to the bottom of the elevators and the conveying equipment used for removing the sugar from the bins. The second part just above, is separated from the basement by a reinforced concrete floor. This part is the storage area for the sugar. Above the sugar storage area and separated from it by a second reinforced concrete floor is the third part which contains the conveying equipment that delivers the sugar from the elevators to the storage area of the bin (Figure 13-9).

These reinforced concrete structures, with wall thickness from 9 to 12 in., are constructed in a variety of sizes, ranging from 35 to 50 ft in diameter, with sugar storage heights from 80 to 200 ft. Capacities of single cylinders range from 50 000 to over 150 000 bags.

These bins are arranged in several ways. A single cylinder may have an outside square or rectangular annex constructed as an integral part, running the full

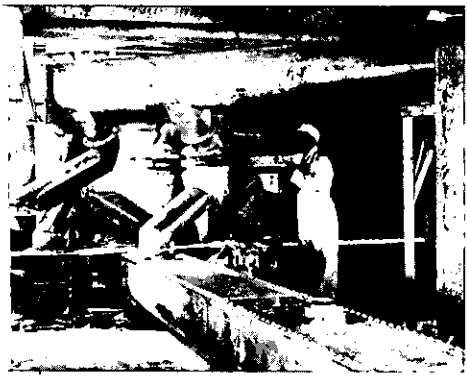

Figure 13-9. Conveyors distributing sugar to concrete bins.

height of the cylinder. This annex may house the elevators and other auxiliary equipment. Single rows of as many as 10 bins in line have a horizontal, rectangular monitor across the top of the bins which encloses the conveyors delivering sugar to storage. Clusters of four, six, and eight cylinders are probably the most common groupings of this type of storage bin. The space between a cluster of four, or "star bin" may be used to house elevators and auxiliary equipment. Clusters of six and eight bins have two and three star bins, respectively. In many cases the extra star bins are used for sugar storage. One star bin has about 60% of the capacity of one cylinder in a cluster. Elevators and other equipment can be housed in the sugar end of the main factory building, freeing all the star bins for sugar storage (Figure 13-10).

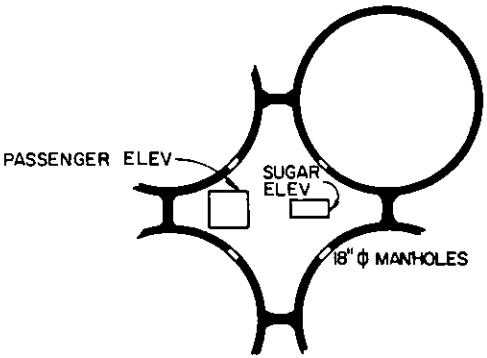

Figure 13-10. "Star bin" in cluster of four concrete bins.

Sugar is removed from the bins through openings in the flat bottom which allow the sugar to flow into screw conveyors housed in the "basement" section of the bin. Early installations have multiple openings which allow self discharging down to a depth of approximately 20 ft. The remainder of the sugar must be manually shoveled to the openings for discharge. Later installations are equipped with mechanical reclaimers that convey the sugar to one center opening for discharge into the screw conveyor beneath the bin floor. The reclaimer consists of an unhoused screw conveyor, approximately the length of the radius of the bin, mounted on a bridge or carrying structure. The bridge is suspended by cables from a track or rail around the top of the bin. This allows the reclaimer to be lowered to the surface of the sugar and to be rotated around the bin as desired. Mechanical reclaimers eliminate most of the manual labor required for emptying a storage bin.

Sugar is stored directly against the concrete walls and floor of the bins in the majority of installations. The interior concrete surfaces are hand-troweled and honed to a smooth, hard surface. Electric surface grinders are also used, followed by a thorough washing down of the new concrete surface to prevent contamination from sand and surface dust.

To prevent moisture migration through the concrete to the stored sugar, some of the early installations were coated with sodium silicate.

The Spreckels Sugar Division, Amstar Corp. has employed a bin-space lining system in all but their most recently constructed concrete bins. This lining is applied to all surfaces in contact with the sugar and provides a space for circulation of warm air up along the walls of the bins, minimizing the influence of moisture migration through the concrete to the sugar. This lining is a three-layer system mounted on 2x3 in. Douglas fir vertical studs, on the bin walls, and horizontal nailer strips on the bin floors. The first layer consists of 0.5-inch thick exterior grade plywood nailed to the studs. The second layer is a six mil thickness of polyethylene film. The third layer consists of 3/16-inch tempered presswood with the smooth surface out, to present the finished surface of the lining.

The SSA-Weibull Bin. Please see Figure 13-11. The first SSA-Weibull steel bin for the storage of refined beet sugar in this country was erected in 1959-60 by Union Sugar Division, Consolidated Foods Corporation at Betteravia, California. The walls of this cylindrical bulk bin are of mild steel plate covered with galvanized iron sheeting which forms air ducts up the outer walls. The sides are insulated, and the insulation is faced with aluminum sheeting. The steel roof is insulated and covered with asphalt roofing paper. An air space is provided under the roof beams with aluminium sheeting. The interior steel walls are protected with an epoxy coating in some cases. A 12-ft diameter center column houses elevators and auxiliary equipment. A traveling crane rotates about the center column, carrying distribution chutes for the sugar and a screw conveyor or reclaimer, which can be raised and lowered, used for carrying the sugar to the center column for removal.

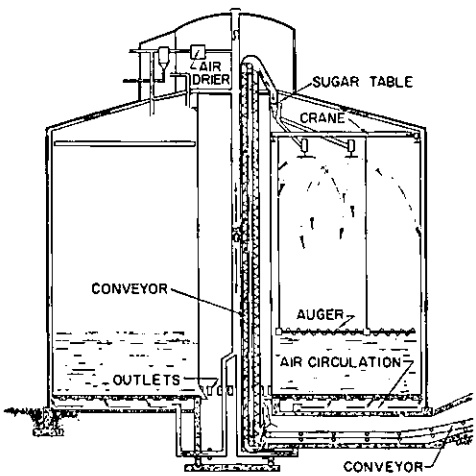

Figure 13-11. Section of SSA-Weibull bin.

Sugar entering the bin can be conveyed beneath the bin to an elevator in the center column, or overhead, entering the dome and feeding directly to the distribution equipment.

The temperature and humidity of the air circulating in the walls, ceiling, and within the bin itself are closely controlled. Capacities of this type of bin range from 110 000 to 880 000 bags, with diameters from 78 to 156 ft.

The Horizontal Bulk Bin. There is one horizontal storage bin in the United States erected for the Michigan Sugar Company at their Caro, Michigan, factory (Figure 13-12). This bin is a steel building 220 × 60 ft and has a storage capacity of 200 000 bags. It is heated with Chromalux MI heating cable in the concrete floor and sides of the building. Conditioned air is circulated through the building, maintaining a temperature of 60°-65°F with a relative humidity of 50-60%. Sugar enters the bin from a steel belt conveyor along the peak of the roof and is removed by a Sauerman drag which moves the sugar to a hopper in one end of the bin for removal by a screw conveyor.

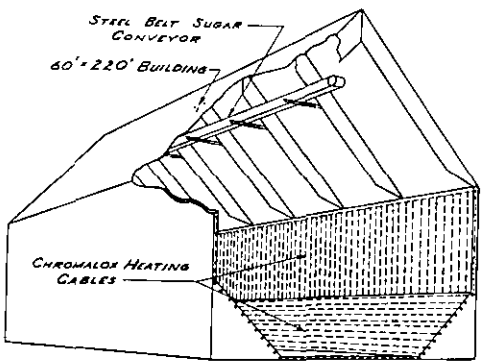

Figure 13-12. Heating cable in horizontal bin of Michigan Sugar Company at Caro, Michigan.

Sugar Conditioning. Sugar in bulk storage will become hard or caked or will become excessively wet under certain conditions of temperature and humidity. Newly produced granulated sugar is said to contain three types of moisture: a) free moisture, b) bound moisture, and c) inherent moisture (1).

Free moisture on the surface of sugar crystals leaving the centrifugal station is readily reduced in the granulators to 0.030-0.040% and the sugar appears to be dry. Bound moisture as described by S. Stachenko, W. Allen, and J. Swan (5) is due to the rapid evaporation rate in the granulators, causing some parts of the outer layer of the syrup film to crystallize at high supersaturations. A glaze is formed, trapping some of the water near the surface of the crystal. This trapped or bound moisture when moved to the surface of the crystal causes the sugar to be wet, or if rapid drying occurs while the sugar is in a static condition, severe caking will occur.

The slow release of bound moisture and the reduction of bound moisture by improved sugar boiling utilizing mechanical circulating vacuum pans is described by T. Rodgers and C. Lewis (4).

Inherent moisture is described as pockets of moisture contained within the sugar crystal, and does not appear to be a major problem in storing bulk sugar (1). One of the most important features of the SSA-Weibull bin is that it is designed to maintain the temperature of the bin and the temperature and humidity of the air within the storage area at 30°C and 70% relative humidity. Under these conditions

it is claimed that refined sugar with a moisture content of 0.07% will be in equilibrium and that caking or excessive surface moisture will not occur during storage (6).

In unlined concrete bins it is not possible to maintain equilibrium conditions of the bin walls and floors, necessitating aging or conditioning of the sugar before it is stored, or within the storage bins during storage.

Holding bins with capacities up to 24 hours retention of sugar production are employed. Bound moisture released during this retention or aging time is removed by blowing air of controlled temperature and humidity through the sugar in the holding bins.

The use of one concrete bin in a cluster for aging and then circulating the sugar to the other bins for storage is another method employed to remove moisture released before the sugar goes to static storage.

The circulation or movement of sugar in storage from one bin to another or from the bottom of a single bin and back to the top of the same bin is a technique employed to keep sugar in a free-flowing condition while in storage. Conditioned air at 65 ° - 70 °F and a relative humidity of 55% is usually circulated through the air space above the sugar in an effort to remove moisture being released from the sugar. In arid climates such as the Rocky Mountain region, humidity control is not as necessary and it is usually sufficient to circulate warm air during the cold weather season.

Explosion Prevention *(3). Before bulk sugar storage and mechanical* handling of sugar came into common use, little thought was given to the safe handling of sugar. Only small and uncomplicated systems were required and the breakage and dusting of sugar in those systems was of little consequence. In the larger and more complicated bulk sugar handling systems of today, sugar dust from the abrasion of sugar crystals in handling has become a hazard of serious proportions, both inside and outside of the equipment.

The conditions that must be fulfilled simultaneously for sugar dust explosion to occur as stated by R. Edwards and E. Cunneen (2) in their study of pneumatic handling systems are: 1) Sufficient oxygen must be present to support combustion, 9% by volume at atmospheric pressure. 2) The concentration of sugar dust must be within explosive limits: a minimum of 20 gms/cu m. 3) The ignition source must release sufficient energy to ignite the sugar dust cloud; a minimum ignition energy of 30 millijoules. 4) Edwards and Cunneen further state that a fifth condition must be fulfilled for an explosion to be initiated by an electrostatic spark. 5) The electrical field strength due to static electric charges on the sugar or the containing vessel must be large enough to cause a spark discharge. This means a minimum field strength of 20 000 volts/cm.

In the past 20 years there have been no less than seven major sugar dust explosions which have resulted in extensive property damage and loss of life. Little has been published in the literature with respect to these explosions due to legal implications, but nevertheless a considerable amount of scientific and investigative work has been carried on, both by the owners and by the insurance carriers.

In each of the above mentioned explosions, the exact location and the exact cause of the explosions have not been determined with certainty. Each, however, followed the characteristic pattern of all dust explosions which begins with a small explosion or pressure rise and proceeds at up to sonic speed until all fuel (dust) in its path has been consumed. This process can originate either inside or outside the

equipment and is supported by dust that is stirred up in the immediate vicinity. Fire follows closely the pressure rise and the result is a number of explosions that increase in magnitude as the process proceeds.

Columbia Basin Factory Installation.[1] In the rebuilding and operation of the Utah-Idaho Sugar Company bulk sugar bins at Moses Lake, Washington, protection against sugar dust explosions and fires was obtained by observing the following: isolation, housekeeping, National Electrical Code Class II 6 for electrical system and equipment, dust collection and control, static elimination, periodic inspections, maintenance and welding regulations, and fire control systems.

Isolation. Each of eight, 50 ft diameter by 130 ft high, bins is completely isolated from each other. Each has its own heating and ventilating system including Pulsaire dust collectors.

The sugar handling equipment serving these bins consists of dust-tight screw conveyors and dust-tight belt type bucket elevators. Each piece of equipment is individualized and designed to "fail safe." It is isolated from other equipment by means of a star-type feeder valve mechanically driven from the conveyor or elevator as the case may be. Each piece of equipment is also equipped with adequate blowout panels which are vented to the outside.

The bucket elevators are all equipped with static eliminators at the head pulleys.

All equipment, including diversion gates and valves, is controlled from an air conditioned control room located outside the bulk bin complex. The bins are operated by one man.

Housekeeping. Perhaps the most important precaution against dust explosions is housekeeping. All floors of the bins, the elevator shafts and the sugar screening and packaging areas are mopped each day. Dust is not allowed to accumulate on the floors, walls, or ledges, including structural steel, and equipment. With this kind of housekeeping an explosion either inside or outside of the equipment cannot travel from one area to another.

Electrical System. All electrical equipment installed in the bulk sugar bins conforms to the National Electrical Code, Class I, Group "G."

Dust Collection and Control. Dust is collected from the circulating air in each bin by means of Pulsaire filters. This dust is removed from the filter each day and returned to the plant for remelting.

A vacuum-type dust collection system using a Pulsaire filter serves the head houses, the basements and the elevator shafts in the bulk bins, as well as the sugar screening and packaging areas of the plant. Sugar and sugar dust collected in this filter is removed each day, and is remelted or stored in bags as sweepings.

Static Eliminators. Static eliminators are used on all bulk elevators and on electrical equipment where needed to permit an electrical discharge that could contain enough energy to set off a dust explosion. It was not found practical to provide static elimination for the space charge that theoretically can develop within the bins if a large movement of sugar should develop a dust cloud.

Periodic Inspections. A periodic system of inspections by the Insurance Department has been developed to insure that maintenance and welding procedures are followed and that other regulations and housekeeping procedures are maintained.

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[1]This factory no longer is operating.

Maintenance and Welding. Rules and regulations governing maintenance and welding in dangerous areas have been put into effect.

Fire Control System. The fire protection system, and fire extinguishers are inspected regularly. A Fire Department has been organized with each man assigned specific duties in case of fire. Fire drills are held regularly.

Bulk Shipments of Granulated Sugar

The granulated sugar stored in bulk bins is used for packaged sugar and liquid products, with a large quantity being shipped direct to customers as bulk granulated sugar. This bulk sugar is shipped in quantities as small as 2000 lbs in Tote bins, up to the largest rail car with capacities of 19 000 lbs.

Rail Shipments. The most common railroad equipment in use today for bulk granulated sugar is the "Airslide" car. The capacities of these cars vary from 120 000 lbs to 190 000 lbs. They are divided into compartments as shown in Figure 13-13.

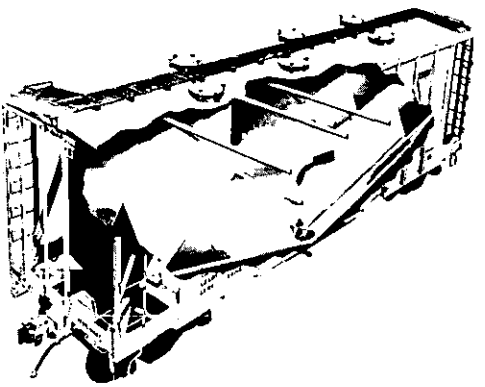

Figure 13-13. Airslide bulk sugar car.

The sugar is loaded through a number of hatches in the roof and a slinger is normally used to distribute the sugar uniformly in the car (Figure 13-14).

The unique feature of these cars is the Airslide type of unloading. The bottom of a compartment is actually a silicone-impregnated canvas membrane which is porous enough to allow air to flow through the membrane into the sugar. The car is connected to unloading spouts. The unloading hatches are opened and sugar is allowed to flow out by gravity. When the flow slows down, low pressure air, of about 6 psig, from a blower with a capacity of 400 cfm is turned into the jacketed area below the canvas membrane. The air flows through the membrane and fluidizes the sugar, causing it to flow to the outlet of the car.

Airslide cars can be unloaded at from 600 to 1200 bags per hour depending on the capacity of the equipment conveying the sugar away from the car. Hopper-bottom cars, which are unloaded entirely by gravity, are also employed. Rail cars are usually conditioned by blowing air at a controlled temperature and humidity through the car before loading begins. Sugar is weighed to rail cars in large steel bins supported on scales, or in railroad platform scales where the car is positioned for loading.

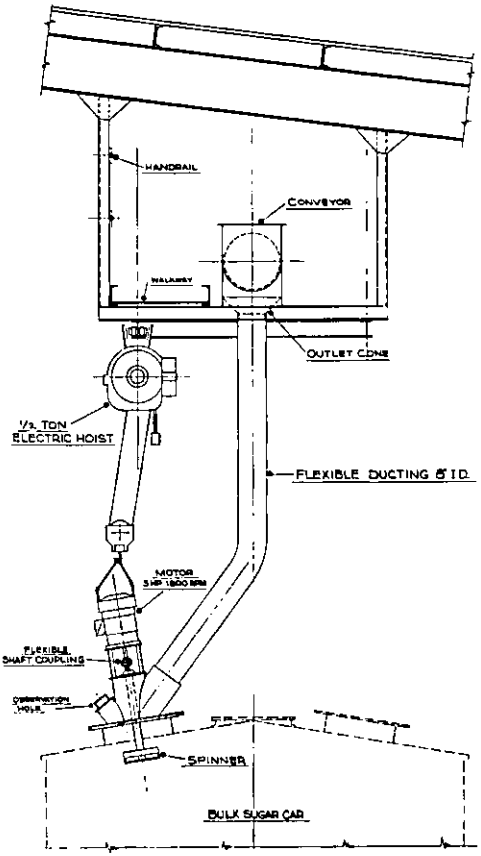

Figure 13-14. Slinger mechanism for loading bulk sugar car.

Sugar shipped in bulk rail cars will arrive at the customer's point of use in a free-flowing, lump-free condition if the proper techniques of storing and handling are followed.

Truck Shipments. Bulk granulated sugar is shipped in trucks using three general types of unloading: hopper-bottom trucks (Figure 13-15) for gravity unloading, mechanical unloading using helicoid screw conveyors running the length of the sugar compartment, and pneumatic unloading employing a compressor that places the customer's storage bin under vacuum and "sucks" the sugar from the truck.

Used air is returned to a sock-type dust collector on the truck. The sugar dust is removed from the dust collector at the loading point for reprocessing. Weighing of sugar to bulk trucks is by the same means as used for railroad equipment. The pneumatic unloading truck is widely used for customers not requiring controlled grain size. (Figure 13-16).

Portable Bins. In addition to rail and truck shipments, bulk sugar is also shipped directly to customers in portable bins commonly called "Tote" bins. These bins hold from 20 to 50 bags of sugar and are constructed to be handled by fork-lift

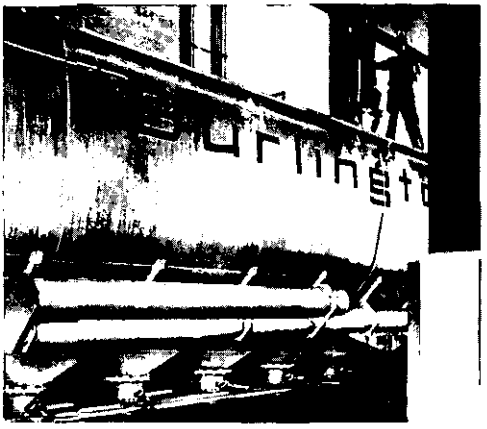

Figure 13-15. Loading hopper-bottom truck.

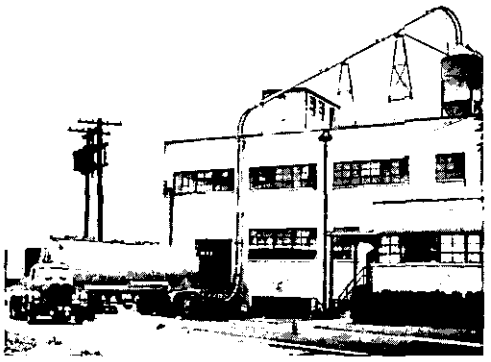

Figure 13-16. Pneumatic truck, unloading sugar to customer's bin.

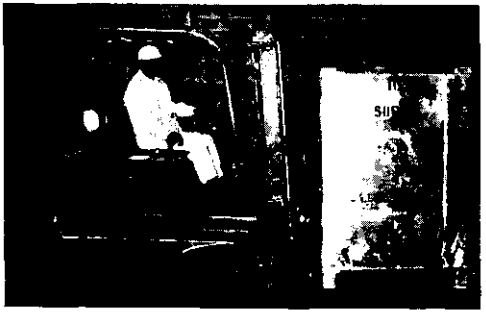

Figure 13-17. Fork-lift truck handling Tote bin.

trucks. Light-weight aluminum alloy bins are most satisfactory for this purpose, although other types of construction are used. The use of portable bins eliminates the necessity of large storage bins for customers. This method of handling and storing sugar is attractive to the customer who uses sugar at a fairly constant rate. (Figure 13-17).

Conveying and Auxiliary Equipment

A sizeable investment is required for auxiliary equipment for bulk-sugar storage installations. This includes conveyors, screens, holding bins, magnets, heating and air conditioning equipment, dust collectors, scales, and lump breakers.

Conveying Equipment. Mechanical conveyors are often used for transporting sugar from the sugar dryers to storage and from storage to sacking stations and bulk loading stations. Helicoid screw conveyors, bucket elevators of galvanized iron construction and belt conveyors are most widely used. Screw conveyors require special sealed antifriction bearings or lubricant-free bearings to prevent contamination of the sugar.

Pneumatic or air conveying systems have been employed in some instances. These are comparatively economical but have the disadvantage of a high percentage of crystal breakage. This breakage creates sugar dust, which, if not removed, affects the flowing and handling characteristics of the sugar. Removal and reprocessing of the dust adds extra cost to the sugar handling. Some pneumatic systems originally installed have been replaced with mechanical conveyors.

Screens and Lump Breakers. Scalping and sizing screens are used for sugar entering bulk storage and in most cases scalping and lump screens are used for sugar leaving storage. These screens are usually vibrating or gyratory types with multiple deck screens being employed where scalping and sizing are done.

Sugar leaving storage bins in many installations is run through mechanical lump breakers ahead of the final screening stations. For further treatment of screening, see pp. 459-464.

Weighing Equipment. Sugar entering bulk storage bins is weighed for inventory purposes using automatic dump scales. These scales vary in capacity from single and double scales of 100 lbs per dump to scales as large as 500 lbs per dump. In order to maintain accurate inventories these scales require constant attention. The system of check weighing at regular intervals is essential for accurate weights from these scales. Signal systems that indicate when a scale is stuck open, reduce the possibility of unaccounted sugar entering storage. Electrical scale control should be housed in dust-proof enclosures to insure proper operation. Dust that boils out with each dump of the scales must be kept cleaned up either manually or by installing dust collection systems. A systematic maintenance program by competent personnel will help insure an accurate accounting of the sugar entering storage.

Other Equipment. Magnets for removal of iron and steel particles are almost universally used in the stream of sugar leaving storage bins and in some cases ahead of storage.

Collecting systems for the removal of dust from conveying equipment in storage areas are necessary. Air is pulled off and the dust collected in dry cyclones, bag collectors, wet rotoclones, and electrostatic collectors. Vacuum cleaning systems for removal of dust from equipment and floors are used at many installations.

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Warehouse Operations*

Sugar storage has changed in recent years with the advent of bulk storage. Previously, all of the sugar was packaged and large warehouses were needed to hold the production at the end of campaign. A modern factory stores sugar in bulk-sugar storage bins, or in the form of thick juice in juice storage tanks. The warehouse is designed to hold only a minimum inventory of packaged sugar. This system has many advantages, such as flexibility in later packaged production, packages that are clean and fresh, lower storage costs, and the possibility of screening to meet customer requirements.

Many older beet-sugar factories still have large package warehouse facilities, usually filled to capacity by the end of campaign. These older warehouses sometimes have piles 65 bags high, which is much higher than normal in other industries. For safety at these heights, paper bags must be carefully stacked. A tie pattern similar to brick laying is imperative, and the four-layer pattern in Figure 13-18 is commonly used. It is important that the edges of the pile be vertical and straight as even a slight lean can cause a shift and the collapse of the pile.

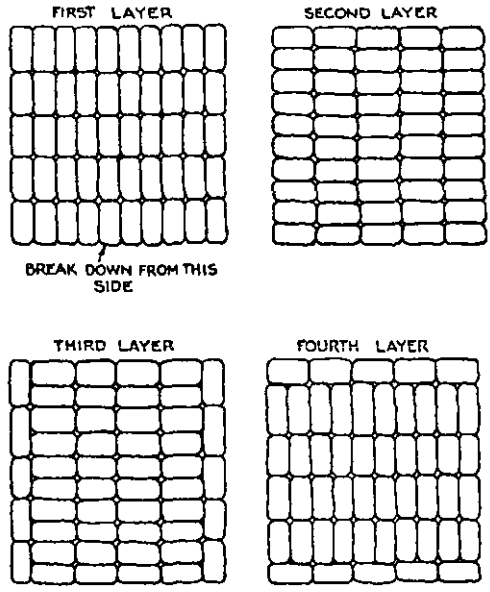

Figure 13-18. Four-layer pattern for piling sacked sugar.

Wooden racks are used on the warehouse floor to hold the sugar away from any moisture on the concrete floor and to allow air circulation under the pile. Moisture-proof paper is sometimes used under the sugar bags. The pile is built with a portable "bag stacker," which carries the bags to workers at the top of the pile. A space of two or three feet is left between the pile and the warehouse wall to permit a passage way around the warehouse walls and to allow air circulation. In cold climates the warehouse should be heated with forced-air circulation to prevent the sugar from turning hard during succeeding summer weather.

Factories with modern warehouses have different storage methods. Instead of high piles requiring manual labor, the sugar is stored on pallets and handled by lift

*by J.R. Corsberg

trucks. Palletized storage is limited in height by lift trucks; four pallets are the normal maximum. Many factories are installing automatic palletizing machines which take the bags from the production line and stack them in a pattern on a wooden pallet. An operator is needed only to supply empty pallets and to remove the loaded ones. Palletized storage can create labor reductions up to 90% of that needed for piling sugar.

Removing bags from a high pile can be a difficult operation. The last pile is left with a low spot where a chute can be placed. Bags are worked down to the chutes, and more chutes are placed as soon as possible. Portable chutes are used, made from wood or metal. Sometimes a screen or a piece of canvas is used on the chutes to provide friction to slow the bags.

Packaged sugar is transported to the customer both by railroad car and truck. Railroad cars are loaded by piling bags in them or by setting pallet loads in the cars. Palletized shipments are being used more because of many advantages, primarily labor savings at each end of the shipment. Man hours required to load or unload can be reduced from 20 hours to about 2 hours. Other advantages of palletized shipments are decreased warehousing costs, increased labor efficiency, better utilization of equipment, simplified inventory control, less damage, and elimination of hard physcial work (see Figure 13-19).

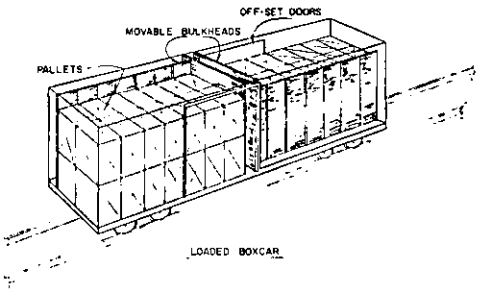

Figure 13-19. Boxcar loaded with pallets.

Special railroad cars have been developed for palletized shipments, such as the DFB cars. These cars have movable bulkheads that lock against the sides and ends of the load to prevent movement, and the cars are equipped with a sliding sill-type cushion underframe device. Palletized shipments in these special cars have been very successful. A standard pallet is desirable for palletized shipping. At present, the most popular pallet is the 48×40 in. as shown in Figure 13-20. Some pallet shipments are sent on special pallets of thick cardboard; however, these require a special lift truck attachment for handling.

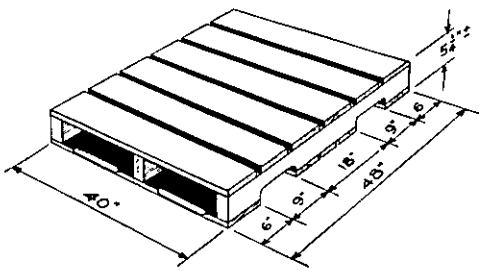

Figure 13-20. Standard pallet.

Packaging

Sugar from bulk storage may be screened, and can be sent either to the bulk truck or railcar loading station, the liquid sugar station, or to the sugar packing station. Packaging is an important part of factory operations, because about half of all sugar produced in North America is packaged (23).

Historical Notes on Sugar Packing*

J. Dedek noted that in 1846 there was a debate over the substitution of bags for wooden stave barrels, but the barrel was not finally eliminated until about 1945. A large part of all the sugar sold, up to the early part of the present century, was in the form of solid cones.

In 1889 Henry E. Smyser developed and patented a semiautomatic packaging machine, which was purchased by John Arbuckle, the sugar and coffee "king." This packaging machinery started a revolution in the food packaging industry, resulted in the building of a great refinery at 122 John Street, New York, and started a sugar war between the industrial giants H.O. Havemeyer and John Arbuckle, which did not subside until about 1900.

At about this time cartons came into use, and by 1905 refinery-filled consumer packages were cutting into the grocers' business of filling kraft paper bags by hand. Beet-sugar consumer packages appeared after 1920, cotton 5 and 10 lb sacks overwrapped with burlap, which had been inaugurated by the American (cane) Refinery in 1897.

The return to paper for sugar packaging was initiated by Benjamin Oxnard, presently retired Senior Vice President of The Great Western Sugar Company, who was then at the Savannah Refinery. In 1937 he announced that henceforth all standard packages of granulated sugar would be available in paper bags packed in a paper bale. Table 13-2 summarizes changes in sugar-packaging distribution over the last 32 years (see also Figure 14-1, p. 527).

Table 13-2 Total U. S. Sugar Deliveries, Beet and Cane, All Grades, All Packages

Calendar Year	Total Cwt.	Household(a) % of Total	Industrial(b) % of Total			
			All	Bulk Dry	Liquid	100-lb Bags
1949	136 928 000	39.0	61.0	2	5	54
1957	159 005 000	33.9	66.1	8	13	45
1958	164 209 000	35.5	64.7	10	13	42
1968	202 121 238	25.4	74.6	25.8	24.8	24
1978	197 400 000	23.0	77.0	44.3	31.5	23.6
1981	185 185 000	25.3	74.7	47.3	27.5	25.1

Notes: Source, U.S. Dept. of Agriculture. This represents 98.5% distribution in Continental United States. (a) Household grades include packages under 50 lbs, excluding 25 lb brown and powdered sugars. (b) Includes fine granulated, canners tested, bottlers, industrial fine, and coarse and powdered sugars.

Packages and Methods of Production*

Sugar packages are produced in many sizes, shapes and constructions to meet the interests of consumers, government, society, retailers, and sugar companies. Small packets holding a teaspoonful of granulated sugar, and small cubes are

*by S.L. Force and P.L. Dupes

favored by institutional-type consumers such as airlines, hotels, and restaurants for individual serving. Larger packages of 1-lb. to 25-lbs are sold by grocery stores and contain granulated sugar, powdered sugar and brown sugar. The largest packages hold from 25-lb to 100-lbs of granulated, powdered or brown sugars in several grain sizes. These packages are sold to industrial-type sugar consumers including soft drink manufacturers, candy makers and bakeries.

Sugar packaging materials, methods, and equipment will be reviewed by types of packages. These are packets, cartons, small and large paper bags, and plastic bags.

Packets

Small paper pouches of granulated sugar in teaspoon amounts, for individual servings, are called packets. The production of sugar packets began in the U.S. in about 1940. Today, billions of sugar packets are sold each year in the U.S. alone (12).

Packets are made from continuous, wound rolls of kraft paper, usually bleached white. The paper is printed on one side with the customer's name or artwork, and coated on the opposite side with a heat sensitive polyethylene to provide sealing characteristics. The preprinted and precoated paper is received from the paper supplier in rolls about 3.5 in. wide.

Packets are formed, filled and sealed on several available types of machinery. On all machines, the paper is first unrolled and folded in half to form a V-shaped continuous web of paper about 1.75 in. wide. The folded edge serves as one side of the packet. Two other sides of the packet are next formed by sealing together portions of the folded, inner-coated paper using a heating plate at a temperature of 250 to 550° F. The heat-sensitive coating seals quickly under heat. Heat does not harm the paper because the length of time the paper is in contact with the heating plate is very short. The resultant three-sided pouch is then filled with a premeasured amount of sugar weighing either approximately 1/6 or 1/4 ounce. Packets are sold by weight. Since the density of sugar is variable, the measured volume of sugar must initially be set high enough on the machine to always assure a given minimum net weight. The alternative of weighing these minute quantities of sugar is too slow. After filling, the top of the paper is heat sealed. The continuous web of packets is then sheared off at the heat seals about every 2.5 in. to form individual packets. The finished 1.75x2.5 in. packet is packed in cartons and shipping containers.

Packet machines vary in design and speed of operation. See Figure 13-21. The intermittent-motion type machine (16) pulls the paper roll, one packet at a time, through the heat sealing, filling and shearing units. The units reciprocate and are actuated by cams driven by various shafts. This machine can produce at the rate of 180 packets per minute. One man can operate several machines. Another machine (6) is based on the continuous-motion principle, in which the paper web is continuously pulled through rotary heat sealing, filling and shearing units, similar to film passing through a motion picture projector. The continuous motion concept results in significantly higher production rates of over 500 packets per minute.

The preprinted, continuous web of paper must be kept "in-register" to assure that each printed design is reasonably close to the center of each formed packet. To accomplish this, the paper supplier prints small, evenly spaced registration marks on the web adjacent to each printed packet face. A photoelectric eye on the machine scans the paper web for registration marks and actuates a registration control system accordingly. Registration is automatically controlled by slightly adjusting the rate at which packet paper is fed through the machine.

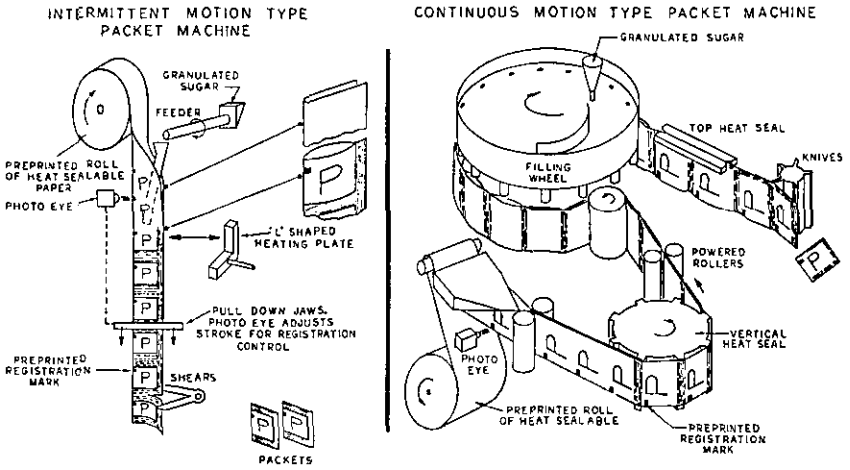

Figure 13-21. Sugar packet production.

Cartons

Although the carton-type sugar container is considerably more expensive than the more commonly used paper bag of comparable size, the carton is popular for holding small 1-lb or 2-lb quantities of granulated, powdered or brown sugars. The carton has several advantages over paper. It has sales appeal because it is easier to grasp and reuse. It is more resistant to punctures and it offers a better moisture-vapor barrier for the sugar. Its stability gives it better stacking characteristics in the warehouse and it is easier than the bag to open, fill and convey on packaging lines.

Numerous paperboards of various weights and constructions are available. One popular board used for sugar cartons is chipboard, made from a mixed-waste paper pulp. A clay coating is applied to one side to provide a surface suitable for high quality printing. Cartons arrive at the sugar plant from the supplier in a flat form, already cut, folded, scored and printed.

Cartoning Granulated Sugar. Granulated sugar is a comparatively free-flowing product. It fills packages more rapidly than powdered or brown sugars and is less vulnerable to caking or lumping. Granulated sugar is cartoned in 1-lb and 2-lb quantities. One firm has marketed granulated sugar in a 4-lb milk carton, but this is not common. An aluminum pour-spout is used on the 1-lb and 2-lb containers as a consumer convenience.

A popular cartoning machine is the continuous motion type machine (17). Chipboard carton flats are first opened and the bottoms are automatically glued. A separate machine inserts pour spouts near the top side of each carton. The cartons are then continuously filled with preweighed amounts of sugar under a series of balance type scales. The carton top is then glued and sealed. Sealed cartons are generally collected in units of 24 or 30 and packed in corrugated, case shippers. Using the net weighing principle, production rates of 180 cartons per minute are noted. Higher rates to 260 cartons per minute (18) can be realized if the sugar is volumetrically measured, or filled into measuring cups and leveled off, instead of being weighed. However, because of rapid density variations of sugar at these high

production rates, package-weight control is seriously sacrificed unless instrumentation is added to continuously monitor density changes and feed back signals to adjust the amount of filling accordingly. For the smaller sugar packager, other lower cost equipment (15) using net weight scales is available at production rates under 100 cartons per minute.

Cartoning powdered and brown sugars. The absorption of moisture by powdered sugar and its subsequent drying creates caking or lumping. Conversely, the loss of moisture by brown sugar results in hardening of the product. Therefore, a liner inside the carton is desirable as a moisture-vapor barrier for these special products. Waxed glassine paper is commonly used as the liner material.

The carton and inner liner are formed on a double package maker (16). See Figure 13-22. The glassine paper liner is first cut from a large roll, and glued and wrapped around a forming block. The machine has eight forming blocks on a turret that indexes to several forming stations on the machine. The liner end is folded on the block and the carton shell is glued and formed around the liner and block. The carton end is then folded and glue-sealed on the block. The lined carton is ejected from the block and conveyed to the filling and gross weighing scales.

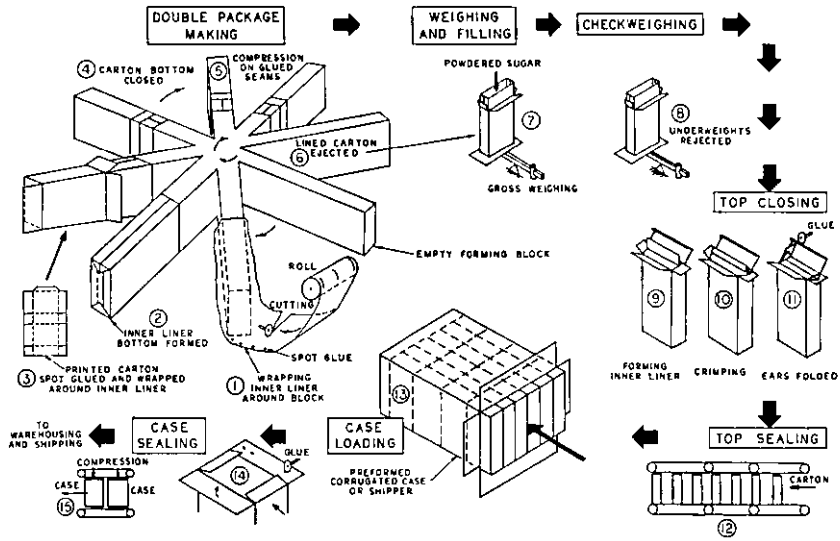

Figure 13-22. Packaging powdered sugar in 1-lb cartons.

The filling equipment (16) includes auger feeders to force the hard-to-handle product into the filling spout above the carton. The carton at time of filling, rests on a balance scale and the product feed is cut off when balance is reached. The scale compensates for the weight of the empty carton. This gross weighing method is slower than the net weighing method used with granulated sugar, but it is necessary due to the poor flowing and sticking characteristics of powdered and brown sugars (4). Further checkweighing, with automatic rejection of underweights, is usually accomplished downstream of the filling equipment. 1-lb and 2-lb carton weights can be controlled within a small fraction of an ounce. The filled carton next proceeds to a top closure machine (16) which closes the inner lining and carton flap. The glued carton travels through a drier conveyor to allow time for the glue to set. Cartons are

then accumulated in groups usually numbering 24, and are packed in corrugated cases or shippers. The shipper is glue sealed and conveyed to the warehouse prior to shipment.

A 1-lb powdered sugar carton line as described can produce 60 cartons per minute. Two men can usually operate the line. The packaging of 1-lb brown sugar cartons is similar except a foil or other type liner material is at times substituted in lieu of glassine paper. The sticky nature of brown sugar makes filling and gross weighing operations difficult.

Small Paper Bags

The widely used small paper bag, holding 5-lb or 10-lb quantities of granulated sugar, is made of two walls of kraft paper. Kraft paper is the most economical and most used flexible packaging material (19). The outer wall is kraft paper bleached white, or a color to provide sales appeal and printability to the surface. The inner wall is unbleached natural brown kraft paper and provides strength to the package.

Bag Making. Unlike packets, cartons and plastic bag packages, empty paper bag containers are not always formed directly on the packaging line. The two-wall bag can be furnished premade by a bag supplier, or it can be fabricated from paper-roll stock on a double bag-maker machine installed on the packaging line. The method selected depends on many conditions. Each bag-making machine is expensive and each machine can produce only one size of bag. However, in-plant bag making eliminates the need of paying a bag maker and no additional packing-line labor is required to operate the machinery. Bag making equipment is generally more suited to the sugar company with a minimum number of 5-lb bag and separate 10-lb bag packaging lines, with each line operating on heavy year-around schedules.

If bags are made by an outside supplier, they must be made to fold flat for shipment to the sugar plant and must be made to open easily on automatic bag filling equipment. If the bag is to be sewn closed after filling, the bag has folded inside gussets to ease forming and pinching of the bag top prior to sewing. (Figure 13-23.)

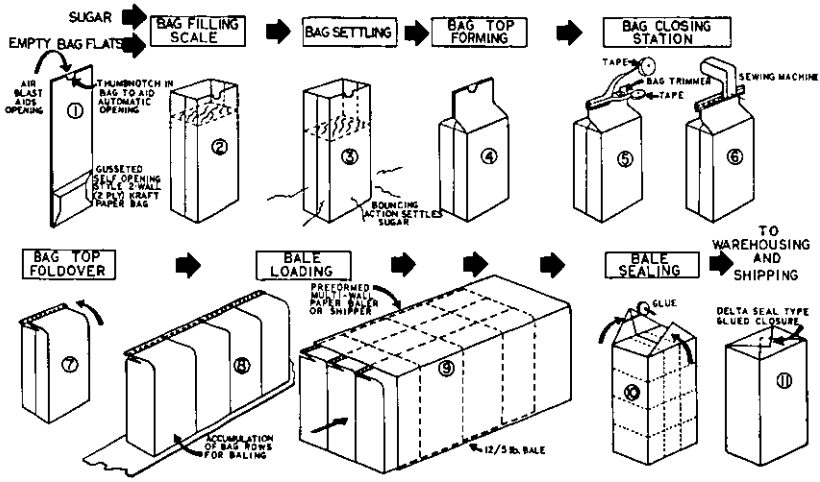

Figure 13-23. Packaging beet sugar in 5-lb or 10-lb paper bags.

Bags that are to be glue sealed after filling do not usually require gussets. When bags are fabricated on the packaging line, rolls of paper are cut, glued and wrapped around forming blocks on a machine (13,20) similar in operation to the double-package maker used to form lined 1-lb cartons. However, pre-printed paper rolls are fed in the machine instead of individual precut carton blanks, and, like packets, registration control is necessary.

Bag Filling and Closing. (Figure 13-23). Formed bags, regardless of design or supplier, are filled under a series of net-weight scales in which a known weight is balanced through a lever against an unknown weight (1,2,4). Weights can be controlled to within a small fraction of an ounce. Filled bags are lightly vibrated to settle the loosely packed granulated sugar and conserve bag materials. The bag top is then glued, folded over, and the glued seam is compressed to aid in setting of the glue. If the filled bag is to be sewn closed, the open top must first be shaped or formed flat by a bag-top-former machiner. Excess paper is then cut off, the amount depending on the height of sugar in the bag, or sugar density. Before sewing, narrow paper tape can be used as shown to reinforce the stitch and decrease the likelihood of any product leaking out. The sewing operation is accomplished by heavy-duty sewing machines (3) that make about five stitches per inch of bag top.

With or without bag making equipment, 5-lb packaging line peak production rates vary from 30 bags per minute to 70 bags per minute depending on number of scales, number of bag closing machines and baling rate. Several men are required to operate the line. 10-lb rates are about 20% lower.

Baling. The closed sugar bags are collected in units of twelve 5-lb bags or six 10-lb bags and are packed inside larger kraft-paper shipping sacks called bales. Similar to the small bag, the bale can either be fabricated and printed by a bag supplier, or made directly on the packaging line. If purchased, the bale is sealed on one end only and is constructed to fold flat for shipment to the sugar plant. The preformed bale is manually opened and the open end is inserted over a loading spout. The accumulated unit of bags is then pushed into the spout and bale. The full bale is ejected from the spout and the open end is glue sealed.

As an alternative, the bale can be formed directly on the packaging line, using an overwrapping machine (9,21). This machine automatically unwinds kraft paper from a large roll, cuts the paper, applies glue to the paper, wraps the paper around the unit of bags and glue seals the paper wrapper. High quality printing is unnecessary on bale shippers and the bale paper is not bleached. Relatively simple, one color printing attachments can be furnished on the overwrapping machine. The sealed bales are conveyed to the warehouse for storage or shipment.

Large Paper Bags

Large, heavy duty paper bags are produced in sizes from 25-lb up to 100-lb. For strength, this group of bags requires three or more walls of unbleached kraft paper. A polyethylene moisture barrier can be sandwiched between the paper walls on bags holding powdered and brown sugars that tend to cake or lump.

It is not practical to fabricate large multiwall bags directly on the packaging line. Two general types of bag construction can be furnished by the bag supplier. These are the open mouth and the valve bag (8). The most common is the open mouth bag that requires closing after being filled with sugar. The most popular closure is the sewn closure similar to that described for the 5-lb paper bag. The open mouth bag can also be supplied with solidified adhesive preapplied near the top of the bag. Closure of this bag is obtained on the packaging line using a heating plate instead of a sewing machine. The plate reactivates the glue and provides a

glued-type closure. This operation is simpler than sewing, but the closure does not open as easily as the sewn closure. The valve bag is completely fabricated by the bag supplier and a closing operation is not necessary. A one-way check valve is incorporated in the bag which allows filling on a special bag filling machine. The machine has a filling tube about 1.5 inches in diameter that penetrates the valve. When removed, the valve shuts tight. Although bag closing operations are eliminated, the valve bag is somewhat more expensive and filling takes longer for each machine because the filling tube has a much smaller cross sectional area than the totally open top or open-mouth bag.

The typical granulated sugar bag packaging line consists of net weigh scales (1) which preweighs the sugar, a filling spout, a bag-holder device, a bag shaker to settle the sugar and a sewing machine (3). A man is usually required to manually form the bag top prior to sewing, similar to that which is done automatically for the small 5-lb sewn bags. Unlike smaller paper bags, large paper bags are not packed in bales or shipping containers. Production rates can exceed ten 100-lb bags per minute. The 100-lb equipment is usually convertible to other smaller bag sizes including 25-lb, 40-lb, 45-lb, and 50-lb.

As mentioned under cartoning, powdered and brown sugars must be gross weighed when filling (4). A powdered rotary feeder (22) delivers the product to the bag until the scale reaches balance. Upon reaching balance, the scale shuts off the product feed. A 100-lb bag of powdered sugar handles like a bag of feathers. To conserve bag material and ease handling, vibration is necessary to settle and deaerate the milled-sugar product before the bag is closed. Bags are checkweighed and sewn closed. One or two men are required to operate the typical line, and production rates on powdered sugar are about two 100-lb bags per minute depending on extent of aeration, and whether product has been freshly milled or stored before packing.

Plastic Bags

There is a small, but growing group of all-plastic sugar containers. The most common bag material is polyethylene. Polyethylene film is transparent, it can be heat sealed, it can be printed and it is a good moisture vapor barrier. The single-wall film package is of simpler construction and is easier to form, fill, and seal than its competitor—the lined 1-lb carton. Poly-film bags, called pillows, include 2-lb powdered sugar, 2-lb brown sugar, 2.5-lb brown sugar and 5-lb granulated sugar. The latter package is not common, however.

The pillow-type bag can be made on a vertical form, fill and seal machine (14) in several ways similar to the intermittent-motion type packet machine. See Figure 13-21. A web of plastic film is wrapped around a filling tube. The seam and bottom are heat-sealed and the formed bag is cut from the continuous film web.

In addition to pillows, polyethylene is used as a heat-sealable coating on sugar-packet paper, and is inserted between paper walls of large paper bags of powdered and brown sugar to provide a moisture barrier. Plastic films are gaining use as a material to overwrap unitized pallet loads of packaged goods. The film shrinks under heat and provides a tight, protective wrap. There are also many new single-wall and multiple-layer, plain or laminated plastic films on the market, including a woven polypropylene fabric that can be sewn closed. Plastics will continue to gain in use in the sugar industry.

Package Weight Control*

The net weight of any material and its container is subject to scale performance variations.

The common, known variables affecting automatic sugar weighing are: a) the grain size of the sugar, associated with MA, CV (see pp 511,512), and percent moisture; b) environment, through dust, vibration, temperature, relative humidity and air currents; c) the sensitivity and condition of the scales; d) the setting and condition of the scales, bulk and dribble (final) feed; 3) the length of fall of the last portion of the dribble feed; f) the mechanical condition of the sequencing devices, linkage, moving parts, and pivots; and g) the shape, size, and location of the scale supply bin. In addition, there are unknown variables.

To protect the interests of the consumer against excessive underfill, and the interests of the producer against excessive overfill, good weight control is required. This means knowledge, effort, time and money, and the closer the control limits, the higher the costs.

Table 13-3 shows the dollar values of various overfills in ounces, pounds, and grams, for 10 million lbs of sugar packed in standard grocery-sized units.

Check Scales. The check scale is a precision instrument, and may be the most valuable single tool in the package line. Most check scales are of the over-and-under type, and accurate check weights are needed. Weight variations must be controlled on the basis of sugar in the package, but the most frequent practice is to weigh the whole finished package.

Tare Weights. The weight of the bag, plus the closing material, minus any "clip-off," is used to determine the tare weight of the bag. Some firms determine the tare weight by averaging the weights of 15 to 20 bags, and subtracting the weights of twine, tape, glue, and correcting for the weight of the clip-off. Some use an empty bag which has been hand-sewn along the same line at which the factory packaging machines sew the bags, changing the tare bag with each lot of bags. Others average the weights of several bags from each lot, making the appropriate corrections for tape, twine, and top clip-off.

Statistical Weight Control

Various statistical weight-control procedures are used. The reliability of the results, however, depends more on how well the procedures are followed, than on the particular technique used. The details of these procedures are readily available in the literature, and their coverage is not possible within the space limitations of this book.[1]

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*by S.L. Force

[1]See refs. (5, 7, 10, 11, 24, 25, 26)

Table 13-3 Packaged Granulated Sugar - Overfill Costs

Overfill Each Package				Cost				
Ounces Avdp.	Decimal Eq. Ounces Avdp.	Decimal Eq. Pounds	Grams	10,000,000 1# Packages	5,000,000 2# Packages	2,000,000 5# Packages	1,000,000 10# Packages	100,000 100# Packages
1/64	.0157	.00098	.445	$ 980	$ 490	$ 196	$ 98	$ 9.80
1/32	.0313	.00196	.889	1 960	980	392	196	19.60
1/16	.0625	.00391	1.771	3 910	1 955	782	391	39.10
3/32	.0938	.00586	2.658	5 860	2 930	1 172	586	58.60
1/8	.1250	.00781	3.544	7 810	3 905	1 563	781	78.10
5/32	.1563	.00977	4.432	9 770	4 885	1 954	977	97.70
3/16	.1875	.01172	5.315	11 720	5 860	2 344	1 172	117.20
7/32	.2188	.01367	6.203	13 670	6 835	2 734	1 367	136.70
1/4	.2500	.01563	7.088	15 630	7 815	3 126	1 563	156.30
9/32	.2813	.01758	7.975	17 580	8 790	3 516	1 758	175.80
5/16	.3125	.01953	8.859	19 530	9 765	3 906	1 953	195.30
11/32	.3438	.02148	9.748	21 480	10 740	4 296	2 148	214.80
3/8	.3750	.02344	10.631	23 440	11 720	4 688	2 344	234.40
13/32	.4063	.02539	11.518	25 390	12 695	5 078	2 539	253.90
7/16	.4375	.02734	12.403	27 340	13 670	5 468	2 734	273.40
15/32	.4688	.02930	13.290	29 300	14 650	5 860	2 930	290.30
1/2	.5000	.03125	14.175	31 250	15 625	6 250	3 125	312.50
3/4	.7500	.04688	21.263		23 440	9 376	4 688	468.80
1	1.0000	.0625	28.350		31 250	12 500	6 250	625.00
1½	1.5000	.0938	42.525					938.00
2	2.0000	.1250	56.700					1,250.00
8	8.0000	.5000	226.800					5,000.00
16	16.0000	1.0000	453.600					10,000.00
32	32.0000	2.0000	907.200					20,000.00
64	64.0000	4.0000	1,814.400					40,000.00

Sugar value = 10¢ per pound

Trends in Packaging*

There are two general trends in packaging. One emphasizes *cost control.* This trend includes a diversity of subjects, the discussion of which is beyond the scope of this text. Package and material testing, statistical analysis and control of package weights, equipment development, maintenance scheduling, spare parts inventory programming, budgeting of packaging operations, packaging personnel training programs, sales forecasting and production planning are tools used in the effort to minimize overall costs. The other trend of increasing importance is toward design of packages with more *function* for consumers and for production, warehousing, distribution and disposal efficiency. Adoption of easy-opening features on packages, strict quality control procedures, moisture barrier materials, the Fair Packaging and Labeling Act and concern over disposability of packages are only a part of this second important trend.

The packaging picture is big and is getting bigger. It is easy to solve one particular packaging problem, only to adversely affect another part of the complex production-warehousing-distribution-consumer-disposition chain. A systems approach of standing back, evaluating and attempting to solve each packaging problem in its entirety, with regard to both cost and function is required. To aid in achieving this responsible goal, an increasing number of companies in many industries, including sugar, have assigned broadly trained packaging personnel to their staff, and have created packaging committees that are made up of or have access to personnel from all departments of the company as well as to consumers, retailers and other groups outside the company.

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Chapter XIV

SPECIAL SUGAR PRODUCTS*

Although crystals remain the basic form of sugar production and consumption, many variations in the marketed products are now widely accepted. Primary and secondary manufacturing facilities convert granulated sugar or partially refined sugar into specialty products found more suitable to industry and the convenience package buyer. These products are delivered to the consumer in packages varying in size from a small cube to 190,000 lb bulk railcars. The products may vary in form from sparkling crystals to dark liquids, and from sucrose syrup to fully inverted syrup. Blends, both liquid and dry, are now obtainable containing sucrose and corn industry sweeteners.

Sugar distribution patterns as shown in Figure 14-1 (6) illustrate the trend from family purchases to industrial purchases and from bagged granulated sugar to specialty products.

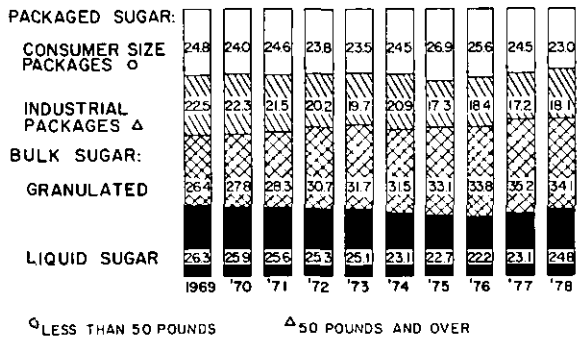

Figure 14-1. Deliveries of sugar in packages and bulk as percentages of total U.S. deliveries, 1969-1978 (6).

Granulated Sugars

Classification by Grain Size. Refined dry sugars are characterized by excellent keeping qualities, high resistance to insect infestation, and exceptional stability to deterioration by microorganisms. There are no rigid industry-wide standards or names for the various granulations. The multiplicity of screen sizes shown in Table 14-1 reflects the sugar industry's experience in meeting customer needs. Missing from the list is Bottlers Special sugar offered by most companies. It may fall in the coarse classification or the manufactureres classification, but it must meet the soft drink industries quality standards (See p. 563).

Large Crystal Specials. There is a small but steady demand for white sugar having much larger crystals than those of fine granulated sugar. This class of sugar, called the large crystal specials, is generally sold at premium prices.

Large crystal specials are rarely produced by beet-sugar companies. Cane-sugar refiners produce them from high-purity, bone-char filtered liquors of low color. The large crystals are grown in the vacuum pan, usually by discharging some of the intitial massecuite, and growing the remainder to the desired size. As the crystals grow, the ratio of surface to weight decreases markedly. Washing during centrifug-

*Original by E.T. Winslow, amplified and rewritten by C.E. Purvis.

Table 14-1 Comparative Sieve Analyses of Various Grain Sizes and Brands

USBS Sieve Size			Large Crystal Specials		Coarse Granulated				Manufacturers Granulated		Fine Granulated		Extra-Fine Granulated		Bakers Special		Crushed Grain		Powdered		
Meshes Per Inch	Openings Inch	Openings Microns	A	B	A	B	C	D	A	B	A	B	A	B	A	B	A	B	A	B	C
12	.0661	1680	5.7																		
16	.0469	1190	59.1																		
20	.0331	840	27.1	9.3	18.0	11.3	5.0	3.1	0.0	0.0											
30	.0232	590	7.3	49.2	49.2	59.8	55.0	31.6	13.4	5.5	1.3	0.3	0.0	0.0							
40	.0165	420	0.5	37.6	24.0	25.6	34.0	56.7	72.7	44.5	18.4	11.9	9.3	3.0							
50	.0117	297			5.0	3.2	5.3	7.8	11.8	37.0	37.4	39.1	39.2	36.0	5.0	1.5					
70	.0083	210							1.9	10.5	29.1	32.6	33.5	41.0	45.0	48.5	30.4	17.3			
100	.0059	149								1.9	12.2	13.7	13.0	15.0	37.0	35.5	40.8	37.1	0.5		
140	.0041	105													10.0	12.0	22.0	19.1	1.8	0.0	
200	.0029	74																	9.2	3.7	0.1
325	.0017	44																	34.2	9.3	1.1
		Percentage finer than last screen reported above	0.3	3.9	3.8	0.1	0.7	0.8	0.2	0.6	1.6	2.4	6.0	5.0	3.0	3.0	6.8	26.5	54.4	87.0	99.8
		Mean Aperture - Coefficient of Variance																			
	MA		1271	660	680	668	625	553	480	420	325	312	300	275	210	165					
	CV		22	21	27	19	21	16	20	26	32	31	32	28	27	28					

ing is so efficient, that the washed crystals are slightly purer than regular granulated sugar. These crystals are then passed through special drying equipment which avoids scratching the surfaces. The dried sugar has a very high luster, and actually sparkles in strong sunlight.

Candy makers buy these sugars because of the low ash content and the resistance to foaming in open-kettle cooking. Some champagne makers still use large grain sugars, but this is believed to be more from tradition rather than necessity (10). Beet-sugar factories could produce large crystal specials from remelted granulated sugar though the cost would be high, the demand low, and the storage and delivery problems severe. A few companies have produced coarse sugar which approaches the smallest of the large crystal specials in crystal size, but is similar to fine granulated sugar in other respects.

Other Granulated Sugars. Each granulation "specialty" is used by a particular type of food processor. The coarse granulated is used for sanding jellies in the confectionry plants, for sanding cookies, and for the manufacture of syrup in the hot process. By this is meant the addition of sugar to hot water in a stirred melter. When the mixing action is severely limited a larger-grained sugar wets more rapidly, gets into circulation more quickly, and shortens the time necessary for complete solution.

Manufacturers granulated is the sugar used by most industrial customers when a specific grain size is unnecessary. It is acceptable for bulk shipments, and is widely used in the baking, canning, and confectionry industries.

Fine granulated sugar is generally used in most retail packages. It is considered to be the granulation for distribution unless a specific grain size is requested.

Extra fine granulated may be preferred for its appearance in individual service packets served in restaurants. Many producers of prepackaged gelatin mixes, cake mixes, or pudding mixes use this type of sugar because it blends more easily with the other ingredients. This is particularly true if a water or alcohol solution of the flavoring is to be added while the mixture is being tumbled in a dry mixer. The demands in this industry are confused because of the preference of some for fine granulated, and of others for extra fine granulated.

Bakers Special is a screened product made from the fines which are present in all granulated sugar. It is usually made from the small percentage passing through the finest screen in the sugar-screening apparatus, and is packed without the addition of starch or other anti-caking agents. It is preferred by some dry-mix manufacturers over the extra fine granulated discussed above. Bakeries use this in making cake batter because of its more rapid solution rate in the sponge dough. It is particularly desirable in the manufacture of high-rising cakes.

Bakers Special may be used in chocolate products where the size of the sugar must be further reduced by milling with the other ingredients. Bakers Special is largely an industrial sugar and is sold in 100 lb bags. Sugar of this type in smaller packages is commonly called superfine, caster (3), or bar sugar. Uses of superfine in the home are in cake batters, in some icings, in mixed drinks, and for sweetening fresh fruit.

The processors making a large grain Bottlers Special are trying to please the soft drink bottlers with a sugar which will dissolve readily in the hot process. Those making a Bottlers Special of fine granulated size are attempting to satisfy the soft drink manufacturers who dissolve the sugar in cold water. In both cases the utmost effort is made to eliminate mesophilic bacteria, yeasts and molds (see p. 688) from

the sugar in order to set at rest the mind of bottlers who attribute many of their failures to the presence of microorganisms in the sugar.

Crushed Grain Sugars. Next in the classification of sugars by grain size are the crushed grain sugars. The largest of these has a grain size distribution very close to Bakers Special, and is sometimes sold under that name. When milling sugar to the size of Bakers Special, it is difficult to avoid a large percentage of fines in the "powdered" range. Since these fines can be removed only with great difficulty the sugar is not as closely controlled in crystal size as Bakers Special.

Powdered sugars include the smallest grain sizes. In the past there have been a great many sizes of powdered sugars, usually made by crushing granulated sugar in a high-speed disintegrator-type mill as shown in Figures 14-2 and 14-3. Over the years it has been found that the finer the powdered sugar, the poorer its keeping qualities. Until it became general practice to add some refined starch, it was impossible to sell a very fine powdered sugar except for immediate use. The addition of about 3% starch or other acceptable conditioner assures the manufacturer that the sugar can be shipped for some distance and arrive at the customer's plant in usable condition. Powdered sugar is, however, at best somewhat perishable under adverse storage conditions. It has been found that even slight differences in its particle size distribution may give it different qualities in storage. The chief use of powdered sugar is in cake batters and icings. It is possible by the addition of water to powdered sugar to make a very inexpensive cake icing.

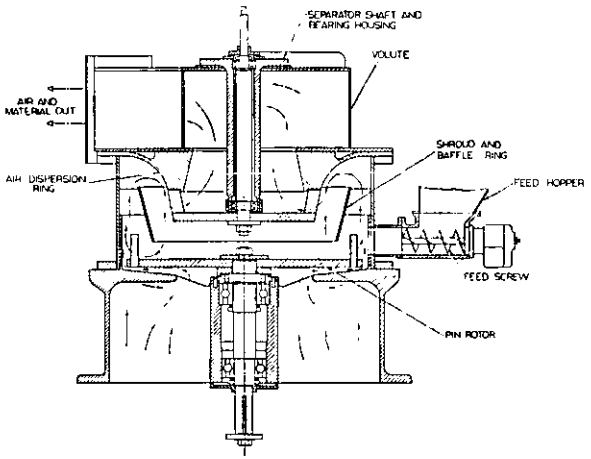

Figure 14-2. Mikro ACM Pulverizer for powdering sugar ***(Courtesy, Pulverizing Machinery, Div. of The Slick Corp.)***

Powdered sugar packages are now frequently marked with 6X or 10X. Presumably X-designations originally referred to the size of the screen or bolting silk through which the powdered sugar was first produced. Current sieve tests recognized by the Biscuits and Crackers Manufacturers Association as typical, are shown in Table 14-2.

Powdered sugar is produced by one of two methods. The first is to pass granulated sugar through a mill which grinds the sugar to the desired particle size in a single pass. The second method is to undergrind the sugar, and bolt or screen out

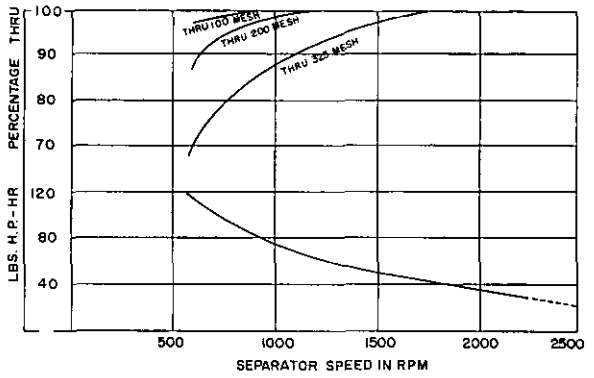

Figure 14-3. Typical curves showing variation of grain size, and of production rate in lbs/hp-h, with variation of the separator speed. ***(Courtesy, Pulverizing Machinery, Div. of The Slick Corp.)***

the unchanged fraction for return to the mill proper. This results in a system consisting of a feeder, mill, screen, receiving hopper and elevator to return the oversize to the mill feeder. While the first method is undoubtedly more economical, the second yields very slightly better control of particle size.

An extremely fine powdered sugar with a maximum grain size of 25 microns (7) is marketed under several names. The label usually contains variations of the words "fondant" and "icing." Most such products are sold at premium prices as specialized equipment is needed to assure elimination of larger sugar crystals. This is best done in high-speed pulverizers equipped with suitable screens or an air classifier. The coarse crystals are retained in the pulverizing chamber until reduced to the desired size. The sugar pulverizer shown schematically in Figure 14-2 uses an internal air classifier.

Powdered sugar has in recent years been packed in multi-wall paper bags containing one impervious layer of a plastic material for the exclusion of moisture. It is standard practice to avoid the excessive pressure which results from piling many bags of sugar on top of each other. Powdered sugar is generally stacked no higher than six bags, and is best kept in a storage place where there is uniform temperature and humidity. With this attention, the sugar should remain in good condition for more than six weeks. It is not unusual to have powdered sugar remain in storage for many months without the formation of lumps.

Table 14-2 Typical Sieve Analyses for Powdered Sugars

USBS Sieve No.	% Retained on Sieve	
	6X Powdered	10X Powdered
on 100	0.5	0.1
140	1.8	1.4
200	6.6	2.4
270	8.2	3.0
325	10.8	7.0
thru 325	72.5	86.1

Brown or Soft Sugars

This class of sugar products consists of fine sugars mixed with brown-colored, pleasantly flavored syrups. The crystal size of the sugar is approximately that of Bakers Special. The term "soft sugar" really describes the feel of this product, which is heightened by the presence of the syrup. It is desirable that the syrup on the sugar base be stable and not supersaturated, so that on aging there will be no crystallization.

There are two basic methods in the manufacture of soft sugars: the first is to boil from a selected syrup a massecuite, which on separation in the centrifugal, will yield a desirable soft sugar without further treatment. This method, described as the "in and in method of boiling," gets the full range of colors and flavors desired (5) and is of necessity a product of a cane-sugar refinery.

The second is to add to a pure, white sugar a treated syrup, usually of cane origin, in the proper amount to give a flavor, texture, and color desired by the manufacturer. Excepting in beet-sugar factories equipped with the ion exchange process, this method, generally called "painting," appears to be the logical choice for the beet-sugar manufacturer.

The special texture of soft sugars should involve a somewhat sandy mass, soft feeling between the fingers, rather than a gummy or grainy mass. The flavor should be pleasant and not too reminiscent of low-grade molasses.

In the past, a great many different colors of soft sugar have been produced by cane refiners. However, a very large percentage of the class is called "Golden Brown," which is a medium lively brown color. A quality largely sought for in soft sugars is a retention of this liveliness or "bloom," without a fading of the syrup during aging. Soft sugars are still sold in many varieties, the two principal classifications being a light brown or "C," and a dark brown or "B," which is often called "Old Fashioned Brown."

Soft sugars are sold to industrial users in 100, 50, and 25 lb bags. For domestic consumption they are packaged in a variety of containers including a 1-lb carton fitted with a moisture-barrier liner, and a 2-lb transparent, plastic bag. Every effort is made in all these packages to prevent the loss of moisture from the sugar to the atmosphere, and to retain the original texture, flavor, and color. These sugars may harden where high temperatures and low humidities are encountered in storage. However, on returning them to a cool, damp place, they usually recover their original textures.

Brown sugars generally have polarizations from 92.0 to 86.0 and corresponding invert percentages from 2.7 to 6.5 depending on the color desired. The moisture level of most brown sugars is about 3.0%.

Bakers and confectioners use soft sugar as a source of color and flavor in many of their recipes for cakes, icings, and candies. It is desirable from their point of view that the sugar be readily miscible with fats and other ingredients.

Another use for soft sugars is in the manufacture of syrup. The flavor is the chief criterion of acceptability, but the clarity of the solution made from the soft sugar may be a deciding factor.

There are many testing problems connected with soft sugar, for which the usual laboratory equipment is unsuited. While the moisture may be determined by drying in the vacuum oven, a special colorimeter which reads the aggregate appearance of the sample, its shade of color, depth of color, and reflectance, is needed. No method except tasting and feeling has been devised which will properly evaluate

the flavor and texture for the trade. This leads to the necessity for experts whose opinion is valued on current production, and on samples which have been subjected to tests under varying storage conditions.

Problems involved in the handling and storage of soft sugars made the development of a free-flowing brown sugar desirable. The first company to produce such a sugar was the American Sugar Company. Both a granular and a powdered form of noncaking brown sugar were offered to customers in 1966 (2,4).

Cubes and Tablets

Molded Sugar. Molded sugar may be classified in two divisions. The first consists of cubes which are made by impregnating a granulated sugar with a sugar syrup or steam, molding this sugar to the desired size, and drying. Mechanical packers have been developed to handle cubes if close tolerances are maintained of both weight and size.

The most modern method of cube production uses the Swedish-conceived Vibro process (see Figure 14-4). Moist sugar, vibrated into and out of molds, is fed into a dielectric oven, which transmits high frequency radio waves through the cubes. The friction created by the radiation heats the cubes uniformly throughout, allowing the moisture to escape evenly and almost instantaneously. The cubes are then cooled and packaged mechanically. The manufacturing and packaging time is reduced to about four minutes. Vibro cubes are lighter, and dissolve more quickly than the conventional cubes (1). See Figure 14-4.

Chipped Slabs. The other type of molded goods is one in which some of the surfaces are not smooth and molded. In contrast, they are the result of chipping the hard, dry basic form made out of the mixture of granulated sugar and sugar syrup. There is a demand for chipped goods in two forms. The first is lozenge-shaped, and is called a tablet. Usually four of the six surfaces are molded or sawed, but the remaining two are the result of chipping with knives, which is the final step in the sizing of the tablet.

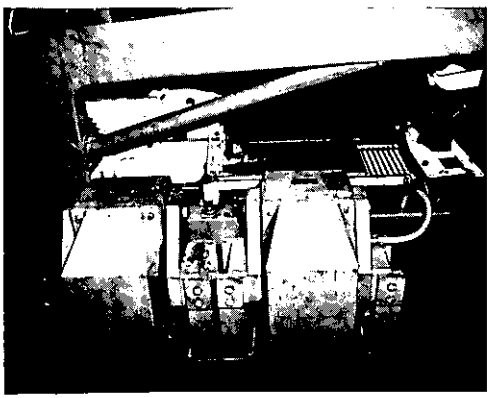

Figure 14-4. (Top) Vibro cube-forming machine. (Bottom) A cube-molding element. ***(Courtesy, Cubelet Press, California and Hawaiian Sugar Co.)***

The second form is a cut-loaf type of sugar, which in effect is a cube made by splitting two or more of the six surfaces. Since most of this type of goods is made from a very large grain sugar, the split surfaces present a ragged appearance, but have embedded in them large crystals which reflect the light and shine like little diamonds. In order to obtain this appearance, it is necessary to have a denser background for the crystals than is obtained in the production of molded cubes.

Miscellaneous Sugar Products

Regrained Sugars. There is another class of sugar products which, though small, is occasionally referred to in the literature. These are truly specialty sugars, being really "regrained." They are made by dissolving the original grain, subjecting the resulting syrup to some transformation, and repackaging for sale. The first of these, called "transformed" sugar, is made by beating a high-density, hot sugar solution at a high speed in a multi-paddled vessel. As the supersaturated solution crystallizes, these crystals grow to a size slightly larger than particles in powdered sugar. The heat of crystallization is sufficient to drive off most of the accompanying moisture, and the product emerges in a fairly dry condition.

A second product in this class is fondant, which may be made and sold in either a wet or dry form. Fondants (9) are made by cooking to a high density a solution of sugar containing either invert syrup or corn syrup added for the purpose of controlling the rate of crystallization of the sucrose. The cooked syrup is cooled to a desired temperature, where it is agitated sufficiently to form very small crystals, many times finer than those in powdered sugar. The individual crystals in fondants cannot ordinarily be felt, either with the tips of the fingers, between the teeth, or with the tongue. Where the fondant is to be sold wet, it is ordinarily placed in sealed cans, and sold directly for the use of the baker or confectioner. This type of sale is usually made by the wholesale supplier rather than the producer. There is a process under which the fondant is dried; that is, the moisture content is reduced to approximately 1.5%. This process has been used by refiners, and gave a sugar which was sold in large packages to industrial users.

A third member of this class is grained invert syrup. In this case, concentrated sucrose syrup, of 75° Brix or higher, is transformed entirely into invert syrup. As a result, the dextrose formed is supersaturated. When the syrup is cooled and seeded with a small amount of crystalline dextrose, a considerable portion of the dextrose present will crystallize out on standing, resulting in an invert "mush." The fructose present will, however, remain in solution, and will not crystallize under such conditions. This type of product is sold both by refiners and by intermediate syrup producers.

Agglomerated Sugar. An agglomerated sugar developed by using a Pillsbury Company (8) patent contains no starch. The product is free flowing and is distributed in consumer size packages. The industrial advantages of agglomerated, powdered sugar over regular powdered sugar apparently do not outweigh the necessary price differential in warehousing problems created by its fluffy condition.

Flavored Sugars. These may be either granular or grain-free. *Sugar and cinnamon* is a simple mixture of an extra-fine granulated sugar and powdered cinnamon. Its distribution is limited to the retail trade and usually in small shaker-type packages.

The grain-free sugars are marketed as "edible" syrups. Flavors are used in virtually unlimited numbers, but the most popular in table syrup is maple. Fountain syrups are widely used in soft drinks and fountain concoctions. The favorites are

cola, root beer, lemon-lime, orange, and chocolate. Edible molasses rightfully falls into the grain-free category.

Colored Sugars. A small volume of sugar is produced in vivid colors for both food and decorative purposes. Colors added to the granular sugars are made from edible food dyes.

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Chapter XV
LIQUID SUGARS*

Liquid sugar is simply a solution of sugar in water. The sugar can be sucrose or invert or a mixture of the two. The mixtures are generally characterized by stating the percent invert on total solids. For example, 50% invert would denote a solution in which 50% of the total sugar solids was invert sugar and 50% was sucrose. In the last 15 years liquid sweetener blends of sucrose, invert sugar, dextrose, and various types of corn syrup have become common and various blends tailored to individual customer's specific requirements are available in many areas. The growth of the liquid-sugar industry is impressive. Starting in New York in 1925 on what was actually an experimental scale, commercial shipments were not made until 1927 (5). By 1968 industrial deliveries of liquid sugar from cane and beets had increased to 43 million cwt (10).

Liquid Sucrose

Liquid sucrose is made from either beet or cane sugar. When made from beet, refined granulated sugar is always used. In the case of cane, the liquid sucrose may be obtained by remelting refined granulated sugar or be produced directly as a liquid in the refining process. In the remelting of granulated sugar, one of three methods is generally employed. The first is batch production, wherein correct weights of sugar and water to produce the required density are placed in a tank equipped with a stirrer and the mixture heated to about 85 °C to dissolve the sugar and pasteurize the solution. It is then filtered or pumped through a fine screen, cooled and pumped to storage. Activated carbon is sometimes used to obtain a complete decolorization. The second method is a continuous system. Many of these have been devised but most are based on the principle of first mixing sugar and water to a concentration slightly higher than that finally desired. An in-line refractometer continuously measures this concentration and signals a control valve adding water as required to reduce the concentration to the desired value. The third method termed "Liquilizing" (6, 7) was devised primarily to be used where the required liquid facility is located too far from a primary source of liquid sugar to ship economically the liquid product. In this system, water, usually in an amount slightly less than that required for the final density, is placed in a tank at the terminal. This water is pumped through a flexible hose connected to a "converter" attached to a railroad car or truck containing bulk dry sugar. The sugar is fed into the converter at a constant rate and the resultant slurry pumped back to the tank. The sugar-water slurry is circulated from the tank to the car or truck until all of the sugar is unloaded into the tank. Solution is completed by stirring the tank contents, and the desired concentration is obtained by addition of a small amount of water.

Liquid Invert

The term invert sugar describes a mixture of equal parts of glucose and fructose (or dextrose and levulose). Sucrose is hydrolyzed readily in acid solutions at rates that increase markedly with increasing temperature and decreasing pH, yielding these two sugars. The hydrolysis takes place at the bond between the pyranose and furanose nuclei, splitting the two apart with adsorption of one

*by Robert S. Gaddie

molecule of water into the free nuclei. This forms one molecule of glucose and one of fructose as shown in the equation:

$$\underset{\text{sucrose}}{C_6H_{11}O_5{-}O{-}C_6H_{11}O_5} + \underset{\text{water}}{H_2O} \xrightarrow{H^+} \underset{\text{glucose}}{C_6H_{12}O_6} + \underset{\text{fructose}}{C_6H_{12}O_5} + \text{Heat} \qquad (1)$$

mol. wt. 342.30 mol. wt. 18.02 mol. wt. 180.16 mol. wt. 180.16

It will be noted that the one molecule of water which enters into the combination of glucose and fructose increases the weight of these combined sugars to 105.26% of the sucrose from which they were produced. The percentages of invert sugar in total sweetening solids in the invert syrups offered for sale vary from 95+% "completely inverted" down to 10%. The one produced in greatest quantity is the 50% invert. In the system, sucrose–invert sugar–water, the most soluble mixture closely approaches 38.5% sucrose–38.5% invert–23% water. For example, the density of safe storage; that is, the density at which none of the three sugars will crystallize at 20 °C, rises from about 67% solids for 10% invert through 70% solids for 48% invert, and then falls to 72% solids for 95+% invert. From this solubility phenomenon, it is easy to understand why the syrup produced in greatest quantity is 50% invert at 77% solids.

This results in lower shipping costs per unit weight of sugar because freight must be paid on the water, more sugar can be stored per unit of tank storage space, and the osmotic pressure of the syrup is so high that very few microorganisms can grow.

Inverting tanks must be of acid-proof construction, either glass lined or of stainless steel. Piping and fittings should be of galvanized iron, stainless steel, or aluminum alloy. Copper and its alloys should be avoided. Plastic-coated mild steel, stainless steel and aluminum alloy are preferred for invert storage tanks.

There are two basic methods used to invert sucrose for industrial use. The first, which finds application to a limited extent, is by means of a strongly acid cation exchange resin. This process operates most efficiently on a diluted syrup, and where this process is used the final Brix is generally obtained by vacuum evaporation after inversion. Because refined granulated sugar is always used in the beet sugar industry, such dilution with subsequent evaporation is costly. In the normal process of refining cane sugar, high purity syrups more dilute than the average density range of the common invert mixtures are available. Therefore, the acid cation exchange resin inversion is more common in the cane industry.

The second method and the one much more generally employed is the hot acid method. In the beet-sugar industry, the batch system is most common and reagent grade hydrochloric acid is universally used. However, the variation between producers in the three variables which control inversion: acid concentration, time, and temperature, covers a wide range. For example, 50% invert is commercially produced at 36 ° to 46 °C in 23 hours. Another manufacturer produces it in 14 minutes at 70 °C, while a third uses 85 °C for 25 to 30 minutes. Sodium hydroxide, sodium carbonate, and calcium hydroxide are used to neutralize the acid and stop inversion. Of these three, sodium hydroxide is most universally used. Sodium carbonate gives a somewhat aerated syrup due to the evolution of the carbon dioxide and may cause foaming difficulties in the customer's process.

There are three batch methods of producing partially inverted syrups. The first is to add part of the sugar to all of the water, heat, acidify, completely invert, neutralize with or without cooling, and then to add the remainder of the sugar and adjust the final density. The second is to produce and store completely inverted syrup which can then be diluted with sucrose and water to obtain the desired percentage of inversion at the desired density. A third is to add all the sugar to the water, heat, acidify, time the reaction, and neutralize at the proper moment. The last method is generally used for invert syrups above 40%.

Besides acid concentration, temperature, and time, inversion rates of these heavy syrups are influenced by the concentration and composition of the ash in the sugar, and the concentration and composition of the solids in the water. The water used for the production of both liquid sucrose and inverted syrups must be of the highest potable quality. It may have to be softened, and soda-lime and zeolite or other ion exchange processes are used. If the water is drawn from a city system, it should be filtered through activated carbon to remove any residual chlorine. If the solids in the water are variable and sugar for inversion is received from several sources, it is sometimes helpful to the beginner to design an inverting system with built-in safeguards similar to the following, in which 4000 gallons of 45 to 55% invert, 77 ° Brix, pH 4.5 to 5.5, and of low color is required.

In the inverting tank 35 000 lbs of sugar is dissolved in 11 450 lbs of water. This gives an initial dry solids content of 75.35%. It is heated with stirring to exactly 70 °C. Sufficient hydrochloric acid is added to bring the melter contents to about pH 4.5. This evens out variables due to water solids composition. Then, 3250 ml of concentrated hydrochloric acid (inverting acid) is added and a timer is started. At the end of exactly 30 minutes, a sample of about 60 ml is drawn in a beaker containing two drops of saturated solution of sodium hydroxide. It is cooled and five grams are weighed out. This is diluted to 500 ml and mixed. It is transferred to a burette and a Lane-Eynon (2) titration using 10 ml of mixed Fehlings solution is made. The burette reading at the end point is referred to Table 15-1 to obtain the estimated stop time. Table 15-1 has been developed from a number of curves, relating inversion rate to time, and correlates the Lane-Eynon titration volume at 30 minutes with the probable moment at which the invert content will pass the 50% of solids point.

At the expiration of the estimated stop time, measured from the time of addition of inverting acid, sufficient NaOH solution is added to bring the pH from 4.5

Table 15-1 Estimated Stop Times as Determined by 30 Minute Lane-Eynon Titration for 50% Invert

Burette Reading	Stop Time Min.	Burette Reading	Stop Time Min.
20.0	44	26.0	61
20.5	45	26.5	62
21.0	47	27.0	64
21.5	48	27.5	65
22.0	49	28.0	66
22.5	50	28.5	67
23.0	52	29.0	69
23.5	53	29.5	70
24.0	55	30.0	71
24.5	56	30.5	73
25.0	58	31.0	74
25.5	59	31.5	75

to 5.5. Eleven to 12 liters of a solution made up by dissolving 5 lbs NaOH in 4 gallons of water should do this. In the neutralization, it is important to avoid overstepping the pH end point. Color production due to decomposition of fructose is very rapid with increasing pH above 6.0, and with increasing temperatures. Finally the density is adjusted to the required value, the syrup is filtered or pumped through a fine screen, cooled and pumped to storage. Here again, activated carbon or a decolorizing exchange resin may be used to completely decolorize the syrup. Both of these materials may change the pH of the syrup and it may be necessary to re-adjust the pH of the final product to obtain the desired value.

Partially inverted syrup production has been automated (1). Automation is based on the fact that the hydrolysis of sucrose is an exothermic reaction. The greater the degree of inversion, the greater the total heat created. By using an analog computer to continuously monitor reaction temperature, compensate for heat lost to surroundings and sum up total heat generated, the degree of inversion can be calculated. To sense accurately the small temperature changes, thermistors are used because of their rapid response and great precision. Pushing the start button adds the hydrochloric acid. The computer's summing unit automatically adds the temperature increase from the reaction and the heat lost to the atmosphere in order to obtain the total heat generated. The output from the summing unit goes to a monitor which adds the neutralizer solution to stop the reaction when the desired degree of inversion is reached.

Typical specifications for liquid sucrose and 50% invert syrup are shown in Table 15-2.

Table 15-2 Typical Specifications for Liquid Sugars

	Sucrose	50% Invert
Dry Solids, %	66.5 ± 0.2	77.0 ± 0.2
Invert, % on Dry Solids	0.02	45-55
Sucrose, % on Dry Solids	99.9	55-45
Specific Gravity (20°/20° C)	1.328	1.3903
Weight per gallon, 20° C, lbs	11.05	11.57
Pounds solids per gallon, 20° C	7.35	8.91
Gallons per 100 lbs Solids, 20° C	13.61	11.22
pH	6.8-7.6	4.5-5.5
Ash, %	0.02	0.06
Color, RBU (4)	35 Max.	35 Max.

In most cases in the production and use of liquid sugar, a refractometer calibrated for sucrose is used to determine the solids content. The refractive indices of invert sugar solutions are lower than those of sucrose of the same concentration. Therefore, the percentage of dry solids indicated by a refractometer calibrated for sucrose must be increased. The error is not linear and Table 15-3 gives these corrections for observed refractometer solids concentrations from 10 to 80%. The data in Table 15-3 have been calculated from NBS tables of refractive indices of pure invert solutions (9) and pure sucrose solutions (3).

Referring to Table 15-3, column B is to be used where the percent invert *in solution* is known. From the observed refractometer solids, column A, note the corresponding factor in column B. Multiply the percent invert in solution by this factor and add to the observed refractometer solids. Column C is used where it is necessary to determine the solids content of invert-sucrose mixtures of known "invert percent on dry solids." To use this column, multiply the correction factor in

Table 15-3 Table for Correcting Sucrose Refractometer Observed Reading for Presence of Invert

A Observed Refractometer Solids	B Factor Where % Invert in Solution is Known	C Correction Factors for Total Invert
10	0.009	0.10
15	0.009	0.14
20	0.009	0.19
25	0.010	0.26
30	0.011	0.34
35	0.012	0.44
40	0.014	0.55
45	0.015	0.68
50	0.016	0.82
55	0.017	0.98
60	0.019	1.15
65	0.020	1.32
70	0.021	1.49
75	0.022	1.66
80	0.022	1.84

column C corresponding to the observed refractometer reading, column A, by *invert percent on dry solids* and add to the observed reading.

Blends

The science of food fabrication has led to the development of syrup mixtures which meet specific functional requirements for different food processors. Such mixtures, which contain carbohydrates other than sucrose and invert, are classed as blends. They are made up to specification and generally on order. Important functional properties of all syrups including blends are sweetness, viscosity, prevention of sugar crystals, prevention of ice crystals, osmotic pressure, bodying, hygroscopicity, humectancy, and freezing-point depression. Blends are made by weighing or level-gauging in batching tanks equipped with stirrers, or the process may be made continuous. A recent in-line, automatic blending system (8) comprises a liquid-type blender connected to two positive displacement meters and a digital control station. The light Brix syrup input is metered and instrument controlled. The heavy Brix syrup, because of its high viscosity, is difficult to meter accurately so its flow-rate is set manually and its volume is the difference between total flow and the light Brix syrup flow, which are metered. The digital control station relates demand (set point) to the output of both meters and signals the light syrup flow controller to maintain the correct proportion of both syrups.

Distribution of Liquid Sugar

Liquid sugar moves from the producer to the customer in tank trucks and rail cars. Tank trucks carry loads up to 4500 gallons, and they may be divided into compartments with separate loading and unloading arrangements (Figure 15-1). The tanks should be of stainless steel, aluminum alloy, or steel coated with plastic lining. Most of these are mounted on a semi-trailer chassis which also carries a positive displacement unloading pump driven by a gasoline engine. If the tanks are baffled, there should be a screw cap or hatch opening in the top center of each baffle compartment. This is to allow for insertion of a high-pressure rotating spray assembly to clean and sterilize each compartment. This washing and sterilization of the truck

tank, pump, pipe lines, and connecting hose should be done once a day if the truck is in continuous use on the same syrup; otherwise each time the truck is loaded. At the customer's plant, the truck driver removes the sanitary cap from the customer's fill line, sprays the connection with a disinfectant and connects his suction hose to his outlet line and to the customer's intake line. After checking all valves to see that they are in proper position, the pump is started and the syrup is pumped into the customer's tank.

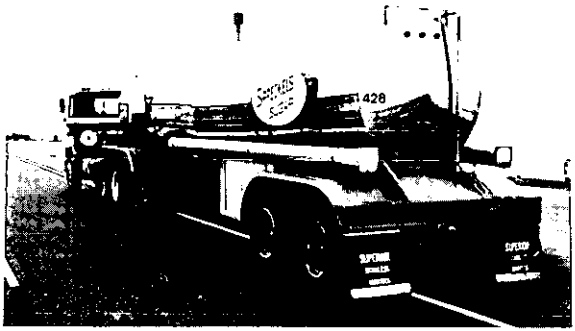

Figure 15-1. Liquid sugar tank truck.

The capacity of tank cars ranges from 8000 gallons up. Normally the sugar producer provides this vehicle either by purchase, by lease from a car rental company, or by loan without charge from a railroad company. It has been found that liquid sucrose can be shipped in plain steel tank cars without appreciable damage to the quality, although this is not to be preferred. For shipment of invert syrups, a specially lined tank car is required. The lining is usually of the sprayed-on plastic type, which has been found acceptable for shipment of wines and such liquids.

Customer Liquid Sugar Handling Systems

Before a food processor can use liquid sugar, he must equip himself with suitable receiving, storing and dispensing equipment. Such systems are nearly always custom designed to meet individual plant requirements, and usually incorporate standardized, proven components, and, where practical, existing equipment. Their versatility makes them well suited for most users, since they can be designed very simply to respond automatically to process demands. Figure 15-2 is a line drawing of a typical customer liquid-sugar system.

Liquid sugar handling systems consist of storage tanks, storage tank accessories, pumps, piping with its valves and fittings, and measuring equipment.

Ideally, each installation should have at least two tanks. Each should have a capacity somewhat greater than the size of an individual delivery. Distance from the producer to the user and the inventory the user wishes to carry for meeting process requirements will influence the size and number of tanks. Single-tank installations are common, but the two-tank system is far superior. With a two-tank installation, sugar can be drawn from one tank until it is completely empty, at which time the other tank is switched into use. The result of this alternate emptying of storage tanks is the assurance that the microorganism count will not significantly build up in the storage tanks, and these tanks can go for very long periods without cleaning and sterilizing. With a single tank, the user must stop his process in order to drain

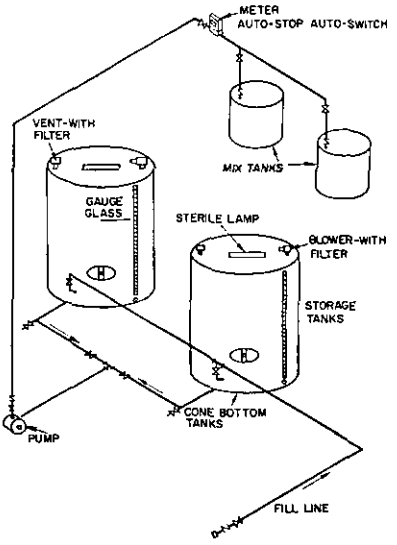

Figure 15-2. Typical customer liquid sugar system.

the storage tank. This is costly, so what actually happens is that fresh deliveries are pumped into the tank containing residual syrup, and any microorganisms in this residual inoculate the new batch. Inevitably, the microorganism count continuously builds up until a danger point is reached and the tank has to be drained, washed and sterilized. Two tanks will also permit delivery at the customer's convenience without making delivery schedules too exacting and without interrupting normal operation at the user's plant. Where possible, storage tanks should be cylindrical, mounted vertically on legs and have a cone or dished bottom. In any event, they should be so mounted that they can be completely drained, preferably to the suction of the pump. Tanks can be fabricated of plastic-coated mild steel, stainless steel, aluminum alloy or laminated, food-grade plastic. Each tank should be equipped with some type of quantity gauge. The simplest and usually most trouble-free is a gauge tube of clear unbreakable plastic, 0.75 to 1 in. in diameter, connected to the tank only at the bottom and extending the full height of the tank. The top of the tube should be open and capped with a filter vent to reduce the danger of water vapor condensing inside the tube, which can occur when the tube is connected into the top of the tank. This would dilute the surface of the liquid, forming an ideal medium for growth of microorganisms.

The top of each tank should be fitted on the outside periphery with a small blower with filter and, on the opposite side, a screened vent. This is to prevent moisture condensation in the head space. As a general rule, ultraviolet lamps are mounted under covered slots in the top of the tank. One ultraviolet lamp is sufficient for about 50 sq ft of liquid surface.

Pumps are usually positive displacement, rotary types and operate at fairly low speeds. Capacities normally range from 15 to 50 gpm, requiring motors of 3 to 5 hp. The systems are designed to supply the liquid product to all processing outlets.

Pipes, valves and fittings may be of galvanized iron, stainless steel, alumnium alloy or plastic. Experience has shown that pipe lines should be pitched so that they are always full of liquid sugar. If they are not so designed, water vapor can condense and form a thin layer of low-density syrup on the surface of the liquid with consequent danger of spoilage by microorganisms. It is, however, necessary to make provision for complete drainage of these lines for cleaning and sterilizing.

Different methods are used to measure or dispense liquid sugar into different processes. One of the best is automatically, by using a meter. Commercial meters, properly calibrated, will maintain a constant accuracy well under 1% and they can be set to deliver a given quantity, to start up when a button is pushed, and to shut off when the predetermined amount has been delivered.

A properly designed liquid sugar system requires very little maintenance, and each liquid sugar user should schedule maintenance according to his need. It is generally recommended that the entire system, including storage tanks, gauge glasses, pumps, pipe lines, and meters be drained, thoroughly washed with warm water and sterilized about every six months. At this time all equipment can be checked. For example, tank linings can be inspected, ultraviolet lamps can be cleaned and checked for radiation efficiency, and filters and strainers can be changed or cleaned.

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Chapter XVI

SUGAR QUALITY*

As the beet-sugar industry has improved the efficiency of its manufacturing processes and the diversity of its products over the decades of this century, the quality of its products has followed a definite upward curve. The two-boiling process—one product and one remelt boiling—which was formerly used in the beet-sugar industry and which raised the molasses purity and lowered the sugar quality, was abandoned for the three-boiling system about 1930.

Sugar as manufactured from the sugarbeet or sugar cane is the cheapest pure organic chemical manufactured, its purity being about 99.96%, of which about half of the nonsucrose or 0.02% is water, and the other 0.02% is nonsucrose solids. For the last 40 years beet and cane sugars have been virtually identical in quality, usable interchangeably by the housewife for every purpose. For the industrial consumer, however, very small quality differences which particularly affect individual uses, acquire abnormal importance. This is magnified in the highly competitive markets and relatively insignificant quality differences may take on inflated importance.

Relative to any other pure food substance manufactured today, the quality defects in modern sugars are conspicuously absent. Thus, this chapter is a "microscopic study," in which 0.04% of the product sugar is enlarged by 2500 diameters to occupy the whole field of interest.

Physical Quality Factors in Granulated Sugar

Crystal Shapes. The monoclinic sucrose crystals which make up granulated sugar are normally of a shape of the type shown in Figure 16-1. Granulated sugar composed of such crystals, and varying with the degree of agglomeration and size

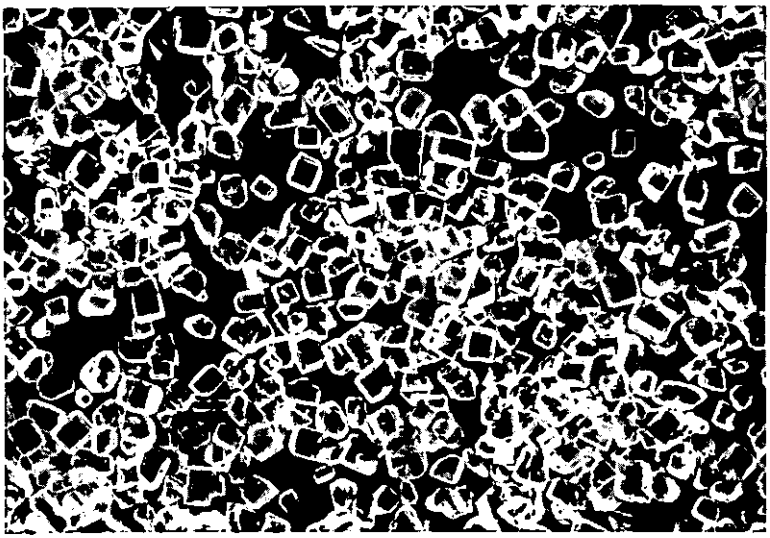

Figure 16-1. Normal crystals of granulated sugar of beet origin.

*by R.A. McGinnis

distribution, as well as the degree of compaction, has bulk densities varying from 54 to 60 lbs per cu ft. In the presence of certain nonsucrose constituents, such as raffinose (5) the crystal habit may become distorted along one axis (see p. 417), or with dextran (68,69) along another axis, to form elongated, needle crystals as shown in Figure 16-2 (69). The bulk density of such crystals may be reduced to as low as 48 lbs per cu ft, and 100 lbs of sugar may no longer fit an ordinary 100 lb bag.

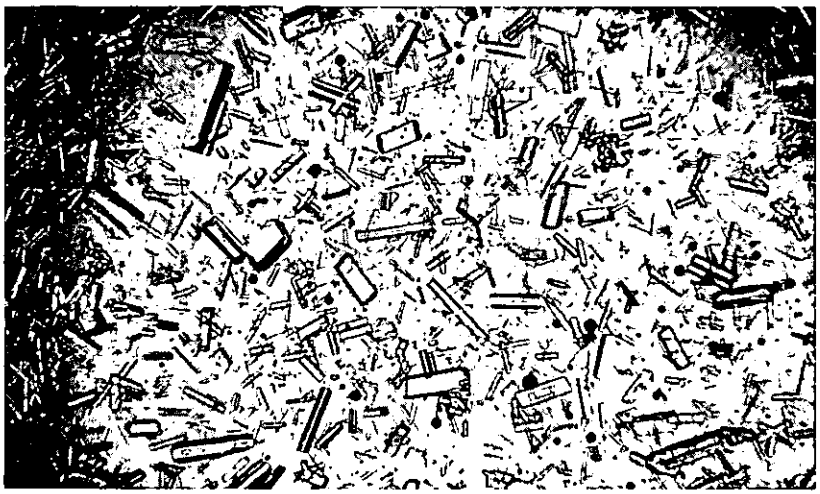

Figure 16-2. Sucrose crystals grown in the presence of 0.5% dextran (M.W. 2000). *D.N. Sutherland and N. Paton (70)*

Agglomeration. Closely associated with grain shape is the degree of agglomeration of the crystals. Agglomeration, or *ball grain,* usually results from errors made in pan boiling.

The highest bulk density is reached by a grain-size distribution in which the voids between the larger crystals are filled by smaller crystals. If there are many agglomerates, there may not be enough small crystals to fill the voids, and the bulk density is decreased. The effects of lower, varying bulk densities are problems for the packager. The effects of occlusions of mother liquor within agglomerates are, however, of concern to the producer and the user. These traces of mother liquor left within the sugar, when not completely removed by the scalping screens, leave small brown specks. In addition to being unsightly, they increase solution color, ash content, and residual organic nonsucrose substances.

The water contained in the occlusions tends to make the sugar unstable in storage. Most degradation reactions, including growth of microorganisms, color formation, and inversion, have in common the requirement for water. In bulk storage the excess water will migrate, through countless evaporations and condensations, toward the cooler sections of the containing bin, and reach relatively high concentrations in these areas, with resulting inversion, and color and odor development partly from microbiological action. It is normal to find that on the side of a bulk bin not reached by the sun, or facing cold winds, there is an inside crust several inches thick of wet, brown, malodorous sugar. As sugar is removed from the bin, the crusts may fall off and break up, loading the mass of sugar with more brown specks, which

may be mistaken for balls of unwashed massecuite, and which degrade the sugar quality in color, turbidity, excessive moisture, and odor.

Under mechanical torque, agglomerates break up far more easily than single crystals will fracture. Thus, after undergoing the mechanical stresses of screw-conveying and screening, plus further drying, the mass of sugar may contain many more fine crystals and much more dust than when it was first sampled as emerging from the granulator. In such cases, the sugar will have more fines in it when sold than when it was made. This leads to failure to meet customer specifications, and to unpleasant relations between production and sales departments.

Degree of agglomeration is sometimes roughly measured by pouring a definite weight of the granulated sugar into a graduated cylinder, and noting the apparent volume. It may be much better observed, however, using a low-power microscope.

In a pioneering paper, Farag *et al.* reported test methods for the measurement of dust in granulated sugar, and physical methods for determining compressive and impact strengths of sugar crystals (22a).

Size and Distribution. Forty years ago beet-sugar manufacturers normally made only one grain size of granulated sugar. The many grain sizes now required to fill special needs are described in Chapter XIV. Grain size is universally specified in terms of the results of sieve tests.

The MA-CV terminology is widely used. This is based on the original work of Powers (60), and as described by Schneider (64e), "is a concise method of reporting the result of a sieve test on a crystalline sugar, which presents a mental picture of the grist and eliminates the need to specify the sieves employed. It is based on the assumption that most samples of sugar have a normal size-distribution [which is found in practice to be true] so that a plot of the cumulative percentages retained by a series of sieves, after shaking, against sieve aperture on 'arithmetic probability' paper will give a straight line. The sieve aperture corresponding to the intercept of this line with the 50% line on the graph is defined as the mean aperture (MA) and a sieve of mesh size equal to the MA will retain 50% of the sample. Please see Figure 16-3. [The MA is reported in thousandths of an inch, or in microns.] The coefficient of variation, CV, a unitless number, is defined as the standard deviation expressed as a percentage of the mean aperture. In the case of a sample having normal distribution, the difference between the apertures corresponding to the 16% and the 84% line is equal to twice the standard deviation. Consequently:

$$\text{CV} = \frac{\text{(aperture corresponding to 16\%)} - \text{(aperture corresponding to 84\%)}}{\text{(mean aperture)} \times 2} \times 100 \qquad (1)$$

[or in an easier form to use,

$$\text{CV} = \frac{\text{(aperture corresponding to 16\%)} - \text{MA}}{\text{MA}} \times 100] \qquad (2)$$

The method is suitable for all granulated sugars having normal or near-normal size distribution. It is not suitable for sugars having abnormal distribution, or for finely powdered sugars." It is also not suitable for sugars made by blending together sugars of different size distribution or for sharply fractionated screened sugar. Improvements suggested by Halden(27) were adopted by the ICUMSA (International Commission for Uniform Methods of Sugar Analysis) (33), which permit the graphical determination of both MA and CV.

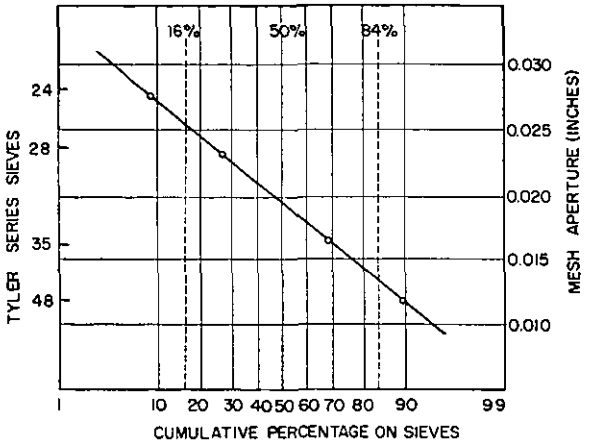

Figure 16-3. M.A.-C.V. test graph. *From H.C.S. De Whalley, "ICUMSA Methods of Sugar Analysis," Elsevier, Amsterdam, 1964.*

Johnson (41) described the use of only two sieves for this analysis, with the use of specially prepared tables. Eis (22) further devised improvements which have been incorporated in the official ICUMSA analysis (64a).

Reflectance. Today, all modern crystalline sugars are sufficiently white so that there would be little advantage for any present use of solid sugar to improve it. Nevertheless, the total reflection of light from the surface of a granulated sugar is sometimes a consideration, far more in foreign countries than in the United States. Subject 22A committee of ICUMSA has been set up to formulate methods for reflectance measurement. In addition to the color of the crystal surfaces themselves, reflectance is strongly dependent on the crystal sizes and their distribution, the amount of moisture in the thin film on the crystal surfaces, or gloss, the degree of packing of the mass of crystals, and physical factors such as the depth of the sample measured. The finer the average grain size, the greater the reflectance, so confectioner's or powdered sugars have the highest reflectance values, and the large-grained specials such as confectioner's A, have the lowest reflectance values, almost regardless of other factors. There are no generally accepted methods of measuring the reflectance of granulated sugars. General-purpose reflectance-measuring equipment is not well adapted for sugar.

Gillett and Meads (26) described the use of a reflection meter. There have been a number of attempts to evaluate the reflectance using tristimulus coordinates (54) as specified by the International Lighting Commission (59,74). A method involving the use of two measurements within narrow spectral bands in the visible region was proposed by Oikawa (53) and elaborated with the use of an integrating sphere by Janofsky (38). Strube (67) reported a differential reflectance photometer using a pair of Ulbrich spheres and matched photocells.

Gloss. Gloss refers to the subjective visual attractiveness of the granulated sugar. A sugar with good gloss has a sparkling appearance, which comes from unscratched crystal surfaces, and sufficient moisture in the surface film to make it shiny. A sugar without a good gloss has a chalky-white appearance. Gloss may develop in dry, dull-appearing sugars, if placed in an environment of high relative humidity, and subsequently in one of low relative humidity. In such cases the dust

particles on the surfaces dissolve and dry to a clear glass. The scratching of crystal faces is, of course, the consequence of abrasion by metal screens and scrolls. Sometimes the special large-grained sugars are screened through bolting silk to avoid abrasion. Gloss is only of importance where the attractiveness of the individual sugar grains is a factor, as where the crystals cover the surface of the food.

No test methods for gloss are known, other than subjective, individual personal evaluation.

Compositional Factors

Water Content. The moisture content of granulated sugar is important both as it affects its ability to flow freely and because of its adverse effect on the stability of sugar in storage. A considerable portion of the trace nonsucroses in the crystals is on the surfaces, in a very thin sucrose-saturated film. This film contains small amounts of invert sugar, and inorganic and organic nonsucroses in solution, or as parts of a heterogeneous phase. The invert sugar, and possibly other nonsucroses are hygroscopic and prevent the film from drying. There is an equilibrium established between this film and the atmosphere surrounding the crystals. As the relative humidity of the atmosphere increases, the percentage of moisture in the sugar increases, and vice versa (45). If the surface film is dried, crystallization takes place, cementing together crystals which are in contact. Thus if sugar is not sufficiently dried when made, drying will continue while the sugar is in packages or bulk storage until equilibrium with the atmosphere is reached, resulting in lumping and caking. Thus the sugar should be dried to the point where it is either below, or about at the equilibrium moisture content at the ambient relative humidity. Conversely, if a caked sugar is humidified by raising the relative humidity of the atmosphere around the crystals, the cementing surface crystals will dissolve, and the sugar will again become free-flowing.

Figure 16-4, reprinted from the first edition, from the work of T.J. Nelson (50a) shows a sigmoid-shaped curve determined by the exposure of small samples of cane granulated sugar at various known relative humidities, which has been found valid in practical use also for granulated sugars of beet origin. "(The curve) shows practically no moisture at low humidities, and that there is a rapid change in the range between 60 and 80% relative humidity. The trace of nonsucrose in the granulated sugars is a component that tends to absorb moisture gradually, and the sucrose theoretically would show an abrupt change. Thus the moisture content of the composite commercial product is, in a sense, an average of that of the two constituents. It may be noted that differences in the nature of these nonsucrose impurities and in the amounts present will influence the humidity equilibrium values over the whole range, and in particular will affect the slope of the curve or abruptness of the moisture increases in the critical storage range between 60 and 80% humidity."

The Codex Alimentarius (the United Nations affiliate which has the task of setting up international food standards) places an upper limit of 0.1% on "loss on drying."

Conditioning. There is a more obscure factor connected with the water content of the sugar crystals. Under normal production conditions a sugar which is put into bagged or bulk storage after being dried to equilibrium moisture content, may evolve further moisture during the next few days. This moisture may originate in occlusions within individual crystals (61), or be sealed within agglomerates, or even be connected with an unstable, amorphous hydrate of sucrose which can form under too rapid drying conditions, and which rapidly crystallizes in the presence of water

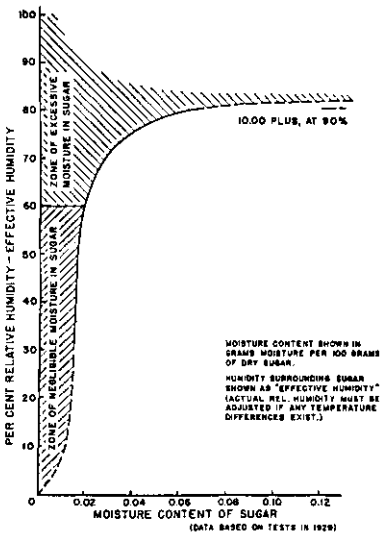

Figure 16-4. Approximate moisture content of granulated sugar at various humidities. *T.J. Nelson (50a)*

vapor (44). This moisture, of course, temporarily dissolves more sugar in the surface film, but after a further period, since there has been no essential change in the amount of the hygroscopic substances present, the additional water will evaporate, cementing the crystals together. If the sugar is sold before this moisture evolution has been accomplished, it may harden in the bag or in bulk transit.

A sugar that is properly *conditioned*, will not harden in storage or transit unless it is subjected to drastic drying conditions, or to strong dampening conditions followed by drying.

Among the many methods of determining moisture in sugar, one general type determines the surface moisture, as by drying with heat, and another permits the determination of additional amounts of moisture present in the crystals or in occlusions. Of the first type are methods of drying in an oven at atmospheric pressure (64c), and under vacuum (80), as listed by the ICUMSA. The use of infrared drying is satisfactory for approximate purposes, providing browning of the sugar is avoided (4,51). Rapid drying of 6-8 g of sugar may be achieved by passing warm air through a sintered disc (48). The methods of the second type use the Karl Fischer titration (7,49,52,63,66), and the vacuum distillation methods (81).

Also of this type is the rapid determination by the use of the "angle of repose" apparatus of the Danish Sugar Company (16), which, within the limits of specific grain-sizes, is said to give a determination in three minutes, with less than 20% error.

There are no reliable methods for the determination of the degree of conditioning of sugar. Some feel that the storage of a small sample of sugar in an airtight glass container, and the observation of its free-flowing condition at the end of the period gives a rough measure. It is possible that a measure of conditioning may be some function of the two factors, surface moisture and internally occluded moisture.

Ash. The content of inorganic material in a sugar is qualitatively defined as the *ash*. Specifically it is measured by the complete oxidation of the organic constituents of the sugar by heat, and the conversion of the residual inorganic substances

to sulfates by repeated evaporations to dryness with concentrated sulfuric acid.

The main inorganic cations in granulated beet sugar are potassium, sodium, calcium, and magnesium, decreasing in that order. The chief anions are phosphates, sulfates and chlorides. The phosphates and chlorides are lost in the sulfating procedure.

The intrinsic importance of ash as a quality factor is that probably, together with the solution color, its amount is regarded as evidence of the degree of purification. Ash limits are specified in the more important sugar standards. The NSDA (National Soft Drink Association) sets a top limit of 0.015%, and the Codex Alimentarius a limit of 0.04%.

There appears to be a relation between the *strength*, or resistance to inversion of sugar and the ash; and this resistance, both in the solid and solution states, may be a direct function.

While the reference method is the sulfated ash determination (79), because of the difficulty of the procedure, ash is almost universally measured through determination of the conductivity of a 28% aqueous solution of the sugar. The conductivity in micromhos/cm is converted to sulfated ash equivalent by multiplying by the "C-ratio factor." The C ratio is the factor by which the specific conductance in micromhos of the test solution, corrected for the specific conductance of the water, is multiplied, to convert the value to percent ash. Unfortunately the C-ratio is not a constant, but varies with the nature of the ash constituents. Values of this factor which have been established and used in practice with beet sugars, are, 0.000432 (Michigan); 0.000714 (Holly), 0.000555 (Tirlemont), and 0.000555 (American Crystal) (19). It can be redetermined for any specific use.

Heavy Metals. Limits for extremely small quantities of iron, copper, lead, and arsenic are listed in various standards. Iron, as ferric ion, is seldom found in measurable quantities in granulated sugar, excepting in the form of fine sediment, where it may be flakes of mill-scale, or other iron oxides from the surfaces of mild steel equipment. These specks are easily removed by passing the granulated sugar over magnetic grids or rollers. Absence of any traces of hard particles is essential for such uses as chewing gum sweetening, and almost complete absence of iron is necessary when the sugar is used in sweetening certain cordials, because of the effect on color. Ferric salts in concentration as high as 1 ppm may be found in inverted syrups of either beet- or cane-sugar origin, where they come from corrosion of steel pipes and fittings, or from solution of sediment particles.

Amounts of cupric ion, well under 1 ppm, may occasionally be found, resulting from erosion or corrosion of brass or copper pipe fittings. Traces of ferric or cupric ions have been said to cause color changes in certain artificial food coloring substances. Copper is also said to catalyze the oxidation of certain fats, and its presence in sugar can cause problems in food mixes (10).

Specifications for limits of lead and arsenic are set in common with all foodstuffs, and the writer is not aware that even traces of these metals have ever been found in granulated sugars.

ICUMSA or AOAC (Association of Official Agricultural Chemists) methods of analysis are standard for these trace metals. For copper and arsenic, most used are the colorimetric determinations with diethyldithiocarbaminate (3,64f); and for lead, the colorimetric determinations with dithizone (64d). Atomic absorption methods have been shown to be very rapid and quite accurate for these determinations (13).

Sulfur Dioxide. Because the organic portion of the nonsucrose materials in beet sugars contains some nitrogenous materials, it is the usual practice to add very small amounts of sulfur dioxide to sugar factory thin juices immediately before concentration in the evaporators, to inhibit catalytically the browning reaction between traces of invert sugar and amino acids, which form colored compounds (see p. 269).

As a result, traces of sulfur dioxide may be found in granulated sugars. In the United States the amounts found in sugar of commerce do not affect any customer use. Excessive amounts, however, can cause pin-hole perforations in cans containing sweetened foods, and other difficulties. Existing limits for sulfur dioxide are: Codex Alimentarius, 20 ppm. Such an amount is clearly excessive for certain uses of sugar.

The official ICUMSA method of analysis for sulfur dioxide traces is the colorimetric method using rosaniline (14). For routine analyses, the iodimetric titration is widely used in the United States industry.

Floc. Aside from coloring materials, the chief organic substances which are important in sugar quality are those responsible for foaming and *floc*. Floc is of two kinds: that appearing in acidified solutions, and that appearing in alcoholic solutions. Acid flocs appear in sugar solutions made from both cane and beet sugars. Alcoholic floc is normally characteristic only of cane sugars.

Highly surface-active, difficultly-soluble solutes owe part of their solubility to their concentrations at liquid interfaces. On acidification, at pH values between 1.5 and 3.0, the molecules may be displaced from the surfaces by the hydrogen ions, and forced out of solution. For example, traces of mineral oil dissolved in water leave solution on acidification, causing a diffuse haze. If certain "triggering" materials are present, however, the oils will form aggregates with the triggering substances, as flocculent precipitates, presenting an unsightly appearance. Thus in carbonated beverages, such flocs may settle to the bottom, or rise to the surface to form a ring in the neck of the bottle.

In granulated sugars of beet origin these triggering substances are known to be traces of saponin coming from just under the sugarbeet skins (see pp. 43-45). In general, amounts of saponin materials over 2 ppm (29) in the granulated sugar will produce visible flocs after standing in acidified solution for up to 8 days.

In existing standards, the carbonated beverage industry specifies that visible flocs be absent.

There are no official analytical methods. For control purposes, either the subjective or objective approach may be taken. The former involves evaluations in which the conditions in actual industrial use are approximated, with qualitative, organoleptic ratings of floc intensity.

The reference subjective method, is the *Coca-Cola test* (64), formulated by C. Gortatowsky, whose pioneering interest furnished the impetus for the discovery of the causative factors by Eis and co-workers (21); and the widely used Spreckels test (1) is an acceleration of the 8-day Coca-Cola test to a one-day interval.

Objective tests have the advantage of better precision, and the disadvantage of poorer accuracy. Visible floc is not measured, but factors which may not be closely related, such as the actual weight of the separated flocs, their hemolytic power, the surface activity of their solutions, or direct colorimetric tests for either the sapogenin (oleanolic acid) or the algycone (glucuronic acid). The weight of the floc is not necessarily related to its visual intensity.

Tests for surface activity assume that the saponin and its parts are the only surface active agents in the sugar, which is not always so. Verhaart (73) has pointed out that other surface-active compounds in beet juices include caramel, cholesterol,

and albumins. Chemical tests for the saponin or its constituents are only useful if these compounds are a constant percentage of the floc-formers, and actually beet-sugar flocs have been analyzed which contain from 20 to 80% of saponin substance, the balance being traces of defoaming materials, natural beet fats, or other, unidentified, surface-active, slightly-soluble nonsucrose materials.

The gravimetric procedure, the Tollens napthoresorcinol analysis, and the hemolysis test are described by Eis (21). Surface activity may be measured with the polarograph, where the measure depends on the degree of suppression of the oxygen maximum (12,72). This has also been proposed as a means for judging the overall quality of granulated sugars. A method depending on surface tension involves titration with solutions of quaternary amines (40). Chemical colorimetric procedures, in addition to the above mentioned Tollens reaction, include the reaction with a saponin of antimony pentachloride (75,76) the α -naphthol–sulfuric acid reaction (6,25), the red coloration with sulfuric acid alone (50), and the Liebermann-Burchard coloration with acetic and sulfuric acids (39).

Foam Test. When a granulated sugar is dissolved in water by pouring it into a tank with cold water, and then applying heat, just before and after the boiling point is reached a surface film of bubbles forms, made of air bubbles from air entrapped between the sugar crystals, and of air previously dissolved in the water and expelled by the heat. Depending on the characteristics of the trace constituents of the sugar, or indeed, sometimes of other components added to the solution by the formulator, the foam may readily break and then disappear, or it may rise up and overflow the tank. In any event the foaming can be avoided by adding the sugar to water which has already been heated to the boiling point. This, however, is inconvenient for some users.

The foam is, of course, a consequence of the presence of compounds in the solution which lower the surface tension, and, as mentioned above, under floc, this is chiefly saponin or its derivatives. A saponin-free beet granulated sugar does not usually present a foaming problem (18a).

While there are no official foaming tests, the procedure in common use in the United States is based on that published by Paine, Badollet, and Keane (55).

Strength. As mentioned in the discussion of *ash*, the strength of a sugar is a measure of its resistance to inversion, especially as accelerated by heat. This is important in the making of hard candies, where a molten mass of sugar may be manipulated under radiant heat at temperatures as high as 177 °C, and undue inversion will cause the final hard candies to be sticky. Strength is also important when a solution of sugar and other ingredients is intentionally inverted to a desired degree by heating with tartaric acid or other source of catalyzing acidity. If the sugar has insufficient, or too much strength, the anticipated degree of inversion under the fixed conditions may not be realized, making it difficult to produce a uniform product without expensive hand control. Historically, most of the tests for strength have involved the making of "barley candy," a hard candy made by heating a sugar-water solution to about 176 °C and then pouring the mass on a slab to cool. The candy is then analyzed, most conveniently for its solution color, which is strongly affected by the colored decomposition products of invert sugar, or for its remaining invert content. The standard ICUMSA method is that of the United States Bureau of Standards (34).

Odor and Taste. Sugar should above all else be sweet in taste, and it is invariably so; there is no difference between the sweetness of different samples of pure crystalline sucrose, regardless of the source. Other odor or taste characteristics are

minor. Sugar is noted for its ability to acquire odors from the atmosphere surrounding it, and it is a common experience to find the sugar with a slight odor derived from the paper or cardboard of the container in which it was packed. Occasionally, granulated sugars will be found with fragrantly perfumed aromas, acquired during storage in a warehouse next to a consignment of perfumed detergent, or less pleasantly, with an odor of kerosene, from storage next to this commodity in a small grocery store. These acquired fragrances can be avoided by packaging in completely air-tight packages, which in some cases can considerably increase costs. Sugar may also reflect the atmosphere of the factory where it was made.

However, for use in sweetening very delicately flavored beverages, minute odors may be the subject of criticism from bottler users, having as sources organic materials volatile at the pH of the existing aqueous film on the surfaces of the sugar crystals. These often are formed as the result of microbial action. For example, Hoffman and Hirschmüller (31) found traces of various aldehydes, glyoxals, and glyceraldehydes in European white sugars. Recent investigations by Mills and associates have shown that certain Amadori compounds resulting from the Maillard or browning reaction are flavor precursors (47a). Of greatest importance for carbonated beverages is the aroma of the acidified solution, where various organic acids become volatile. Gas chromatography has made possible identification of a number of the lower members of the fatty acid series (58,62,65).

Of necessity, methods of analysis for odor are subjective. To examine the odor of a dry sugar it is usual to place the sample in a sealed jar in a warm place for 30 minutes, and then to remove the cover and make a quick olfactory judgment. Considerable work has been done to develop the method of "threshold dilution," in which a sugar solution is diluted in a series of steps, continuing until no difference can be detected between the diluted sample and the taste or odor of a sample of carefully deodorized water (2,68,77). In general threshold methods have not been found to correlate well with the full-strength evaluations. For tests of acidified aroma, the sugar solution is commonly acidified to about 1.5 pH with phosphoric or other acid, and is organoleptically judged before or after heating.

In all odor and taste analyses the utmost care must be taken to clean and deodorize sample containers, beakers, and water, and the analyst must avoid eating or smoking for a time before the test. Frequently such tests cannot be made in the usual atmosphere of the sugar-factory laboratory. It is customary to provide individual booths for members of the panel, to avoid distracting or biasing influences. It is usual to deodorize the air circulated through the room.

pH. For some purposes the pH of white sugars may be of importance, and this must be measured in solution. The ionization constant of pure sucrose has been reported as, $K = 2.4 \times 10^{-18}$ at 18 °C(47). Kunin has published results of determinations of pH at various concentrations, which are shown in Table 16-1 (43).

Table 16-1 pH of Pure Sucrose Solutions at 18°C

% Concentration	pH Measured	pH Calculated
1	6.9	6.9
5	6.7	6.7
15	6.4	6.5
60	6.0	6.0

In practice these values are affected by the influence of trace impurities. The ICUMSA method (37) requires determination in a 60° Brix solution, using water of specified purity, as developed by Dubourg *et al.* (20).

Purity. The fact that in the early part of the century, sugars were sold, both of beet and cane origin, containing excessive quantities of impurities, led to industrial users establishing various criteria of purity, or more exactly, criteria of the degree of purification involved in the manufacture of the sugar. Thus the confectioners acquired a prejudice in favor of large-grained sugars, which, because of their smaller crystal-face area per unit weight contained smaller traces of mother syrup.

Other arbitrary indicators of "purity" sometimes applied, are: color; sulfur dioxide, an excess of which could indicate a "plantation white"; or ash. These are all, to some extent, measures of purity; however, they are usually quite misleading. Activated carbon can selectively remove coloring matter; many highly purified sugars contain relatively large quantities of sulfur dioxide, and variations of ash content with the same degree of refining, may reflect rather the beets or raw cane materials than the degree of refining.

The use of a polarographic method for "purity" measurement has been mentioned above (p. 553), but this is biased toward surface-active materials to the near exclusion of sensitivity to other nonsucrose materials.

Pasetti (56,57) uses a general method for evaluation of sugar purity, an oxidation by potassium permanganate under controlled conditions; i.e., 1 ml of 0.1*N* $KMnO_4$ and 0.5 ml glacial acetic acid, added to 100g of a 50° Brix solution at 70-71 °C. The time of color change from violet to yellow depends on the sugar, varying from 8 to 80 seconds.

Properties of Solutions

Color and Turbidity. The "color" of sugar has traditionally been used as one of rough measures of the purity of the product. As before mentioned, the visual appearance of granulated sugar is affected by the color, the surface gloss, and the crystal size. Since gloss and crystal size affect the reflectance markedly, it is difficult to measure objectively the color of granulated sugar in its solid state. When grading on the basis of visual appearance is considered adequate, the product is compared visually with carefully prepared standards under prescribed viewing conditions. Since the gloss of the crystals may change with time due to loss or gain of moisture, or as a result of abrasion, visual appearance is not a reliable measure of product purity.

The development of highly accurate optical instruments now permits the measurement of the optical properties of sugar in solution. Such measurements eliminate the variables of surface gloss and grain size. In solution colorimetry, two optical properties may be determined: the absorption of radiant energy due to the presence of colorants, and the scattering of radiant energy; i.e., the turbidity due to refractive inhomogeneities. The ICUMSA (35) recognizes the significance of these two properties in its recommendations: "1. the basis for the specification of sugar color shall be the absorption of visible radiation by a solution of the sugar. 1a. The basis for the specification of sugar turbidity shall be the scattering of visible radiation by a solution of the sugar."

The development in 1962 of the "sphere photometer" (9) made it possible to measure sugar color and turbidity rapidly and accurately by purely optical means.

Before the sphere photometer became available as a laboratory instrument, a number of approximate methods for the estimation of color and turbidity were developed (36). In the purely optical methods, the light transmission of the solution is determined at two wavelengths, 420 and 720 nm (mμ), the latter being used to correct the measurement at 420 nm for the effects of turbidity (25). In other methods, it is attempted to remove the impurities which cause the turbidity by filtration, prior to the measurement of light transmission. The accuracy of the two-wavelengths methods is adversely affected by the variability of the correction factor related to the turbidity estimation (46). The methods employing filtration suffer inaccuracies due to the removal of significant amounts of colorants together with the suspensoids. The presence of turbidity itself causes further inaccuracies in the transmission measurements because the light is scattered predominantly in the forward direction. Unless suitably modified to exclude forward-scattered light from the detector, colorimeters and spectrophotometers will indicate transmissions significantly above the true values (8).

The ICUMSA recognizes only method 4, the filtration method for white sugars, using 420 nm, adopted by the Codex Alimentarius.

In 1970 the U.S. National Committee of ICUMSA recommended that methods 1a and 1b be removed from the ICUMSA-approved listing.

The color limits placed by the Codex Alimentarius are 60 maximum ICUMSA units; and by the NSDA, 35 Bottlers reference basis units (ICUMSA method 1a).

Foreign Materials

Microorganisms. In addition to freedom from pathogenic organisms, which requirement sugar shares with all other foods, the absence of spoilage or fermenting organisms is required for certain uses. The NSDA has specific limitations involving sugar to be used for making carbonated and other soft drinks, for mesophilic bacteria, and for yeasts and molds (1) (see p. 733). Even though many of the drinks are at low pH, dilute sugar solutions are particularly susceptible as likely media for growth. The National Food Processors Association likewise has limits for thermophilic organisms (see p. 734), which could survive the heat treatment involved in canning. Microorganisms are discussed in detail in Chapter XXIII.

Sediment. Specifically, sediment refers to insoluble material collected on a filter surface through which a solution of the sugar under test has been passed. Such material, extremely small in quantity, may be made up of small particles of paper or other fibers from package materials, scale or oxide fragments from equipment, small metallic particles from metal equipment erosion, traces of particles from filter aid, small carbon particles from activated carbon treatment, "fly-ash" from boilers, or small pieces of dried beet pulp.

In the United States sediment is usually determined using equipment similar to the "milk-sediment tester," and such a procedure is specified by the NSDA (1), in which the sediment from 300 g of sugar is collected on a 1-3/16 in. diameter filter disc, and visually compared with a standard sediment disc furnished by the Association. The standard contains the limit, or 2 ppm. Various procedures and equipment for sediment determination using membrane filters have been described in publications by Hibbert and Phillipson (30), Devillers (17,18), Friml (23), and Gruszecka (24). However, no method is universally accepted.

Specks. The word refers to colored or black and possibly soluble particles,

such as massecuite balls, large enough to be quite distinct to the eye and visible in granulated sugar.

Insoluble specks in sugar may be determined by a test using comparison standards as described in the 1962 ICUMSA proceedings (32). Both soluble and insoluble specks may be determined by visual inspection of a given weight of granulated sugar spread out on a surface, which is tiresome and time-consuming. Equipment to make such visual examination easier has been reported by Kregel (42).

Results of examinations for specks are frequently expressed in terms of number of visible specks per weight of sugar: for example, 50 g. Specks are sometimes additionally graded as to size and visual intensity. There are no specifications of specks of record.

Rubber. Small pieces of rubber dust may be eroded from conveyor belts, rubber hoses, gaskets, or, to a minor extent from rubber balls used to prevent blinding of moving screens. These rubber particles are objectionable to certain beverage bottlers, since they float on the surface of beverage concentrates, and may react with some of the colloidal constituents (11). Thus, very close tolerances of rubber particles are set by these consumers. To meet these requirements, in many cases rubber must be removed as a material for equipment construction.

Practical Maintenance of Sugar Quality

This section is designed to furnish a check list in convenient form, for important factors in the control of sugar quality.

Check List of Sugar Quality Imperfections and Their Causes and Remedies

A. **Pan Scale.** These are constituents of the sediment and the ash of the sugar, which come from precipitation of lime salts in the vacuum pans. The primary cause is the presence of slightly soluble lime salts in the standard liquor.

1. Diffusion. Caused by poor quality cossettes, degraded beets, over-diffusion from such factors as too-high temperatures or too-long cossette detention times, low-quality diffuser supply water, and excessive degradation from microorganisms. Remedies are sharp knives, beets free from trash, optimum diffusion conditions, sound beets, microbial control measures such as sterilization by heat and chemical agents, and good quality diffuser supply water.

2. Defecation and First Carbonation. Caused by insufficient lime, too-high temperature in mild processes, too-low first carbonation alkalinity, or other errors in juice purification. As remedial measures, use enough lime, hold the alkalinity at the optimum point, keep the temperature of first carbonation in defeco-carbonation processes as low as consistent with the setting of other variables, and still permit satisfactory separation of first carbonation sludge.

3. Second Carbonation. Caused by alkalinity not at the optimum, lime salts in the thin juice too high, or temperature of carbonation not high enough. To remedy, the alkalinity should be held at the routinely determined optimum, the temperatures kept as high as possible, adequate holding time should be provided for the reduction of the supersaturation of the calcium carbonate after carbonation, too-low natural or effective alkalinity should be restored with soda ash, and additions of small amounts of lime to second carbonation may be found effective.

4. Second Carbonation Filtration. The filtration should completely remove the precipitated calcium carbonate to avoid solution of the particles in sulfitation, with consequent increase of the dissolved lime salts.

5. Evaporators. Inversion can occur from low pH values in juices in passage, particularly since the pH is considerably lowered at the higher temperatures in the first effects. In such cases, measures should be taken to increase the pH of the thin juices. Sulfitation of thin juice may be reduced to raise the whole pH pattern, and if necessary, split addition of sulfur dioxide may be used to both thin and thick juices. Watch for microbiological action in low-pressure evaporator bodies.

B. High Sugar Color. Basic causes: presence of colored nonsucroses from invert decomposition, the browning reaction, incomplete purification, and rarely from caramelization.

1. Diffusion. See A. 1.

2. Defecation and First Carbonation. Low alkalinities are common causes of high color, since destruction of invert at low pH results in much more color. Also all factors in A. 2. are important.

3. Second Carbonation. See A. 3.

4. Sulfitation. There should be a minimum of 20 ppm of sulfur dioxide in all sugar-end juices and liquors to prevent the browning reaction.

5. Evaporators. See A. 5.

6. Standard Liquor Filtration. All suspensoids passing through these filters will enter the product. Constant control is required of filtered liquors, whether by clarity meter or sediment testing.

7. White Sugar Boiling. Adequate feed-liquor purity is primary; avoidance of agglomeration, and use of minimum boiling times and temperatures is necessary to avoid sugar degradation, caramelization, and invert-color formation. Boilings should be carefully scheduled to eliminate the necessity of holding, awaiting receiving room in the mixers.

8. White Centrifuging. Complete separation of the mother liquor from the crystals must be made by correct setting of wash amounts, quality, spray patterns, temperatures, and times of application; proper basket loading; adequate basket cleaning between loads, periodical washups to clean the centrifugal spider; elimination of massecuite leaks to product; and diversion of the sugar to remelt if anything goes wrong.

9. Remelt Boiling and Centrifuging. Keep all massecuite pH values over 7.0, as acid massecuite will steadily invert and form color. Remelt sugars must be of high purity and low color, since major quantities of color, ash, and floc substance enter the white massecuite from the remelt sugars. For this purpose, maintenance of the massecuite purities at proper levels is essential.

10. Drying and Granulating. Avoid temperatures high enough to caramelize; do not pass sugar with high moisture to the product, as the color will increase in storage. Sugar to storage should be as cool as possible; color increases in storage approximately double for each 10 °C increase in temperature.

11. Screening. Scalp off all coarse particles, which may include colored massecuite agglomerates, as these strongly increase the color. Do not overload the screens; keep the screens from blinding; watch closely for tears in the screens; do not recycle coarse scalpings over the screens.

12. Contamination by Equipment. Send to remelt the first sugar through equipment: after being empty for an appreciable period; after repairs; after "hammering"; and after boiling out. Avoid syrup drips into product under centrifugals and elsewhere. Do not send high moisture product to storage. Avoid using the sugar in the very bottom of a storage bin for product purposes.

C. **Solution Turbidity.** Primary causes are: slightly soluble lime salts, and standard liquor filter leaks.

1. Diffusion. See A. 1.

2. Defecation and First Carbonation. Insufficient lime addition is an important cause of high lime salts and turbidity. See A. 2.

3. Second Carbonation. Too-high alkalinity is a major cause of turbidity. See also A. 3.

4. Second Carbonation Filtration. Tight filtration is essential to prevent solution of leaking calcium carbonate particles in sulfitation.

5. Standard Liquor Filtration. See B. 6. A few drops of concentrated hydrochloric acid added to a white sugar solution will show if the turbidity is due to acid-soluble lime salts, or to leaking filter aid. Breakdown of polyphosphate additives to form primary phosphates, with resultant formation of calcium phosphate may be a contributing factor to acid soluble lime-salt turbidities.

6. White Sugar Boiling. Turbidity-forming lime salts are formed from sucrose degraded in the pans. See B. 7.

7. Foam-Breaking oils. May cause turbidity in sugar produced.

D. **Odor.** Primary cause: improper purification and failure to remove all traces of mother liquor from the sugar.

1. Defecation and First Carbonation. An adequate amount of lime is essential. See A. 2.

2. White Sugar Boiling. Occlusions of mother liquor in agglomerates must be avoided. See B. 7.

3. White Centrifuging. Wash water should be treated well water, rather than odorous condensates. Wash the water to the ash minimum rather than to a satisfactory color level.

4. Remelt Boiling and Centrifuging. Remelt sugar purities should be high to minimize return of molasses nonsucroses to the white sugar. See B. 9.

5. Drying and Granulating. High moisture sugar in storage is susceptible to both odor absorption and development. Development of high-moisture pockets due to migration from temperature differentials increases the difficulty. Odors may also be enhanced by microbial infection in wet sugar. See B. 10.

6. Contamination from Atmosphere. Sugar will absorb odors in the factory atmosphere from sources such as kilns and waste-water ponds, as well as normal factory "sugarbeet aroma."

7. Contamination from Equipment. Syrup drips into sugar product, or condensed moisture from equipment can result in odor increase. Sugar in the bottom portions of bins may be more strongly odored, and should be remelted.

E. **Floc.** Primary causes: the beets, improper juice purification, any factor returning nonsucroses to the white massecuite, or any factor making the floc less soluble in the standard liquor.

1. The Beets. Beets vary in their extractable saponin contents. Saponin is more easily extracted from degraded beets, as from those frozen and thawed, or from long-term storage.

2. Defecation and First Carbonation. Good purification will remove 97% of the floc. See A. 2.

3. Second Carbonation. A small lime addition in second carbonation is frequently helpful in reducing floc.

4. Supplementary Juice Purification. Activates carbon, either powdered or granular, or ion-exchange treatment are positive remedies for floc.

5. White Sugar Boiling. Floc is carried in occlusions of mother liquor in agglomerates. See B. 7.

6. White Centrifuging. Floc is tightly adsorbed to crystal surfaces. The syrup removal should be clean and complete. See B. 8.

7. pH of Massecuite. Keep the massecuite pH at the desired high level to prevent increase of floc in the sugar due to the lower solubility of the floc at lower pH values.

F. **Ash.** Primary causes: any factor increasing lime salts, traces of mother liquor in the product, inadequate centrifugal washing.

1. Defecation and First Carbonation. Operate to produce minimum lime salts. See A. 2.

2. Second Carbonation. See A. 3.

3. Second Carbonation Filtration. Lime salts are greatly increased by incomplete filtration.

4. Sulfitation. Formation of imidosulfonates is sometimes a major factor in ash formation. This should be suspected if the known amount of sulfur dioxide added to the juices is not found in the analysis of the sulfured juices. The remedy is to eliminate the sources of nitrate-forming infection.

5. Standard Liquor Filtration. See B. 6.

6. White Sugar Boiling. See B. 7.

7. White Centrifuging. The conductivity ash content of the wet white sugar provides an excellent means of wash-water control, and of final sugar ash control. See B. 8. Be sure Zeolite-type wash-water softeners do not leak salt-brine after regeneration.

8. Contamination from Atmosphere. Any insoluble inorganic materials entering from the atmosphere are measured as ash constituents. A few specks can greatly increase the ash of a sample. Open centrifugals, scrolls, and pan-storage tanks should be closed. Dust should be kept out of the sugar end, from such sources as pulp driers, lime kilns and lime pulverizers, fly-ash, and carbon kilns. Air filters or dust-precipitators used in cleaning air to the driers must be kept effective.

G. **High Sulfites in Sugar.** Primary cause: too much sulfur dioxide used, or in the wrong places. Sulfur dioxide addition should be for catalytic color inhibition only, so that the quantity in the white fillmass should not exeed 25 ppm.

H. **High Sugar Foaming Test.** This is chiefly a function of the amount of saponin in the sugar. See E.

I. **Sediment.** Primary causes: improper standard liquor filtration, or contamination from the atmosphere or equipment.

1. Standard Liquor Filtration. See B. 6.

2. White Centrifuging. The wash water must be filtered quite free from sediment.

3. Drying and Granulating. The air used in the units must be adequately filtered.

4. Screening. The very-fine fraction, containing minute dust particles, should be screened out and reprocessed. See also B. 11.

5. Contamination by Atmosphere. See F. 6.

6. Contamination by Equipment. See B. 12. In addition, the use of stainless steels and other appropriate corrosion-resisting metals or plastics is important to avoid specks of rust, other oxides, or mill scale. Cast iron, which has reached the stage of porous disintegration, is a serious offender.

J. **Microorganisms.** Primary causes: imperfect standard liquor filtration, contamination from air and equipment, and high moisture in product.

1. Standard Liquor Filtration. A tight filtration will remove all yeasts, molds, thermophiles, and most mesophiles.

2. White Centrifuging. Improper sanitation around the centrifugals, syrup leaks, fermented paddle-cleaning water, and contaminated air at the centrifugal station will increase the microbial count. An open centrifugal draws air through the sugar layer, and the solid contents of the air are retained by the sugar.

3. Drying and Granulating. Production of sugar with too high a moisture content assists in the continuation of bacterial life. Most microorganisms, other than spores, in properly dried sugar will die from dehydration within a few weeks time. Air used in driers must be properly filtered.

4. Contamination by Atmosphere. See F. 8.

5. Contamination from Equipment. Serious microbial contamination can result from drips of syrup or condensed moisture into the product sugar. Avoid using the very bottom sugar in the bins, which frequently has a high bacterial count.

K. **Poor Color Stability in Storage.** Primary causes: use of sulfur dioxide as a bleach, high moisture or invert in the product, or high storage temperatures.

1. Sulfitation. The practice of using sulfur dioxide in quantities large enough to actually bleach juice results in return of color in the stored sugar by oxidation of the sulfite-containing compounds by the oxygen of the air. Sulfur dioxide should be used only as a browning reaction inhibitor.

2. Drying and Granulating. Sugar must be dried to a low moisture content. Water is required for all sugar degradation reactions, including oxidation, inversion, color formation, and bacterial viability.

3. Screening. Presence of agglomerates with syrup occlusions results in sugar which will degrade in color in storage, due to high moisture and amino acids which will enable the browning reaction to proceed. See B. 11.

4. Storage Temperature. Many factories have no sugar coolers at all, and many that do, do not send the sugar to storage at a low enough temperature. Color increase in storage from most causes is greatly accelerated by higher temperatures.

L. **Agglomerates (ball grain).** Primary causes: leaks of syrup or water into product, techniques of pan boiling, inadequate centrifuging, and inadequate screening.

1. White Sugar Boiling. Agglomerates of crystals can be produced by too-high supersaturation during the period of "setting the grain," by air leaks into the pan, by too great a degree of "tightening" of the massecuite before discharging the contents of the pan.

2. White Centrifuging. Inadequate washing of the sugar in the centrifugals, whether due to improper spray-nozzle adjustment, or just not enough water, as well as inadequate basket cleaning and syrup drips into the product are frequent sources.

3. Screening. Inadequate scalping to remove filmass balls, or recirculation of scalpings causing small, broken, colored particles of agglomerates to remain in the product, are common causes.

4. Contamination from Equipment. See B. 12.

5. Formation in Storage Bins. High-moisture sugar in storage bins will result in moisture migration to the cool side of the bins, with local high moisture, inversion, color formation, and colored agglomerate formation.

Existent Sugar Standards

There are four specifications of standards for granulated sugar quality applicable to the United States: those of the Codex Alimentarius, the U.S. Pharmacopeia, the National Soft Drink Association, and the National Canners Association. Following are abstracts and excerpts from these standards.

Codex Alimentarius (15)

Polarization	99.7% minimum
Invert sugar	0.04% maximum, by wt.
Conductivity ash	0.04% maximum, by wt.
Loss on drying (3 hrs at 105 °C)	0.1% maximum, by wt.
Color (ICUMSA units)	60 maximum
Sulfur dioxide	20 mg/kg maximum
Arsenic (As)	1 mg/kg maximum
Lead (Pb)	2 mg/kg maximum
Copper (Cu)	2 mg/kg maximum

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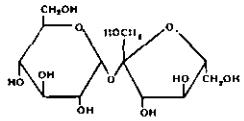

$C_{12}H_{22}O_{11}$ 342.30
α-D-Glucopyranoside, β-D-fructofuranosyl-.
Sucrose [*57-50-1*].

Sucrose is a sugar obtained from *Saccharum officinarum* Linné (Fam. Gramineae), *Beta vulgaris* Linné (Fam. Chenopodiaceae), and other sources. It contains no added substances.

Packaging and storage—Preserve in well-closed containers.

Specific rotation [781]: not less than +65.9 °, determined in a solution containing 2.6 g in each 10 ml, the sample having been previously dried at 105 ° for 2 hours.

Residue on ignition [281]: not more than 0.05%.

Chloride—to 10 ml of a solution (1 in 10), acidified with 2 *N* nitric acid to a pH of 1, add 1 ml of silver nitrate TS: no opalescence is produced within 1 minute.

Sulfate [221]—A 5.0-g portion shows no more sulfate than corresponds to *0.30 ml of 0.020 N sulfuric acid (0.006%).*

Calcium—To 10 ml of a solution (1 in 10) add 1 ml of ammonium oxalate TS: the solution remains clear for at least 1 minute.

Heavy metals [231]—Dissolve 4.0 g in 15 ml of water, add 1 ml of 0.12 *N* hydrochloric acid, and dilute with water to 25 ml: the limit is 5 ppm.

Invert sugar—Dissolve 20 g in water to make 100 ml, and filter if necessary. Place 50 ml of the clear liquid in a 250-ml beaker, add 50 ml of alkaline cupric tartrate TS, cover the beaker with a watch glass, and heat the mixture at such a rate that it comes to a boil in approximately 4 minutes, and boil for 2 minutes, accurately timed. Add at once 100 ml of cold, recently boiled water, and immediately collect the precipitated cuprous oxide on a tared filter crucible containing a sintered-glass disk of medium pore size, or suitable equivalent. Thoroughly wash the residue on the filter with hot water, then with 10 ml of alcohol, and finally with 10 ml of ether, and dry at 105° for 1 hour: the weight of the cuprous oxide does not exceed 112 mg.

National Soft Drink Association (1)

Standards for "Bottlers" granulated sugar. (For microbiological standards, see p. 734).

Ash: not more than 0.015%

Color: not more than 35 reference basis units (ICUMSA Method 1a).

Sediment: The sediment (means the insoluble or suspended matter such as pan scale, rust particles, filter aid, dirt, calcium deposits and other insoluble material) content shall not be more than shown on prepared sediment disc (approximately 2 ppm) available from National Soft Drink Association upon request).

Taste and Odor: shall have no obviously objectionable taste or odor in either dry form or in a 10% sugar solution acidified to pH 2.5 with U.S.P. phosphoric acid.

Floc evaluation of beet sugars: shall not show a positive floc test by the Spreckels Qualitative Floc Test Procedure. Universally accepted methods of testing for turbidity in cane and beet sugars and floc-producing substances in cane sugars have not been developed. As soon as accepted test methods and tolerances have been established they will be added to the standard.

Sampling: "Bottlers" sugar shall be adequately sampled by the producer immediately prior to packing to assure compliance with these standards.

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Chapter XVII

LIME AND CARBON DIOXIDE PRODUCTION*

The beet sugar factory requires a supply of lime (CaO) for carbonation and for the Steffen process, if used. The former also requires a supply of carbon dioxide (CO_2) gas. Both of these reagents are manufactured at the factory site by calcining calcium carbonate, which when heated decomposes to form the two desired products. The source of the calcium carbonate may be either limestone or carbonated lime waste from the sugar factory process. Calcination is performed in a direct-fired furnace called a lime kiln.

Quality of Lime and Carbon Dixoide. For satisfactory operation of the factory the lime and carbon dioxide must meet certain specifications. For the non-Steffen factory the most important requirements of the lime are: a) it should be relatively pure; i.e., upwards of 80% CaO; b) it should slake at a reasonably rapid rate; c) the non-CaO constituents should be relatively harmless to the factory process and non-toxic. Some impurities commonly found in limestone such as magnesium, iron, aluminum, and sulfate compounds are relatively harmless as they will be largely precipitated and eliminated from the juice in carbonation. Others such as silica can cause problems or inefficiencies in the factory process.

For Steffen work the following additional properties are required: a) the lime must be grindable to a powder with high specific surface. It is especially important in this regard that the lime not be over-calcined so as to produce shrinkage (30). See also pp 575-576; b) it must be nearly free from recombined CO_2; c) it should be cool.

The carbon dioxide gas must be of sufficient purity to react efficiently in the carbonating equipment being used. Carbonation equipment is usually designed for gas of at least 28% CO_2 content. It is practical, however, to design carbonators to operate with gas containing as little as 12% CO_2 by increasing the juice depth over the gas distributors or the cross-sectional area of the carbonating tanks and distributors or both. Regardless of equipment design the CO_2 content of the carbonating gas should be uniform to facilitate control of carbonation. The gas should also be free of unburned fractions of coal or petroleum, which are potential contaminants to the products of the factory.

Equipment for Limestone Calcining

The overwhelming majority of beet-sugar factories produce the necessary CaO and CO_2 by calcining limestone which is found in abundance throughout the world. Of the many types of kilns which have been designed for calcining limestone, the beet-sugar industry uses predominantly a type called the "vertical shaft" or "Belgian" kiln, because it is relatively inexpensive, simple and efficient. Modern vertical shaft kilns fall into two general categories: 1) *mixed feed with solid fuel*; 2) *gas fired* by direct injection of gaseous fuel or producer-gas generated from oil or coal in external gas generating chambers.

Mixed feed and gas- and oil-burning kilns will be described. Firing with coal producer-gas is not practiced in the beet-sugar industry.

Mixed feed kilns are fired internally by a solid fuel of high carbon content, coke or anthracite coal (15), which is charged into the kiln in a mixture with the limestone. Most common in the industry is the Belgian kiln illustrated in Figure

*by E.M. Hartmann. Revised by L.W. McCosh

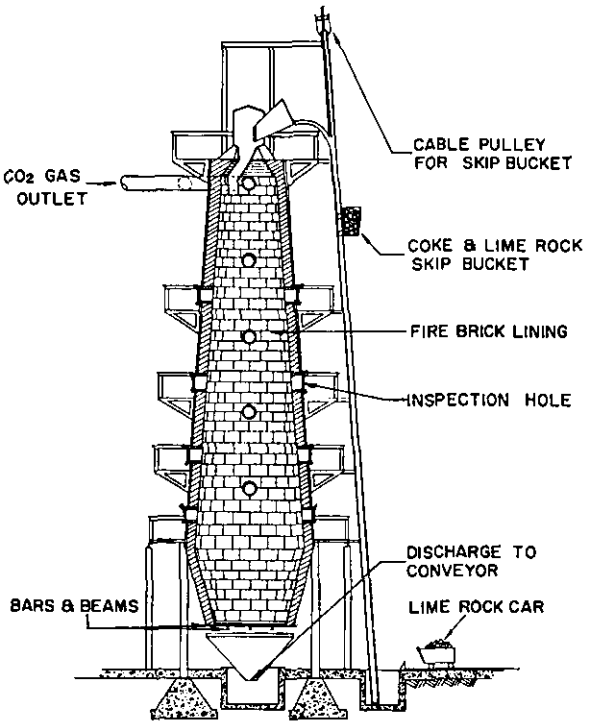

Figure 17-1. Vertical, or Belgian kiln.

17-1. It consists of two truncated cones fastened together at their bases. The lower cone is much shorter than the upper cone and is called the "bosh." The cones are made of sheet steel and are lined with refractory brick, usually from 9 to 14 in. thick. The upper cone is provided with several rows of holes for inspection of the contents during operation. These holes have tight-fitting steel doors or plugs. Typical design criteria for a Belgian kiln would call for a volume of 65 to 85 cu ft per ton of CaO produced per day, with a height-to-maximum-diameter ratio of 3.7 to 4.5.

The bottom of the kiln is provided with a fixed or movable grate arrangement which will control the removal of the burned product while allowing the continuous admission of combustion air. A conical hopper and conveyor are usually located under the grate or discharge mechanism for removal of the discharged lime.

The top of the kiln is closed by a steel hood in which a well-fitted door is provided for admission of the mixed limestone and fuel. Below the door is located a distributing arrangement to spread the charges of stone and fuel evenly over the cross section of the kiln and to reduce coning in the center. This distributor may consist of a stationary cone with apex up, or an offset swivel chute which can be turned in a different direction for each charge.

Large ports are provided below the door for removal of the gas produced by the kiln. The gas is piped through scrubbing equipment to a compressor which pro-

vides induced draft for the kiln and a pressurized supply of CO_2 gas for the carbonation. To the hood itself is usually connected a small centrifugal exhauster discharging to the atmosphere. Its purpose is to remove the air which enters when the door is opened for charging, and also to provide a small draft when first starting the kiln or when an emergency shutdown of the factory occurs.

An accurately proportioned mixture of limestone and solid fuel is introduced in batches to the top of the kiln. This charge is kept moving down through the kiln by withdrawal of the calcinated lime from the bottom. The product gas is continuously withdrawn at the top by the compressor, causing fresh air to enter the kiln at the bottom.

As the solid feed moves down through the upper third of the kiln, the preheating zone, it absorbs heat from the gases moving up from the fire zone. At the bottom of the preheating zone, the fuel reaches the kindling point and begins to burn.

Here starts the fire zone, which occupies approximately the central third of the kiln. In this zone, heat is generated continuously by combustion of the fuel and the oxygen supplied by the draft air. Under the influence of this heat, the calcium carbonate in the limestone dissociates into the two desired products while the combustion of the carbon in the fuel produces additional carbon dioxide.

At the bottom of the fire zone the fuel is exhausted and the cooling zone begins, occupying the remaining length of the kiln. Here the calcinated lime is cooled by the incoming draft air, which in turn is preheated for combustion in the fire zone.

Mixed feed shaft kilns have substantial advantages:

1. They are mechanically very simple.

2. They require low capital investment and moderate mechanical attention relative to other types of kilns.

3. They are thermally efficient; i.e., in the region of 70% as compared with 30% to 60% for some other types (16). A modern highly insulated German mixed feed kiln achieves an efficiency of 85% (16).

4. The CO_2 content of the gas product is high; i.e., 32% to 37%, requiring minimal investment in gas scrubbers, compressors, and carbonating equipment.

5. The ratio of fuel to limestone, which is of critical importance, is predetermined in the charge. This simplifies the problem of controlling the kiln under conditions of varying operating rates or emergency stoppages.

Mixed feed shaft kilns have disadvantages, too:

1. They are critical of limestone quality, requiring a stone that is well size-graded, nondecrepitating, not easy to melt, and able to yield an active lime when calcined over a wide range of temperature. It seems impossible to achieve precise and uniform distribution of temperature throughout the fire zone of a mixed feed kiln (28).

2. They are sensitive to solid fuel quality, requiring for good results a hard, high purity coke or anthracite of controlled size gradation. Such fuel is expensive in many places.

3. Their operation is somewhat of an art, requiring subjective judgment of such factors as temperature and heat distribution, degree and uniformity of calcining, uniformity of charge movement, and incipient melting. Such judgment must be largely based on visual inspection through the portholes. The transit time of the

solids is on the order of 24 hours, so that problems created by operating errors, automation failures, or changes in feed quality are not quickly rectified.

4. Fuel ash and unburned fuel become mixed with the lime.

Mixed feed shaft kilns have received attention from modern technology, resulting in lower costs and more satisfactory operation. Following are some features of modern kilns as described by Annis (1):

1. Automation: The mechanical grates which discharge the calcined lime are controlled by the liquid level in the lime milk tank. As the fill level in the kiln subsides, this is detected by a nuclear level gauge at the top of the kiln, which activates the charging mechanism. The latter is programmed electronically to weigh out the proper proportions of limestone and fuel into the charging skip and hoist it to the top of the kiln, properly activating the kiln door and charge distributor. Temperatures at various points of the kiln periphery are indicated by thermocouples encased in hollow blocks of carborundum. Figure 17-2 shows a modern control panel for a mixed feed kiln and lime slaking plant.

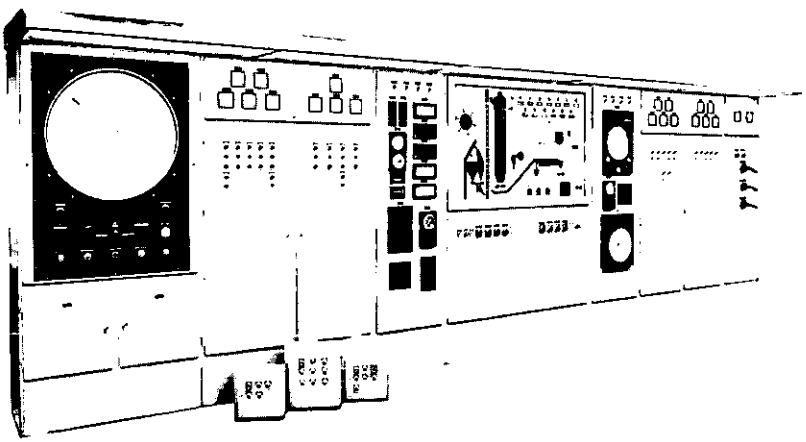

Figure 17-2. Control panel for a modern, automated, mixed-feed shaft kiln and slaking plant. *Courtesy of Cocksedge & Company, Ltd., Ipswich, England.* The instruments on this panel are as follows: 1) Scale dial for rock and coke weighing hopper. 2) Signal lamps for weighing hopper door and skip hoist motor. 3) Alarm for excessive down time of skip hoist motor. 4) Counters for skip hoist trips (daily and total). 5) Signal lamp for position of rotary charger door. 6) Signal lamp for turning of rotary charger. 7) Pyrometer indicators for lining temperatures. 8) Recorder of charging time. 9) Kiln draft pressure indicator. 10) Exhaust gas temperature recorder. 11) Exhaust gas % CO_2 recorder. 12) Signal lamps for motors on discharger mechanism, quicklime conveyors and slaking plant motors. 13) Recorders for milk of lime storage tank level and density. 14) Indicator for temperature of slaking sweet water. 15) Motor starters and interlock sequence controls.

2. Improved refractory linings: Renewal of the kiln lining, at least in the fire zone, used to be a regular feature of each intercampaign's maintenance program. Improved refractories have now lengthened the life of the lining to as much as six years, with minor patching. Durable plastic refractories are becoming available whereby patching can be effected by driving anchor studs into the steel shell, and then spraying or plastering the refractory material. Full linings of plastic refractory are a future possibility.

The suppliers of refractory materials should be consulted as to specifications for lining materials. Generally the more durable refractories are higher in price and

their relative economics must be evaluated in the light of local labor prices for installation, duration of campaigns and other economic factors.

3. Improved design details:

a. Cylindrical instead of conical kilns are common. They are probably less expensive to design and construct than tapered kilns. The theoretical advantages of tapering; i.e., freer downward movement of the charge and less abrasion to the lining, are not clearly demonstrable in practice.

b. Structural support is greatly simplified by a chimney-like design in which the shell of the upper cone or cylinder is continued past the bosh to foundation level. This replaces the stanchion and beam support at lower cost. It also provides an enclosure for the discharging machinery, sometimes eliminating the need for a building.

c. Pneumatic scales are used for weighing the limestone and fuel. They are said to be more durable and accurate than beam scales under the shock-loading and dusty conditions of kiln charging.

d. A double-decked weighing hopper for the limestone and fuel results in a more uniform mixture when the charge is dumped into the kiln.

e. A special charging bucket for the initial filling of the kiln is lowered by a hoist into the kiln to deposit the initial charges gently on top of the fill. This eliminates the shattering caused by the long fall of the feed when charging an empty kiln, reducing the amount of shattered fines in the initial fill. Start-up problems are thereby reduced. After the kiln is filled to operating level, the special bucket is withdrawn from the kiln.

Gas- and Oil-Fired Shaft Kilns

Development of these has been stimulated in the last three decades by the increasing availability and decreasing cost of fuel gas and oil relative to coke and anthracite. Basically, the gas-burning type consists of a shaft similar to the mixed feed kiln, but equipped with stationary burners at various points for the injection of gaseous fuel. In oil firing, external gasification chambers are provided to convert the fuel oil to fuel gas before it enters the kiln (6). Thus they are all really "gas fired." Modern gas-fired kilns have provisions for injection of the gaseous fuel into the center of the kiln, as well as the periphery, for more uniform temperature distribution. Some have provision for the recirculation of exhaust gases to produce a tempering effect and to control the length of the fire zone. Some also have provision for bypassing a portion of the secondary combustion air around the level of fuel injection to temper the fire and control its length. Figures 17-3 through 17-6 show three types of gas-burning shaft kilns in use in the North American beet-sugar industry, and one type of oil-burning shaft kiln.

Gas- and oil-fired shaft kilns have these advantages:

1. High thermal efficiency, although not so high as mixed feed kilns: i.e., about 55% for gas firing (29,39) and 60% for oil firing (22).

2. Reduced fuel cost compared to mixed feed kilns where fuel gas or oil are enough cheaper per heat unit than coke or anthracite to offset the difference in thermal efficiency.

3. Instant control of fuel quantity and distribution.

4. Control of flame tempering and length.

5. Reduction or elimination of ash contamination by the fuel.

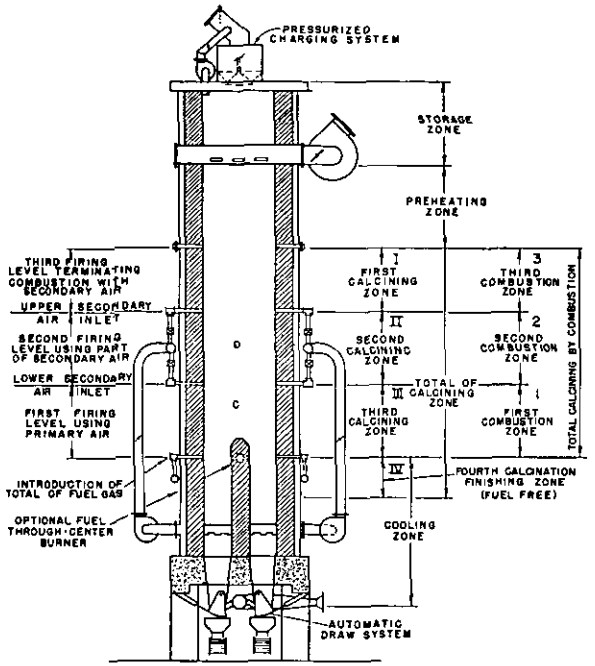

Figure 17-3. The Azbe "Ultimos" gas-fired kiln. *Courtesy, The Azbe Corporation.* This kiln is approximately square in cross section, but the cooling section is divided into two rectangles by a central refractory wall. This wall contains burner ports for the injection of fuel into the center of the kiln at the bottom of the fire zone. A portion of the secondary combustion air is withdrawn from the cooling zone by the lower blowers and bypassed to the "second firing level." This elongates the firing zone and tempers the fire by starving it at the level of fuel injection. Azbe has designed a further refinement called "Maximus" (7) in which the central wall is extended up to the level of the upper secondary air inlet. Ports in this wall enable the bypassed secondary air to be injected into the center as well as the periphery of the fire zone.

6. They are possibly not so demanding of limestone quality as mixed feed kilns, owing to the opportunity to control the temperature and temperature distribution more accurately.

7. They can tolerate smaller-sized limestone, which is often cheaper than the 2 in. or larger sizes required for mixed-feed kilns.

Disadvantages of gas- and oil-fired shaft kilns are:

1. Higher investment and mechanical attention because of the combustion equipment and controls, as compared with mixed feed kilns.

2. The internal burner arrangements in some cases tend to obstruct the flow of limestone through the kiln and increase the difficulty in the event of clinker formation.

3. The critical ratio of fuel to limestone is not predetermined. Thus, almost instant overburning, and sometimes clinkering, or underburning can occur if there is an unplanned change in the flow of limestone or if channeling takes place in the kiln.

To counter this problem, some gas-fired kilns are equipped with hopper scales under the kiln. Thus as each mechanical draw grate is activated, it is required to

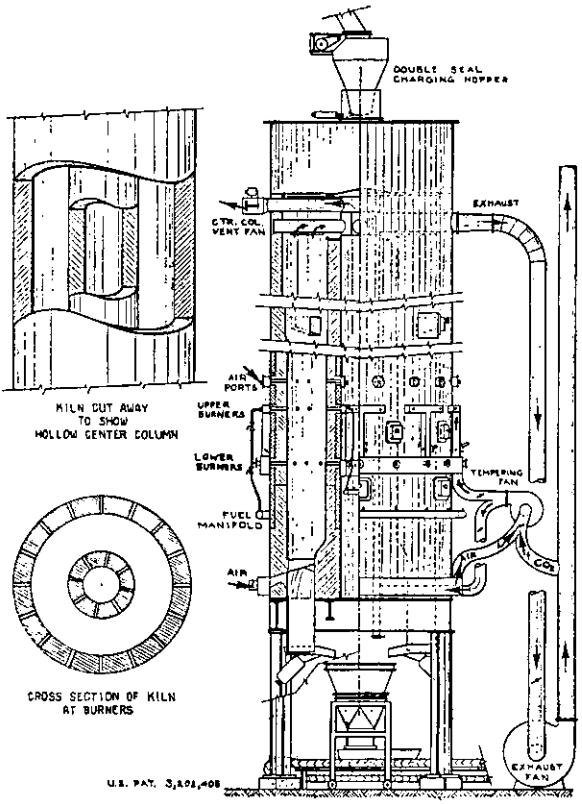

Figure 17-4. The Macin "Limelighter" kiln. ***Courtesy, Macin, Inc.*** **A central refractory tower makes this gas-fired kiln annular in cross section and provides means for the internal injection of gaseous fuel. All surfaces in contact with the charge are vertical. The auxiliary blower system is equipped for bypassing of secondary air as in the Azbe Kiln as well as for recirculation of a portion of the exhaust gas. A hopper scale is located under the automatic draw mechanism for precise control of the draw.**

continue in operation until a predetermined weight of lime has been drawn from its section of the kiln. If such predetermined weight is not drawn within an allotted time an alarm sounds. This system provides considerable assurance against potentially serious, unexpected variation in the fuel:limestone ratio. However, it increases the requirements of investment cost and mechanical attention.

4. The CO_2 content of the kiln gas is lower than that from mixed fed kilns because of the higher hydrogen:carbon ratio in the gas and liquid fuels. Typical round values worked out from Azbe's chart (5, 17) of thermal efficiency vs. exhaust gas analysis are as in Table 17-1.

Table 17-1

	Thermal Efficiency %	M Btu per ton CaO	% CO_2 (vol) in gas, assuming 2.5% O_2 and no CO
Coke firing	70	4000	35
Bunker C oil firing	60	4600	32
Texas type natural gas firing	55	5000	29

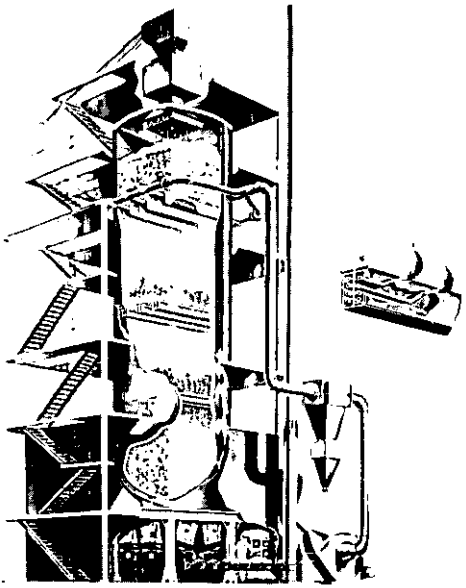

Figure 17-5. The Union Carbide Gas-Fired Kiln. *Courtesy of Union Carbide Corporation.* Distribution of the gaseous fuel is accomplished by water-cooled steel burner beams arranged in two tiers. Pipes within each beam carry the fuel to a series of evenly spaced ports on either side of the beam. Sight ports give access to the void space under each beam and provide means of inspecting the charge completely across the width of the kiln. Means are provided for renewing any beam without shutting down or emptying the kiln. Means of bypassing or recirculating gases is not provided and is apparently unnecessary. A hopper scale is provided for precise control of the draw.

This may call for a somewhat larger or more elaborate carbonating system than is required with a mixed feed kiln.

Design criteria and operating know-how for kilns of these types are, at this writing, largely concentrated in patents and in the proprietary knowledge of the firms that supply them. Consultation with such firms is strongly recommended to those considering their installation.

Theory and Practice of Limestone Calcining

The basic chemistry of limestone calcining is simple, but the physical and physicochemical factors are so complex that in the present state of knowledge some features of lime production are more of an art than a science.

Chemistry

The fundamental chemical reaction is:

$$\underset{100}{CaCO_3} + 43.3\ \text{Cal.} \rightleftarrows \underset{56}{CaO} + \underset{44}{CO_2} \qquad (1)$$

A simple stoichiometric calculation based on this equation shows that under conditions of perfect efficiency the production of one ton of CaO requires 1.79 tons of $CaCO_3$ and 2.78 million Btu of heat. These are minima and can be used for calculating the thermal and material efficiencies of the lime manufacturing station by comparing them with the actual operating results. Furthermore, if the efficiencies of an existing or proposed installation are ascertained or estimated then the raw material and fuel requirements can be forecasted for that system on the basis of the above values.

The reversibility of the basic reaction is very important. Thus for successful calcining, one must maintain the conditions which drive the reaction to the right, as

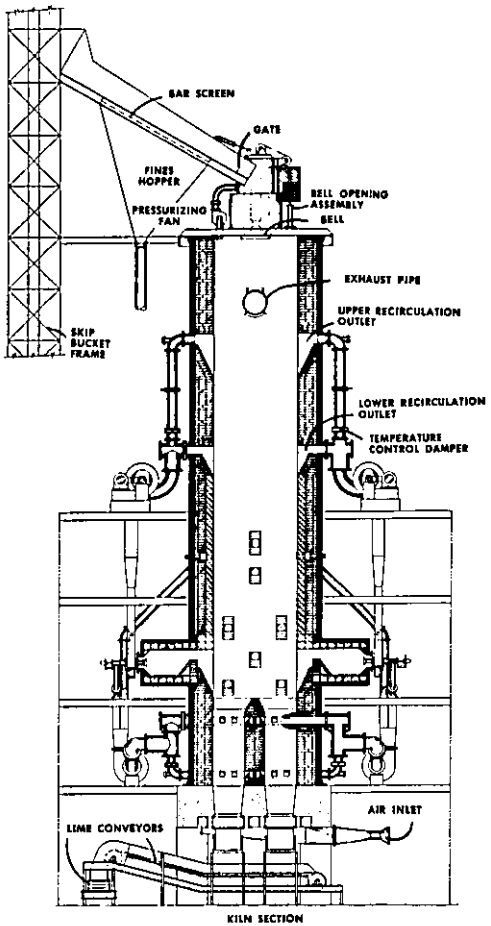

Figure 17-6. An Azbe Oil-Fired Kiln of the "Ultimos" Type. *Courtesy of the Azbe Corporation.* Fuel oil is gasified in the side chambers. Hot gases are recirculated back to these chambers from the levels above the fire zone. These provide part of the heat needed to vaporize and crack the atomized fuel oil. More heat for this purpose is provided by partial combustion of the oil vapors in the gasifying chamber. The gasified fuel passes on into the kiln at the level of the gasifying chambers. Secondary air enters at the bottom of the kiln, but a portion of it is withdrawn by the lower blowers and bypassed around the level of fuel entry, as in the "Ultimos" gas-fired kiln, to lengthen and temper the flame.

written above, and must allow conditions to develop which would cause it to reverse. The equilibrium conditions have been shown by J. Johnston (21) to depend entirely on the temperature of the dissociating $CaCO_3$ and the partial pressure of CO_2 in the atmosphere surrounding it. Johnston determined that the equilibrium line is defined by the equation:

$$\log p = \frac{-9340}{T} + 1.1 \log T - 0.0012T + 8.882 \qquad (2)$$

where p is the partial pressure of CO_2 in mm of Hg and T is the temperature in degrees Kelvin. Johnston's findings have been reconfirmed by Azbe (2). Figures 17-7 and 17-8 are plots of Johnston's equation. If the temperature and CO_2 pressure conditions are in the region above the equilibrium line then $CaCO_3$ will dissociate. If conditions are in the region below the equilibrium line, then CO_2 and CaO will combine to form $CaCO_3$. Such recombination will occur rapidly at temperatures near the equilibrium temperature (3).

Thus to satisfy the fundamental chemisry of calcining $CaCO_3$, the following steps are necessary: 1) Heat the $CaCO_3$, to the equilibrium temperature; 2) Supply

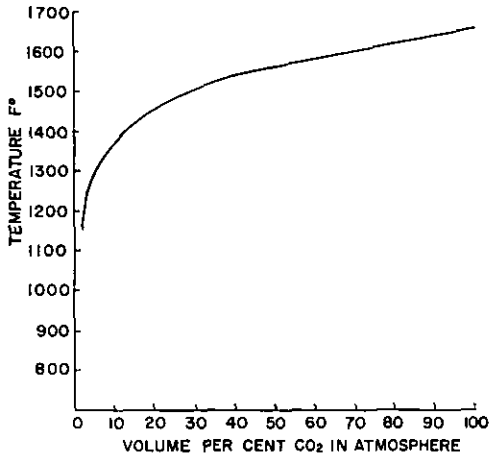

Figure 17-7. Equilibrium conditions for thermal dissociation of calcium carbonate, at one atmosphere total pressure. ***Values of J. Johnston*** **(21)**

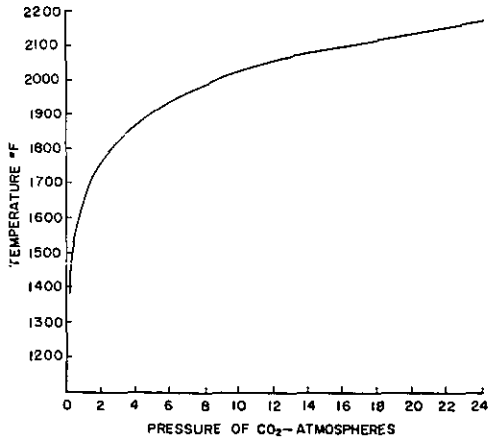

Figure 17-8. Equilibrium conditions for thermal dissociation of calcium carbonate at various partial pressures of carbon dioxide. ***Adapted from values of J. Johnston*** **(21)**

the heat that is absorbed by the dissociation reaction; 3) Remove the CO_2 as it is generated; 4) Provide an atmosphere of low CO_2 content during the cooling of the CaO.

Avoiding the recombination of CO_2 with the finished lime is of paramount importance for Steffen work. Such recarbonation, even in minute amounts, apparently spoils the lime-surface conditions necessary for the precipitation of cold saccharate. One can easily demonstrate this by calcining powdered $CaCO_3$ in a laboratory muffle and then exposing it briefly to a CO_2 atmosphere as it cools through redness. The resulting lime, although it may contain as little as 1% CO_2, will be almost completely incapable of precipitating cold saccharate. Nevertheless, it may slake vigorously in water or sweetwater and make a lime milk which is quite acceptable for straight-house carbonation. Extensive recarbonation is, of course, undesirable even for straight-house work as it increases the limestone costs by reducing the net yield of CaO.

Recarbonation in calcining can occur in two ways: 1) from CO_2 in the atmosphere during the cooling stage of the calcining process; 2) from incomplete calcination of limestone such that the interior core of the stone is still hot and emitting CO_2 while the exterior is cooling. The latter occurrence is illustrated in Figure 17-9 and 17-10.

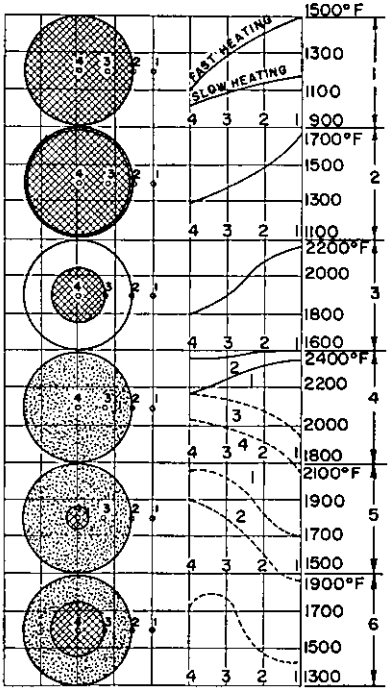

Figure 17-9. Probable stages in the manufacture of lime in a vertical kiln: 1) preheating stone; 2) surface calcination; 3) internal calcination; 4) completion of calcination and incipient cooling without recarbonation; 5) cooling with slight recarbonation; 6) cooling with considerable recarbonation. ***V.J. Azbe. 1947*** **(3)**

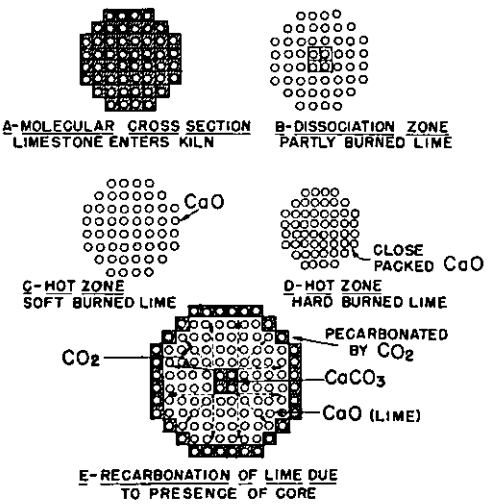

Figure 17-10. Spatial configurations in lime burning. ***V.J. Azbe, 1947*** **(3)**

Complete and steady removal of the CO_2, from the kiln as well as complete calcination of the limestone are therefore important factors to successful lime manufacture. In making lime for Steffen some overdrafting of the kiln may be justified to assure rapid cooling of the calcined lime with a minimum of exposure to CO_2. The resulting sacrifice in thermal efficiency of the kiln may be well repaid in improved Steffen efficiency.

Practically all the lime produced in the kiln is recombined with CO_2 in the factory carbonation. Chemically reaction (1) read from right to left is the simplest possible statement of this process. Although there may be intervening steps, nevertheless, the stoichiometric quantities are those shown in the equation. Since the carbonation process is not perfectly efficient in using the CO_2 supplied to it, an additional supply is necessary. This is accomplished by performing the calcination in a kiln that is fired internally with a carbonaceous fuel; i.e., coke, coal, oil, or fuel gas. The CO_2 from the combustion thus supplements that from the limestone. The amount of such supplemental CO_2 generated will, of course, depend on the thermal efficiency of the calcining process and the ratio of carbon to other combustibles in the fuel used.

Considering the lime industry as a whole, bituminous coal, anthracite coal, coke, producer gas (from coal), natural gas, fuel oil and wood are commonly used to provide the necessary heat and the supplemental CO_2. In the sugar industry, the most predominant fuels are coke and natural gas. In order to clarify the relationships of CO_2 production, equation (1) is restated as follows with the assumption of one ton CaO to be produced:

100				56		44	
$CaCO_3$	+	Heat	$\rightleftharpoons$	CaO	+	CO_2	(3)
1.79 ton		2.7 M Btu/ ton CaO		1 ton		.79 ton	

The reaction of the combustion of coke is stated as follows (assuming 8% coke on rock):

12		32		44		14000 Btu/lb	
C	+	O	$\longrightarrow$	CO_2	+	Heat	(4)
.143 ton		.381 ton		.524 ton		4.00 M Btu	

Analysis of these two reactions reveals that the amount of CO_2 available to supplement that being produced from the limestone is:

CO_2 from	Ton	% of total
Stone	.79	60
Fuel	.524	40
Total (per ton CaO)	1.314	100

Considering the reaction of natural gas as a fuel, and making the assumptions that the gas is 100% methane, and that the fuel input is 4.8 M Btu/ton CaO, the fuel combustion reaction can be stated:

16		2 (32)		44		2 (18)	23900 Btu/lb	
CH_4	+	2 O_2	⟶	CO_2	+	$2H_2O$ +	Heat	(5)
.1004 ton		.402 ton		.276 ton		.226 ton	4.8 M Btu	

Analysis of equations (3) and (5) shows that the amount of CO_2 available to the process is:

CO_2 from	Ton	% of total
Stone	.79	74
Fuel	.276	26
Total (per ton CaO)	1.066	100

It is apparent from reading formula (3) in the reverse direction that, chemically (in a 100% efficient system) only the CO_2 which comes from the rock is necessary for the carbonation of the CaO back to $CaCO_3$ in the factory carbonations. The efficiency of absorption in the carbonators ranges widely (from less than 50% to 90%), but all require some additional CO_2 from the fuel to complete the carbonation process in the factory.

One might ask the question, "Why the emphasis on high CO_2 content in the kiln gas?" The answer to this usually lies with the size of the equipment used to pump the kiln-off gas to the carbonator. Excessive air leaks into the top of the kiln will dilute the CO_2 content and require the handling of much more gas to obtain the same amount of CO_2. Obviously, if the system is mechanically designed to operate with a 15% CO_2 gas, it is entirely possible chemically.

Proper operation of any type of kiln requires that the rates of limestone feed, fuel supply and withdrawal of calcined lime and CO_2 gas be held relatively constant and not be subjected to hour-to-hour or minute-to-minute variations in the demands of the Steffen or carbonation. Some surge storage of the product lime or lime milk is therefore necessary, as is some buffering of the CO_2 gas supply. Surge storage of the gas is impractical, but buffering of the demands of gas can be provided by maintaining the absorption efficiency of the carbonation at a point where the process does not normally need all of the CO_2 supplied by the calcining plus combustion reactions. The surplus CO_2 gas is then normally vented to the atmosphere. When the carbonation requires a temporary over- or under-supply of CO_2 the rate of venting the surplus can then be adjusted, and the rate of withdrawal from the kiln need not be disturbed. Only by so insulating the temporary operating variations of the process from the kiln can both be maintained at maximum efficiency.

Physical and Physicochemical Considerations

Some forms of limestone and quicklime, even though they may be of the highest purity, are nevertheless quite useless for sugar factory work because of their physical structure (11). The more important physical and physicochemical factors, which are interrelated in their effects, are (11):

Physical properties of the limestone: crystal size and size distribution, density, degree and type of agglomeration, if any, tendency to decrepitate, quantity and nature of impurities and types of occlusion, and particle sizing and size distribution.

Physical conditions of the calcination process: rate of calcination, temperature, and duration.

Physical properties of the quicklime: porosity, density, effective surface area per unit weight, and particle sizing and size distribution.

Most limestones were originally beds of marine shells and skeletons. After their original deposition such beds may have been subjected to a variety of geological processes, such as leaching and recrystallization, pressure, heat, fracturing, uplifting and erosion. Their physical natures vary widely depending upon their geologic history, and their behavior in calcining will vary with their physical structures. Some will calcine satisfactorily in all types of kilns, some are suitable only for calcining in rotary or hearth kilns, and some are very difficult or impossible to calcine satisfactorily in any commercial kiln.

The types of physical behavior which must be avoided or minimized through proper selection of limestone and proper calcining technique are: 1) shrinkage, 2) melting, and 3) decrepitation.

1) **Shrinkage.** A piece of good quality limestone, when calcined carefully at just above the equilibrium temperature and cooled immediately thereafter, will retain its original dimensions. The resulting lime will have a porous structure resembling chalk, with great internal surface. Liquids will penetrate it readily. It will be highly reactive, easy to slake, easy to grind, and efficient for Steffen use, if not recarbonated.

If, however, it is kept above the calcining temperature for an excessive time, or is overheated, the internal surface will be reduced and the above desirable properties correspondingly diminished. Such lime is called "hard burned." With sufficient overcalcining of this nature, the resulting lime will increase in density by shrinkage and will ultimately become almost inert. Such lime can, for example, remain immersed in water for several days without slaking. In this almost inert condition, it is called "dead burned."

Boynton (9) has summarized the work of several researchers, showing that such loss of internal surface and ultimate shrinkage are caused by the clumping of the CaO crystals at high time-temperature into agglomerates of "crystallites." This process is illustrated by Boynton, whose drawings are reproduced as Figure 17-11.

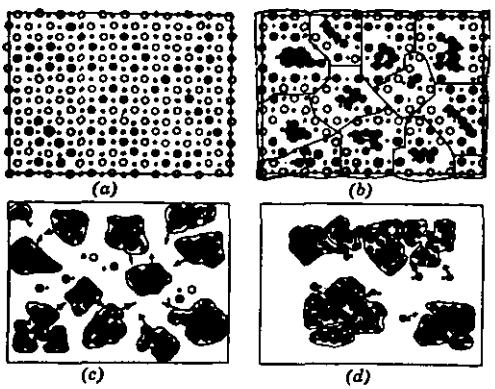

Figure 17-11. Graphic display of how lime oxides gradually form much larger crystallites as calcination temperature increases. Gradual agglomeration of small crystals into larger crystallites is illustrated sequentially in a) through d). (R.S. Boynton) (9). *Courtesy Interscience Publishers, division of John Wiley and Sons.*

The density of the limestone has an important bearing on its tendency to lose surface or shrink during calcination. Figure 17-8 shows that the equilibrium temperature of the calcining reaction continues to increase with increasing CO_2 pressure. As calcination proceeds toward the interior of a piece of limestone, considerable pressure is generated by the escaping CO_2 and this tendency is increased in

limestones of high density and low porosity. Thus the time-temperature requirements for calcining high-density limestones are greater than for those of low density. The closer molecular spacing in high density stones undoubtedly further facilitates the formation of crystallites. Low-density limestones are much more tolerant of the natural variations of time-temperature exposure that occur in calcining processes, and are to be preferred to high-density stones.

A measure of the relative susceptibility of different limestones to hard burning or dead burning can be obtained in the laboratory by calcining pulverized samples at various temperatures over the range 1500° to 2400°F for four hours. The resulting lime can then be evaluated by rate-of-slaking tests or Steffen-efficiency tests. Desirable limestones will retain a high degree of reactivity over a wide range of calcining temperatures. Undesirable ones will exhibit a peak reactivity when calcined at a temperature near 1800° to 2000°F, with reactivity falling off rapidly at higher as well as lower calcining temperatures (30).

In work done in the mid-1950's Murray (27) observed a definite trend of lower shrinkage of lime produced from a stone that had been treated with salt or soda ash. Those limestones that exhibited a tendency of high shrinkage and the resulting reduction in reactivity contracted less and demonstrated a higher degree of reactivity than the same stone which had not been treated with the salt or soda ash. Limestones that exhibited a low shrinkage tendency were unaffected by the salt or soda ash addition. Boynton (10) points out that lime manufacturers in several countries have experimented with small additions of sodium chloride either dry or in solution in the range of 0.2 to 1.0 percent (2-10 lb/1000 lb rock) and that contradictory results have been reported. In the past few years there has been considerable interest in the sugar industry in this phenomenon, where similar experiments have been conducted and similar results obtained. If salt or soda ash is effective on a particular limestone, it produces a lime that has less shrinkage, and, therefore, greater porosity and greater reactivity. The degree of improvement is the variable that is not predictable between stones of different origins. This must be determined experimentally. There have been no documented adverse effects of using salt or soda ash; however, some operators feel that salt has a possible detrimental effect on the gas handling equipment. Since more dusting is observed from a stone that has been treated, it would appear that additional gas washing may be advisable in some cases. There is some indication that Steffen losses have been reduced by a factor of ten by using salt in the lime kiln. The primary negative result has been excessive dust and in some cases a tendency to decrepitate.

2) Melting. The melting point of pure CaO is 4400°F, a temperature which should never be reached in a calcining operation. However, the melting point of lime can become much lower than this under the fluxing influence of certain impurities. Such impurities can be contained in the limestone or supplied by the fuel as well as by the refractory lining of the kiln. Of the common impurities in limestone, i.e., SiO_2, Al_2O_3, Fe_2O_3, and $MgCO_3$; SiO_2 has the greatest fluxing action, since it easily combines chemically with CaO (31). Melting is, of course, increased by conditions of greater time-temperature. When melting is allowed to occur it reduces the reactivity of the lime in the same way as does shrinkage. Also when the CaO combines chemically with impurities such as SiO_2 during melting, it thereby becomes unavailable for its mission in the beet process.

A further serious consequence of melting is the fusion of the limestone pieces to each other and to the kiln refractory to form clinkers. Such clinker formation interferes with the flow of both solids and gases through the kiln, reducing its capacity

and heat economy besides injuring the quality of its product, as well as damaging the refractory lining.

Proper selection and sizing of the limestone, avoidance of fuels high in ash, and good kiln design and operation will minimize or avoid melting.

3) Decrepitation. This is defined as the tendency for the limestone to shatter during calcination. It results in blocking of the draft in a shaft kiln by fines. In all types of kilns it generates undesirable fines and dust. When decrepitation occurs it becomes difficult or impossible to calcine the larger pieces of limestone without overburning or dead burning the decrepitated fines and dust. Decrepitation is characteristic of limestones composed of large calcite crystals and of some stones of striated or fractured formation. It can be abetted by poor calcining techniques; i.e., thermal shock from too rapid heating and physical shock from rough handling.

A good preliminary test of the decrepitating tendency of a limestone can be made by calcining pieces, say 1 in. cubes, in a laboratory muffle at 2200 °F. If the limestone does not decrepitate during such laboratory calcining, a further evaluation can be made by drop-testing the calcined pieces and comparing their behavior with that of calcined cubes made from a limestone that is known to perform satisfactorily in full-scale practice.

Selection of Limestones for Calcining

It is evident from the foregoing that the selection of a suitable limestone is of great importance in calcining practice. Ususally the noticeable physical characteristics of suitable stone are: fine crystals, low density, high porosity, a minimum of striations and fractures, and a minimum of impurities, especially SiO_2.

Although some preliminary screening can be obtained from chemical analyses and laboratory calcining tests, as described above, nevertheless the ultimate suitability of a limestone for calcining cannot be known except by actual trial in a full-sized kiln (11). In evaluating a potential new source of limestone the insurance value of running a trial carload or two through an operating kiln is usually well worth the cost.

Limestone Sizing. This is another important physical consideration. Since calcination proceeds from the exterior of a limestone particle toward the center, obviously the time-temperature effort required for complete calcining increases with the increasing size of the limestone. Excessively large pieces may require more of such effort than is available at the desired throughput rate of the kiln; or they may become overburned on the exterior before the interior is calcined. Excessive variance in size gradation tends to result in simultaneous over- and under-burning.

Azbe (4) has proposed a mathematical expression of the relationship of the size and shape of a given piece of stone vs. its behavior during calcination:

$$K = \frac{S \cdot \Delta T \cdot t}{V \cdot P} \qquad (6)$$

where K = a calcination constant, S = the surface, sq in.; ΔT the temperature difference in °F between the flowing gas and the core of stone; t = the time, min; V = the volume, cu in.; and P = the mean penetration, in.

Calling the calcining effort $E = \Delta T \cdot t$ and equating P to the average thickness of the piece of stone in question, then the minimum requirement for complete calcination is expressed as:

$$E = \frac{V \cdot P \cdot K}{S} \quad (7)$$

It is apparent from the above that the shape factor is as important as the size factor. The average thickness is as important as the volume of a stone. Thus slabs or irregularly shaped rocks require less calcining effort than those of the same volume but approaching cubic or spherical shape.

In the above study, Azbe has proposed a graphic representation of the calcining effort vs. the size and shape factors. He has further suggested that in a given kiln feed the greatest and least required calcining efforts for individual particles should be related by a ratio of 5:1 or less.

The average size and size variation have another important effect in a shaft kiln; i.e., they cause resistance to the flow of gases through the kiln. Excessively small-sized rock, or rock with excessive size variance, will limit a given kiln's capacity by preventing the adequate introduction of combustion air and removal of gas products. Sizing variance can also cause channeling of the gas flow, producing such detrimental effects as local over- and under-burning and recarbonation.

Small sizing or size variance has another disadvantage in limestones which are susceptible to melting. Because more points of mutual contact are presented, the tendency toward clinker formation is increased.

Obviously there is an optimum average size and shape of stone for each combination of kiln design, operating rate, and stone composition. However, it appears that kiln performance will always be improved by reducing the size variance. The economy of sizing and size variance, however, must also be considered. Ultrauniform sizing with its attendent processing cost and the rejection of a large fraction of the limestone may be less economical than acceptance of more size variance or a less desirable average size. Each combination of kiln installation and limestone source will obviously have its own economical specification for stone sizing, and this must be determined by local study and experience. Some general guidance may be obtained from the knowledge that shaft kilns are using feeds from 2 to 8 inches in average particle length.

Equipment for Waste Lime Reclamation

The cost of high quality limestone is great enough in some areas to justify the increased investment and processing costs of reclaiming and reusing the waste lime from the factory carbonation. Two types of kilns have been used successfully for such a process: the rotary kiln and the multiple hearth kiln (20,23,25,26). The Oxnard and Spreckels, California, factories for many years used small gas-fired rotary kilns to provide 10% to 25% of their lime supplies by calcining some of the sun-dried waste lime from previous campaigns. The gas from these small kilns was too low in CO_2 content to be utilized in the carbonators. Thus the quantity of reburned waste lime that could be utilized was limited to the amount of surplus CO_2 that was available from the shaft kilns. The Alvarado, California, factory for some 25 years used a multiple-hearth kiln to supply nearly half of its lime and CO_2 gas needs by reburning sun-dried waste lime. Equipment was also provided for the direct drying and calcining of fresh carbonation filter cake. The Mendota, California, factory was constructed in 1963 with a large rotary kiln as its only source of lime and CO_2 gas, reburning dewatered fresh carbonation cake plus a make-up of waste lime from other factories.

Experiments are under way at present on the briquetting of waste lime so that it can be calcined in shaft kilns (8).

Rotary kiln. This consists of a cylindrical shell lined with refractory brick, mounted at an angle of 3 ° to 5 ° on trunnions and mechanically rotated (see Figure 17-12). The internal diameters of commercial rotary lime kilns range from about 5 to 13 ft, and the ratio of length to diameter ranges from about 30 to 40 (13). Typical criteria for size vs. capacity are 120 to 150 cu ft of volume per ton of lime produced per day from lime sludge (24).

Figure 17-12. A 10.5x275 ft rotary kiln designed for calcining precipitated $CaCO_3$ sludge. ***Courtesy Fuller Company/General American Transportation Corporation.***

The raw material is fed into the upper end of the kiln and flows slowly toward the lower end under the influence of the slope and rotation. Heat is supplied by a burner at the lower or discharge end and travels counter-current to the charge under the influence of an induced draft fan or compressor, protected by gas-scrubbing equipment, which withdraws the product gas at the feed end. About the upper 40% of the kiln's length is occupied with preheating the charge and the next 55% with calcining (38). Only about the last 5% is available for cooling, because of the proximity of the heat source to the point of product discharge. Thus the product lime may leave the kiln at about 2000 °F (38). Most rotary lime kilns are therefore equipped with some sort of auxiliary heat exchanger to cool the product and simultaneously preheat the secondary combustion air (14,38).

Heat transfer in the calcining zone of a direct-fired rotary kiln is almost entirely by radiation and in the preheating zone it is mostly by convection and conduction. To improve the efficiency of the preheating zone there is usually provided some sort of packing or baffling to increase the contact of the feed with the hot gases and gas-heated surfaces (14,38).

Kilns designed to handle a wet feed, as from dewatered carbonation cake, may be equipped with loosely draped chains as part of the packing to provide additional heat transfer surface.

Successful heat transfer in the calcining zone requires a long luminous flame, a dust-free atmosphere, and a *rolling* bed of calcining solids. Absent any of these conditions, a rotary lime kiln will be unsatisfactory in efficiency, capacity and product quality.

The desired luminous flame conditions are obtained by proper burner and hood design and operation. These should be such that the supply of primary air is minimized while the secondary air flows as a laminar sleeve around the flame, being gradually diffused into it and thus gradually used up by the combustion as the flame proceeds into the kiln.

The necessary rolling bed and absence of dust when burning limestone in a rotary kiln are brought about by feeding a nondecrepitating stone of relatively uniform sizing and not larger than 2 in. or smaller than 0.25 in. in average length; also by proper rotational speed of the kiln, usually between 1 and 2 rpm.

Such proper bed and dust conditions when calcining sugar-factory waste lime are not easily achieved. This material is, of course, a fine precipitated chalk mixed with organic and inorganic impurities from the beets. When damp, about 30% moisture, it readily assumes an angle of repose of 90° or more. When dry or partly calcined it can be very dusty, and when agitated it may form a fluid-like bed with almost no angle of repose. Calcination in a rotary kiln under such dusty and fluid conditions would be virtually impossible.

To resolve this dilemma it is necessary to cause the waste lime to agglomerate into nodules of the optimum size for rotary kiln work; i.e., 0.25 to 1 in. in diameter. This is accomplished by maintaining a high temperature at the end of the drying zone and by adding a fluxing substance to the feed such as soda ash. Excess draft with some sacrifice in combustion efficiency may be required to produce the proper temperature pattern.

The optimum rotational speed for calcining waste lime may be slower than the optimum for calcining limestone.

The advantages of a rotary kiln for waste lime reburning are:

1) Fuel economy is moderately good when equipped with an air-preheater, lime cooler (14,38); probably about 50% (16).

2) Maintenance problems are relatively uncomplicated; i.e., there is only one burner location, the refractory layout is uniform and is largely secured by wedging without mortar, and the control system is relatively simple.

3) The nodulized product is relatively easy to handle, slake and grind for Steffen use. It resembles pebble lime, but is softer.

Disadvantages of the rotary kiln are:

1) Control of the temperature pattern needed to produce nodulization, simultaneously with the optimum degree of calcination, is critical and difficult. Very uniform operation is required to maintain such control. The pattern can be disturbed by variations or interruptions in feed rate, firing, draft, kiln rotation, and feed composition. It is sometimes difficult and time-consuming to re-establish the optimum pattern, once it is disrupted.

To minimize this problem large storage bins or tanks must be provided for the kiln feed and the product, the kiln draft must be completely protected from variations in gas flow to the carbonation, and all equipment and controls associated with the kiln must be subjects of a good preventive maintenance program.

2) The investment cost is high as compared with a shaft kiln, but the shaft kiln alone will not reclaim waste lime from its natural state. The combined investment cost of a shaft kiln and briquetting equipment for waste lime, if the latter can be worked out, may not be very different from the cost of a rotary-kiln plant.

3) The CO_2 content of the gas product when calcining waste lime at 35% moisture is low; 16 to 20%, requiring larger or more elaborate carbonators, scrubbers, and gas compressors than for a shaft kiln.

Multiple Hearth Kiln. Figure 17-13 shows a cutaway view of a modern multiple-hearth kiln. It is a vertical cylinder lined with refractory and containing a multiplicity of horizontal hearths or trays made of refractory brick. These are designed to be slightly conical in shape to make them structurally self-supporting.

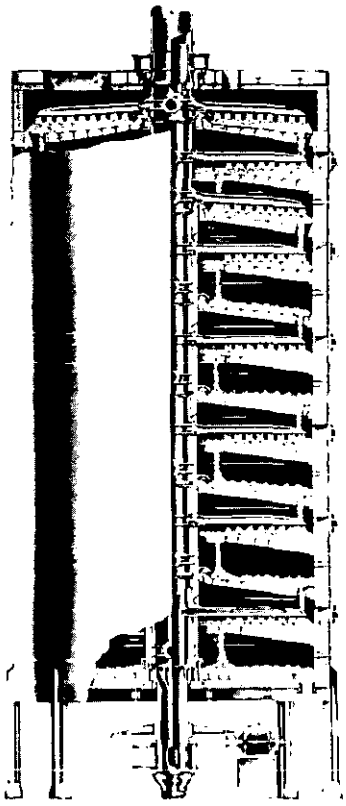

Figure 17-13. Multiple Hearth Kiln. *Courtesy BSP Corporation*

There is a central air-cooled metal shaft carrying a set of rotating rakes or rabble arms for each hearth. These arms may also be air cooled. The rabble teeth are so pitched that they rake the charge in opposite directions in the successive hearths. Thus on the top hearth in the illustration the incoming raw feed is raked toward the periphery where it discharges through a port hole onto the second hearth. Here it is raked toward the center where it discharges onto the third hearth, and so on.

The lower hearths are each equipped with a number of burners, usually for gas firing. Both primary and secondary air are admitted at each burner. This makes it impractical to exchange heat between the calcined product and the secondary air, so this source of heat economy is usually sacrificed in this type of kiln. The calcined product leaves the kiln from the bottom hearth at the calcining temperature and must be cooled by some type of air- or water-cooled heat exchanger for process use.

The upper hearths are not equipped with burners and serve as the preheating zone.

The product gas leaves the kiln through a large port in the top hearth. If the gas is to be used in the carbonation, scrubbing equipment and a compressor are provided.

Design criteria for the multiple hearth kiln will usually be based on effective hearth area per unit of feed or product. The effective area of each hearth is the rabble area, and will be on the order of 70% of the hearth area. A round-number ratio of effective hearth area in sq ft to tons of lime produced per day is 25:1; e.g., assuming 12 hearths in a 22 ft, 3 in. diam kiln with about 3000 sq ft of effective area, producing 120 tons of lime per day from oyster shells (24). A somewhat higher ratio may be needed for the calcining of damp waste lime.

Industrial kilns range in diameter from about 4 to 22 ft and from 4 to 17 in number of hearths (24). Structural considerations for the self-supporting refractory hearths put a practical limit of 22 to 25 ft on the diameters of these kilns.

The advantages of the hearth kiln are:

1) The precise temperature pattern is easily designed and controlled by the placement and throttling of the burners.

2) Precise control of the kiln atmosphere is likewise easily designed and controlled by means of the secondary air flows and it can even be made oxidizing in one part of the kiln and reducing in another part, if necessary.

3) The rabbling action is very gentle and does not, for example, tend to fluidize waste lime during calcining. This action is not sufficient to agglomerate the waste lime, even with the addition of soda ash, but agglomeration is apparently unnecessary in the absence of any tendency to break down or fluidize the waste lime.

4) Heat transfer is largely by conduction through the rabble teeth and is not as affected by dusty conditions as in a rotary kiln.

5) The temperature pattern and proper ratio of fuel to charge flow are simply and precisely controlled by separate automatic temperature controls for each fired hearth. The kiln thus keeps itself adjusted to changes in the charge flow.

6) Starts and stops are easily accomplished.

Disadvantages are:

1) A low thermal efficiency, probably comparable to a rotary kiln without a heat-reclaiming aftercooler; i.e., about 30% (17).

2) A low CO_2 content, about 15 to 20% when calcining waste lime at 30% moisture, requiring large or elaborate carbonators and large scrubbers and compressor.

3) A high investment cost, similar to a rotary kiln of equal capacity.

4) Relatively high maintenance cost due to the complexity of the burner system, controls, and refractory work.

5) The maximum available size is limited to about 100 tons of CaO output per day.

Technology of Reclaiming Waste Lime

Following are some of the important factors to be recognized in considering, planning, or operating a system for reclaiming waste lime.

Composition: Waste lime, of course, consists of the precipitated $CaCO_3$ chalk from the carbonation, plus the nonsucroses removed from the beet juice in carbonation, plus any filter aids that are discarded with the waste lime. Included in the beet juice nonsucroses that reach the waste lime is all the field dirt and sand from the beets that gets into the diffusion juice. Table 17-2 shows a typical analysis of waste lime and of calcined waste lime.

Properties of Calcined Waste Lime. Properly calcined waste lime; i.e., soft-burned and not underburned, is light yellow in color and contains no black or grayish evidence of incomplete combustion of the organic matter; nor does it contain hard burned cakes or nodules that will not crush easily. It is much more effective for Steffen work per unit of CaO content than lime made from limestone.

The higher concentration of inorganic impurities in reclaimed waste lime appears to have no important chemical effect on either its Steffen or carbonation uses. They are all relatively insoluble under the alkaline conditions of these processes. They do have an important physical effect, however, in that they create an added solids load which must be reckoned with in the conveying, grinding, settling and filtering operations of these processes. Furthermore the settling and filtration rates may be reduced, especially if the lime is high in colloidal or combined SiO_2.

Table 17-2 Typical Analysis of Beet Factory Waste Lime and of Calcined Waste Lime

	Waste Lime (Dry Basis)	Calcined Waste Lime*
SiO_2	3.06	5.39
Al_2O_3	2.44	4.30
Fe_2O_3	0.54	0.95
CaO	47.59	83.77
MgO	1.62	2.85
K_2O	0.08	0.14
Na_2O	0.18	0.32
SO_3	0.16	0.28
CO_2	39.17	2.00
Organic and undetermined	5.16	
Total	100.00	100.00

*Computed from waste lime analysis at 2% ignition loss, which is a typical value of good soft-burned reclaimed lime.

Inorganic impurity buildup. The inorganic impurities will, of course, continue to build up in a recycling system of waste lime reclamation since they are continually augmented by inorganic constituents of the beets and process water as described previously. Some form of blowdown and makeup of the lime supply must therefore be provided. The percentage of blowdown required is easily estimated by a material balance computation based on the analysis of the waste lime and the material available for makeup. If "virgin" waste lime is used for makeup, about 20% blowdown may be required to maintain a reasonable CaO content of 80% in the calcined product.

Sources of makeup. A logical source of makeup is virgin waste lime, as suggested above. Established factories may have many thousands of tons of this material stockpiled as a waste product. At 20% makeup rate such stockpiles may provide many years of supply to a waste lime reclaiming plant.

The addition of crushed limestone to the feed of a lime reclaiming kiln seems on the surface to be a reasonable way of making up, but it is unlikely to prove successful. Unless such limestone is of very porous nature, it is not likely to calcine satisfactorily in the time-temperature pattern required to produce a good, soft-burned product from beet-factory waste lime. Finely pulverized ordinary limestone might work but is likely to be very expensive. If seriously considered it should be tried in practice, preferably in full scale, as it could interfere with nodulization in a rotary kiln or have other unexpected bad effects.

Making up with purchased quicklime or with quicklime hauled from another factory that has surplus kiln capacity may be a more economical and satisfactory method. Some saving features are: 1) Less blowdown and makeup will be required than with virgin waste lime because of the higher purity of the makeup. 2) Fuel cost at the lime reclaiming factory is reduced. 3) Freight cost per unit of CaO hauled in quicklime at over 90% CaO is apt to be much less than in hauling limestone at 50 to 55% CaO, or damp waste lime at 30 to 40% CaO. 4) In a Steffen factory imported quicklime makeup can be used entirely in the slaker for wet lime addition. The full high efficiency of the reclaimed lime for precipitating sugar can then be realized by using it entirely for the dry lime addition.

Effect of organic impurities. These are available as fuel for the reclaiming operation. Kiln design and operation should allow for their combustion by providing sufficient secondary air. Such combustion will occur in the late preheating and early calcining zones under proper operation. As much as 45% of the total fuel requirements for drying and calcining in a reclaiming plant may be provided by the organic matter in the waste lime itself.

Physical nature of waste lime. Its physical properties at various moisture contents must be well recognized in designing and operating a reclaiming system. At moisture contents of 50% or more, it can be handled as a fluid. Carbonation cake direct from the filters at 50% moisture will puddle if agitated, and can be pumped. At moisture contents from about 35 to 45%, it is a sticky putty which can be handled by screw conveyors. Between about 25 and 35% moisture, it is friable and relatively nondusty. It can be handled in this condition by front-end loaders, bucket elevators, and belt conveyors. It will slide down properly designed chutes. It arches readily, however, and will not handle well in bins or tapered chutes and transitions.

Below 25% moisture content, it is dusty and at very low moisture content it is easily fluidized and difficult to handle. Practical logistics dictate that the moisture content of the kiln feed will be 20% or more. Waste lime from carbonation may be pumped to the reclaiming plant at 50 to 60% moisture and dewatered to 35 to 40% by centrifuging before being fed to the kiln. Sun-dried waste lime may be harvested at 20 to 30% moisture to avoid unreasonable handling problems and fed to the kiln in that state. There will, therefore, be a substantial drying load in a waste lime reclaiming operation and kiln capacity should be provided accordingly.

Generation of dust in the kiln will be considerable as the feed approaches dryness, even under the best agglomerating conditions. In fact as much as 30% of the feed solids may be entrained by the kiln exhaust gas. Scrubbing equipment should be designed accordingly. This entrainment is likely to take care of all the blowdown requirements and perhaps an unwanted additional waste. Supplementary equipment to reclaim and recycle a portion of the entrained dust may be required. This could take the form of a small settler for the scrubber waste water or a dry cyclone ahead of the scrubbing equipment.

Lime Milk Manufacture

For use in the carbonation or for "wet lime" in the Steffen process, the quicklime is converted into an aqueous slurry called "milk of lime." This is actually a suspension of calcium hydroxide which is formed by the reaction between quicklime and water:

$$\underset{56}{CaO} + \underset{18}{H_2O} \rightleftarrows \underset{74}{Ca(OH)_2} + 15.5\ Cal \qquad (8)$$

This is performed in an apparatus called a slaker, which is designed to agitate a mixture of quicklime and hot water or hot sweetwater, composed of carbonation filtrate and washings, in order to promote the above reaction. There are various designs of slakers, but a common one is a horizontal drum mounted on trunnions and equipped with an internal broken-flight helix or spirally mounted paddles. The ends are annular or conical and serve as weirs to cause liquid retention in about 25% of the total volume of the drum. Commonly, the lime and liquid are fed in together at one end while the scroll or paddles agitate the mixture and finally discharge any unreacted solids at the other end. The milk of lime slurry overflows the annular weir at the far end and both it and the unreacted solids fall upon a cylindrical coarse screen attached to the drum which separates the two. The screened solids at this point are considered to be waste and the liquid slurry contains the $Ca(OH)_2$ for further use in the process.

Annis (1) suggests that the drum should be rotated most of the time in the direction such that the paddles throw the solids backwards; and should then be reversed for five minutes out of each hour to discharge the dross. Alternately, the liquid and solids can be fed at opposite ends and made to flow countercurrently through the drum. The unreacted solids can then be discharged in a drained condition without screening, and the slurry can be discharged unscreened by overflowing at the opposite end.

As to sizing of the slaker drum, Silin (35) suggests a criterion of 0.5 cu m total volume per 100 metric tons of beets sliced per day. This would be about 16 cu ft per 100 short tons of daily beet slice and appears to agree closely with practice in North America. A Steffen factory preferably should have slaking equipment of sufficient capacity to operate as a straight house, even though only about one-fourth of such slaking capacity is needed for Steffen wet lime. Delays and breakdowns do occur in the Steffen process, during which substantial savings can be realized if milk of lime is provided for the factory to continue operating as a straight house.

The liquid from the slaker contains the dissolved and suspended $Ca(OH)_2$. It will also contain some sand-like particles of uncalcined limestone, unreacted lime, brick spall, unburned coke, and impurities. These should be removed by further screening or classifying. Vibrating screens with 30-mesh media, hydrocyclones, or mineral rake classifiers or screw classifiers are commonly used for this purpose.

A storage tank with stirrer should be provided between the classifier and the lime milk pumps to the process. The recommended surge capacity of this tank is 30 minutes supply of lime milk or more (1). Its purpose is threefold: 1) to buffer the kiln from variations in the lime demands of the process and 2) to provide capacitance in the control of the $Ca(OH)_2$ concentration of the lime milk. This concentration should be held constant to the greatest practial degree to facilitate accurate control of the carbonation or Steffen processes as the case may be. 3) To provide additional time for aging or slaking which increases the effective surface area of the hydrate. The German industry regularly provides retention capacities of an hour or more.

Automation of the slaking operation is usually by the following scheme: A level detector in the lime milk surge tank starts or stops the lime drawing mechanism of the kiln and also the flow of sweetwater or water to the slaker. The flow rate of liquid is preset to give a $Ca(OH)_2$ concentration greater than is finally wanted in the lime milk. A liquid density meter, continuously monitoring the contents of the surge tank, controls a secondary flow of sweetwater or water to the tank. This trims the lime milk density to that corresponding to the desired $Ca(OH)_2$ content. Milk of lime for the beet-sugar factory is usually standardized at about 30° ap-

parent Brix when made with 10° Brix sweetwater, or at about 20° Brix when made with plain water, giving, in either case, a CaO content of about 10%.

Technology of Lime Milk Manufacture

It is important to remember that milk of lime is not a liquid reagent, but rather a liquid suspension of a solid reagent. The solubility of $Ca(OH)_2$ in water is very low, and its solubility decreases with increasing temperature. Its solubility is increased by the presence of sugar because of the formation of saccharate — a loose compound of sucrose with CaO (33). Table 17-3 shows the solubility expressed as weight-% CaO at various temperatures and concentrations of sugar (12).

Table 17-3 Solubility of Lime in Water and Sugar Solutions (g CaO/100g Saturated Solution)

	Sugar Conc., g/100g Saturated Lime Solution			
Temp °C	0	10	15	20
10	0.133			
25	0.122	1.40	2.63	4.08
40	0.106			
60	0.088			
80	0.070	0.19	0.23	0.37
100	0.054			

Obviously at operating temperatures of about 80 °C, over 98% of the lime is in the solid phase in a milk of lime containing 10% CaO and 10% sugar. Silin (33) points out that even in the dilute conditions of carbonation at 2% CaO on juice, only about an eighth of the CaO added can dissolve directly. The remainder must either react directly in the solid or colloidal phase, or must await the removal of lime from solution by the carbonation reactions before it can enter the solution phase.

To perform its task effectively the solid phase in the lime milk should be finely dispersed and have a high surface area per unit of weight. The design and operating conditions of the average beet-sugar factory's slaking equipment are well suited to the production of this type of lime milk; however, both designers and operators should be aware of the technology of good slaking practice and the pitfalls to be avoided. Stowell (37) and Boynton (18) have written excellent reviews of this subject and a brief summary of the factors important to beet factory work are given below.

Good slaking conditions are characterized by vigorous disintegration of the quicklime stones or pebbles. This is caused by the change in crystal structure and also by the boiling of the slaking water within the lime particles as the heat of reaction is evolved. There is a substantial increase in solid particle volume during slaking, not only because the total weight of solids is augmented by the reacting water, but also because the absolute density of the $Ca(OH)_2$ (2.2 sp gr) is much lower than that of CaO (3.32 sp gr). This literally explodes the solids into the desirable, very finely divided state. In addition, there is considerable evidence that hydrated colloidal micelles of the type $[Ca(OH)_2n(H_2O)]_x$ are formed, increasing the tendency of the solid phase to remain in suspension (37).

The proper conditions that produce the above type of slaking in making milk of lime are: a medium soft burned quicklime; a temperature in the slaker of 90° to 100 °C; a sufficient supply of slaking liquid so that the solids are completely im-

mersed at all times; ample agitation so that the submerged solids are constantly turned and not allowed to lie dormant at any time; and the presence of sugar in the slaking liquid.

Water burning results when a starvation of slaking liquid occurs, as from incomplete immersion of the slaking solids or inadequate agitation. It is caused by local overheating at centers where there is insufficient opportunity for the heat of reaction to dissipate. This results in the formation of an inactive gritty substance similar in properties to dead-burned lime. It is commonly known as "water burned" lime. According to Stowell, there is some evidence that this substance may be a compound of the formula, $CaO \cdot Ca(OH)_2$, but it may also be a dense form of CaO that is produced by a reversal of the slaking reaction, which occurs at 580 °C.

Silin (35) and Schneider (32) point out that another bad consequence of local overheating is the caramelization of the sugar in the sweetwater used for slaking.

Water burning may be hard to avoid when slaking extremely active soft-burned quicklimes, and in such cases hard burning may yield a wet hydrate with more surface (19). Therefore in some rare cases, i.e., with limestones that produce extremely "hot" lime when soft burned, a non-Steffen factory may find it advantageous to hard burn the lime to a moderate degree.

Drowning results when the slaking liquid is present at low temperature and in excessive amounts. The rate of slaking and the vigorous physical action described above are reduced. The surfaces of the lime particles become hydrated, but tend to become impervious to water penetration to the interior, and incomplete or delayed slaking results. The remedy is to use hotter water or sweetwater, to reduce the flow of water or sweetwater to the slaker, or to try to make softer-burned lime.

Both water burning and drowning conditions may result in the rejection and waste of potentially useful lime by the grit separating equipment. In addition, either of these conditions tends to reduce the specific surface of the hydrate. This is illustrated by the work of Staley and Murray (19,27,36) who compared the specific surface of dry lime hydrates produced under normal and abnormal conditions, shown in Table 17-4.

Table 17-4 Method of Hydration

Method of Hydration	Surface area of hydrate, sq m/g
Slaking under cold conditions (5°C), ice added	6.67
Hydrating with steam at 113°C	8.05
Normal slaking (excess water, boiling conditions)	13 - 32
Slaking with 2% sugar solution	37 - 46
Slaking with ethanol solution	36 - 46

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Chapter XVIII

THE STEFFEN PROCESS*

Sucrose will, under proper conditions, form insoluble products with the hydroxides of barium and strontium and with the oxide of calcium. Hence, by properly treating beet molasses with one of these reagents and filtering, most of the sucrose can be retained in the filter cake while most of the nonsucrose is left in the filtrate. The sucrose-containing precipitate can be decomposed by carbonation, forming an alkaline-earth carbonate that can be filtered off, leaving the sucrose in solution. The latter can then be recovered in pure form by crystallization.

This process, when employing calcium oxide, is widely practiced throughout the industry for recovering sucrose from beet molasses. In America it is called the Steffen process after its inventor, Carl Steffen (14).

The practically sugar-free filtrate from the Steffen process can be used for the recovery of monosodium glutamate and fertilizer-grade potassium sulfate or it can be used as a stock-feed supplement, either in mixed feed or as an additive to the beet pulp before drying. In either case it is first partially refined by carbonating with CO_2 gas and settling or filtering to remove lime; then concentrated to about 60% solids by multiple-effect evaporation.

Choice of Reagents for Sucrose Precipitation. Apparently the efficiency of the reaction increases with increasing atomic weight of the elements in the alkaline-earth group. Thus magnesium, and probably beryllium, oxide and hydroxide, will not precipitate sucrose to any appreciable extent. Insoluble lime-sucrose products are well known and readily prepared, but the practical use of the reaction requires unusually active calcium oxide and carefully controlled conditions. Strontium and barium in the hydroxide form will produce the desired insoluble products with sucrose under much less exacting conditions; the recovery of sucrose is not as good as with calcium oxide, but the separation from the molasses nonsucrose is more complete. Practical consideration, however, dictates the use of calcium oxide in most cases. Calcium oxide, or quicklime (CaO) is much less expensive than strontia and baryta. Calcium ion is nontoxic, whereas strontium and barium ions are poisonous. The factory requires lime for carbonation in any event, and in the Steffen process the lime gives double service in recovering the molasses sucrose and purifying the diffusion juice.

Outline of the Steffen Process. Figure 18-1 is a flow diagram to supplement the description.

1) The molasses is diluted with water to produce a solution containing 5% to 12% sucrose.

2) Finely powdered CaO is added to this solution with agitation. The amount of CaO used is generally 100 to 130% of the quantity of sucrose present, or 6 to 8 moles of CaO per mole of sucrose. Heat is liberated during this reaction, and the temperature is held down, preferably below 18° C, by mechanical cooling.

3) The mixture is now filtered. Under good conditions, about 90% of the sucrose will be found in the washed precipitate ("cold-saccharate cake"); the remaining 10% passing through with the filtrate. About 65% of the nonsucrose from the molasses also stays in the filtrate.

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*by E. M. Hartmann

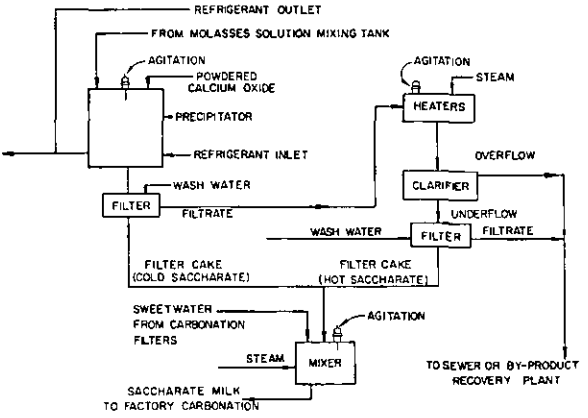


Figure 18-1. Steffen process.

4) The filtrate is now heated to about 90° C. A precipitate forms which is removed by settling and filtration. The washed precipitate ("hot-saccharate cake") contains about 6.5% of the sucrose originally present in the molasses. The remaining 3.5% of the molasses sucrose, and 65% of the nonsucrose are contained in the final filtrate, which is discarded, or concentrated for monosodium glutamate production or for use as a food supplement or dried-pulp additive.

5) The two filter cakes are dispersed in an approximately 10% sucrose liquor (sweetwater from the factory) and heated to about 85° C. This ("saccharate milk") mixture is proportioned into the factory carbonation, along with the raw juice from the beets, replacing the milk of lime used in a non-Steffen sugar factory.

6) In the carbonation the combined actions of elevated temperature, carbonic acid, and the acidic constituents and sucrose of the raw juice cause the lime-sucrose products in the saccharate milk to decompose. The resulting products are dissolvd sucrose, some dissolved nonsucrose and lime. The latter serves as the purifying agent for the raw juice and, under proper conditions, probably also eliminates a portion of the nonsucrose from the saccharate, as will be discussed later. The syrup from the saccharate commingles with that from the raw juice and proceeds to the evaporators and sugar end.

Under good conditions only about 20% of the nonsucrose from the Steffenized molasses may ultimately reach the thin juice, along with 96% or more of the sucrose.

Equipment for the Steffen Process. As can be seen from Figure 18-1, the essential units for the Steffen process are 1) a molasses solution mixer, 2) a *refrigerated precipitating unit*, 3) *cold saccharate filters*, 4) *a cold filtrate heater*, 5) a hot saccharate clarifier, 6) hot saccharate filters, and 7) a saccharate mixer. The equipment used for each of these operations will be discussed.

Batch Cooler. The classical and still widely used unit for the precipitation operation is the batch cooler, which is illustrated in Figure 18-2. It consists of an open, calandria-equipped tank, with a propeller agitator in the calandria well. In some units there has been installed a fixed and a rotating screen of about 6 meshes per inch in the well, to increase the intensity of the shear-mixing. The calandria chest is supplied with cold water or other refrigerant. Above the tank are mounted a

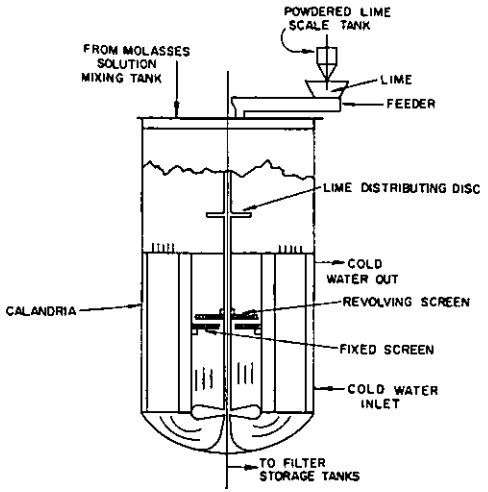


Figure 18-2. The batch cooler.

molasses scale, a solution-mixing tank, and a lime feeder. A lime-spreading disc is often mounted on the propeller shaft above the liquid level. The powdered lime is discharged onto this disc which casts it over a broad area of the moving liquid surface. The cooler tank has a bottom discharge equipped with a shutoff valve, which is connected to the supply tank for the cold saccharate filters. To operate the cooler, a charge of molasses is weighed into the solution-mixing tank, usually one-half to one ton, and diluted to the desired degree with water. The charge of cooler solution is then run into the cooler.

While the cooler is being filled with molasses solution, the proper quantity of lime is weighed out and dumped into the hopper of the lime feeder. As soon as the cooler is ready, the lime feed is started. The feeder is set at such a rate that the solution temperature never rises above a predetermined value, usually 13 to 18 ° C. The admission of the lime requires 10-20 minutes.

When all the lime has been added, the operator filters a sample of the mixture and determines the approximate solids content of the filtrate by means of a Brix hydrometer. This gives a rapid indication of the effectiveness of the sugar precipitation, enabling him to adjust the amount and rate of addition of lime to the batch, or to succeeding batches, for maximum economical recovery without having to wait for a laboratory analysis.

If the batch is satisfactory the discharge valve is opened, the product discharged to the filter supply tank and the cycle repeated.

The batch cooler operation is readily automated by the addition of modern program controls so that it will cycle without manual attention.

Spreckels Continuous Saccharate Process. This is a system which performs the work of the batch cooler in a continuous operation. It is illustrated schematically in Figure 18-3.

The dilution of the molasses may be performed batchwise in a solution mixer as in the normal batch process. A charge of molasses, usually 1 to 1.5 tons, is weighed out and dropped into the mixer. Next a quantity of milk of lime equivalent to about 22% CaO on the sucrose in the molasses is dumped in from the lime-

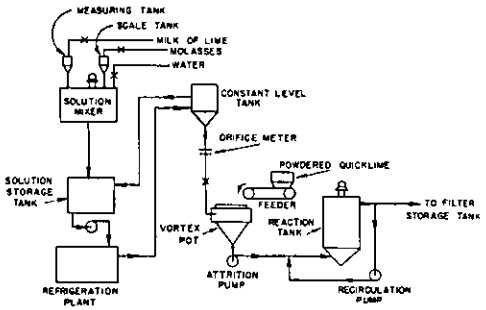


Figure 18-3. Flow sheet for Spreckels continuous saccharate process.

measuring tank. The agitator is started and cold water is added to a predetermined level to produce a solution of predetermined concentration, say 6-9 grams of sucrose in 100 ml. The batch of solution is discharged to the solution storage tank and the mixing cycle repeated. In a modern installation (6a) this stage of the process is performed continuously.

The solution is pumped continuously through a refrigerated heat exchanger which cools it to about 5 ° C. It now passes to a constant-level tank which overflows back to the storage tank. From the constant-level tank the refrigerated solution flows through a regulating valve to a funnel-shaped vortex pot, which is shown in Figure 18-4. The constant head supplied by the constant-level tank provides for a very uniform rate of flow of solution to the vortex pot.

Figure 18-4. Vortex pot (simulated).

Entering the pot tangentially, the whirling solution forms a vortex with a foam-free surface. This makes an ideal condition for the necessary rapid wetting of the powdered quicklime, which is fed uniformly into the vortex.

The mixture of lime and dilute molasses now passes through a specially designed mixing pump which produces the necessary fine dispersion of the lime and pumps the dispersed mixture to a reaction or curing tank. This tank provides about three minutes detention with gentle stirring, allowing the precipitation to become complete. In order to produce the proper particle size for good filtration a portion of the finished product from the reaction tank is recirculated to the discharge line from the mixing pump. This provides nuclei on which the newly precipitated sac-

charate can grow. From the reaction tank the product passes to the filter-supply tank, or directly to the filter trays.

The Spreckels process produces highly satisfactory results with a minimum of labor and floor space. One operator per shift can easily handle the entire unit as illustrated, with manual controls, or it can easily be automated to run without an operator.

The efficiency of the process may be still further increased by dividing the powdered-lime addition between two stages. In this case the first lime addition is made exactly as shown but in a reduced amount, and the discharge from the reaction tank passes to a second vortex pot, mixing pump, and reaction tank of equal size. No recirculation is needed for the second stage of lime addition. Best results are obtained by adding 70% of the powdered lime in the first stage and 30% in the secind stage.

R.T. Continuous Sucrate Process[1]. This author's description of the process in *SUGAR TECHNOLOGY REVIEWS* is repeated below with minor changes:

This process was developed by Vandewijer (16a) for the Raffinerie Tirlemontoise in Belgium, hence the name "R.T." The first industrial plant was installed at Tienen and was tested during the 1971 campaign. A second installation was made at Origny, France in 1972. Installations have followed in Iran, Italy, Morocco and Japan. The first North American installation, at Tracy, California, began operating in 1975.

Pieck and Vandewijer (8c) have described the 1972 version of this system in considerable detail. Figure 18-5 is a correct orientation of the flow sheet that appears in that reference.

Following the usual calcining and grinding A, the powdered quicklime is transported pneumatically to a storage hopper and metering scroll B. Molasses storage and weighing are shown at C, and the continuous dilution and wet prelming at D.

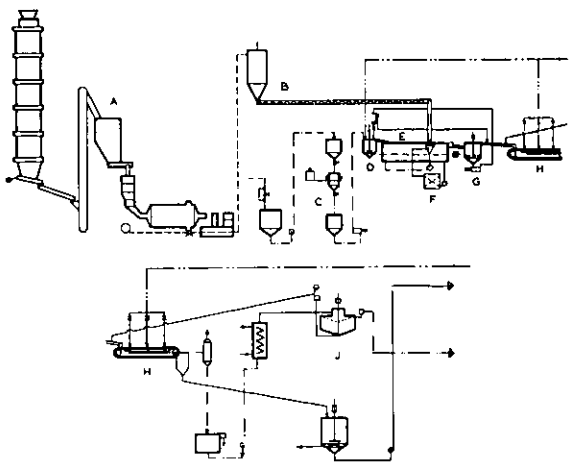


Figure 18-5. The R. T. Continuous Sucrate Process, courtesy of Raffinerie Tirlemontoise.

[1]See refs. (4e,8c,16a,16c)

The reactor E is a U-shaped trough equipped with paddles mounted on a horizontal shaft to provide gentle agitation. It is designed to provide about 20 minutes detention. From the discharge end a recirculating pump withdraws about five volumes of throughput and forces this stream through a refrigerator F to the dry-liming device. This consists of a short trough or flume located beside the reactor at about ⅔ of the distance from the inlet to the outlet of the latter. An overflowing stream from the reactor joins the refrigerated stream at the liming trough, making the total flow through the latter about 15 volumes per volume of throughput. The refrigerator is controlled to maintain a temperature of 13 ° C in the reactor.

Overflowing the discharge end of the reactor, the throughput stream passes to a decanter G, wherein the unreacted lime fractions are separated by sedimentation. The underflow slurry is returned to the molasses-dilution stage, where it largely provides the preliminary liming in lieu of lime milk.

The overflow from the decanter passes to horizontal-belt, vacuum filters H for separation and washing. These are each equipped with three separate vacuum chambers below the belt surface, to provide the desired levels of vacuum for draining, washing and drying, and also to permit the separation of the filtrate and wash liquors. There are also surface dams above the belt, to enable flooding of the cake surface with water during washing.

A conventional hot-saccharate process is shown on the flow sheet at J, with the thickened hot-saccharate slurry being returned to the belt filters, for separation together with the cold saccharate.

The developers of the R.T. system claim that its reactor produces a cold saccharate of unusually good filterability, enabling not only the use of efficient belt filters with a low ratio of active surface to throughput, but also the processing of a more concentrated molasses solution than the other systems. It is said to operate satisfactorily with molasses solutions containing up to 12 g sucrose/100 ml.

Great Western Continuous Saccharate Process (1). The appealing feature of this process is the use of conventional batch-cooler vessels as the continuous reactors, which enables relatively simple and inexpensive conversion of existing batch Steffen houses to continuous operation. Such conversions have been made as of this writing at five U.S. factories.

The basic nature of the process is illustrated in Fig. 18-6. The molasses to be

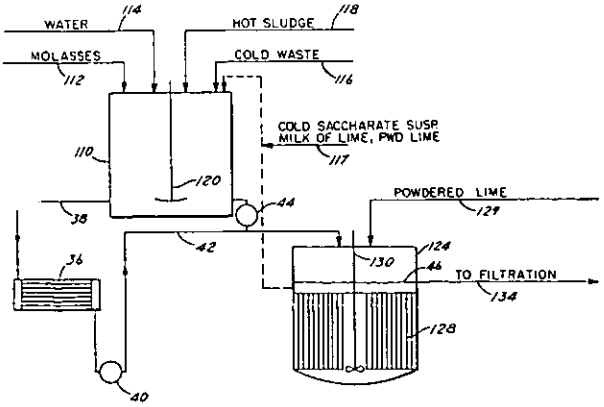


Figure 18-6. The Great Western Continuous Saccharate Process, courtesy of The Great Western Sugar Company

processed is diluted continuously in mixer 110 and is prelimed to the extent of about 1.2 moles CaO per mole of sucrose, or 19 CaO/100 sucrose. In the industrial installations the preliming is done in a separate continuous mixer. Recycled saccharate slurry from the reactor and recycled underflow from the hot-saccharate thickener are generally used for this purpose. Claim is made that the process will work successfully with molasses-solution concentration as high as 8.1 g sucrose/100 ml.

From the mixer the solution is pumped continuously through pre-cooler 36 to the reactor 124, a conventional batch-cooler vessel equipped with an overflow outlet. Some recirculation of the precooled solution may be allowed through valve 44. Since the industrial installations employ multiple reactor units, these are operated in parallel with a weir-splitting device to divide and control the flow of solution among them.

Powdered lime 129 is added at a controlled rate by a continuous feeder. The finished saccharate slurry overflows to a surge tank, not shown in the sketch, supplying the filters.

Other continuous processes. Schoenrock et al. (11a) have patented a novel system in which the lime is pulverized in a non-aqueous liquid medium and is fed continuously into the molasses solution as a slurry in the grinding medium. A static, pipeline mixer serves as the reactor. When isopropanol is used as the non-aqueous medium it is recovered by vapor fractionation coincident with the hot-saccharate process. It is claimed also that water-immiscible media can be used and recovered from the filtrate by decantation. Claim is made that this process reduces the CaO requirement to about 80% of that needed with conventional dry-grinding processes.

Toth (14b) has patented a reactor which is essentially a modification of the Spreckels process wherein the vortex pot and means of agitation have been placed within the reaction tank. The agitation is provided by a type of static mixer whose energy is supplied by an external recirculation pump.

As of this writing neither the Schoenrock nor the Toth process has been reduced to industrial practice.

Comparison of cold-saccharate processes. All of the continuous processes appear to offer advantages over the batch process in terms of improved sucrose recovery or reduced CaO requirement, better filterability, higher saccharate purity, reduced dilution of the filtrate and better adaptability to automation.

Of those which have been reduced to industrial practice, the Spreckels process appears to offer the advantages of compactness, low detention time and possibly minimal investment cost for a new installation in a warm climate where mechanical refrigeration would be required for any of the processes. It does, however, require the precooling of the molasses solution to the order of 4 ° C.

Since both the R.T. and Great Western processes provide for the exchange of heat during the reaction they can, like the batch process, operate without mechanical refrigeration in locations where a supply of very cold water is available.

As has been mentioned, the outstanding advantage of the R.T. process appears to be its ability to operate with minimum dilution of the filtrate, and that of the Great Western process the opportunity to convert existing batch Steffen houses to continuous operation at minimum cost.

Cold and Hot Saccharate Filters. Rotary drum vacuum filters have been standard equipment for this service since the 1930's; however horizontal-belt filters have now become standard in installations of the R.T. process and are beginning to be used with the Great Western process.

The use of separate filters for the hot saccharate can be avoided by a modification in flow devised by Cottrell and Jensen (1a). The underflow from the thickener is mixed with the feed to the cold-saccharate filters and the hot-saccharate filters are eliminated. Tests by the author have shown that the overall recovery of sucrose is less with this modified flow than with separate filtration of the hot saccharate. Local economic conditions will determine whether the saving in investment and operating cost will economically offset the increased sucrose losses or lime consumption.

Another variation sometimes practiced is to recycle the thickener underflow to the molasses-solution mixer. Although this also eliminates the cost of separate filtration, it nevertheless must result in either increased lime consumption or reduced sucrose recovery. The excess sucrose in the molasses solution decomposes the trilime-sucrose in the hot saccharate, releasing its sucrose into solution. This sucrose, which had been precipitated and made ready for recovery by filtration with three moles of CaO per mole sucrose, must now be reprecipitated as cold saccharate which requires six or more moles CaO per mole sucrose.

A modern Steffen house (6a) using the Spreckels process has an installed rotary-vacuum filter (RVF) area of 0.81 m^2 per metric ton of daily molasses processing capacity (TMD) (4e). A Steffen batch plant had 0.90 m^2 of RVF area per TMD (4e). Specifications for an R.T. Sucrate plant called for only 0.10 m^2 of active horizontal-belt filter area per TMD, which would probably require 0.30-0.40 m^2 of total belt area, including ends, pulleys and return surface (4e).

When the hot saccharate is filtered separately, the installed filter area requirements will be divided in the range 85-90% for cold saccharate and the balance for hot saccharate.

Cold-Filtrate Heater. The Shafor continuous heater is widely used. This consists of a cylindrical tank with bottom inlet and overflow outlet. Its volume is sufficient to provide about 15 minutes detention. Heating is done with a perforated coil for the injection of steam or vapor. A propeller agitator recirculates at a rate of about 40 volumes per unit of feed, providing the necessary seeding for the production of a fast-settling, filterable precipitate. The perforated steam coil tends to scale, but this can be remedied in a minimum of shutdown time by shutting off the steam temporarily and pouring several gallons of 10% muriatic acid into the steam coil inlet, downstream from the shutoff valve. The used acid mixes into the filtrate with negligible economic consequences.

Older designs of cold-filtrate heaters are in use, some consisting of compartmented tanks in which the liquid is heated stepwise by steam injection. Recirculation of a part of the heated slurry is usually practiced to improve the settling and filtration characteristics.

The final temperature is automatically controlled and is rather critical. The optimum point varies with different molasses, lime, and cold precipitation units, but is usually near 85° C. The optimum should occasionally be determined by laboratory tests. Below the optimum temperature, formation of the insoluble lime-sucrose may not be complete; furthermore the product becomes more soluble at decreasing temperatures. Above the optimum temperature the product begins to decompose.

Heating by injection steam or vapor has been standard practice because the strong scaling tendency of the heated filtrate has discouraged the use of surface heat exchangers; however it has appreciable disadvantages. First, the hot-saccharate precipitate has a certain degree of solubility, so that the dilution caused by the injection heating results in a proportional increase in the sucrose loss. Secondly this same

dilution causes an increase in the energy requirement when recovery of the final filtrate by concentration is practiced.

Some success has been had with spiral or plate heat exchangers for preliminary heating of the cold filtrate to about 40 °C, which is below the temperature at which precipitation and rapid scaling begins. The heating medium was the hot, final filtrate, so that some recovery of its waste heat was effected, along with a reduction in the sucrose loss. Some scaling did occur, but it was minimized by interchanging the hot- and cold-filtrate flows in the exchanger several times daily.

More recently, Pieck (8b) has patented a scheme for the full heating of the cold filtrate by steam or vapor in surface exchangers. The success of the scheme, which is illustrated in Fig. 18-7, is dependent upon its three basic features: 1) A high (10:1) ratio of recirculation is maintained through the heat exchanger with an intervening detention period in two tanks, where the major part of the hot-saccharate precipitation occurs; thus deposition on the heating surface is minimized. 2) Since fouling is not completely eliminated, two exchangers are provided which are used alternately. 3) Since the deposit is mostly sugar-soluble, the cleaning is done by circulating the solution from the molasses-dilution station through the idle exchanger.

The first installation of this process operated in Italy in 1978.

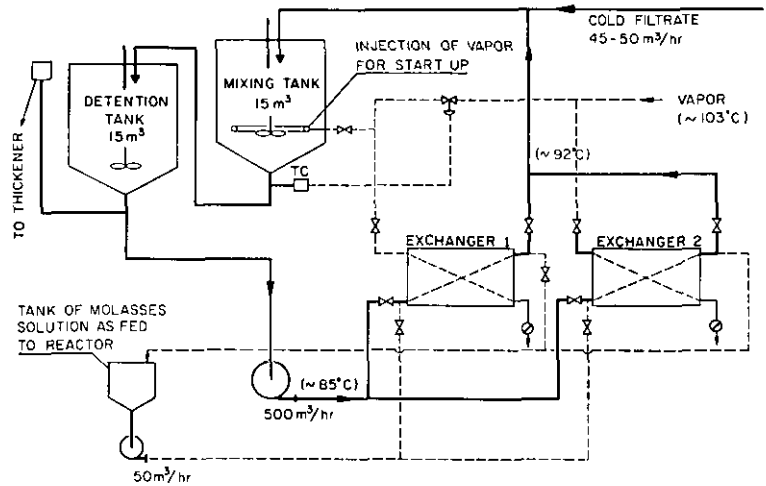


Figure 18-7. Pieck's system for the full heating of cold filtrate in heat exchangers, courtesy of Raffinerie Tirlemontoise.

Hot-Saccharate Thickener. Most U.S. installations use the Dorr multi-tray thickener. A typical U.S. installation has a total installed tray area of 0.48 m² per ton of daily molasses-processing capacity. One U.S. plant (6a) uses the highly efficient Enviro-Clear Settler (1d) with an installed area of only 0.058 m² per metric ton.

Saccharate Mixer. This consists of a cylindrical tank, usually covered, equipped with an overflow outlet, a steam injector, and a stirring mechanism. The saccharate cakes from the filters are conveyed directly to the tank, where they are continuously diluted with sweetwater from the carbonation or other filters to the optimum density for factory carbonation, usually about 25 ° Brix. The temperature is maintained at, or slightly above, the temperature of carbonation.

The production of saccharate milk at a uniform CaO content is important for good control of first carbonation. Close attention by the operator to this detail, or the use of an automatic density controller to regulate the sweetwater flow, is essential.

Technology of the Steffen Process

Chemistry. The chemical basis for the Steffen process is the formation of insoluble combinations of lime and sucrose. The industry has mistakenly dubbed these "saccharates," whereas in English terminology a saccharate is actually a salt of saccharic acid. The term will, however, continue to be used by this author to refer to the insoluble industrial products of the Steffen process, i.e. cold saccharate and hot saccharate. For the true compounds of lime and sucrose this text will use the terminology of Soubeiran (13a) and Mackenzie and Quin (6c), i.e. "lime-sucrose."

It is probable that only three lime-sucroses can be formed in the system sucrose-lime-water, viz. monolime-, dilime- and trilime-sucrose (6c,7a,8d,8f). It has, however, recently been shown (8c) that an alternate form of trilime-sucrose can probably be formed in the system sucrose–$CaCl_2$-NaOH. This will be discussed later. The probable nature, properties and mechanisms of formation and decomposition of the lime-sucroses involved in the Steffen process are summarized below:

Monolime-sucrose exists in aqueous solutions of sucrose and lime as a true salt or alcoholate, $C_{12}H_{20}O_{11}Ca$. It could rightly be called monocalcium sucrate. It is highly soluble, at least in the cold, and is also appreciably ionized at low concentrations of $Ca(OH)_2$, i.e. $<0.1N$. At higher concentrations of $Ca(OH)_2$ its ionization is suppressed and it is then probably in equilibrium with dilime-sucrose and $Ca(OH)_2$.

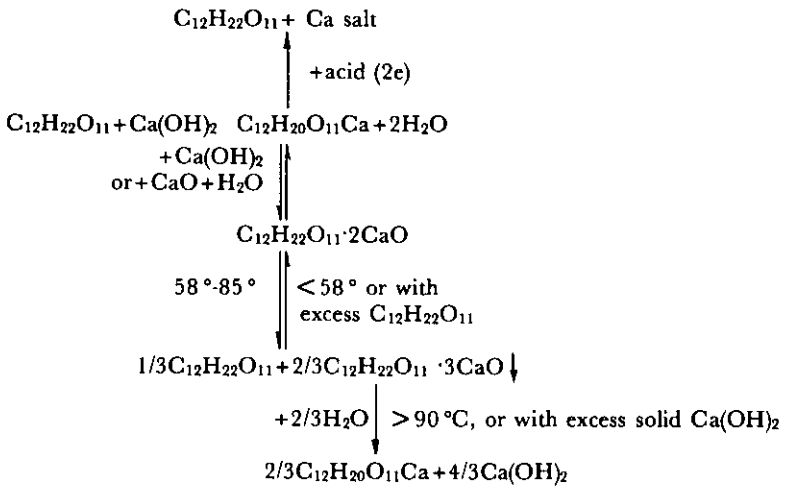
Dilime-sucrose exists in cold sucrose solutions saturated with lime. It is less soluble than monolime-sucrose. At temperatures $<58°C$ it is apparently in equilibrium with sucrose and sparingly soluble trilime-sucrose, with the equilibrium shifted toward the latter by increasing temperature. It is probably an addition compound or adduct of CaO and sucrose with the formula $C_{12}H_{22}O_{11}\cdot 2CaO$.

Trilime-sucrose is a sparingly soluble, amorphous adduct of CaO and sucrose with the formula $C_{12}H_{22}O_{11}\cdot 3CaO$. It is somewhat stable in the presence of water at elevated temperature, e.g. 85° C, and also in the absence of water at ambient temperature. In water at low temperature, probably $< 58°C$, or in the presence of excess dissolved sucrose at any temperature, it dissolves with conversion to an equilibrium mixture of dilime-sucrose, monolime-sucrose, $Ca(OH)_2$ and sucrose.

In the presence of water at high temperature, e.g. $>90°C$, or at lower temperatures in the presence of solid Ca $(OH)_2$, trilime-sucrose decomposes to precipitated $Ca(OH)_2$ and a liquid phase of unexplored composition. It is likely that the latter decomposition is caused by the crystallizing affinity of $Ca(OH)_2$, triggered by the presence of seed, exceeding the strength of the adduct's coordination bonds.

On the next page is the proposed reaction scheme for the formation and decomposition of the lime-sucroses involved in the Steffen process (4b):

Vandewijer et al. (16c) found that an insoluble lime-sucrose could be formed in the cold by adding NaOH to a solution of sucrose and $CaCl_2$. Francotte et al. (2a) determined by Raman spectroscopy that this is not the same substance that is formed in the cold-saccharate reaction, but is probably an alternate form of trilime-sucrose, namely tricalcium sucrate, $C_{12}H_{16}O_{11}Ca_3 \cdot H_2O$. Subsequently, Francotte,



Vandegans and Jacqmain (2b) have studied the system NaOH - sucrose - $CaCl_2$ in great detail and have concluded that such formation of insoluble $C_{12}H_{16}O_{11}Ca_3$ is the result of a double-ion-exchange in which the exchangeable hydrogen of the sucrose is first replaced by Na^+, this being followed by an exchange of the Na for Ca^{++}.

Representing the sucrose by H_8R, this study proposes:

$$H_8R + 4NaOH \rightleftharpoons Na_4H_4R + 4H_2O$$

$$Na_4H_4R + 2Ca^{++} \rightleftharpoons Ca_2H_4R + 4Na^+$$

$$Ca_2H_4R + 2NaOH \rightleftharpoons Ca_2Na_2H_2R + 2H_2O$$

$$Ca_2Na_2R + 2Ca^{++} \rightleftharpoons Ca_3H_2R + 2Na^+$$

Theories of Cold Saccharate Formation. The above chemistry does not, so far, explain the efficient precipitation of sucrose by lime at low temperature which occurs in the Steffen process. The formation of "cold saccharate" when cold dilute sucrose solution is treated with powdered quicklime is still not well understood and several different theories are currently available:

a) Dedek (1b), Waterman and Van Aken (16d), Hartmann (4a,4d) and Gerasimenko (3) have proposed that it is an adsorption process wherein sucrose, or more probably dilime-sucrose, is adsorbed upon finely divided or coloidal CaO.

b) Hungerford (6) proposed that the precipitate is dilime-sucrose which is forced out of solution by simple mass action.

c) Silin (12) believes that the minute particle size of the powdered quicklime gives it a super-solubility so that it tends to remain in solution while trilime-sucrose is precipitated preferentially.

d) Schneider (10) suggests that another trilime-sucrose, different from the one precipitated by heat, is formed in the cold-saccharate process.

e) Golovin et al. (4) suggest that the CaO removes the envelope of hydration from the sucrose, or from the monolime- and dilime-sucrose, thus enabling their conversion to insoluble trilime-sucrose.

f) The Service Chimique of Raffinerie Trilemontoise (8e) expressed doubt of the adsorption theory on the grounds that they were unable to cause the adsorption of the soluble lime-sucroses upon such powerful adsorbents as Al_2O_3, fullers' earth, Bentonite and colloidal silica. Recognizing that sucrose could be precipitated very efficiently in the cold by adding NaOH to a sucrose-$CaCl_2$ solution, they hypothesized that this was the same precipitate that is formed in the cold-saccharate process. Accordingly, they proposed a chemical reaction of sucrose with "nascent" Ca^+OH, formed during the hydration of the CaO in the cold-saccharate process, to yield insoluble $C_{12}H_{22}O_{11}{\cdot}3CaO$ preferentially to $Ca(OH)_2$.

g) Francotte et al., as previously mentioned, determined by spectroscopy and subsequent experimental studies that the precipitate formed in the cold system NaOH - sucrose - $CaCl_2$ is probably an alcoholate of sucrose, into which Ca has been introduced by a double-ion exchange requiring the participation of Na^+. This mechanism is surely different from that occurring in the cold-saccharate process, which can readily be duplicated with pure sucrose, water and CaO.

Francotte et al. further determined (2a) that in the cold-saccharate precipitate the CaO molecule appears to remain intact, although the structure of the sucrose molecule appears to be appreciably altered. They theorize that it is a polymeric adduct of CaO and sucrose.

Discussion. Despite the observations and hypotheses reported in f) and g) above, there is little doubt in this author's mind that the cold-saccharate reaction is a surface phenomenon. It is well known that the efficiency of sucrose precipitation by this reaction is directly correlated with the specific surface of the powdered quicklime (14a) and is even more dependent on the nature of that surface (4c).

Thus it is found, for example, that a highly efficient lime powder for this purpose can be manufactured by calcining the waste lime from the carbonation, which consists principally of precipitated $CaCO_3$. Such reclaimed lime, although much less pure than that from high-quality limestone, has much more specific surface. Furthermore, it is also found that any Steffen-effective lime can be rendered virtually impotent for cold-saccharate precipitation, without significantly altering its analysis or other important properties such as slaking rate or ability to purify raw juice, by lightly coating its surface with $CaCO_3$. This effect is readily demonstrated by dividing a sample of a satisfactory Steffen lime powder, heating both portions to about 870 ° C and then exposing one portion for a few seconds to a stream of CO_2 as it cools through redness, while the other portion is cooled in a normal dry atmosphere or in a desiccator. When the two portions are then compared as to their abilities to precipitate sucrose as cold saccharate, as well as to their analytical compositions, slaking rate, and other familiar properties, the results will be quite startling.

There is much other evidence to support an adsorption theory of cold-saccharate formation (4d,8,15,16), however the experiments of the Tirlemont group with adsorbents other than lime (8e) make it clear that this is not the mere physical type of bonding that occurs, for example, on the adsorption of helium upon charcoal. Rather it must be the type of adsorption ("chemisorption") that occurs through a true chemical bonding at the sorbent surface (8a) and therefore requires a specific reagent, or type of reagent, as the adsorbent.

The adsorption theory appears not be at odds with the spectroscopic findings of Francotte et al., i.e. that the cold-saccharate precipitate has the nature of an "*amorphous* or polymerized *substance without well-defined structure*" in which the CaO molecule is intact (emphasis added).

The mechanism proposed by this author for the cold-saccharate reaction is the formation of dilime-sucrose which is adsorbed onto the surface of the properly prepared lime powder or a colloidal dispersion thereof, forming the floc which is the cold-saccharate precipitate:

$$CaO + H_2O \rightarrow Ca(OH)_2$$

$$Ca(OH)_2 + C_{12}H_{22}O_{11} \rightarrow C_{12}H_{20}O_{11}Ca + H_2O$$

$$C_{12}H_{20}O_{11}Ca + Ca(OH)_2 \rightleftharpoons C_{12}H_{22}O_{11}\cdot 2CaO$$

$$C_{12}H_{20}O_{11}Ca + CaO + H_2O \rightarrow C_{12}H_{22}O_{11}\cdot 2CaO$$

$$C_{12}H_{22}O_{11}\cdot 2CaO + xCaO \rightarrow (C_{12}H_{22}O_{11}\cdot 2CaO)\ldots xCaO\downarrow$$
(adsorption product)

As to the hot-saccharate process, it is proposed that the residual dilime-sucrose in the cold filtrate reacts when heated according to the previously proposed reaction scheme to form sparingly soluble trilime-sucrose, resulting in a filterable yield of about 2/3 of the sucrose in the cold filtrate:

$$3C_{12}H_{22}O_{11}\cdot 2CaO \rightleftharpoons 2C_{12}H_{22}O_{11}\cdot 3CaO\downarrow + C_{12}H_{22}O_{11}$$

Practical Aspects of Lime-Sucrose Chemistry. The need for certain precautions to assure successful and efficient operation in a Steffen house is readily explained and understood in the light of the chemistry and theory propounded above:

1. The quality of the lime powder is the most important single variable in promoting efficient sugar recovery. A large amount of calcium-oxide surface, uncontaminated by recombined carbon dioxide, is essential for good results.

The most critical factors in producing good lime for Steffen are the proper selection and calcination of the limestone. Although good grinding is very important, nevertheless it is actually in the calcining that the potential surface of the powdered lime is created or destroyed. The secret is to produce a lime that is fully calcined, yet free from the shrinkage that can occur in the kiln, and from the recarbonation that can occur either in the kiln or in subsequent handling. The best grinding available cannot make good Steffen lime from a kiln product whose internal surface has been destroyed by shrinkage or coated by recarbonation.

The nature of the limestone, both as to chemical adulterants and physical structure, is as important as the calcining technique. These facts are well illustrated by Rorabaugh, Orleans, and Norman (9). The selection of limestone and the technique of calcining are discussed in detail in Chapter XVII.

Waste lime from the carbonation when reclaimed by proper calcining and grinding has very high specific surface and makes an extremely efficient reagent for the Steffen process. Reclamation of waste lime is discussed in detail in Chapter XVII.

2. A low temperature must be maintained up to the point where the cold saccharate is filtered off. This promotes the colloidal dispersion of the lime and improves the adsorption equilibrium. Generally speaking, the lower the temperature the better the sugar recovery.

3. Immediate agitation is required with the addition of the powdered lime, to promote proper dispersion and contact of the reactants.

4. The molasses solution must be kept free from foam while the powdered lime is being added. Foam on the surface of the solution traps and floats the lime powder. The floating lumps are slowly slaked by the moisture in the foam. Thus the CaO is not brought into immediate contact with the sucrose or lime-sucrose; furthermore local heating probably occurs during this slaking which is unfavorable to the formation of colloidal lime as well as to the adsorption equilibrium.

5. The presence of nuclei during both the cold and the hot precipitation is beneficial, both to filtration and to sucrose recovery. This is a well-known principle in all precipitation and crystallization processes. Substances flocculated or crystallized in the presence of nuclei of the same substance tend to form on these nuclei rather than to initiate new particles. As a result the product so made has a larger and more uniform particle size and thus filters more readily.

In the cold-saccharate precipitation this condition may be produced by adding the lime slowly or in steps, if a batchwise precipitation is made. If the process is continuous, "seeding" may be accomplished by stepwise addition of the lime, or by recirculating a portion of the finished mixture, or by a combination of the two.

In the precipitation of hot saccharate, proper particle size may be produced by slow or stepwise heating or by recirculating large volumes of the finished mixture within a continuous heating system.

6. $Ca(OH)_2$ will react with sucrose under the conditions of the cold-saccharate process to the extent of about 1.5 moles CaO/mole sucrose, which corresponds to an equilibrium mixture of monolime- and dilime-sucrose. Thus milk of lime can be directly substituted for powdered quicklime in the Steffen process to that extent, i.e. 24 CaO/100 sucrose, without loss of efficiency in sucrose recovery.

This enables a considerable saving in many Steffen process installations, because milk of lime is easier and cheaper to produce than high quality lime powder. Capacity of, and maintenance on the grinding equipment is reduced. Stored lime from the lime kiln can be reclaimed in this way without loss of efficiency. Coarse lime particles which do not react in the cold saccharate process can be recovered by sedimentation and used as milk of lime.

Lastly, if the lime milk is cooled after hydration a good percentage of the heating effect in the cold-saccharate precipitation can be eliminated, and a considerable saving made in cooling-equipment cost. For this reason the use of lime milk in place of a portion of the powdered lime actually improves the sucrose recovery in some Steffen processes; notably the Spreckels process.

7. Variations in proportioning the lime and sucrose result in reduced efficiency. If the sucrose left in the solution is plotted against the ratio of lime to sucrose for a given cold-saccharate precipitation process, there is found in all cases a hyperbolic curve which is characteristic of adsorption processes. Figure 18-8 shows such a graph. It will be noted from the example shown on this plot that the recovery of sucrose per unit of lime used always diminishes when the lime addition is allowed to vary.

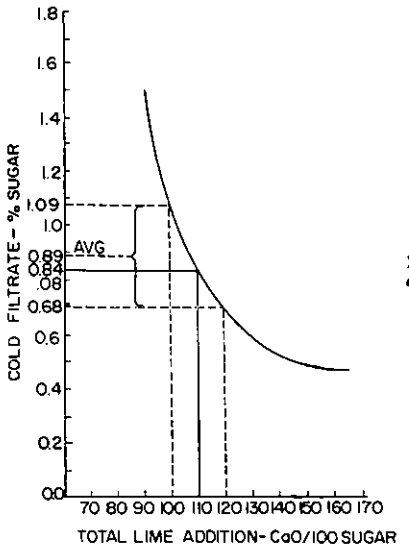


Figure 18-8. Typical precipitation curve.

Thus, in the example given, a uniform lime addition of 110% on sucrose will reduce the concentration of sucrose in solution to 0.84%. On the other hand a lime addition which shifts back and forth from 100% to 120% on sucrose, but averages 110%, will reduce the average concentration of dissolved sucrose to only 0.89%.

Saccharate Purity, Molasses Degeneration, and Discards. It has been mentioned that the saccharate precipitates contain some of the molasses nonsucrose. It is not possible to make a perfect separation of the sucrose from the molasses nonsucrose. Some of the latter is precipitated by direct reaction with the lime or by adsorption, and so becomes part of the filter cake. Among such substances are sulfate and phosphate ions, invert sugar, raffinose, and doubtless several organic substances.

By the purity of the saccharate cake is meant the purity of the syrup it will produce after it has been decomposed in the carbonation. This purity is a direct index of the degree of nonsucrose elimination achieved in the Steffen process and the carbonation. It is usually determined by treating the saccharate cake with CO_2 in the laboratory, filtering off the calcium carbonate so produced, and analyzing the remaining syrup.

Schoenrock (11) has shown by material balances of the molasses and filtrate that the purity of the delimed saccharate *per se* is best estimated by deliming it with $(NH_4)_2CO_3$ and removing the resulting ammonia by vapor displacement before analyzing the syrup. The result so obtained is substantially lower than that given by the conventional CO_2 deliming, indicating that in the latter method some of the nonsucrose is retained with the precipitated $CaCO_3$. Schoenrock et al. (11b) demonstrated that under the conditions of their experimental carbonation of raw juice with saccharate the $(NH_4)_2CO_3$ method better represented the purity of the syrup ultimately released by the saccharate into the thin juice.

Degeest et al. (1c) confirmed Schoenrock's findings that the saccharate *per se* contains more nonsucrose than is shown by the conventional CO_2-deliming method;

however under the conditions of their experimental carbonation the CO_2 method was shown to yield a conservatively low estimate of the purity of the syrup released by the saccharate into the thin juice. Thus in Degeest's tests there apparently occurred a substantial elimination of the saccharate nonsucrose during its carbonation with raw juice.

The conflicting results obtained by these two respected authorities may well be due to the effect of differing agricultural conditions on the nature of the nonsucrose, or to differences in their cold-saccharate processes or carbonations techniques, which were considerable. The matter is, however, of prime importance to Steffen processors. As will be seen, the purity of the saccharate as defined in the second paragraph of this section has a direct and powerful bearing on the ultimate yield of refined sugar from the molasses Steffenized. Accordingly, in evaluating the economics of a Steffen operation it is very important to know this purity as accurately as possible. Because of the conflicting reports described above, it appears that no processor can be confident, without experimental confirmation, that the conventional CO_2-deliming method accurately represents the real saccharate purity under his local conditions. Stated differently, it is probable that the nonsucrose content of the saccharate *per se* is appreciably higher than that estimated by the CO_2 method, and confirmation is needed as to whether or not this additional nonsucrose is eliminated in the factory carbonation.

Schoenrock and Degeest each answered these questions for his own conditions by a relatively simple experimental procedure which every processor using the Steffen process would be well advised to follow:

a) The purity of the saccharate is determined by the processor's favorite method.

b) An experimental first and second carbonation is carried out in a replica of the factory's process, using a sample of the saccharate and a sample of the factory's raw juice. The purity of the resulting thin juice is determined.

c) A second experimental carbonation is carried out with an equal portion of the same raw-juice sample, but using milk of lime equivalent in amount to the CaO provided by the saccharate in b). The purity of the resulting thin juice is determined.

d) By actual mixing, or by material-balance computation, the purity is determined of the thin juice that would result from mixing the syrup from a) with the thin juice from c) in the same proportions that occur in b).

If purity b) is essentially the same as purity d), then the processor's favorite purity method has reliably estimated the real saccharate purity. If purity b) is lower than d) when a) is determined by the CO_2 method, then examination of the factory's carbonation and Steffen practices are in order to uncover ways of improving the elimination of the nonsucrose from the molasses Steffenized.

Under favorable circumstances the purity of the saccharate as estimated by the CO_2 method will reach 90 and that of the hot saccharate 95. Such circumstances occur when the Steffen house is well operated and is working "virgin" molasses, i.e. molasses produced by a factory without an operating Steffen house. Some Steffen factories in the U.S. operate in this way continuously, working only virgin molasses imported from a non-Steffen factory and not recycling any of their own molasses production to the Steffen house.

When a factory does recycle molasses from the sugar end to the Steffen house a degeneration of the saccharate purity occurs. Under these circumstances the nonsucrose which is returned to process by saccharate milk will eventually become part of the factory molasses and so return to the saccharate process again. Since its

nature is to precipitate readily with the saccharate there will be negligible elimination of this particular nonsucrose during such second working. Moreover its proportion will now have been reinforced by additional nonsucrose of the same type, which will have been introduced by the raw juice; thus the saccharate produced during this second cycle will be of considerably lower purity. It is apparent that as this recycling process continues, the precipitable nonsucrose will constantly accumulate in quantity, and the saccharate will become lower and lower in purity.

This continual accumulation of nonsucrose in the factory reduces its production capacity and increases costs.

The theoretical relationship between the saccharate purity and extraction of refined sugar from the molasses is expressed by the following equation:

$$E = 1 - L - \left(\frac{(1-P_s)\ (1-L)\ P_m}{P_s\,(1-P_m)} \right)$$

where E = refined sugar recovery, lbs/lb sucrose in molasses worked; L = loss of sucrose Steffenized, lbs/lb sucrose in molasses worked; P_s = purity of saccharate, expressed as a decimal fraction; and P_m = purity of molasses produced, expressed as a decimal fraction.

Table 18-1 gives some theoretical extraction rates for various saccharate purities, under typical factory conditions of sucrose loss and molasses purity.

Table 18-1 Theoretical Extraction of Refined Sugar from Steffenized Molasses, Assuming 5% Loss and 60% Purity Molasses Produced.

Saccharate Purity	92	90	88	86	84	82	80
Extraction %	82.6	79.2	75.6	71.8	67.9	63.7	59.4

This downtrend in purity must eventually be stopped, otherwise the operation becomes uneconomical. To stop it, a molasses "discard" is made. This can be accomplished either batchwise or continuously. In a batch discard the Steffen process is temporarily fed with fresh (virgin) molasses procured from a non-Steffen, or straight-house factory; or else the Steffen process is shut down during the discard period. Meanwhile the molasses production of the factory is directed to separate storage tanks until the recycling nonsucrose derived from the saccharate cakes has been cleared out. In a continuous discard, only a fraction of the molasses produced by the factory is recycled to the Steffen process and the balance is continuously discarded. A makeup supply of virgin molasses may be provided to the Steffen house.

Local conditions will dictate the economics of discarding and makeup. As has been mentioned, many Steffen factories find it economical to work only pure virgin molasses and discard their entire molasses productions. A few, located in isolated areas, with virgin molasses not cheaply available and no good market for their own molasses, may find it economical to operate a saccharate process at a moderate rate, recycling a portion of the molasses produced with no virgin molasses makeup and discarding the surplus. Others find their best economy to lie in practices somewhere between these extremes.

The discarded molasses is almost equal in value to virgin beet molasses for feeding or fermentation processes, and is usually sold for these purposes or dried with the beet pulp.

Control of Saccharate Purities. Some control over saccharate purities may be achieved by proper precautions in the Steffen operation, as well as by judicious discarding.

1. Cake washing. Obviously if the nonsucrose-laden filtrate is not thoroughly washed from the saccharate, the purity of the saccharate will be reduced. On the other hand, over-washing is also detrimental. Saccharate cakes have definite solubilities or adsorption equilibria and tend to dissolve in the wash water, increasing the sucrose loss. Furthermore overwashing of the cold saccharate produces excessive amounts of filtrate which require extra heat for the precipitation of the hot saccharate and also for evaporation, if the final filtrate is concentrated for by-product recovery. There exists, therefore, an optimum quantity of wash water which must be determined for each installation, and which usually must be adjusted from time to time to meet changing conditions. The customary method of controlling the wash-water usage is through the so-called "perfectly washed" purity of the saccharate cake. To determine this, a sample of the factory saccharate is given an additional, thorough washing in the laboratory and its purity determined. If the factory saccharate has been properly washed, its purity should not be more than 1.5 units below the "perfectly washed" purity.

Determination of the chloride content of the liquid phase in the filter cake has been found a simple and rapid means of estimating the effectiveness of the washing.

Proper distribution of the wash water on rotary-vacuum filters is also very important. Application of water should begin upon emergence of the drum from the liquid in the tray and continue uniformly and without interruption to the point of beginning final drainage. Any hiatus in washing up to this point not only wastes useful washing area on the filter but also may allow the cake to crack before reaching the next available wash. Such subsequent wash water will then tend to bypass through the cracks and lose its effectiveness for displacing the nonsucrose-bearing filtrate.

The point of beginning final drainage, or terminator of washing, should be sufficiently above the beginning of the blow-off to allow adequate elimination of the free liquid from the cake before it is discharged from the drum. When all is properly adjusted one should not be able to observe free liquid being blown out through the cloth and running down the discharge apron.

2. Control of filtration pressures and wash-water distribution. Generally rotary vacuum filters are used for separating the saccharate precipitates. The pickup and wash vacua must be controlled to give sufficient filtration capacity without detriment to the purging of the cake.

The optimum pressures to be used will vary among different installations. However, good results should be achieved if the pickup or cake-forming vacuum is set to obtain a cake from 0.5 to 0.75 in. thick with clean filters, and the wash vacuum set as high as possible without causing appreciable cracking of the cake under the sprays.

3. Concentration of the molasses solution. As the concentration of molasses solution is increased, the purity of the saccharate cake tends to decrease. This is due to the effect of concentration on the chemical and adsorption equilibria. It is also due to the increased viscosity of the filtrate which makes the cake harder to purge. On the other hand, excessive dilution of the molasses solution results in increased sucrose loss and heat consumption, due to the solubility of the saccharate and to the increased quantity of filtrate to be heated or concentrated.

The optimum concentration of the molasses solution varies with the equipment used and with local conditions. In terms of g sucrose/100 ml it may lie between 5 and 7 for the batch process, between 6 and 9 for the Spreckels process and Great Western process, and between 7 and 12 for the R.T. process.

Vandewijer et al. (16b) have found that the addition of $CaCl_2$ to the molasses solution in the proportion of < 50 meq/100g sucrose increases the filterability of the resulting saccharate and permits, at least in the R.T. process, the working of a 12 pol. molasses solution containing a substantial proportion of recycled cold filtrate.

4. Recycling of filtrate. Recycling of a portion of the cold filtrate has been practiced by a number of Steffen plants, to reduce heating and by-product recovery costs. The recycled filtrate is substituted for a portion of the dilution water in making the molasses solution.

The recycling of filtrate results in lowering of the saccharate purity, thereby reducing the yield of refined sugar from the molasses worked. Local economic conditions should be evaluated to determine if the reduced yield offsets the fuel saving.

Raffinose. Raffinose is a troublesome factor with Steffen operations, particularly when working beets grown in the cooler climates, such as the Rocky Mountain area. Raffinose is a sugar with the formula $C_{18}H_{32}O_{16} \cdot 5H_2O$ (see pp 33-34). Upon complete hydrolysis it yields three simple sugars—glucose, fructose, and galactose. It is practically tasteless and has little commercial value.

Like sucrose, raffinose is precipitated by lime in the Steffen process, and most of the raffinose introduced in the beets tends to be retained in a Steffen factory if the production molasses is recycled. Since raffinose has a specific optical rotation approximately 75% higher than that of sucrose, its presence causes the apparent purity of a syrup to be higher than the actual purity.

Because of this error in apparent purities, in a Steffen factory it is necessary to make determinations of the true purity of factory materials, particularly the saccharate cakes and raw massecuite, for operating guidance.

Raffinose reduces the crystallization rate of sucrose and also causes the sucrose crystals formed in its presence to be elongated in shape (see p. 33). Mantovani and Faglioli (7) have shown that a raffinose concentration of 2% on water reduces the rate of sucrose crystallization to less than half of that at 0% raffinose. They found an appreciable concentration of raffinose in crystalline sucrose derived from solutions containing raffinose, and concluded that the raffinose is adsorbed on the surface of the growing sucrose crystals and finally becomes trapped to some extent in the crystals.

Because of its retarding effect on sucrose crystallization raffinose, rather than total nonsucrose, can be the criterion for the rate or timing of molasses discards. The maximum tolerable concentration of raffinose in the factory syrups will depend upon the individual factory design and the local economic conditions and is likely to lie between five and ten parts of raffinose per 100 parts of solids in the final molasses.

Developments within the last decade have made it possible to ameliorate the raffinose problem, thereby reducing the proportion of molasses that has to be discarded to control the raffinose build-up, while simultaneously recovering some sucrose from the raffinose itself.

Hudson and Harding (5) pointed out that the enzyme melibiase will, in the absence of invertase, partially hydrolyze raffinose, the products being sucrose and galactose. The Fermentation Research Institute of Japan (16e) developed a commer-

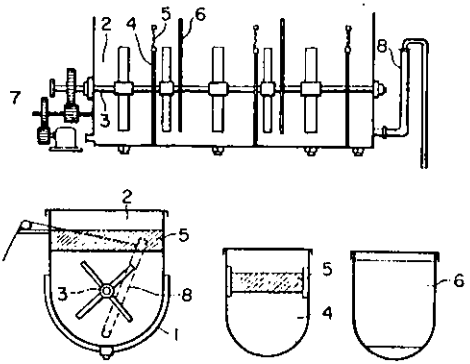


Figure 18-9. Shimizu & Toshio's reactor for the enzymatic hydrolysis of raffinose to sucrose and galactose, patent sketch.

cial application of this process, wherein a similar enzyme which has been named α-galactosidase is produced on a large scale by the mold *mortierella vinacea*. The molasses from the sugar end is treated with this enzyme before Steffenizing.

The process has been patented (11c). Its basic principle is illustrated in Figure 18-9, the patent sketch of the original reactor, together with the following description: The reagent is a solid-bed enzyme catalyst. It is prepared by culturing the mold *mortierella vinacea* in a medium consisting mainly of lactose, then harvesting, washing and centrifuge-drying the resultant mycelium, which ends up in the form of pellets rich in α-galactosidase.

The continuous reactor is a horizontal U-shaped trough with a retention time of one to three hours. The sketch shows it to be equipped with a paddle agitator, but modern versions use portable-type propeller stirrers as shown in Figure 18-10. The reactor in the sketch is divided into a series of compartments by baffles of two alternating types, 4 and 6. Type 4 extends to the bottom of the vessel and is surmounted by a removable screen 5. Type 6 extends to the top of the reactor and leaves a passageway at the bottom.

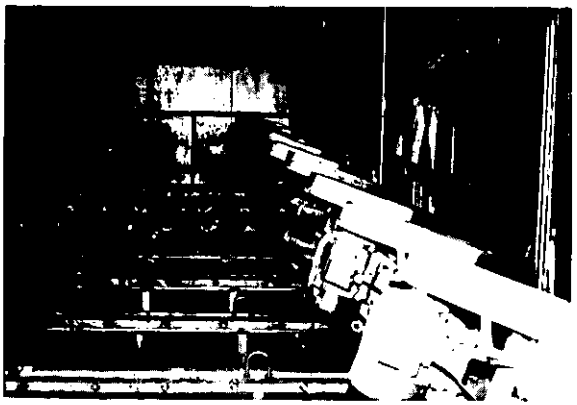


Figure 18-10. Top view of a reactor for the enzymatic hydrolysis of raffinose to sucrose and galactose, courtesy of The Great Western Sugar Company.

In operation the reactor is filled with enzyme pellets to the upper part of screens 5. The molasses to be treated is diluted in a separate mixer to 30° Brix, warmed to 50° C and adjusted to pH 5.2 with H_2SO_4. This solution is allowed to flow continuously through the reactor, entering at the bottom inlet at the left of the sketch and leaving by the adjustable overflow 8 at the right. It then passes over a screen to remove any entrained enzyme pellets, and on to the Steffen house. As the pellets become exhausted, the screens are periodically lifted, allowing the bed or a portion thereof to flow downstream and out with the treated molasses solution, and the bed is recharged with fresh pellets.

In modern versions (7b) the enzyme pellets are discharged and replaced completely about every 25 days. It is reported that 0.12 tons of pellets are required for each ton of raffinose hydrolyzed.

The economics of the process are discussed in detail by Meguro and Konishi (7b). It has been in use in Japan where the raffinose content of the beets is very high, e.g. 0.16%, since 1968. An installation has been in service at Billings, Montana since 1973. The raffinose content of Montana beets is on the order of 0.08%. A second U.S. installation has been made more recently at Torrington, Wyoming. The use of the process has not been justified in California, where the raffinose content of the beets is generally less than 0.03%.

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Chapter XIX
BY-PRODUCTS
Molasses*

Molasses is the final syrup spun off after repeated crystallizations in the extraction of sugar. It is so low in purity that further crystallization from it is impractical. It is therefore discarded from the sugar end, carrying with it all of the nonsucroses of the beet not eliminated by the juice purification process, the soluble impurities added in processing, and the degradation products formed during processing. It also carried out about one and one-half pounds of sugar with each pound of impurities. It represents therefore an important loss of extraction, amounting to 12 to 16% of the sugar in the beets. The amount of molasses produced depends upon the impurities left in the juice after purification and upon the degree of exhaustion of the final syrups. In 1970 molasses production in straight houses ranged from 4 to 6% on beets; at Steffen houses, 5 to 7%. The estimated production in 1970 was, for straight houses, 235 000 tons for desugaring processes; 165 000 tons shipped for other uses; and 368 000 tons of discard molasses, which was mixed with pulp for cattle food, etc., for a total molasses production of 768 000 tons. In 1979, molasses production was 684 000 tons. Figures are not available for an accurate breakdown of this production, but the distribution was probably similar. A typical approximate analysis of beet molasses is: dry substance 81-84%; sucrose 50-52%; nitrogenous compounds 12-13%; nitrogen-free extract 9-10%; and ash 11-12%. The nitrogen content of molasses is about 2%.

Betaine is the most abundant nitrogenous compound found in molasses. Carruthers *et al.* (3) found 3.26 to 4.25% of betaine in British molasses, increasing somewhat toward the end of the campaigns. The next most abundant nitrogen compound is glutamine and its hydrolytic products, pyroglutamic acid and glutamic acid. This group constitutes more than half of the total amino acid content of molasses. Aspartic acid occurs as asparagine in the beet. Like glutamine, asparagine is partially hydrolyzed during liming and carbonation; hence, it is found in molasses as an alkali salt of aspartic acid. Leucine, isoleucine, alanine, valine, glycine, and other amino acids are also found in beet molasses in small amounts (see chapter II). About 5% of the nitrogen in molasses is in the form of purine and pyrimidine bases, principally adenine, adenosine and guanosine (3). Found in amounts ranging from near traces to 5% or more of the nitrogen in beets is inorganic nitrogen; i.e., nitrogen as nitrates and nitrites.

The nonsucrose carbohydrates of molasses consist of invert sugar, usually less than 1% in straight house molasses, raffinose, galactinol, inositol, kestoses, and possibly others. In beet growing areas where beets are stored in piles for protracted periods prior to processing, the raffinose content increases, often from 0.5% on dry substance at the beginning of campaign, to 2% or more at the end. Steffen factories show corresponding increases in raffinose content of molasses ranging upward to 5% or even more, on dry substance.

Other nitrogen-free organic impurities in beet molasses are the acids—lactic, malic, acetic, oxalic, citric, and others—formed in part by bacterial action or by destruction of invert in the purification process.

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*by E. H. Hungerford (Revised by R. A. McGinnis)

Uses of Molasses

Molasses is a valuable by-product of beet sugar manufacture. About 60% of straight beet house molasses produced in the United States is shipped to Steffen factories for the recovery of sugar by precipitation with lime.

Most of the discard molasses and some straight-house molasses is used for cattle feeding, either directly or in mixed feed production. Thousands of tons are mixed with beet pulp and dried to produce feed for cattle or sheep.

Molasses, because of its high content of sugar and certain nitrogenous constituents, is a valuable raw material for the fermentation industries.

Alcohol Production

Ethyl alcohol was a very important by-product of molasses 50 years ago with a renewed interest being generated in the late 1970's and early 1980's due to the rapid rise in foreign crude oil prices. Ethyl alcohol is being encouraged for use as a 10% mixture with gasoline to produce gasohol and diesohol for internal combustion engines. The sugarbeet and sugarbeet products are being considered as one of the biomass feedstocks for alcohol production along with other farm and forest products. Several commercial and private producing plants are under development in the Western United States.

The basis of alcohol production from molasses and sugarbeets is the yeast fermentation of the molasses sugars. Sucrose is hydrolyzed by the invertase enzyme of yeast to glucose and fructose which are, in turn, transformed by another enzyme, zymase, to alcohol and carbon dioxide. The theoretical yield of alcohol is four moles of alcohol to one mole of sucrose, or 53.8% by weight, corresponding to about 81 gallons per ton of molasses. Actual yields are lower, of the order of 65-70 gallons per ton. Raffinose is hydrolyzed to fructose and to the disaccharide, melibiose, by invertase. The latter is not fermentable but can be hydrolyzed to glucose and galactose by melibiase, an enzyme found in bottom beer yeast, and is thus made fully fermentable. Molasses must be diluted to about 25% solids before fermentation. The temperature is held at 20-25 °C. After completion of fermentation, the alcohol is distilled, leaving a residue (slop) which is concentrated and used as a fertilizer.

Yeast Production

Molasses is the main raw material used in the production of baker's yeast. Yeast cultures are selected and propagated under carefully controlled conditions. A medium for the commercial production is prepared by adding to a dilute molasses solution the necessary supplementary substances required for the growth of yeast. Molasses contains no phosphate and its nitrogen content is relatively deficient. Less than half the nitrogen of molasses can be used by the yeast. Superphosphates, urea, and ammonium salts are therefore added. The mixture is aerated and stirred by a strong current of air. Nutrients are added, as required, to replace those taken up by the yeast cells. Under the conditions of low concentration of yeast food, and of strong aeration, alcohol formation is suppressed and proliferation of yeast accelerated.

Glutamic Acid

Production of glutamic acid in the form of monosodium glutamate by microbiological fermentation of molasses (not entirely beet molasses) now accounts for virtually all production in the United States. Originally it was produced from Steffen filtrate. The production of glutamic acid by microbiological processes was

discovered almost simultaneously by workers in Japan and the United States. The microorganisms used are not identical but apparently are similar (*Brevibacterium spp*). Diluted molasses, or other carbohydrate material, and required supplemental ingredients for growth of the organism, including growth-promoting substances such as biotin, are adjusted to a suitable pH and inoculated with a culture of the organism. Recovery of glutamic acid after fermentation follows roughly the process used in the production of glutamic acid from Steffen filtrate.

Citric Acid

Citric acid, formerly manufactured entirely from citrus fruits, chiefly lemons, is now almost exclusively produced by the action of selected molds of the aspergillus type. Dilute molasses, to which mineral salts containing nitrogen and phosphates are added, is inoculated with the mold culture and placed in shallow pans arranged in a large incubator, through which a current of sterile air is passed. Fermentation proceeds at about 30 °C. At the conclusion of the growth period the liquid is separated from the mold mycelia by draining and pressing. The citric acid is precipitated by milk of lime and washed free of molasses impurities. Citric acid is readily obtained by treatment of the precipitate of calcium citrate with sulfuric acid, separating the insoluble calcium sulfate, and subsequently crystallizing the citric acid.

Molasses is used in numerous other fermentation processes.

Molasses Formation

The sugar carried out of the beet-sugar factory in molasses is its largest single loss. This large loss of extractable sugar has prompted study and effort since the beginning of the industry to find means of reducing it. Obviously, more complete exhaustion of the final syrups can be achieved by boiling to high density, cooling to low temperature, and allowing longer periods of time for crystallization. Molasses purities of approximately 50% have been obtained in this way by numerous workers. As a practical matter, however, there are limitations to this approach. As the density of low raw massecuites increases, the viscosity rapidly increases, making separation of crystallized sugar slow and unsatisfactory. The viscosity of molasses approximately doubles with each increase of 2 in percentage dry substance (1). (Please see Table 19-1.) Similarly, lowering the temperature by 10 °C doubles the viscosity. As the purity of the syrup is lowered, the rate of crystallization decreases rapidly so that many months may be required to even closely approach the actual solubility limit of sucrose in these syrups. Molasses, therefore, cannot be defined as the syrup from which no more sugar ultimately can be crystallized, but rather a syrup from which "no more appreciable amounts of sugar can be recovered by crystallization at economical cost."

The nonsucroses of molasses vary markedly in composition. These nonsucroses influence the crystallization of sugar in two ways: first, by affecting and, usually increasing, the solubility of sugar in the water, and second, by affecting the rate of crystallization of sucrose, either directly by impeding deposition on the surface of the crystals, or indirectly by the effect on the viscosity of the syrup, and the consequent decrease in the mobility of the sucrose molecules in the syrup surrounding the crystals. The viscosity of impure beet sugar solutions is lower than that of pure sugar solutions of the same percentage dry substance. The high viscosity of low purity massecuites is due to their high dry substance content—not the presence of nonsugars.

Herzfeld determined the solubility of sucrose in water and the influence of in-

Table 19-1 Viscosity (in centipoises) of Beet Factory Syrups (1)

True Purity	Dry Substance, % 74	76	78	80	82	84	86
				At 40°C			
60	250	435	806	1690	3960	11200	40000
70	261	456	863	1830	4340	12400	
80	273	480	919	1990	4710	13600	
90	284	503	977	2120	5080	14800	
100	295	526	1030	2260	5450	16000	
				At 50°C			
60	131	210	376	726	1570	3880	12000
70	135	221	400	777	1700	4200	
80	139	231	424	828	1830	4520	
90	144	242	448	879	1950	4840	
100	148	258	470	929	2050	5160	
				At 60°C			
60	77	121	200	359	719	1630	4310
70	79	125	207	375	756	1710	4610
80	81	129	214	392	792	1780	4920
90	83	132	221	407	829	1850	
100	85	135	228	423	866	1920	
				At 70°C			
60		74	115	199	361	756	1810
70		75	119	205	374	785	1880
80		76	123	210	387	807	1940
90		77	127	215	401	833	
100		78	131	220	414	858	
				At 80°C			
60		49	73	115	199	383	844
70		50	73	118	205	396	883
80		51	76	120	210	408	922
90		51	78	123	216	421	
100		52	78	125	222	434	

organic salts on the solubility about 80 years ago. Other workers have studied the effect of the addition of pure substances on the solubility of sucrose. In general, most substances in small quantities and at low temperatures, under 40 °C, depress the solubility, but at higher concentrations and at higher temperatures, above 60 °C, the solubility of sucrose in water is usually increased. The substances thus increasing the solubility of sucrose are referred to as *melassigenic*. Alkali salts are the most melassigenic: i.e., they appear to "bind" more sugar in an uncrystallizable form than do other substances. This has influenced many observers to attribute molasses formation principally to alkali-sucrose compounds. Dedek postulated that one mole of potassium or sodium binds one mole of sucrose in molasses. The mole ratio of sugar to the K+Na therefore indicates the degree of exhaustion of molasses, and it should be 1.0 on molasses from properly crystallized raw massecuite. This ratio has been found by other workers to range from about 0.8 to more than 1.2. Carolan found that, at least in Ireland, calcium had to be included as an alkali in the sucrose-alkali ratio. The mole ratio of sucrose to K+Na+Ca was found by him to lie between 1.0 and 1.10. The calcium content of molasses is usually so low compared to the content of potassium and sodium that its inclusion does not materially affect the ratio.

Calcium and magnesium are normal constituents of beets, but only about 10% of the calcium and 50% of the magnesium is extracted in diffusion. Almost all of the magnesium is ultimately eliminated in carbonation. Most of the calcium is also eliminated except when processing deteriorated beets (3). Calcium and particularly magnesium salts tend to decrease the solubility of sucrose. Magnesium salts, indeed, have been found to depress the solubility of sucrose in water to such a degree that they may be said to be "negatively melassigenic" when they replace other salts present. Quentin (4) devised a process which, with the use of ion-exchangers, replaced the strongly melassigenic alkalies in intermediate syrups with magnesium. The purity of molasses produced from these syrups is considerably lower than the molasses obtained from the untreated syrups (see pp 326-330).

Nitrogen compounds, which constitute one third to one half of the nonsucroses in beet house syrups, decrease the solubility of sucrose at all concentrations at which they normally occur. Invert sugar is never found in beet molasses in amounts large enough to have an appreciable effect on molasses purity. In cane molasses, however, the invert sugar content is high and the sucrose purity of the molasses correspondingly low: i.e., 35 to 45% sucrose on solids. The raffinose content of beet molasses from Steffen houses is often high enough to exert a measurable lowering effect on molasses purity (2). Often the potential lowering of purity by raffinose is not obtained in practice because of the marked reduction in rate of crystallization of sucrose in the presence of raffinose.

Despite its failure to provide highly accurate estimates of the probable loss of sugar in molasses, the sucrose: K+Na ratio is possibly the best measure obtainable by chemical analysis. Spectrographic determination of Na and K is rapid and the analyses can be made a part of routine laboratory control.

$$\text{organic factor} = \frac{\text{nonsucrose} - \text{ash}}{\text{ash}} = \frac{\text{organic nonsucrose}}{\text{ash}} \qquad (1)$$

As might be expected, high organic factors accompany molasses of lower purities. Schneider (7) has modified this relationship by correcting the ash for its content of CaO;

$$f = \frac{\text{nonsucroses} - 1.9\ (A - 2.35\ CaO)}{1.9\ (A - 2.35\ CaO)} \qquad (2)$$

He found a linear relationship between the purity of various molasses at the same nonsucrose: water ratio after long periods of crystallization, and the quantity, f. "A" in this equation is conductivity ash, and CaO means the lime and magnesia determined by ethylene diamine tetraacetate titration. The ash component of the equation, as corrected by Schneider, consists essentially of potassium and sodium salts, hence the factor f is actually another relationship measuring the sucrose binding power of the alkalies.

P. M. Silin (9) defines as "normal" a molasses of 82% dry substance which is saturated with respect to sucrose at 40 °C. To produce such normal molasses, it is necessary to hold the massecuite at a lower temperature—30-35 °C—long enough to reduce the purity, and then reheat to 40 °C. The viscosity of normal molasses, as defined by Silin, is 44 poises at 40 °C for 1100 rpm batch centrifugals. For 1800 rpm centrifugals the viscosity range is 90 – 120 poises.

In the United States molasses at many factories is spun off at 83-84 ° dry substance. However, the centrifuging temperature is usually raised above 40 °, often to about 48 °, so the viscosity is still only about 44.

With this concept of a "normal" molasses it is possible to determine the melassigenic coefficient, m_o, of the impurities. If the purity found under these standard conditions is:

$$P_o,\ m_o = \frac{P_o}{100 - P_o} \cdot m_o$$

represents the pounds of sugar retained in the molasses by a pound of impurities in that molasses. Z. A. Silina (9) has used this method to determine the melassigenic coefficients of a number of individual nonsucroses. She first determined the normal purity, P_o, of a molasses, and then added a nonsucrose, for example NaCl, in an amount equal to n% of the nonsucroses in the molasses mixture. The normal purity, P_1 and the coefficient, m_1 of the molasses mixture was then determined. The melassigenic coefficient, m, of the added nonsucrose could then be calculated from these data as follows:

$$100\ m_1 = nm - (100\text{-}n)\ m_o, \text{ and } m = m_o - \frac{100}{n}(m_1 - m_o) \qquad (3)$$

Each determination of m was repeated using different molasses. The "m" values are shown in Table 19-2.

Table 19-2. Melassigenic Coefficients of Nonsucroses (9,9a).

Nonsucrose	m	μ
NaOH	4.61	1.69
K_2CO_3	3.38	1.10
Na_2CO_3	2.88	0.87
K-acetate	2.85	0.84
NaCl	2.58	0.72
KCl	2.48	0.67
Betaine	1.03	−0.028
K-lactate	1.02	−0.038
K-glutamate	0.99	−0.038
KNO_3	0.96	−0.056
Na-glutamate	0.93	−0.070
K-tyrosinate	0.090	
Na-lactate	0.81	−0.13
K-products of decomposition of invert sugar	0.70	
$CaCl_2$	0.56	−0.25
Na-products of decomposition of invert sugar	0.55	
$NaNO_3$	0.42	−0.34
Invert sugar	0.19	
Ca-glutamate	0.18	−0.48
Ca-tyrosinate	0.11	
Ca-lactate	−0.14	+0.58
Ca-acetate	−0.55	−0.78
Ca-products of decomposition of invert sugar	−0.66	
$Ca(NO_3)_2$	−1.14	
$MgCl_2$	0.65	−2.12
$Mg(NO_3)_2$	1.03	−2.06

(Results for Mg salts by T.P. Hvalkovsky)

Silin's daughter, N.P. Silina, (9a) noted that the above relation ignores the fact that water is itself a very melassigenic nonsucrose. She calculated the melassigenic properties of specific nonsucroses in terms of the specific melassigenic coefficient, μ, which is specific for a given nonsucrose composition, and indicates changes in sucrose solubility:

$$\mu = m_n - 2.37\,(m_n + 1)\,(100 - ds_n)\,/ds_n \qquad \textbf{(3a)}$$

where m_n is the melassigenic coefficient of a given nonsucrose, 2.37 was the accepted value for sucrose solubility (the later value of 2.35 involves only slight changes) at 40 °C, and ds_n is the dry substance content of the "normal" molasses.

Comparison of the two sets of values shows that changes in sucrose solubility per gram of nonsucroses are much smaller than the quantity of sucrose solubility in water. Thus a reduction of the amount of water in molasses is of prime importance in reducing molasses purities.

Rorabaugh and Norman (5), determined the inhibiting effect of several individual impurities on the crystallization of sucrose from molasses. They found potassium carbonate and potassium chloride have greater inhibiting effects than any other constituents of molasses. Betaine and potassium sulfate have the least effect per unit weight of the impurity. Betaine, however, because of its relatively great abundance, exerts an important total effect. Glutamic acid has a greater inhibiting effect per unit weight than any of the other organic constituents except leucine and isoleucine, and because of its relatively great abundance is an important inhibiting agent.

It is evident that it is impossible to attribute molasses formation to a single or even to a selected few nonsugars. The results show the strong melassigenic effects of the alkalies, the smaller effects of nitrogenous compounds, and the relatively slight effects of calcium salts.

Each constituent of molasses is melassigenic in the sense that it adds to the total dry substance of the syrup, and, whether it acts to suppress or to increase the solubility of sugar in water, it adds to the viscosity of the syrup, and thus influences the crystallizability of sugar from the syrup. The only alternative to the chemical approach appears to be the determination of the actual solubility of sugar in each syrup, considered at concentrations and temperatures within the practical operating range of the factory.

The solubility of sucrose in impure solutions has been studied by many investigators, including Herzfeld, Grut, Claassen, Wiklund, Silin, and Brown and Nees. Their results, although strictly applicable only to the particular syrups studied, are used widely to calculate pan and crystallizer supersaturations. Indeed, there is no alternative; the time required to achieve saturation in syrups of low purity at low temperatures—often several months—precludes routine determinations of solubility in factory syrups.

O. Wiklund found that the coefficient of saturation, or saturation, C, as it is referred to by Claassen, remains constant with changes of temperature for syrup of given nonsucrose:water ratio. This finding, known as Wiklund's rule, permits the calculation of the solubility of sucrose in impure solutions from determinations made at higher temperatures, where attainment of solubility equilibrium is accomplished more quickly—often in a few hours. The saturation number, C, is defined by the equation $C = Z'/Z$. Here, Z and Z' are the solubilities in g sucrose/g water in pure solution and in impure solution, respectively, at the same

temperature. The Wiklund rule states that the saturation number, C, determined at temperature, t_1, is equal to the saturation number determined at any other temperature, t_2, in syrups of the same nonsugar:water ratio, N/W. Thus:

$$(C_{t1} = C_{t2})\ N/W = \text{a constant} \quad (4)$$

Wagnerowski, D. Dabrowska, and C. Dabrowska (11) describe a quick method and the apparatus for determining solubilities in impure solutions, and a method of calculation of results based on the Wiklund rule. Briefly, molasses or raw pan mother liquor is adjusted by concentration or dilution to a nonsucrose:water ratio between 1.6 and 3.0 g nonsucrose/g water, and is placed in a metal solubility vessel containing a mechanical stirrer and a thermometer. A quantity of screened coarse sugar is added, and the apparatus is tightly closed and placed in a thermostatically controlled bath. The temperature is held at a point where sugar will be dissolved. Saturation equilibrium is reached in from 10 to 26 hours, depending on the nonsucrose-water ratio. A sample of crystal-free liquid is drawn through a filter tube, and the sucrose content and dry substance are determined. Wagnerowski and co-workers made solubility determinations in a syrup at four nonsucrose:water ratios. The saturation numbers thus determined, when plotted against the nonsucrose:water ratios, fall on a straight line. It is obviously necessary then to make the solubility measurements at only two ratios. Vavrinecz (10) tested the validity of the Wiklund rule by plotting published solubility data of 11 investigators in Europe and America, and found nearly complete confirmation. The linear relationship between saturation number and nonsucrose:water ratio holds only at ratios above about 1.5. This fact does not limit the usefulness of the method, since the nonsucrose:water ratio of raw massecuite must, in practice, be held between about 1.8 and 3.0. At ratios below 1.5, massecuite must, in practice, be held between about 1.8 and 3.0. At ratios below 1.5, molasses purities cannot be reduced to desirable levels. At ratios of 3.0 or higher the molasses becomes increasingly viscous and difficult to centrifuge satisfactorily.

With a knowledge of the actual solubility of sucrose in the final low purity syrups the exhaustibility of a molasses can be determined without recourse to uncertain calculations based on the effects of various individual nonsugars. Wagnerowski (11) developed equations relating "normal" purity of molasses to the constant of their solubility equations. He also shows a method for calculating optimum purity and dry substance of the final boiling massecuite. The laborious calculations are simplified by nomographs.

K. W. R. Schoenrock and J. R. Johnson (8), using the Wagnerowski method, have shown that the solubility of sucrose in the low purity syrups from beet factories in Idaho and Oregon is considerably higher than the solubility taken from the tables of either Grut or Brown and Nees for syrups of the same purity. The difference, in part, may be attributed to differences in analytical procedures used. Schoenrock and Johnson determine sucrose in their syrups by single acid, Clerget inversion, and the dry substance with refractometer. Brown and Nees determined sucrose by double-enzyme inversion and dry substance by oven drying. Grut also determined dry substance by oven drying. It is apparent that Schoenrock's solubility data are completely satisfactory for his purposes. He was able to set up criteria for the density of massecuite in the crystallizers and for the temperature of massecuite at the centrifugal station. Dilution and reheating during various crystallizer stages were scheduled with minimum danger of redissolving crystallized sugar, or inadequate exhaustion of the molasses.

A detailed review of molasses formation and composition was published in 1978 (3a.) G. Vavrinecz covered the same subject in a series of 14 papers, published between 1965 and 1974 (10a).

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DRIED PULP*

A Brief History

Since the earliest days of the beet-sugar industry, beet pulp remaining after the extraction of the sugar has been recognized as a valuable cattle food, containing valuable proteins, minerals, and carbohydrates. To the European farmer in the last century, the exhausted beet pulp was often as valuable as the revenue from the sugarbeets.

It was then a common sight to see a farmer waiting for his wagon to be filled with beet pulp produced from beets he had previously delivered. Farming was not as specialized as it is now, and the feeding of cattle was just as important as growing beets. This led to the practice of storing the "green" or wet beet pulp in large pits or silos, where it remained until it was needed by the farmer. Tremendous losses are incurred in the wet storage of pulp. Some tests have indicated that as much as 65% of the original weight of beet pulp and 50% of its food value is lost during a six months' storage period. In addition, the decomposition and fermentation products of wet beet pulp stored in open silos are offensive, and often damage the relationship of the sugar company with the surrounding community.

As time passed, the trend towards scientific specialized farming and less cattle raising by individual farmers reduced the value of beet pulp to the grower. This forced the beet processors to seek additional outlets for pulp. Since water is a major

*by Fred F. Coons, revised by James M. Buchert

component of wet pulp, transportation over long distances was not economical. This led to the practice of drying the pulp. A greater part of the moisture is removed from the wet pulp by mechanical pressing, and the remainder is then evaporated by heat to produce dried pulp, a highly concentrated cattle feed. When molasses is added ahead of the evaporation step, "molasses-dried" beet pulp is produced; without molasses addition, "plain-dried" beet pulp is the product. Some processors substitute up to 50% of the molasses solids with concentrated Steffen filtrate solids or liquid protein concentrate to produce a "molasses-CSF" beet pulp.

Table 19-3 shows typical analyses of the two types.

Table 19-3 Typical Analyses of Dried Beet Pulp* (Pacific Coast Beets)

	Plain Pulp	Molasses Dried Pulp	*CSF and* Molasses Dried Pulp
Crude Protein	8.31	7.61	10.16
Crude Fat	0.66	0.54	0.66
Crude Fiber	23.05	18.34	18.59
Nitrogen-free extract	65.59	68.96	64.27
Ash	2.39	4.55	6.32

*Percentages reported on dry basis.

Outline of the Drying Process. The wet beet pulp is either pumped with added water or mechanically conveyed to the pulp drying station, where the free water is removed by screening. From the screens, the pulp is fed to presses which mechanically force the pulp into smaller and smaller volumes, the water escaping through screens. Please refer to Figure 19-1 for a cross section of the vertical single screw, pulp press, and to Figure 19-2 for a cross section of a horizontal twin screw, pulp press.

Roughly 81% of the total water that is removed in the process is removed in the pulp presses, and the resultant pressed pulp contains from 72 to 85% water, on a wet basis. When producing molasses-dried beet pulp, either beet or cane molasses is sprayed on, or otherwise mixed with the pressed pulp.

The pressed pulp is fed to a direct-fired, induced-draft, parallel-flow, rotating-drum drier, which reduces the moisture content to approximately 5 to 12% in the final product. The drum contains a large number of baffles which drop the pulp through the hot flue gases from the furnace as the drum rotates. These drier drums are usually mounted perfectly level, so that as the drum rotates, alternately lifting and dropping the pulp, the pulp is progressively moved forward through the drum only by the flow of combustion gases.

The capacity of the pulp drier drum depends on its dimensions. Driers with drums as small as 7.5 ft in diameter by 31.5 ft long are in use. Average size units have a drum 10.5 ft in diameter by 48 ft long, which will handle pulp from about 2000 tons of beets a day. The larger units, 12 ft in diameter by 56 ft long, are becoming more common, and these will handle pulp from about 3500 tons of beets a day, all figures conservatively stated. In practice, the average unit has been known to handle pulp from 3500 tons of beets a day and the large unit has been known to handle pulp from 5000 tons of beets a day.

Various fuels, including oil, natural gas, and pulverized or slack coal can be used in the furnace. The products of combustion are mixed with a cooling gas prior to entrance to the drier drum to obtain an entering temperature of 900-1700 °F, depending on the load handled relative to the size of the drier. As a general rule,

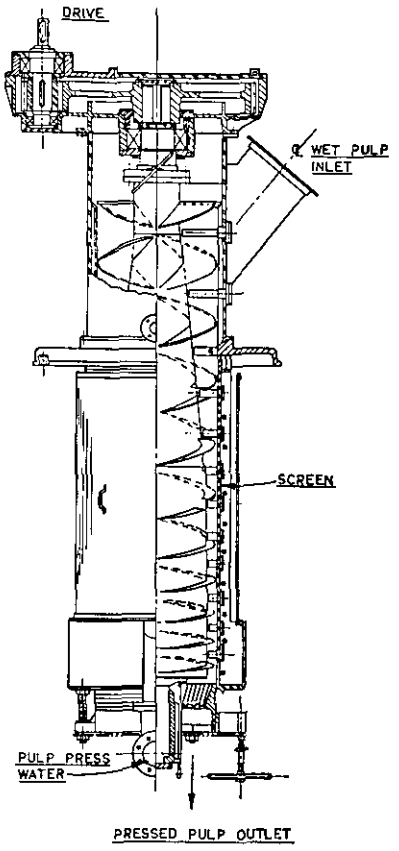

Figure 19-1. Cross section of single-screw, vertical pulp press.

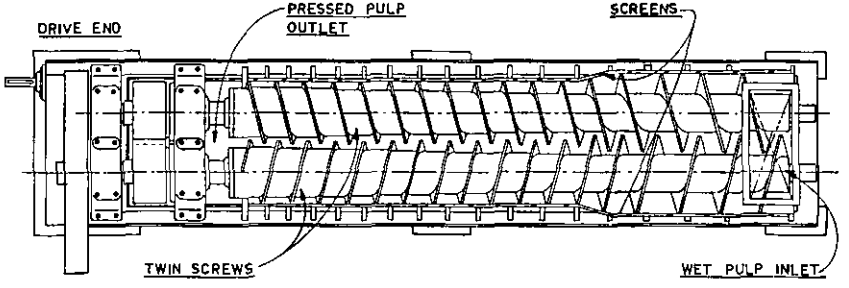

Figure 19-2. Cross section of twin-screw, horizontal pulp press.

losses from combustion of the product increase as the temperature from the furnace increases. The induced draft fan located at the discharge end of the drum draws air from the revolving drum and discharges it through a cyclone separator to recover small particles of pulp from the exhaust gases. The temperature of the exit flue gases varies from 190 to 280 °F, depending on the drum design and load.

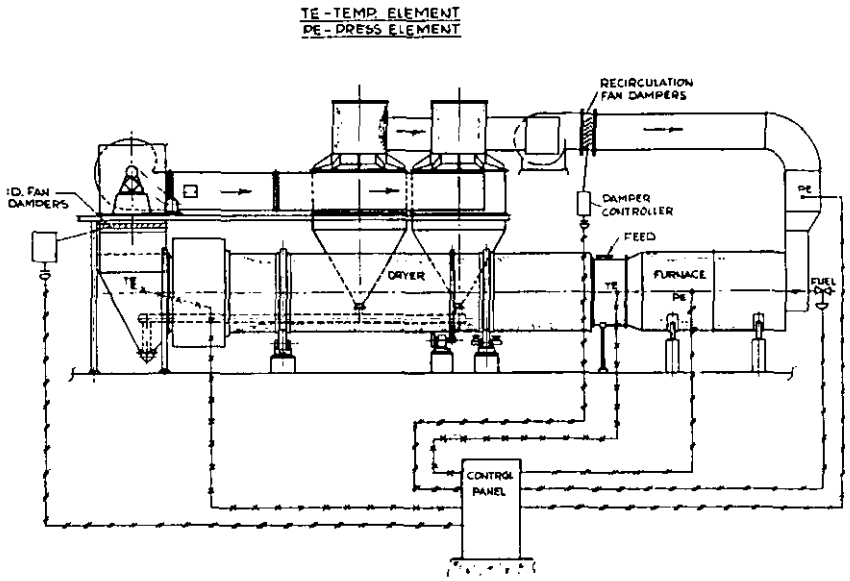

Figure 19-3. Pulp drier with exhaust gas recycle system. *(Courtesy, Stearns-Roger Corp.)*

A number of processors have installed systems for recycling stack gases back into the drier furnace. (See Figure 19-3.) A special connection to the stack removes most of the fine dust not caught by the cyclones, together with a portion of the stack gas. This mixture is forced by a recirculating fan or by back pressure created by the cyclone stack restrictors through a duct to the hot air furnace to supply the necessary cooling gases. For proper combustion of the fuel, flames should be relatively hot. For proper drying, the drying gases should be cooler than the combustion temperature. This is accomplished by introducing the recirculated stack gas into the furnace after the burning zone, to cool the mixture of gases to a temperature of approximately 1200 °F preparatory to entering the drier drum. The fines that were removed from the stack are subjected to high heat and are usually converted to ash. Since only a small amount of excess air need be used for primary combustion, and the system is essentially closed, the oxygen content of the gases contacting the pulp may be very low when using recycled gas. It has been reported that the oxygen content in recycled systems is approximately 0.5 to 1.0%, as contrasted to the normal 15% of excess oxygen, in an air-cooled system. Since a nearly inert atmosphere is created, degradation of the product by oxidation is minimized. Moreover, since the oxygen content of the gas is low, the possibility of accidental fire in the drier drum is reduced. Also, less fuel is used. Usually the very hot furnace gases are cooled with secondary air at room temperature, say 70 °F. When the stack gases are recirculated, the secondary cooling air is about the same temperature as the stack gases, namely, 230 to 280 °F. Return of these hot stack gases means fuel savings of approximately 5 to 8%.

The importance of fuel used in the pulp drier operation is paramount. Of the total fuel required by the entire beet factory, approximately 30% is used in drying pulp. Any savings in fuel by astute operations, use of recycled gas, or by lowering the moisture content of the pressed pulp will yield substantial savings in fuel consumption.

Present day technology provides a substantial opportunity to decrease product quality variances through greatly improved control systems and processing equipment. In most cases spontaneous heating and fires in storage can be controlled or eliminated through better moisture and temperature control of the product. Efficient product cooling is a major controlling parameter toward this end (6b). New process control systems have demonstrated a capacity to sharply reduce the range or variation in output product moisture (6a,6b).

Substantial efforts in increasing efficiencies in the areas of pressing, fuel consumption, product moisture optimization, and electrical power consumption are easily justified at today's increasing fuel costs. The exhaust gases from gas turbine electrical generators may provide heat energy for drying in addition to electrical energy for the dehydrator if appropriate fuels are available. Recent experience with additives and physical techniques which enhance pressability offer additional benefits (6b,7a,7b). Given the normal operating life and amortization parameters for capital equipment, primary emphasis in systems design and operation must be given to energy conservation and efficiency. Performance in the energy area may well determine not only corporate profitability, but corporate survival.

Table 19-4 is a typical theoretical material balance for a pulp drier. It contains several assumptions which are self-explanatory.

In the following section, the theoretical aspects of the drying process, losses encountered and various factors that affect the process will be considered in greater detail.

Factors Affecting Pressing. The pressing operation is mechanical and is affected by the physical characteristics of the pulp. The physical condition of the wet

Table 19-4 Theoretical Balance for a Pulp Drier

	Station	% Sugar	% Water	Sugar	Soluble Nonsugar	Total Nonsugar	Total Solids	Total Weight
	Pulp Flume to Drier							
1.	Wet pulp		93.47	1.31	.72	5.22	6.53	100.00
2.	Flume water			.40	.40	.40	.80	450.00
3.	Total			1.71	1.12	5.62	7.33	550.00
	Separator Screen							
4.	Feed			1.71	1.12	5.62	7.33	550.00
5.	Flume water			.40	.40	.40	.80	450.00
6.	Pulp to presses		93.47	1.31	.72	5.22	6.53	100.00
	Pulp Presses							
7.	Feed	1.31	93.47	1.31	.72	5.22	6.53	100.00
8.	Pulp press water	1.39		1.06	.27	.27	1.33	76.37
9.	Pressed pulp	1.06	78.0	.25	.45	4.95	5.20	23.63
	Mixing Scroll							
10.	Feed		78.0	.25	.45	4.95	5.20	23.63
11.	Molasses (60 apc, 30% ds, on total ds)		16.0	1.34	.89	.89	2.23	2.66
12.	Drum feed		71.7	1.59	1.34	5.84	7.43	26.29
	Drier Drum							
13.	Drum feed		71.7	1.59	1.34	5.84	7.43	26.29
14.	Water evaporated		-------	-----	-----	-----	-----	18.03
15.	Loss (10% of feed ds)		10.0	.16	.13	.58	.74	.82
16.	Dried pulp	19.2	10.0	1.43	1.21	5.26	6.69	7.44

pulp from the diffuser is a function of the condition of the beets before processing, the size of the cossettes, the temperature and duration of the diffusion process, the pH of the diffusion operation, the mechanical handling of the cossettes in the diffuser, and the conveying system from it.

If the pulp is too firm, the load on the presses is increased to a point where the capacity of the press drives is exceeded, and the moisture is removed with great difficulty.

If, on the other hand, the pulp is too soft or mushy, the load on the presses is considerably reduced. However, the pulp may be so soft that a large portion passes through the screens with the pulp press water. As in the case of pulp that is too firm, the pressed product may also have a high moisture content. It has been contended that addition of molasses to the wet pulp before pressing makes it possible to press out more water (3).

Pulp with physical characteristics between these extremes will involve a normal load on the press drive and will pass a minimum of pulp through the screens with the pulp press water. Fresh beets which have been properly diffused will yield a pressed pulp moisture content of 75-80%. In the case of horizontal twin-screw presses and also in vertical presses, it is possible to vary the speed to improve performance and alter the capacity of the presses. In general, the slower speed will produce the lowest moisture content of pressed pulp, but the capacity is reduced almost lineally.

Conditions which mean good operation in the diffuser are also generally conducive to good pulp pressing operations. If the pulp is too firm, this usually means that the diffuser temperatures were too low, or the retention time too short, with the consequently high diffuser losses. Likewise if the pulp is too "mushy," from soft beets, from too-high diffuser temperatures, or from too-long diffuser retention time, low purity of diffusion juices results. When conditions are such that low pulp losses and high purity diffusion juices are obtained, the pulp drier operations are usually satisfactory.

A linear correlation has been found (7) between the diffusion battery temperature and the pressed pulp moisture content. In the range studied, the pressed pulp moisture increased as the average battery temperature increased. This has, of course, been observed and taken into practical account by many operators in the past.

A pulp which presses better is also secured as the pH of the last few cells of the diffuser is lowered. This result may be achieved by permitting enough microbiological action to take place in the diffuser to acidify the pulp. This causes loss of sugar, but in some cases this loss of sugar may be less than either a) the cost of the fuel to evaporate the additional water in the pulp drier, or b) the cost of the addition of acid to adjust to the same pH. Recent research provides greater understanding of and accurate procedures for controlling diffuser acidity for maximum energy savings while minimizing sugar losses in the diffuser (5a). Substantial and continuous effort in this area is mandated by rapidly changing energy economics. From a practical point of view, 5.5 pH appears about optimum for the liquid in the last cell when considering this factor.

Therefore, with physically softened beets, both higher purity diffusion juice and drier pressed pulp result from lowering the diffuser temperature. Thus, the general statement can be made that the diffuser and the pulp drier operations are closely connected.

Factors Affecting Drying by Heat. Drying is an evaporation step, in which the latent heat of the evaporation of moisture in the pulp is supplied by the sensible heat of the hot gases in the drier drum. (Figure 19-4).

In the feed end of the drier where the pulp is wet, as the evaporation proceeds from the surface of the pulp, a gradient is established between the moisture at the surface and in the interior of the pulp particles. When there is considerable water present, the rate of diffusion from the interior to the surface is rapid enough to keep the surface wet, and evaporation takes place at a temperature below the wet bulb temperature. As the hot gases and the pulp pass down the length of the drum, the temperature of the gas decreases, and simultaneously the temperature of the pulp increases. At the discharge end, the pulp temperature tends to rise, approaching the temperature of the effluent gases. However, actual tests sometimes show the pulp temperature may be as much as 100 °F below the wet bulb temperature. In a direct-fired rotary drier such as is generally used for drying pulp, the wet bulb temperature may be in the range of 180-200 °F. Thus, if the above conditions are met, the temperature of the pulp may be as low as 140 °F, even though the entering flue gases are at 1200 to 1500 °F.

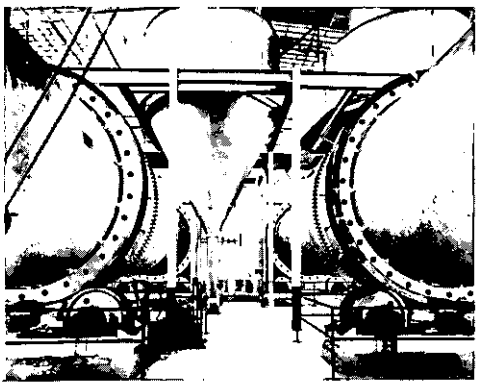

Figure 19-4. Pulp drier drums, Carlton factory, Brawley, California. Stearns-Roger manufacture. ***(Courtesy Holly Sugar Corp.)***

As long as the pulp remains sufficiently wet to be below the wet bulb temperature, the evaporation is at a more or less constant rate. When the water content becomes so low that the surface is not wet, the evaporation rate decreases, and the temperature of this surface increases. However, at this point in the dryer drum, the temperature of the flue gases is now much lower than on entrance, and as a result, the temperature of the pulp increases only slightly.

It is apparent that small, thin particles of pulp will lose their moisture much more rapidly than large particles constituting the main bulk. If this occurs close to the point of entry to the drum, the small particle will be heated nearly to the temperature of the combustion gases, which may be more than 1000 °F, and at this point some burning may occur. This is to some extent counteracted by the rapid drying and reduced weight of the finer particles, which are selectively transported toward the cooler zone at the discharge end of the drum.

An important means by which the operator can increase the drying rate is to increase the amount of fuel fed to the furnace. This affects the rate of evaporation both by increasing the amount of available heat and by increasing the temperature difference between the pulp and the flue gases.

Great fuel economy is realized when the pulp drier drum is operating close to its maximum capacity. This is probably because the increased fuel requirements per unit of air fed to the drier causes a greater temperature difference, and in turn, increases the rate of evaporation. However, as will be shown, the total losses in the drum increase as the rate of evaporation increases.

As a general rule, the drying drums have ample capacity to meet the normal fluctuations in pressed pulp moisture content. Nevertheless, increasing the pressed pulp moisture content from 79 to 80% will increase the load on the drier drum by approximately 6%. Hence, in the interest of economy, every effort should be made to keep the pressed pulp moisture as low as possible. This becomes increasingly important if the drums are operated near maximum capacity.

Further tests (11) have indicated that the pressed pulp treated with molasses is more susceptible to losses in the drier drum than plain pulp. However, the use of molasses does not greatly alter the rate of drying, but it does extend the constant drying-rate zone. Because of its higher dry substance, closely approaching that of the dried pulp, molasses addition actually reduces the load on the drum per bag of dried pulp produced.

Losses. Losses occur at several points in the water-removing operation. A large portion of the dissolved solids within the wet pulp is lost with the press water in the pressing operation, of which about 50% is sugar. In an effort to recover this sugar, and also to reduce the waste disposal problem, most factories treat the water for return to the diffusion operation. This factor, in addition to factors previously stated, makes the operation of the diffusion process and the drying process very closely related to each other, so that the operations must be integrated to obtain the optimum results.

The pressing operation is accountable for considerable loss of small particles of pulp which are forced through the screens and the presses. These particles can be recovered by either returning to the diffuser or separately screening and returning to the pulp press feed. In installations having a separate screen for press water, operators can learn whether or not there are holes in the press screens by observing the quantity of material separated on the screens. While these particles are often recovered in the process, they are susceptible to eventual loss in the drier drum. As was mentioned, they may be burned in the drier drum. Furthermore, when the very small particles are dried, they become fine dust, which is very difficult to separate efficiently from the waste gases.

Plant tests have shown that when the stack gases are not recycled to the furnace, the stack losses of particulate matter range from 0.15 to 0.50% of the total dried material. When the stack gases are recirculated, on the other hand, the stack losses are reduced by about 40 to 60%. Measured total drier losses range from 4.5 to 10%. The difference between the total and the stack losses is, of course, the loss by combustion. The amount lost by combustion depends on the temperature of the hot gases entering the drier drum, the amount of pulp being dried and the amount of molasses or other additives being used.

Losses within the drum have been found to increase with increased molasses addition, increased operating rates, increased moisture content of the drum feed, the size of the pulp particles in the feed, and an increase in the firebox temperature. For minimum losses within the drum, it appears inadvisable to operate at a furnace exhaust gas temperature above 1200 °F.

Control of the Drying Process. For reasons of economy, it is advantageous to produce dried pulp containing only very slightly less moisture than the guaranteed

maximum. If the moisture content is much less, not only is the processor delivering more dry matter than he is paid for, but he is also out the cost of the additional fuel used in drying the pulp to the lower moisture content. If operations are such that the final moisture content has an uncertainty of ±3%, the aim should be for a moisture content of 3% below the desired value in order to be sure that the pulp meets specifications. If operations can be improved to the point where the limits of fluctuation in moisture content are no greater than 1%, then the goal can be set 1% below the guaranteed maximum, reducing the fuel cost and increasing the amount of production.

However, control of the drying operation is particularly difficult in the rotating direct-fired drum, because of the long process lag which may amount to 45 minutes. Some of the particles move much more rapidly and some much more slowly, but the average retention time is so great that direct control of the moisture content of the product being discharged from the drum has usually been considered too slow to be used for direct control of the fuel rate to the drier. The pulp drier operators have relied principally on "feel" for primary control. A surprising amount of skill can be developed in detecting by "feel" small changes in the dried pulp moisture content. Certain radiant-heat drying devices provide moisture tests in 10 to 15 minutes and are used in many factories.

Most of the more recent drier installations use temperature controllers to regulate the amount of fuel. The process lag in the gas phase is relatively short, being only a few seconds. As the load on the drier drum increases, the temperature of the flue gases leaving the drum decreases and the controllers, by increasing the quantity of fuel, bring the temperature back to the control point. However, this system of automatic control is defective inasmuch as the exit flue gas temperature is not directly related to the pulp moisture content. A temperature controller that alters its control point in direct relation to a change in load is now available (6a).

Some drier drums are also equipped with an additional controller bulb, placed in the inlet to the drum, to prevent the furnace temperature from exceeding some predetermined maximum value.

Dried Pulp Storage

Warehousing Dried Beet Pulp. In some installations, the dried beet pulp is bagged directly as produced, usually in 50 and 100 lb burlap or paper bags, for direct sale or warehouse storage. The bags are then put in warehouse storage. This practice is rapidly diminishing in North America at the present time in favor of bulk storage.

A considerable number of factories have bulk storage in which the dried pulp from the production line is stored until packaged or sold in bulk. This method has several advantages. First, delays in the packaging station do not delay production of the dried pulp. Second, in bulk storage the moisture content of the dried pulp from different operating conditions is readily equalized. Third, it is possible to adjust the temperature and humidity of the circulating air to alter the moisture content of the dried pulp to a desired value. All of these advantages in bulk storage systems tend to produce a more uniform quality of dried beet pulp.

Other factories are pelletizing dried pulp from the drier drums and storing the pellets in bulk warehouses. Pulp pellets range in size from ¼" to ¾" diameter

depending on the ultimate use of the dried pulp in the market area surrounding the affected factories (See Figure 19-5 and 19-6). The advantage of storing pelletized product is that the density is increased approximately three-fold over bulk dried beet pulp, thereby reducing warehouse volume requirements for storage.

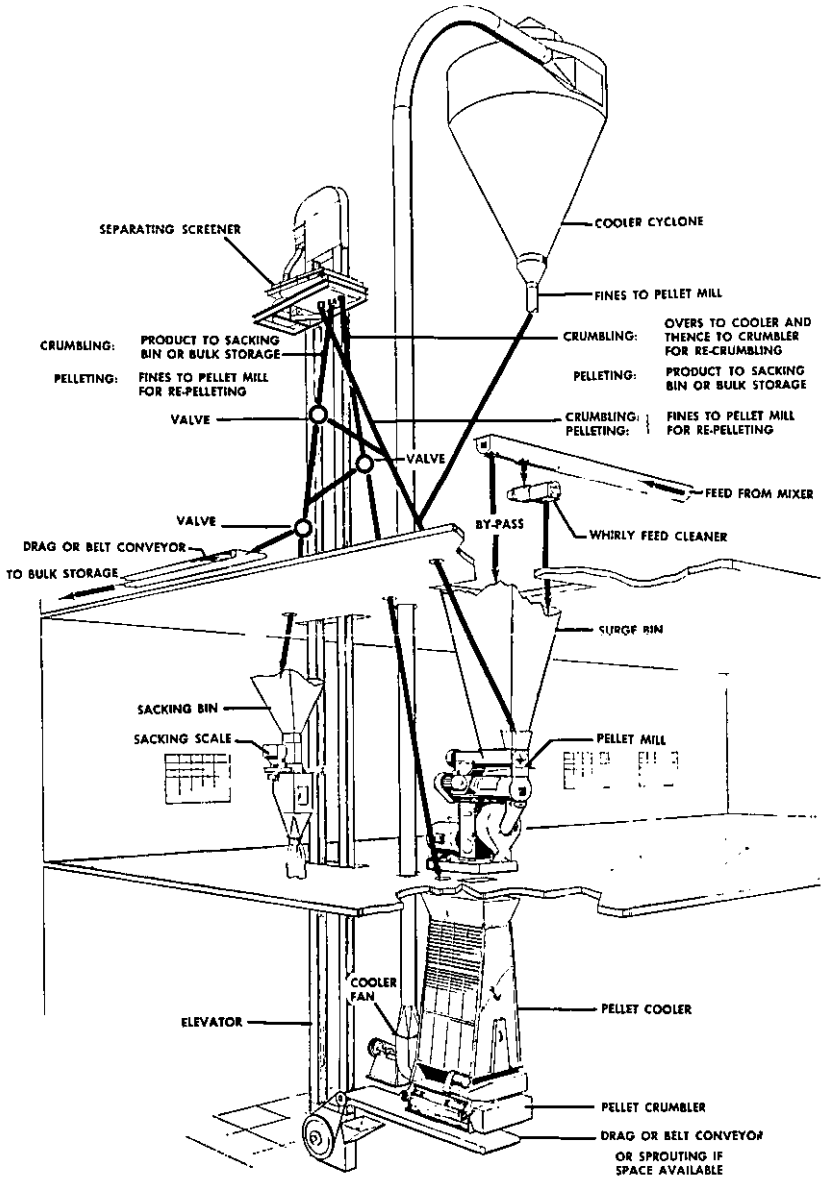

Figure 19-5. Pelleting system flow diagram. *(Courtesy, California Pellet Mill Co., San Francisco.)*

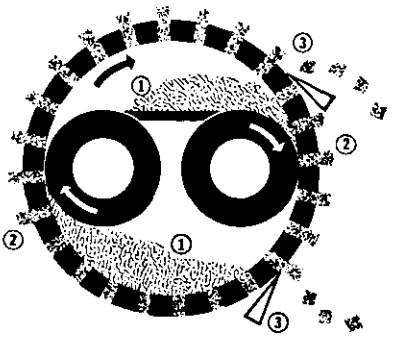

Figure 19-6. Typical die and roller assembly used for producing hard pellets. 1) Loose dried pulp is fed into pelleting chamber. 2) Rotation of die and roller pressure forces material through die, compressing it into pellets. 3) Adjustable knives cut pellets to desired length. *(Courtesy, California Pellet Mill Co., San Francisco)*

Spontaneous Heating of Dried Pulps.* In many agricultural products like hay, grains, and beet pulp, a spontaneous temperature increase is often observed during storage. Such spontaneous heating may be caused by the metabolism of microorganisms or by chemical reactions in the material (2,4,5,6). Microorganisms do not proliferate at the relative humidity and moisture levels encountered in normal pulp storage. In cold climates, however, moisture migration within the stored mass can raise the moisture levels in localized areas, and in these areas rapid microbial growth can occur with eventually resulting hot spots. It is indicated that spontaneous heating of dried beet pulp is mainly due to oxidation reactions, and that these reactions occur at all practical storage temperatures.

At normal storage temperatures below 50 °C, the oxidation reactions in beet pulp are not particularly harmful to the quality of the product, but elevated temperatures lead to degradation of the pulp. When temperatures rise unchecked, ignitition of the pulp may result.

The principal factors which affect spontaneous heating rates in dried pulp were investigated extensively by the research department of Spreckels Sugar Company (1). Pulp samples with and without a variety of additives were produced in a small pulp drier under precisely controlled conditions and their spontaneous heating rates were determined at a number of moisture levels and storage temperatures in a specially designed adiabatic calorimeter. Calorimetric data collected over a period of 12 years show that the spontaneous heating increases with moisture content, level of additives, and storage temperature. It was further shown that the type of additive has a large bearing on the heating rates and that significant variations in heating rates occur from year to year.

Data taken with the adiabatic calorimeters during 1967 with pulp from beets grown in the Sacramento Valley of California may serve to illustrate the effects of production and storage variables on spontaneously generated heat, and are shown in Table 19-5.

The heating data obtained by the use of calorimeters permit the determination of conditions for safe storage of pulp. Safe storage is assured when the rate of heat removal from the storage pile equals or exceeds the rate of heat generation by the spontaneous process within the pulp pile. In large storage piles, natural heat losses through radiation, convection, conduction and evaporation of moisture may not suffice to prevent deleterious temperature increases in the interior of the pile, unless the

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Table 19-5 Heating Rates in Btu/(ton)(day)

Storage Temp. °C.		40	50	60	70
Additives % on Beets	Moisture %				
None	8.6	86	140	338	1260
2% molasses	8.8	191	299	814	2560
2% CSF	8.7	364	850	1955	4583
2% CSF	12.1	634	1512	4241	6336

pulp is put into storage at reduced temperatures, and the storage time is kept short. When the natural heat losses are inadequate to maintain safe storage temperatures, forced cooling is necessary. In most beet growing areas, the relative humidity and temperature of the air permit cooling of the storage pile by forced ventilation. It is advantageous to draw the ventilating air from the surface to the bottom of the pile because this permits determinations of the enthalpies of the ambient and exhaust air. When the air flow and enthalpies are known, the heat removed from the pile can be calculated. Conversely, when the weight of the storage pile and the heating rate of the pulp are known, cooling requirements can be calculated.

Practical Methods of Combating Spontaneous Heating. In summarizing the indications in the preceding section, the pulp introduced into bulk storage should be cooled to as low a value as is practical. Some companies use rotary drum coolers, while others simultaneously screen the resultant product while cooling. It is important to avoid excessively high moisture content of the dried pulp, say, over 12%, as this leads to excessive heat development during storage. The type of additive and the quantity added also affect the rate at which heat is generated. Exhaust fans can be installed in warehouses to pull ambient air through the pulp storage pile and out through the exhaust fan to dissipate the heat as it is generated. It is also important to use common sense in spreading the pulp over the entire periphery of the pile to ensure that no concentrations of high moisture pulp are accumulated and to offer the maximum opportunity for the pulp to reach equilibrium with the ambient air prior to being buried by additional product.

It has been found that pelletized pulp is capable of dissipating the heat in storage to a greater degree than bulk pulp, mainly because of the voids between particles which allow for greater movement of air and cooling.

Monitoring of the pulp during storage is usually accomplished by the olfactory senses, since some of the products of decomposition are acetic and other volatile, pungent organic acids. Once detected, the most satisfactory method of combating spontaneous heating is to rapidly isolate and remove the affected area from the storage pile to allow a rapid dissipation of the heat generated. Care must be exercised in this operation to prevent the accidental ignition of the volatile gases which could result in a major fire. As mentioned earlier, the presence of high-moisture pulp adversely affects the heating rate of the resultant product, which means that all care must be exercised to avoid the inclusion of small beet tails or slabs from the drying operation. Some factories practice screening of the pulp to remove these large-sized particles prior to storage.

Bulk Density of Dried Beet Pulp

The bulk density of plain dried beet pulp is approximately 8 to 16 lbs/cu ft, and that of molasses-dried beet pulp is about 10 to 20 lbs/cu ft, depending on the molasses addition, the character of the beets and the cossette size. For 33% molasses

solids on product solids, the weight per cu ft is about 16 lbs, corresponding to an apparent density of 0.25. With this low apparent density, very large and cumbersome bags are required for sacking the product. Pellets have a bulk density of 30 to 40 lbs/cu ft.

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Chapter XX
THE BOILER*

Introduction. Extraction of beet sugar and the several by-products from sugarbeets requires large quantities of heat, since the starting material contains about 75% water, most of which must be removed during the processing. In the beet-sugar industry water and steam are singularly suited as media to absorb heat from the combustion of fuel in a boiler and to transmit the heat from the boiler to the process, where water is removed from process materials by evaporation.

Steam is singularly suitable because: 1) boiler steam and condensate systems can easily be integrated with evaporator vapor and condensate systems; 2) power to operate factory machines can be obtained at low cost by expanding boiler steam through prime movers, usually steam turbines, on its way to the evaporators; 3) modern boilers and associated equipment have a relatively high efficiency; 4) steam is in common use, is economically transported in pipe lines, and is easily controlled.

Boilers. A boiler is a pressure vessel wherein water is evaporated to steam by the application of heat. It contains a furnace, which is an enclosed space for the combustion of fuel.

Boilers used in the beet-sugar industry almost without exception: 1) are water-tube boilers with steam and water on the inside of the tubes and with heat applied to the outside of the tubes; 2) have natural circulation of water and steam resulting from differences in density (temperature); and 3) operate at pressures below 700 psig. The boiler industry considers boilers of about 600 psig or less to be "low pressure" boilers.

An ideal beet-sugar factory boiler is: 1) designed and constructed for safe operation; 2) of high quality design, materials, and workmanship; 3) competitive in price; 4) one that has adequate facilities to separate steam from water and thus deliver clean steam; 5) of a high thermal efficiency; 6) easily and economically maintained, cleaned, and laid by; 7) responsive to sudden changes in loads; and 8) suitable for economic instrumentation and control.

Some years ago an attempt was made in the engineering profession to initiate the term *steam generator* for a modern boiler. This attempt has failed, and the term *boiler* is generally accepted.

A boiler can be located indoors or outdoors in almost any climate. More boilers are being located outdoors, particularly those that are substantially automated, but in colder climates usually the front face of the boiler is located indoors.

Until about 30 years ago most new beet-sugar factory boilers were assembled on the job, in the field. With such *field-assembled boilers,* the larger the capacity the lower the installed capital cost per unit of steam generated, and this resulted in fewer boilers being installed. In fact several beet-sugar factories have been equipped with but one boiler. In recent years the trend with field-erected boilers is to have portions of them shop assembled.

Starting about 30 years ago use of *shop-assembled boilers* or *packaged boilers,* where almost complete assembly of the boiler is done by the manufacturer in his

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***by F. H. Ballou, Jr.**

shop, began to gain favor in the beet-sugar industry because of their relatively low initial cost. Since, at that time, shop-assembled boilers could only be built and shipped in relatively small sizes, a beet-sugar factory equipped with these boilers was likely to have up to six or seven units. Boiler manufacturers continue to design and build ever larger shop-assembled boilers. In 1969 a packaged boiler was built with a capacity of 325 000 lb/hr of steam (1). Now, with more frequency, beet-sugar managements are choosing large shop-assembled boilers.

To select the boiler steam pressure the design engineer will first determine the exhaust steam pressure to be used in the evaporator first effect. Next he will determine the amount of power to be generated by the prime movers. Then he will select a boiler steam pressure which will allow generation of the required power, using about 85% of process steam requirements. The remaining 15% of the boiler steam will then be "made up" by throttling steam from boiler pressure into exhaust steam.

In recent years, there is a tendency toward boiler furnaces with *pressurized firing* and away from *balanced draft*. With balanced draft a slightly negative pressure, or draft, is maintained in the furnace by the action of a stack alone, or by an induced draft fan and a stack. With pressurized firing there is a positive pressure in the furnace, and since there is no induced draft fan, the total fluid friction in all of the air and gas passages through the entire boiler is overcome by the forced-draft fan. Thus the positive pressure in the furnace equals the sum of all friction losses between the furnace and the stack, less any draft created by the stack.

In a balanced draft furnace any leakage results in air leaking in, whereas in a pressurized furnace any leakage is outward. With balanced draft the furnace needs to be only reasonably tight, as a small amount of leakage is insignificant. With pressurized firing, special precautions must be taken to keep leakage to an absolute minimum. Frequently the furnace is sealed with a welded steel or aluminum casing, and special attention is given to sealing soot blowers, damper shafts, and fuel-oil guns, where they penetrate the casing. Usually compressed air is piped to observation ports to keep the sight glass cool and to act as a jet pump into the furnace when the observation port must be opened. Pressurized firing reduces the initial and maintenance costs of the boiler and simplifies design, operation and control.

Boiler Fans. Probably all modern boilers in North American beet-sugar factories use one or more draft fans. Forced draft fans supply air for combustion. Induced draft fans remove the gaseous products of combustion and discharge to a stack. With certain coal stokers a separate fan provides overfire air. With pulverized coal several additional fans may be incorporated in the pulverizing system. Fans are a vital part of any modern boiler, and their selection and application is very important but outside the scope of this book.

Fuels. *Coal* has always been a major fuel for boilers. The amount of coal used in the United States, as a percentage of all fuels used, gradually declined from around 1900 until recently. A few years ago a cartel was formed by a number of the large oil producing countries. This cartel has been the major factor in limiting the supply and effecting large price increases for petroleum products and natural gas. Actions by the cartel are resulting in significant increases in U. S. coal production and use. Deposits of coal are widely distributed over the world. In North America major deposits are known in 25 states of the United States, in five provinces of Canada, and in at least one state of Mexico.

Coal is classified into four broad rankings: anthracite, bituminous, subbituminous, and lignite. These terms cannot be completely defined. Anthracite is

commonly considered to be the hardest coal, and lignite the softest. By definition, anthracite must contain 86% or more of fixed carbon on a dry basis; lignite must have a moist Btu content of less than 8 300 per lb, subbituminous 8 300 to 11 000 moist Btu per lb and bituminous more than 11 000 moist Btu per lb and less than 86% dry fixed carbon. The heat content of coal can be up to 15 000 Btu per lb or more.

The suitability of a particular coal for a particular boiler can best be determined by an actual trial.

Coal is commonly fed to the furnace by a *stoker* or a *pulverized-coal firing system*. With the inception of the beet-sugar industry in North America, coal-fired boilers were generally equipped with *chain-grate*, or *traveling-grate, stokers*. Coal is fed to a hopper located on the front of boiler with the front end of the chain-grate forming the bottom of the hopper. Coal feeds from the hopper onto the chain-grate as it moves into the furnace, after passing under an adjustable gate, which regulates the thickness of the fuel bed. Chain speed and depth of coal on the chain can be varied to meet changes in demand on the boilers. At the back of the furnace the grate turns around the rear sprockets, thereby discharging the ashes by gravity and returning the endless chain-grate to the front of the furnace.

About 1940 the spreader, or overfeed, stoker was developed and became popular for burning bituminous and subbituminous coals. By 1950, probably 75% of all stokers in the United States were of the spreader type (2). Coal hoppers on the front of the boiler discharge to a feeder which regulates the rate of fuel flow to the furnace over a wide range. The feeder can be a traveling chain, a specially designed revolving drum, or other device. Following the feeder is a distributor which "throws" the coal into the furnace and onto the grate. A rotating shaft equipped with blades usually serves as the distributor and is designed to give, alone or in combination with other distributors, a uniform distribution of fuel onto the grate, which may be of the dumping or continuous discharge type. In boilers with spreader stokers and traveling grates, the ash must be discharged under the spreaders. If the grate discharges opposite to the spreaders, large pieces of coal that have longer trajectories may leave the grate only partially burned. Spreader stokers combine some of the principles of pulverized-coal and chain-grate stoker firing, as fine coal particles are burned in suspension and larger particles are burned on a grate. Almost every type of coal can be successfully fired with a spreader stoker. Interestingly, they can also be used with such fuels as sugar-cane bagasse, sawdust, and coffee grounds.

Practically every type of coal mined is being successfully burned in pulverized form. *Pulverized coal firing* of boilers was first successful in the 1920's and since 1930 has been the most common method of burning coal in the United States. Pulverization of coal can be accomplished in any one of several types of mills: ball, tube, ball-race or ring-roll. The latter two types are the most common, and the last type is commonly used in the beet-sugar industry to pulverize quicklime in the Steffen process. Pulverized coal is carried in an air stream out of the pulverizer to burner fuel-nozzles, penetrating the boiler furnace wall.

Ash is defined as incombustible solid matter in the fuel. All commercial fuels have some ash. Coal can have a great deal, usually within the range of 4 to 20%.

The ash from coal, known as fly-ash or particulate matter, is carried by the combustion gases and if not removed will be emitted from the stack as an air pollutant. Sulfur in coal results in emission of sulfur dioxide. In many areas coal cannot now be burned because of emission of particulate matter or sulfur dioxide or both,

unless means are provided for their removal from the combustion gases prior to discharge to the atmosphere (for detailed treatment see Chapter XXI).

In some boilers, the ash collected in the first and second passes may be reinjected with the overfire air to improve efficiency by burning carbon remaining in this ash, as opposed to allowing it to pass on to the ash disposal system. This practice must be carefully studied since the recycling of the ash may result in much greater abrasion of economizer or air heater and the induced draft fan, plus greater stack emission. Higher cost of maintenance and repair may offset the improved combustion efficiency.

Residual fuel oil, commonly called grade No. 6, is the type and quality normally burned in a beet-sugar factory boiler. In certain areas, particularly where transportation cost from an oil refinery is low, fuel oil can be the lowest priced fuel. Typical properties of a residual fuel oil are shown in Table 20-1.

Residual fuel oil must be heated prior to atomizing and burning. The oil burner, or gun, can be designed to atomize the oil mechanically, or to do so with the use of steam. Steam atomization is the more popular, even though it reduces boiler efficiency, because it produces good atomization.

The higher cost for lighter and less viscous oils may be justified in cold climates where the oil is used as standby for gas by eliminating or reducing the cost of heating the more viscous oils to the required temperature for proper combustion.

Residual fuel oil contains most of the solid impurities which were present in the crude oil. Although ash content is low, a trace to 0.2%, if it contains alkaline salts, the ash can fuse at a low temperature and stick to boiler and superheater tubes, flux with firebrick, and cause corrosion of metal parts. When sulfur is also present these effects are accentuated. Vanadium is another ash constituent that can result in severe coating of boiler tubes with V_2O_5, which has a melting point of only 1275 °F.

Table 20-1

Specific gravity, 60/60°F	0.986
Lbs per U. S. gallon, 60°F	8.21
Viscosity, Saybolt Furol, 122°F	170
Temperature for atomizing, °F	200
Temperature for pumping, °F	100
Btu per gallon	150 000
Ash, %	0.08
Sulfur, %	0.5-3.2

Sulfur in fuel oil, as with coal, can also result in unacceptable levels of sulfur dioxide in the combustion gases. A number of oil refineries have installed equipment to allow production of low-sulfur fuel oil. This too has increased the price.

Natural gas is the easiest and cleanest common fuel to burn and control. However, when not at the proper combustion control point, natural gas is far more subject to explosion than coal or oil. Usually natural gas has between 950 and 1150 Btu per standard cu ft. Methane, ethane, and propane are the primary hydrocarbon constituents, and nitrogen and carbon dioxide can be important detrimental constituents. The specific gravity varies from 0.56 to 0.79.

Natural gas is sold in several different ways: by volume, as defined in the purchase contract, per therm of 100 000 Btu, or in some arbitrary combination of volume and heat units. It is an exacting task to measure accurately the Btu content

of natural gas flowing past a particular point in a pipe line as there must be known the Btu content, the flow in cu ft, and the flowing temperature and pressure of the gas. Many gas utility companies in the United States deliberately adjust their measuring and billing system to give the customer more Btu's than he pays for; thus preventing accurate measurement of boiler efficiency based on the utility meters and invoices.

Ash in natural gas shows up as a light, loose deposit on the tubes.

Contamination of natural gas by hydrocarbon liquids, ranging from heavy oil to gasoline, and by salt water does occur on rare occasions. These can be detected by observing changes in the flame, such as small moving yellow spots.

Combustion. Combustion is the rapid chemical reaction and combination of oxygen with the combustible elements of a fuel. Coal, oil, and gas, which are fossil fuels, have only three elemental constituents, carbon, hydrogen, and sulfur, which will react with oxygen in air to produce heat. Sulfur is usually a very small percentage of the fuel and thus contributes a negligible amount of heat. This leaves carbon and hydrogen as the main elemental fuels.

Carbon can combine with oxygen to form carbon dioxide directly:

$$C + O_2 = CO_2 \quad (1)$$

or it can combine in two steps with carbon monoxide as an intermediate:

$$2C + O_2 = 2CO, \text{ and} \quad (2)$$

$$2CO + O_2 = 2CO_2. \quad (3)$$

Generally all three of these reactions take place in a boiler furnace. With satisfactory combustion there are no more than trace amounts of carbon, hydrocarbons, and carbon monoxide in the products of combustion discharged to the atmosphere.

Hydrogen combines with oxygen to form water vapor:

$$2H_2 + O_2 = 2H_2O. \quad (4)$$

Sulfur combines with oxygen to form either sulfur dioxide or sulfur trioxide:

$$S + O_2 = SO_2 \quad (5)$$

$$2S + 3O_2 = 2SO_3 \quad (6)$$

Much of the carbon and hydrogen exists in the three fuels discussed herein in chemical intercombination as hydrocarbons, and can combine directly with oxygen: for example,

methane $CH_4 + 2O_2 = CO_2 + 2H_2O$ (7)

ethane $2C_2H_6 + 7O_2 = 4CO_2 + 6H_2O$ (8)

As shown above, hydrogen in a fuel burns to water, which is emitted by the boiler stack in the form of gaseous water vapor. As pointed out in Chapter XI, water vapor, or steam, gives up a good deal of heat when it condenses. Thus the heat obtained from the water-evolving reactions will be one value if the water vapor is condensed and cooled to the initial temperature of the fuel, and another if it is not. The first value is called the *high heat value*, and the second the *low heat value*. Fuels are purchased in the United States on the basis of the high heat value, even though combustion gases emitted by boilers contain water vapor. Under normal conditions it is not economical to recover the heat in the water vapor.

Heats of combustion for some of the reactions given above are listed in Table 20-2.

Table 20-2

Substance	Btu per lb	
	High	Low
Carbon, to CO_2	14 100	14 100
Carbon monoxide	4 340	4 340
Hydrogen	61 100	51 600
Sulfur, to SO_2	3 980	3 980
Methane	23 900	21 500
Ethane	22 300	20 400

Nitrogen and other inert gases, 76.85% by weight of air, pass through the furnace and absorb heat which is lost to the atmosphere. Oxygen is 23.15% by weight of air. Since perfect intermingling of oxygen molecules from air and fuel particles cannot be achieved in the boiler furnace, a 10 to 20% surplus of air must be supplied to the furnace to obtain the most efficient combustion.

Boiler Accessories. It is unusual to see a modern boiler in a beet-sugar factory without a *superheater*, which consists of tubes located in the boiler-furnace structure. Steam from the boiler at near saturation is piped to the superheater tubes, wherein its temperature is raised above the saturation temperature, or "superheated." Superheat of 100 to 130 °F is common in the beet-sugar industry. With superheat each pound of steam then carries more energy, which results in dry steam through the steam turbines. Far less erosion of the turbine blades takes place with dry steam than with wet steam.

Air preheaters are a commonly used boiler accessory to improve boiler heat recovery—boiler efficiency. The air heater reclaims heat from the combustion gases leaving the boiler and adds it to the air used for combustion. Two types are in common use, the tubular and the regenerative.

Economizers are also used to improve boiler efficiency. An economizer also removes heat from combustion gases leaving the boiler but adds it to the boiler feedwater. Tubular heat exchangers are used with plain or finned tubes. In a beet-sugar factory it is unusual to use an air preheater or an economizer.

Tall *stacks* are no longer installed, and their place is being taken by fans, to create the flow of air to, and combustion gases through, the boiler. When new boilers are placed in an older plant with a tall stack the stack can be retained as it will reduce the fan power requirements. In new plants short stacks are used.

Soot blowers are used on coal and oil fired boilers, on air preheaters and on economizers to remove ash from the heating surfaces. Soot blowers are lances, and use compressed air or steam as the cleaning medium. They come in a variety of shapes, sizes, and designs. In very high temperature areas they must be retractable to prevent damage by heat. Stationary installations can be made in lower temperature areas. They are not successful in removing certain types of ash which can be deposited on the tubes by residual fuel oil.

Many *boiler valves* are used in critical services. Selection of these should be carefully made for reliability of operation and low maintenance.

Centralized *instrumentation control* of the steam generation area of the beet-sugar factory in North America has been practiced for many years. It has resulted in improved boiler efficiency, economy of labor, one-man operation by a skilled technician, higher average operating rates, better matching of steam production to factory

process needs with less blowoff of steam to atmosphere, less boiler outage, and safer operation.

More important functions for instrumentation control are: control of the water level in the boiler, combustion control, flame monitoring, and control of boiler steam pressure.

Other functions which are frequently controlled or monitored are: feedwater pressure, frequently this is part of the boiler water level control system; temperatures of steam, feedwater, materials to and from air preheaters and economizers, fuel, oil, natural gas, and stack gas; pressures of fuel oil, natural gas, compressed air; draft or pressure in furnace, air heater, economizer, stack and windbox; flows of steam, air, and water; levels in supply tanks and bins of water, coal and oil; deaerators; pH of feedwater, boiler water, and treated water; caloric content of fuel; sugar content of various waters; conductivity or concentration of salts in various waters; rate of carryover from the boiler; and the alkalinity and other chemical characteristics of boiler water and boiler feedwater.

Careful study should be given to the location of the instrument panel and to the arrangement of instruments and monitors thereon to obtain the fullest measure of performance from the operator and the installation.

Fuel is ususally one of the three or four most expensive factors in the manufacture of a pound of beet sugar, and thus good combustion control is of importance. The purpose of combustion control is to regulate the amounts of fuel and air to the furnace to achieve optimum combustion, and to do so at such a rate as to maintain the desired output of steam at the desired pressure, within a small tolerance. This can be achieved by controlling: 1) oxygen in the combustion gases, or, 2) the ratio of flow of fuel to flow of air. Other systems control the ratio of flow of steam to flow of air or the CO_2 content of the combustion gases.

Measurement of unburned hydrocarbons in the combustion gases also can be helpful.

Monitoring of operation or nonoperation of important pumps, fans, and compressors on the main boiler control panel with visual and audible alarms is important. These can be tremendously helpful tools for the boiler operator.

Boiler Feedwater and Boiler Water. *Boiler feedwater* may be selected from two basic sources: 1) condensate from the first effect evaporator with makeup condensate from other evaporator effects, vacuum pans, or heat exchangers, or 2) raw water. A third selection can be a combination of the two basic sources, such as first effect condensate with raw water makeup.

The first selection has the advantage of economical recovery of the heat in the condensate and low solids content water. With the second, some device is required to recover the heat in condensate such as heat exchangers or a flashing system or both. An advantage of the second system is that contamination of the boiler water with sugar is a remote possibility, but unless the raw water is very pure it must be treated as an expense for capital cost of equipment, plus operating cost for chemicals, heat for deaeration (4) and labor. Obviously such treatment must be tailored to fit each particular raw water. The third system obtains the advantages of each selection in part.

Water in the boiler should ideally maintain all internal boiler surfaces clean and free of scale or other deposits, be noncorrosive to all internal boiler surfaces, and have no oxygen content; and be nonfoaming so as to minimize carryover of

boiler water from the boiler to the superheater, steam turbines, first-effect evaporator, and other heat exchangers. With suitable feedwater these ideals can be accomplished by chemical treatment of the boiler water and proper control of blowdown. Continuous blowdown gives far better control than intermittent blowdown and is now in common use. Heat is frequently recovered from the blowdown stream by flashing or by a tubular heat exchanger.

A number of chemical treatment systems for boiler water are available. Frequently such reagents as caustic soda, sodium phosphate, potassium phosphate, hydrazine, sodium sulfite, lignins, and defoamers are used. It is beyond the scope of this book to discuss specific systems of boiler water treatment, but information may be found in the literature (3,5,6).

Separation of steam from the boiler water to give a low carryover is very important. Even a small amount of continuous carryover can build a deposit inside the superheater tubes. Greater carryover or carryover of silica can result in deposits of material on the turbine blades with resulting problems. Treatment to maintain a nonfoaming boiler water, and proper drum internals to physically aid the separation, are necessary.

Contamination of boiler feedwater and boiler water by a sugar-bearing material creates a problem peculiar to the sugar industry. The first procedure is to prevent the occurrence, but even with the best preventive measures, it does happen on occasion. The second procedure is to monitor the waters going to boiler feed for sugar. Instruments which will detect low sugar concentrations are available. With such an instrument and timely action, sugar can be kept out of the boiler. If sugar does reach the boiler it will break down into acids, cause foaming and massive carryover, corrosion, and deposits inside the boiler, and damage to the boiler and prime movers can occur. Acidity should be counteracted by generous doses of caustic soda, but to prevent damage to the boiler it should be shut down.

Many beet-sugar factories use an outside consultant to specify treatment of boiler feedwater and boiler water. A number of reliable firms are available.

Management and Operation. In choosing a site for a new factory the local cost of fuel should be taken into account as one of the factors. Once a plant is built plant management must closely control steam production and use, to obtain minimum operating costs. Boiler efficiency is one of the important figures to be measured and controlled, because only a small deviation can be of significant value over a period of time. Measuring devices should be installed so that the boiler efficiency can be obtained at least with daily frequency, on each boiler, to allow prompt corrective action.

Integration and coordination of steam generation and processing operations is necessary. Specifically, uniformity and economy of steam generation and steam usage in the process will reduce costs. All those concerned should be aware of the limitations of the process and the steam generation operations.

Routine operation of a boiler is far easier than operation in an emergency. Improper boiler operation during an emergency can result in costly damage to equipment and hazard to personnel. The boiler operator should know exactly how to recognize a problem in the early stages and what steps to take. Procedures can be set up for such problems as lack of feedwater, a tube rupture, carryover, the dropping of a generator off the line, and flame failure (8).

Boiler efficiency is the ratio of heat output to the high-heat value of fuel burned, expressed in percent.

$$\text{Overall Boiler Efficiency} = \frac{W_s (h_2 - h_1) + W_{BD} (h_3 - h_1)}{W_F Q_H} \times 100 \quad (9)$$

where W_s is the pounds of steam per unit time delivered by the boiler, h_2 is the heat content of the delivered steam in Btu per lb, h_1 is the heat content of the feedwater in Btu per lb, W_{BD} is the pounds of blowdown per unit time, h_3 is the heat content of the blowdown in Btu per lb, W_F is the quantity of fuel burned per unit time, and Q_H is the heat value in Btu per unit quantity of fuel. All authorities do not give credit for incremental heat in blowdown (7,9) even though it has been transmitted through the boiler heating surface. In many plants a large portion of the heat in the blowdown is recovered before it is discarded. Certainly in this case the boiler should receive credit for the heat.

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The Electrical Power System*

Processing of sugarbeets to refined sugar requires about 1.2 kW of electrical energy per ton of beets sliced per day. This electrical energy demand is affected by factors such as: the type of beet receiving and cleaning facilities, whether or not a Steffen house is provided, the lime production method, the drying and pelletizing of beet pulp, and the number of steam drive units, compared to electrical motor drives, particularly in the higher horsepower units.

The electrical energy consumption per unit of product output has continually increased over the years, and this trend appears unlikely to change in the foreseeable future. Among the primary reasons for increased demand are the extensive mechanization of the process, higher lighting illumination levels, and new practices; i.e., waste-water treatment, requiring additional electrical power for circulation pumps and aerators.

The electrical power system may be divided into two parts, generation and distribution. Power generation involves the conversion of heat and mechanical energy into electrical energy. Power distribution involves the transmitting of the electrical power from the point of generation to the point of utilization.

*by Aurand L. Munn, Jr. and Arnold J. Daglia

Power Generation

The sugar industry, because of its need for relatively large quantities of low-pressure process steam, usually finds it economical to generate its own electric power. The power plant normally uses a noncondensing steam turbine generator which will exhaust steam at the pressure required by the process This power can be generated for about half the fuel required in a condensing steam-turbine generator plant used for power generation only.

Regardless of the source of electrical power, steam-boiler facilities must be provided to supply the process steam requirements. With in-plant generation, the fuel chargeable to power is the additional fuel needed over that required for operation with purchased power. Figures furnished by the General Electric Company show that the cost of fuel chargeable to electric power generation using a noncondensing steam turbine is 0.425 mils per kW-hr for each 10 cents of fuel cost per million Btu. Thus, using $2.80 fuel, and with a cost of purchased power of 35 mils per kW-hr, with an assumed load of 4000 kW, the factory could pay for the entire installation cost of a noncondensing steam-turbine generating set in approximately three years, without considering taxes.

Figure 20-1 shows a typical noncondensing steam-turbine generator. Normal practice is to install one large unit rather than two half-capacity units because of the lower installed costs.

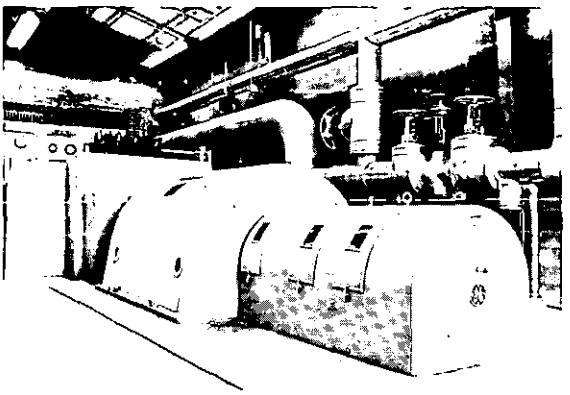


Figure 20-1. Turbine rated 5000 kW capability with 400 psig throttle, 605 °F and 45 psig back pressure, 3600 rpm, synchronous generator rated 4000 kW at 0.80 power factor, 4160 V, wye-connected, and provided with direct-connected d-c shunt-wound exciter, 25 kW, 125V, 200 A., Manteca, California.

The inlet steam to the turbine will normally be between 140 psig, 365 °F, to 600 psig, 725 °F, with the back pressure or exhaust varying between 17 and 55 psig. The most modern power plants are operating in the range of 400 to 600 psig with superheat, and turbine exhaust at about 45 psig. The trend to the higher steam pressures is necessary because they affect the efficiency and amount of power that can be so generated.

As is shown by Table 20-3, if the process requires 100 000 lbs per hour of 45 psig steam and the inlet steam conditions are 600 psig, 720 °F, the power generated is 4750 kW. However, with inlet steam of 200 psig, 388 °F, the power generated is only about 1800 kW.

Table 20-3 Steam Rate. Pounds of steam per kW-hr for a 5000 kW noncondensing steam turbine at various loads with different inlet steam conditions with the exhaust constant at 45 psig

kW Load	200 psig, 388°F	400 psig, 605°F	600 psig, 720°F
5000	44.0	23.00	21.01
4000	44.2	23.29	21.12
3000	47.8	24.60	22.35
2000	55.0	27.25	24.70

The reliability of the main steam supply system and the need for process steam has made it normal practice to power the large-horsepower individual loads with mechanically-driven noncondensing steam turbines. Typical of such units are the carbon-dioxide and Steffen-refrigeration compressors. Selecting several individual loads, usually over 300 hp, to be turbine-driven allows the steam designer further flexibility in balancing out the steam requirements in the whole plant. In addition, a pressure-reducing station will be provided between the main steam supply and the process steam header for flexibility in meeting steam demands under varying flow conditions, electrical load, and also to hold the process level constant.

Purchased Power

Almost all sugar factories purchase some outside electrical power, which is needed for standby usage when the factory is not in operation and the power plant is shut down. During such nonoperating periods, power is required for factory maintenance, liquid-sugar production, bulk-sugar handling, packaging operations, lighting, and office-machine operation. In the event of power-plant disturbances and loss of factory-generated power, the standby power provides for the electrical needs of critical loads, such as emergency lighting, and boiler plant and water systems. It is not usually economical to size the utility-company purchased-power standby source to meet the factory's total electrical demand, and this is generally sized for about 20% of the total factory demand.

Electrical Power Distribution

Modern beet-sugar factories use the load-center philosophy of power distribution. In load-center distribution systems electrical power is generated or purchased at a high primary voltage, and is distributed at this primary voltage in feeders to areas of high-load density. At these locations, load-center unit substations are used to step the voltage down to that required by the equipment and devices served. See Figures 20-2 and 20-3. Distribution to the load at utilization voltage is made through feeders from the load-center unit substation, to motor control centers and then to the motors. A motor control center is a grouping of motor control devices for a group of motors in a given load area.

A one-line diagram of a typical electrical distribution system for a modern beet-sugar factory is shown in Figure 20-7. Power is distributed from the main switch gear at a primary voltage of 2300 V or 4160 V, 3-phase, 60 Hz. Load-center unit substations in the range of 225 kVA to 1500 kVA step the voltage down to 480 V for direct utilization by motors of 250 hp or less. Larger motors may be served directly at 4160 V with high-voltage motor starters. Induction motors rated 200 hp and below, and other loads such as control, lighting, welding, and heaters, are served from the 480 V system, or at their special voltages, through individual, step-down transformers.

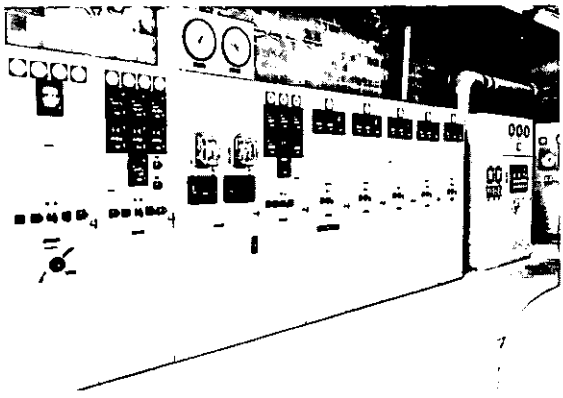


Figure 20-2. Metal-clad switch gear rated 4160 V, 250 mVA, 3-phase, 3-wire, and left to right consisting of: generator and exciter control, utility incoming line and metering unit, and five radial feeder sections.

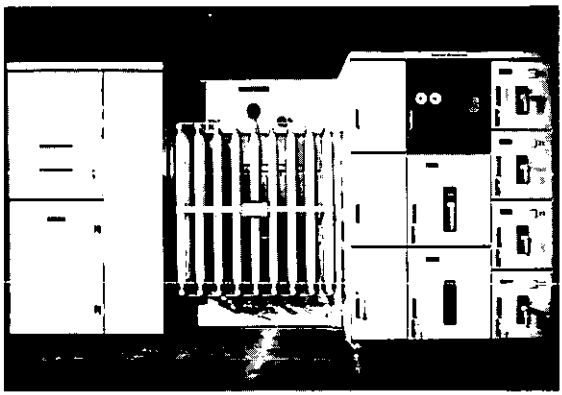


Figure 20-3. Load-center unit substation, rated 1000 kVA, 4160 to 480/277 V, consisting of primary switch, transformer, and low-voltage switch gear and metering.

Some pertinent facts related to the choice and rating of circuit elements and arrangements shown in Figure 20-2 and the influence of the particular requirements on the beet-sugar industry are:

1. Load-center distribution systems have four major advantages over systems where large blocks of power are generated and distributed at utilization voltages, namely; a) lower installed cost of equipment and cable; b) short-circuit or fault duty on the low-voltage systems is maintained within reasonable and economical values of equipment ratings; c) expansion of the system for increased loads is handled easily with new high-voltage feeder and load-center unit substations without increasing the burden on existing equipment; d) voltage drops between the main switch gear and the load are minimized, since for a given load, fewer amperes are required at higher distribution voltages.

2. The choice of 4160 V for the primary distribution as compared to over 2400 V is based upon lower equipment costs, limitations and load handling as well as fault-interrupting capability at lower voltages, and also upon the relative conve-

nience for future expansion. The primary distribution at 4160 V appears to be optimum for plant loads from 2000 to 10 000 kVA. Where all power is purchased at 2400 V or where most of the load is concentrated using short feeders to motors in the 200 to 700 hp range, the 2400 V system may be desirable.

3. Generation and distribution at 480 V as compared to 4160 V is more costly due to the larger conductor sizes required. A 5000 kVA, 480 V system requires a bus-conductor rating of 6000 A, whereas at 4160 V the rating is only 695 A of load and would be handled easily with standard 1200-A rated equipment. A voltage of 480 is a standard secondary voltage rating for load-center unit substations, and has the advantages of lower cost, size and flexibility per transformed kVA, when compared to a lower secondary voltage such as 240 or 120/208.

4. Beet-sugar factories universally use a *radial* distribution system because of its simplicity, reliability, lower first cost, and lower maintenance. With the radial system, a given load is served by only one primary feeder, load-center transformer, and secondary feeder, and failures with quality equipment are extremely low, particularly when there is adequate maintenance. There is no duplication of service equipment or tie cables as there would be with the more extensive primary or secondary selective systems.

5. Load-center unit substations, each consisting of a 4160 primary disconnect switch, a step-down transformer, and one or more 480 V secondary feeder breakers are common in the sugar industry in the range of 750 to 1500 kVA. The 1000 kVA unit with a standard impedance is popular for small and medium size systems, since short-circuit current levels on the secondary side are compatible with the interrupting capabilities of standard design, grouped, motor-control centers and other low-voltage distribution equipment. For large systems, special impedances are available to permit compatability. For large spot loads at 480 V, or where there are special voltage regulation problems, or where diversity of load is involved, ratings up to 2000 kVA may often be justified, in spite of higher short-circuit current levels and the need for more expensive feeder and motor-control equipment.

Power Distribution Equipment at the Utilization Voltage

Power can be supplied to motors or other load devices by three methods: a) direct service from the primary system at the primary voltage to large motors, normally rated above 200 hp at 2300 V or 4000 V; b) direct service from 480-V circuit breakers for motors 100 hp and above at 460 V, using the electrically operated circuit breaker as the motor starter and the branch circuit protective device; or c) service from feeder circuit breakers of combination motor starters as part of a grouped motor control center.

It is usually more economical to purchase 2300 V or 4000 V motors and controls for motors rated above 200 hp, when these higher voltages are available. If capacity at existing 480 V substations is not available, there is sometimes justification for choosing 2300 V or 4000 V motors at 200 hp and below.

When 460 V motors rated at 100 hp and up are necessary, direct service from the load-center unit substation or switchboard feeder is economical as long as frequent motor starting is not necessary, as for boilerfeed pumps, Electrically operated, low-voltage, air circuit breakers with properly selected, direct-acting trip devices are used for motor-starting duty.

About 65% of the beet-sugar factory electrical load consists of 3-phase induction motors with ratings of 100 hp or below. These motors are served at 480 V from

combination motor starters as part of grouped motor control centers. In addition, the control center may be arranged with branch-circuit and feeder-circuit breakers for other loads such as lighting transformers, welding receptacles, and process control. The main bus-conductor of the motor-control center is available in 600 A and 1200 A ratings with short-circuit bracing for 22 000 A, symmetrical, as standard. Direct connection to load-center unit substations for systems of 1000 kVA or less is thus possible. Bracing for higher levels of fault current is available as an option for increased cost.

Figure 20-4 shows a comparison of costs of induction motors and starters for 460 V, 2300 V, and 4000 V ratings up to 500 hp. Approximate 460 V motor and control costs are plotted for two cases: where 480 V is available and served at that voltage, and where substation capacity must be installed to serve the motors and control at 480 V from the primary system. The break-over point at which high voltage motors and equipment are preferable over 460 V equipment is between 200 and 250 hp.

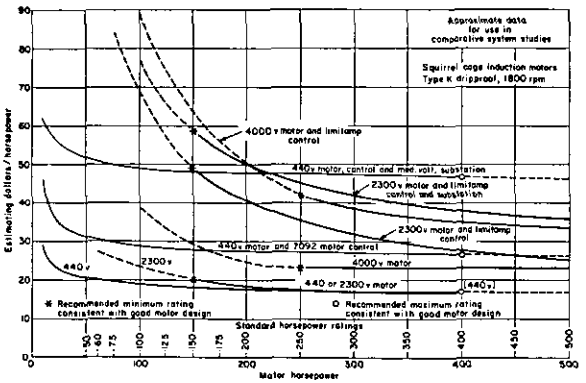


Figure 20-4. Cost comparison for induction motors and controls through 500 hp.

Grounding

The word "grounding" is commonly used in electrical power system work to cover both *system grounding* and *equipment grounding*. These terms are defined by the National Electric Code as follows: a system ground is a connection to ground from one of the current-carrying conductors of a distribution system or of an interior wiring system (system neutral ground); an equipment ground is a connection to ground from one or more of the noncurrent-carrying metal parts of the wiring system or of apparatus connected to the system. As used in this sense, the term "equipment" includes all metal parts such as conduits, raceways, motor and generator frames, and enclosures of switch gear and motor controllers.

The primary purpose of equipment grounding is to limit the voltage between noncurrent-carrying parts of the factory and ground to a safe value (6). With a uniform voltage throughout the grounding system, the chances are reduced of large differences of voltage within reasonable reaching distances of an individual, great enough to shock or injure him when short circuit or other abnormal conditions occur. It is important that adequate equipment-grounding be provided for the safety of personnel and property.

The primary purposes of system neutral grounding are to: reduce operating and maintenance expense through reduction in magnitude of transient voltages, improved lightning protection, simplification of ground-fault locations, and improved system and equipment fault protection; improved service reliability; and greater safety for personnel and equipment.

System neutral grounding is generally done at the neutral of wye-connected generators or supply transformers of primary voltage systems, and at the neutral of the load-center unit substation of lower voltage systems (4). Low-resistance grounding is generally used above 600V and the value of the resistance is chosen to limit the maximum phase-to-ground fault current to some value between 200 and 800 A, as determined by the overcurrent relays and current transformer ratios used on the feeders. A 10-second, time-rated resistor is used to keep the physical size small, assuming properly coordinated relays are used to remove the ground fault before the thermal limitation of the resistor is reached.

Historically, low-voltage systems have been operated either ungrounded or solidly grounded (5). Proponents for either system base their decisions upon whether it is desired to trip a feeder, for a ground fault on the feeder. Continuous process industries have favored the ungrounded system since a detection system will permit alarming a ground fault without tripping open a circuit breaker. Then location and correction of trouble can be delayed if necessary, until it is necessary to shut the system down. However, an ungrounded system is susceptible to transient overvoltages or a severe phase fault, if a ground develops on another phase during the waiting period. A solidly grounded system, on the other hand, while drawing enough fault current to trip a protective device, does not usually permit extensive electrical-burning damage to the equipment at the point of fault. Factors such as personnel safety and shock hazard are also important in the decision as to the grounding method selected.

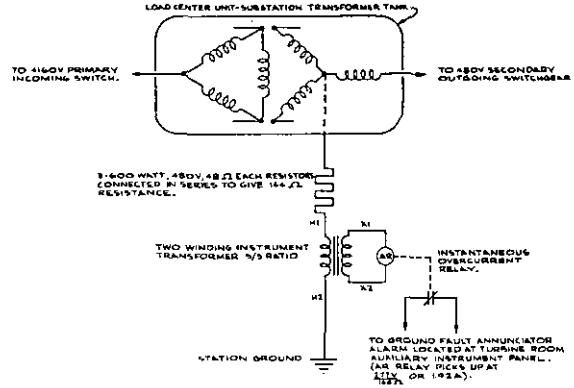


Figure 20-5. High resistance grounding of 480 V load-center unit substation with alarm functions. Spreckels Sugar Company, Spreckels, California.

In recent years, the sugar industry has favored the high-resistance grounding method for low-voltage systems, in which a high value of resistance is used between the system neutral and ground (Figure 20-5). The value of resistance is chosen so that the maximum ground fault current is somewhat higher than the normal capacitive-charging current of the system; i.e., a few amperes or less. The high-

resistance grounding eliminates transient over-voltages, yet retains the favorable non-tripping feature of the ungrounded system. The low value of current which flows in a ground fault that develops in the high-resistance grounding system may be monitored and alarmed. Also, traceable signals may be superimposed on the system without interrupting the service, to permit easy location of the point of fault. High-resistance grounding has the disadvantage that a fault in one phase permits the other phases to be elevated above ground by the full line-to-line voltage. In addition, a phase-to-ground fault must be quickly located and isolated to prevent a severe phase-to-phase fault developing as the result of a ground fault in another phase developing elsewhere in the system (Table 20-4).

System Overcurrent Protection

Three basic devices are used to detect overcurrents resulting from faults in an electrical system. These are relays, direct-acting trips on circuit breakers, and fuses.

Relays. Relays are devices installed in the system to detect trouble and complete a circuit to electrically trip their associated circuit breakers to isolate the trouble spot. Relays may be simple overcurrent devices, or they may use a combination of current and voltage, or current and current coils, to detect the direction of current flow, current balance, differences in the current at two ends of a circuit, or distance. Relays provide the highest degree of overcurrent protection. They can be built to much more accurate tolerances than fuses and direct acting trips, and they have adjustable time and current characteristics. At 2300 V and above, relays are commonly used for generator, utility company incoming line, and feeder overcurrent protection. In these applications, the relay current and voltage coils are connected from the secondaries of instrument transformers that are installed in the power lines. Their contacts are then connected into the tripping circuits of their respective power circuit breaker.

Direct-acting Circuit-Breaker Trips. Direct-acting trips are mounted directly on a circuit breaker, and they trip the breaker by direct mechanical action in response to the magnitude of current in the circuit. At 600 V and below, their trip coils are connected in series with the power circuit. Although direct-acting trip coils are much less accurate than relays, they are entirely satisfactory for most low-voltage power system applications, and are commonly used for overcurrent protection at 600 V and below. The justification for direct-acting trip coils at lower voltages is strictly an economic one, because of their lower cost and the larger number required.

Fuses. Fuses are thermally operated devices combining the function of fault detection and circuit clearing. They are used on both high- and low-voltage systems. Fuses have the disadvantages of being nonadjustable and quite slow in operation for moderate values of fault current. They are less accurate than relays, but comparable with direct-acting, low-voltage circuit-breaker trips for high current, and they are superior to direct-acting circuit-breaker trips on low-level short circuits or overloads. Another disadvantage of the fuse is that it can cause a single-phase condition, with resulting damage to motors or other 3-phase units, unless the fuse is so arranged to trip the other phases. For this reason, many engineers are reluctant to use fuses or other single-pole interrupters in applications for main feeder-circuit, overcurrent protection. However, a fuse of the current-limiting type has the advantage of being able to interrupt high short-circuit currents.

Figure 20-6 summarizes commonly used fault devices for the sugar-industry power distribution system.

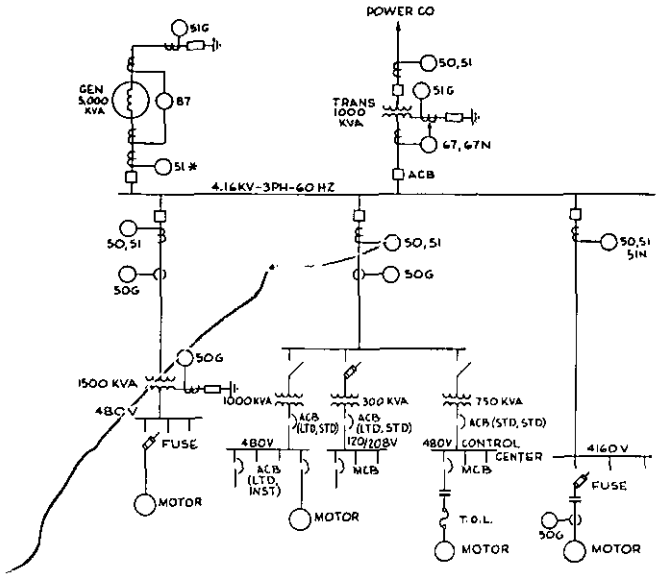
Table 20-4 Principal Characteristics of Major Methods of Grounding Low Voltage Systems

System Property	Type of System Grounding		
	Solid *	High Resistance **	Ungrounded
Immediate Shutdown of Faulty Circuit on Occurrence of First Ground Fault	Yes	No	No
Control of Transient Overvoltages Due to Arcing Ground Faults	Yes	Yes	No
Control of Impressed or Self-generated Steady-state Overvoltages	Yes	No	No
Flash Hazard to Personnel During Ground Fault (No Escalation of Fault)	Severe	Essentially zero	Essentially zero
Arcing Fault Damage to Equipment During Ground Fault (No Escalation)	May be severe unless fault is promptly removed	Usually minor unless fault removal is so prolonged as to cause fault escalation	Usually minor but transient overvoltages may cause fault escalation or multiple insulation failures
Shock Hazard, Unfaulted Phases to Ground, During Ground Fault	Line-to-neutral voltage	Approximately line-to-line voltage	May be several times line-to-neutral voltage

Table 20-4 (continued)

Shock Hazard, Equipment Frame to Ground During Solid Internal Line-to-Ground Fault	Moderate	Minimum	Small
Detection of Arcing Faults	L-L or L-G arcing faults readily detected, esp. with ground fault relaying	Ground detectors and fault locating equipment required for L-G arcing faults. L-L faults readily detected by phase overcurrent devices unless fault current is severely limited	Ground detectors and fault locating equipment required for L-G arcing faults. Transient over-voltages may meanwhile cause additional insulation breakdowns. L-L faults readily detected by phase overcurrent devices unless fault current is severely limited
Suitable for Four-wire, Three-phase Service	Yes	No	No

*For optimum results, use of solid grounding method should include sensitive ground fault relaying.
**For optimum results, use of high resistance grounding method should include equipment and procedures for alarming, tracing and removing the ground fault promptly.



Definition of Device Numbers and Abbreviations

50	Instantaneous overcurrent relay (phase protection).
50G	Instantaneous overcurrent ground relay.
51	Time-delay overcurrent relay (phase protection).
51*	Time-delay generator overcurrent relay with voltage restraint.
51N	Time-delay overcurrent residually connected ground relay.
51G	Time-delay overcurrent ground relay.
67	Directional overcurrent relay (phase).
67N	Directional overcurrent relay (ground).
87	Differential relay.
ACB	Air circuit breaker.
LTD	Long time-delay direct-acting trip.
STD	Short time-delay direct-acting trip.
Instant	Instantaneous direct-acting trip.
MCB	Molded-case breaker with nonadjustable thermal and instantaneous trip
T.O.L.	Thermal overload

Figure 20-6. Typical beet-sugar plant electrical system, one-line diagram showing fault protective devices commonly used.

Coordination of Protective Devices

The final step in the design of an electrical distribution system is a study to be sure that the various protective devices chosen are selective or coordinated. Thus short-circuit and relay coordination studies must be made. These studies are beyond the scope of this text, but the reader is referred to references (1,3).

The overcurrent devices; i.e., relays, breakers with direct-acting trips, and fuses, should be selective in operation in order to localize the disturbance. This means that the one nearest the fault on its power-source side should have the first chance to operate. If this protective device fails to function as intended, the next device in the chain should take over the task, and so on, in successive steps to the power-source circuit breaker itself, if necessary. When coordinated-selective tripping of protective devices is provided, the damage to equipment and delay to production resulting from short-circuits will be held to a minimum.

Overall Planning

The power system should be planned to obtain maximum service reliability with minimum first cost. While the electric system may be installed in discrete parts,

it must function as a completely integrated and coordinated unit. The best way to get an overall picture of the probable performance of a power system is to start with a well-planned, one-line diagram of the entire system. Please refer to Figure 20-7. Some of the more important aspects to be considered are:

1. *Safety.* One cannot place too much emphasis on safety. Electricity is becoming more and more an integral part of the processing equipment. It is also used in all of the unit process areas through which many workmen must pass. Enclosures that are marked "for authorized persons only," are no guarantee. Authorized personnel make mistakes, too. Three fundamental ideas help to build safer power systems: inclose all live conductors in grounded metal, or provide a ground conductor in a non-metallic raceway; use only adequate circuit-protective and switching equipment; and design the system so that working on energized conductors is not necessary.

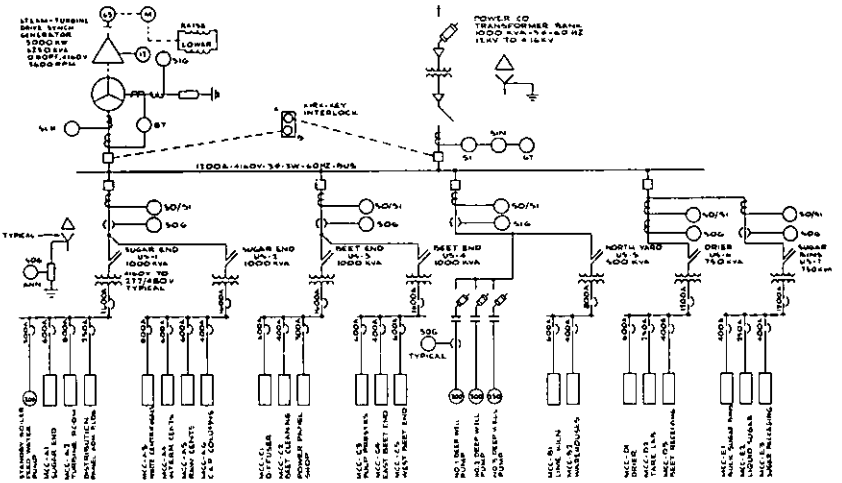


Figure 20-7. Electrical one-line diagram showing typical power distribution scheme for factory rated 4200 tons per day slice production capacity.

2. *Plan for a load growth.* Probably one of the greatest mistakes made in planning the power system is not allowing for future expansion. Some important guidelines applicable to existing and new facilities are: provide steam-turbine capability to drive the generator at the rated kVA and a power-factor loading of at least 0.92; review thoroughly the selection of turbine throttle steam pressure in order not to exceed the processing steam needs at increased generator loadings; buy main switch gear with a margin in interrupting rating to allow for additional power sources to the factory; allow some reserve substation capacity over predicted present loads. Provide a reserve margin of current-carrying capacity at main feeder cables and, perhaps more important, select the proper voltage; i.e., one high enough to permit growth without bottlenecks in current ratings or interrupting capabilities.

3. *Simplicity.* Make systems simple to operate and maintain. Many total shutdowns have resulted from systems with complicated and unnecessary interties, since operators are not involved in emergency switching practice every day. If the system is complicated, experience proves that workmen are apt to make mistakes in

an emergency. These errors usually cause greater outages than would be obtained with simple systems. Simple systems are easy to understand, operate, and maintain both under normal and emergency conditions. The growing difficulty of obtaining adequately trained personnel also makes a simple power system desirable.

4. *Flexibility.* The electrical utilization load in the beet-sugar factory is not static. Historically, it has experienced growth in the areas of sugar storage, liquid-sugar production, and beet-receiving facilities. In addition, other phases of the process are modified to improve quality as well as production. The trend for most process industries is towards higher use of electricity per unit of production. In order to permit the addition of blocks of electrical load, flexibility and expandability must be designed into the electrical distribution system. Adequate floor space for expansion of main switch gear and motor control centers should be considered. The installation of more, but smaller load-center unit substations can often provide this flexibility.

5. *Service Reliability.* Service reliability in any factory is important. The production of sugar is a continuous process, so that a shutdown in any of the unit process areas from the beet end through the sugar end can result in complete production stoppage. High service reliability generally can be obtained through installing only the highest-grade electric equipment available, using the best installation methods, and by the development of good overall preventive maintenance programs.

6. *Selection of Equipment.* The best available electrical equipment adequate for the application should be selected. It costs no more in the long run and pays dividends in service continuity and lower maintenance. Some widely accepted practices are: use only metal-clad equipment; consider the flexibility of 4160 V and below drawout construction switch gear and similar construction for motor control equipment; choose noninflammable transformer coolants and wye secondaries for the load-center transformer so that neutral grounding may be used; use factory-assembled equipment for easier field installation and better coordination; and be sure equipment ratings are adequate in every respect, such as voltage, current, and interrupting rating.

7. *Modernization and Expansion.* When the factory power facilities are modernized, the engineer is afforded the opportunity to design the ideal electrical system. First a one-line diagram should be made of the system that he would have if the plant were starting as new. Having made this plan, it should be used as a guide for all future modernization and expansion. Existing equipment obviously cannot all be retired at once. But, as additions and replacements of equipment are made, they should be on the basis of integration into the ideal plan, and not merely as replacements of equipment in the old system. One of the most significant parts of the new program is the selection of the proper voltage.

Typical Power System

Figure 20-7 is a one-line electrical diagram of a typical sugarbeet processing factory. The system has suitable generating capacity to provide for the electrical production demand. "Kirk-key" interlocks are provided between the generator-main, and the power company incoming main breakers. The interlocks prevent both circuit breakers from being closed at the same time, assuring that the factory generating system and the utility system will not be operated in parallel. The possibility of their union out of synchronism when switching from factory to the outside power source is prevented. Sometimes, in order not to interrupt the power

to the load during transfer, synchronizing equipment, not shown, will be provided for short-time, momentary paralleling. When operating in parallel, either on a temporary or permanent basis, it is necessary that device 67 (directional power relay) be provided to separate the two systems in the event of a power-company system interruption. Tripping the incoming line circuit breaker assures that the systems will not be reunited out of synchonism. In the event the factory electrical-load demand exceeds its generator capacity, a permanent parallel-tie between the factory and the power-company systems may be arranged. If so arranged, a tie-line controller is often used to optimize the purchased power. The controller senses tie-line kilowatt and kilovar flow, and if these values differ from the contracted levels or set point levels, corrections are made to the governor and voltage regulator settings for the plant generator, until the tie-line kilowatts and kilovars are at their correct values. Under-frequency relaying would normally be provided to prevent serious generator overload. The generator should also have directional-power and negative-sequence, current relaying. Directional-power relaying protects the generator from motoring on loss of its turbine drive throttle pressure. Negative-sequence current-relaying protects against heavy overloads resulting from a power company single phase.

The main 4160 V generated power is distributed to seven load-center unit substations through five 4160 V main feeder-breaker positions. Large hp, deep-well pump motors are fed directly at 4160 V with high voltage, motor controllers.

Load-center substations are limited to 1000 kVA with standard impedance in order to limit short-circuit currents on downstream equipment to a maximum of 30 000 A, symmetrical standard. Large 460 V motors are started across the line with an electrically operated substation secondary-feeder breaker, since infrequent starts are required.

The 480 V secondary switch gear feeder breakers serve motor control centers and power panel-boards. This secondary distribution serves the factory utilization load at 480 V.

The generator and load-center substations, secondary-winding neutrals are resistance grounded and the power company transformer bank is solidly grounded. If the power company was operated in a continuous parallel tie with the factory generator, it would also be resistance grounded. Selective tripping between the 4160 V incoming, power-company main, and the 4160 V feeder breakers is provided for ground faults on any 4160 V feeder.

The system shown in Figure 20-7 is fully rated, and is provided with coordinated-selective tripping for overcurrent protective devices. "Fully-rated" means that all circuit protective devices have sufficient interrupting capacity to safely clear a 3-phase, bolted, short circuit.

The entire system may be expanded readily with a minimum amount of additional investment.

The reader will find the references (1-6) useful in further studies of this subject.

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Chapter XXI

THE ENVIRONMENT*

Introduction:

The United States Environmental Protection Agency (EPA) was created in December of 1970 to consolidate and strengthen federal efforts to protect the health and welfare of the American people, by rehabilitation and protection of the environment. The EPA was designed to bring under one federal agency the authority and the means to control air, water, and solid waste pollution. The most important function of EPA was to assist states and local governments in their own efforts, and while this system of state and local administration lends flexibility in handling the wide spectrum of conditions and problems, it places the beet-sugar industry in a very complex system of regulations. This does not allow for across-the-board solutions to environmental problems in an industry spread over 5 federal regions, 16 states (and 2 Canadian provinces) that include climatic, geographic, social-economic and political variances, resulting in a wide range of regulation limits and control methods.

The ultimate goal of the EPA is to transfer administrative authority to the state, when the state has presented a program that is at least as, or more restrictive than the federal guidelines. This means a state or local government may impose more stringent regulations than the federal regulations, and results in a maze of regulations which must be carefully researched and complied with when environmental programs are contemplated in a factory area. This also results in numerous permit applications that must be properly filed before construction can begin, and failure to comply with this condition adds to the complexities of environmental problems.

The EPA envisioned a basic system of three segments; air, water and solid waste, but as the program evolved and more trouble spots developed, the categories were expanded.

Following is a list of environmental subjects which are designated as chapter sections: 1) air; 2) odor; 3) noise; 4) water, a) ground water, b) liquid storage; 5) solid and hazardous waste; 6) toxic substances.

Under each of these sections will be discussed: 1) regulations, 2) sampling and analysis, 3) control methods and equipment, 4) results obtained by various systems, *and 5) future status of the program.*

Air*

The air around us has never been completely pure. There has always been some natural pollution which nature's own system could accommodate. However, with the industrial revolution and population explosion, the natural system became overloaded.

A knowledge of the earth's atmosphere or troposphere is essential to understand the problems of air pollution.

The troposphere is about 18 miles thick and is composed of the following elements: nitrogen, *78%*, oxygen, *21%*, and argon, *0.9%*. The remaining *0.1%* (or 1000 ppm) consists of: carbon dioxide, 330 ppm; neon, helium and krypton (all in-

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* Written by C.H. Iverson, revised by D.L. Stewart

ert gases like argon), plus the pollutants. The above is for dry air. The water content can vary from 900 to 0.9 ppm. Therefore we are concerned with a miniscule fraction of the atmosphere that is constantly changing in composition, influenced by sunlight and varying amounts of water vapor(1).

The Clean Air Act of December, 1970, did not achieve the intended goal of attainment of health-protecting standards by July 1975, and was amended in 1977 to allow states to draw up specific plans for bringing non-attainment areas into compliance. The law requires that if a state fails to implement and enforce the act, EPA must do it. This follows other congressional efforts to preserve and enhance the authority of state and local government while setting forth guide lines for state actions.

The Clean Air Act originally established standards for six pollutants: 1. particulate, 2. sulfur dioxide, 3. carbon monoxide, 4. hydro-carbon, 5. nitrogen dioxide, 6. ozone.

The intention of the standards was to protect the health (primary standard) and welfare (secondary standard) of the citizens of the United States.

The compound lead was added to the original list in 1978.

The following list makes up the Ambient Air Quality Standards as now effective:

Table 21-1
National Quality Standards for Ambient Air (15)
(in micrograms or milligrams per cubic meter · μ/m^3 and mg/m^3 and in parts per million · ppm)

Pollutant	Averaging Time	Primary stds (Health)	Secondary stds (Welfare, Materials)
Particulate (matter)	Annual	75 μ/m^3	60 μ/m^3
	24 Hour	26 μ/m^3	150 Σ/m^3
Sulfur dioxide	Annual	80 μ/m^3 (.03 ppm)	
Sulfur dioxide	24 Hour	365 μ/m^3 (.14 ppm)	
	3 Hour		1300 μ/m^3 (.5 ppm)
Carbon monoxide	8 Hour	10 mg/m^3 (9 ppm)	Same as Primary
	1 Hour	40 mg/m^3 (35 ppm)	
Hydrocarbons (non-methane)	3 Hour (6-9 AM)	160 μ/m^3 (.24 ppm)	Same as Primary
Nitrogen dioxide	Annual	100 μ/m^3 (.05 ppm)	Same as Primary
Ozone	1 Hour	240 μ/m^3 (.12 ppm)	Same as Primary
Lead	3 Month	1.5 μ/m^3 (.006 ppm)	

The two air pollutants that are of main concern to the beet-sugar industry are particulate and sulfur dioxide. A study conducted by EPA (1) indicated that industrial processes are responsible for 47% of the total particulate emissions, while combustion accounts for 34%. The burning of sulfur-bearing fuel produced 82% of the sulfur dioxide and certain industrial processes contributed 15% of the total.

Beet-sugar processing plants are located in a wide variety of geographic and climatic conditions, and air quality standards are designed to control each of their

individual problems. Data submitted by industry members show a wide range of emission limits imposed by the various air quality control agencies.

Thus, source-point regulations require careful study of local, regional, state and federal laws before final decisions can be made on clean-up programs and equipment.

Source Point Regulations

Beet-sugar processing plants are typical stationary sources of air pollution. The control of existing sources is primarily under state control. Each state must develop a plan devised to fit the region's needs, and the technical and economic feasibility of controlling emissions.

For selected categories of new industrial plants, and for those that are substantially modified, the law requires EPA to set emission limits for certain pollutants. EPA is to set standards for all major stationary sources.

These new standards are the new "Source Performance Standards" and are specific for each industry. They should not be confused with ambient air quality standards. The new source standards set maximum limits for each pollutant which can be emitted from the new source stack, vent or process in the facility.

Boiler house emission limits are established by two systems. One system is based on the total heat input in millions of Btu/hr. The allowable emissions under this system vary over a wide range of values as illustrated in the industry report, where one company indicates a range from 0.7 lbs/10^6 Btu to 1.8 lbs/10^6 Btu allowable emissions.

The allowable emission rate can also be calculated from Table 2.

Table 21-2.
Standard Gas Volume Table

Source Gas Vol, SCFM*	Concentration GR/SCF**	Source Gas Vol, SCFM	Concentration GR/SCF
7 000 or less	0.100	120 000	0.040
8 000	0.096	140 000	0.038
9 000	0.092	160 000	0.036
10 000	0.089	180 000	0.035
20 000	0.071	200 000	0.034
30 000	0.062	300 000	0.030
40 000	0.057	400 000	0.027
50 000	0.053	500 000	0.025
60 000	0.050	600 000	0.024
80 000	0.045	800 000	0.021
100 000	0.042	1000 000 or more	0.020

(Note: The data submitted by the industry members indicate both of these systems are used to establish the allowable emission rates, but the heat input method is more often applied.)
*(SCFM = standard cubic feet per minute)
** (GR/SCF = grains/standard cubic foot)

Stack Analysis

Boiler-house stack analysis is an exacting procedure because of the nature of the stack and boiler-house equipment. Several sampling systems are available, but approval of the local agency is generally required. Work platforms and ladders to carry out the sampling may vary from region to region, but the following arrangement is basically acceptable:

The sampling ports should be located 8 to 10 stack diameters above the last obstruction and at least 2 stack diameters below the stack exit. If the stack diameter exceeds 10 feet, 4 sampling ports at 90° spacing around the circumference are

required. If the diameter of the stack is less than 10 feet, then 2 sampling ports spaced at 90° will suffice. The length of the probe is a significant factor.

The work platform should be at least 4 feet wide and capable of holding 2 people plus the weight of equipment. The platform should be 360° on a 10-foot or greater stack diameter but less than 360° for a 2 port system.

OSHA (Occupational Safety and Health Administration) rules and regulations should be observed and local requirements checked on the construction of the platform, railing and ladder.

In the case of low profile stacks the location of the ports is often a matter of local preference, but a general rule is 1/5 of the stack height down from the stack exit, when the diameter rule cannot be applied. Boiler-house stack sampling equipment usually is selected on the basis of agencies' requirements or recommendations. Stack sampling crews must be trained in the techniques, and in the case of opacity evaluations many agencies require that the opacity value be determined by a certified observer.

When stack emission loadings have been determined, selection of control equipment can be projected on the basis of information obtained from the survey. Basically four systems were found to be in use when the industry was surveyed. The four types of control equipment were:

1) electrostatic precipitators, 2) water scrubbing, 3) cyclonic and fall-out chambers, and 4) bag houses.

Both of the electrostatic precipitators reported were used with lignite-burning boilers, burning lignite of relatively low sulfur content. A Venturi scrubber at the Wahpeton, North Dakota, factory of Minn-Dak Farmers Cooperative is also used with lignite, and both systems are satisfactory.

The use of some mechanical collector preceding any device, to remove larger particles, seems desirable and reduces loadings of the final unit. The operation of any control equipment has some disadvantages. The electrostatic precipitator functions more effectively on a higher sulfur fuel but seems to perform reasonably well on lignite.

Scrubbing has some desirable features in that it cleans up the stack and reduces odor and sulfur emissions. Argabrite describes wet scrubbing for the removal of sulfur dioxide and particulate from boiler stacks (1).

The greatest objection to scrubbing is the possible emission of odors from stored scrub-water, but proper pH control largely prevents this. However, in extremely cold regions where ponded scrubbing water is carried over winter, this is a problem.

1. *Fly-ash.* This is an emission common to all coal burning units. Fly-ash is composed of the ash and unburned combustibles which become air-borne in the firebox and find their way to the atmosphere because of the velocity of the flue gas through the boiler and up the stack. The type of stoker equipment has much to do with the amount of fly-ash emitted. *Pulverized coal* units with dry bottom furnaces are the worst fly-ash emitters because the only way to remove the ash is to carry it out with the flue gas. Collecting systems for fly-ash must be installed for this type equipment.

The *spreader stoker* is a lesser offender, but still a serious one, because the coal is thrown into the combustion chamber where much of it is burned in suspension. The ash of these finer particles never reaches the grate and is carried out with the flue gas.

Chain grate and *underfeed* stokers are the least offenders, in that order. Fly-ash is generated in these types due to the air velocities through the coal bed, causing some particles to become air-borne.

The type and screen analysis of coal are also factors in the generation of fly-ash. A high volatile, or coking type of coal generates a light-weight ash particle which is more easily air-borne. Excessive fines result in a larger quantity of fly-ash.

Smoke emission problems from a boiler plant stem from many sources. Some of the main causes and solutions are:

(a) *Type of coal.* High volatile content coals generally burn with more smoke than low volatile coals. This problem can generally be solved with a good overfire air system.

(b) *Load on boiler.* Too often boilers are operated at loads well beyond the manufacturer's rating. At these overloaded conditions draft and overfire air auxiliaries are apt to be deficient, high gas velocities tend to entrain more fly-ash, and high combustion-chamber temperatures often result in slagging problems. The solution is obvious.

(c) *Distribution of coal on grate.* It is necessary for complete and efficient combustion that each piece of coal get just the right amount of air. Uneven thickness of coal on the grate creates a condition where too much air goes through the fuel bed where it is thinner, and too little where it is thicker. This starving for air in the thicker fuel bed is probably the most common source of smoke. Proper maintenance and adjustment of the stoker should minimize this problem. With spreader stokers, a change in coal screen-analysis will change the distribution on the grate, therefore frequent adjustments of rotor speed and spill plate position are necessary. Also, size segregation of coal in coal bunkers and spouts will affect grate distribution.

(d) *Overfire air.* The principal purpose of overfire air is to create sufficient turbulence above the fuel bed so that a more intimate mixture of air and the unburned volatile combustibles distilled out of the coal is realized. This mixing aids the burning of these volatiles and reduces the amount of smoke generated by poor distribution of air through the grate.

Overfire air systems can be inadequate, as a result of improper nozzle design, nozzle location, inadequate fan capacity, or improper air duct sizing (1). If there are smoke problems, it would be well to review the overfire air system. If operating above the manufacturer's load rating, the overfire air system is probably inadequate.

(e) *Fuel-air ratio.* Particularly if the boiler is overloaded, it may be operating with an inadequate air supply. An Orsat analysis can detect this condition. The fuel-air ratio may be improperly adjusted, and even a natural gas flame can smoke if it does not have enough air.

(f) *Fuel oil atomization.* The improper atomization of fuel oil is the main cause of smoke from an oil burner. The more finely divided the oil droplets are, the better will be the combustion. Oil atomization is a function of several factors: temperature of the oil, nozzle pressure, and steam pressure, if the oil is steam atomized. At a given load the burner-tip orifices may be too large, forcing operation at an oil pressure too low to provide adequate atomization. Remedy—change to a smaller tip.

(g) *Grate and setting air seals.* For most efficient and smoke-free operations, all the air that enters a boiler should pass through the fuel bed, burner, or overfire air nozzles. Therefore, it is very important that the air seals around and below the

grate and in the ash flume be in good condition. Any air that bypasses the fuel will reduce efficiency and promote the generation of smoke. Air entering the firebox from the ash flume over the end of the grate is a major source of "tramp" air.

Pulp Drier Emissions

The limits are based on process weight or source gas volume. While the process-weight system is most widely used, the gas volume is employed in some areas.

The beet-sugar industry process-weight for pulp driers is generally defined as the weight of the pressed pulp plus the weight of solid fuel, and the allowable emission is computed from Table 3 on that basis:

Table 21-3.
Process Weight Table

Process Wt. tons/hr	Rate of E* lbs/hr	Process Wt. tons/hr	Rate of E lbs/hr
10	19.2	45	43.6
15	25.2	50	44.6
20	30.5	60	46.3
25	35.4	70	47.8
30	40.0	80	49.0
35	41.3	100	51.2
40	42.5	500	69.0

(*E = allowable emission in lbs/hr)

Pulp-drier stack sampling requires essentially the same equipment and instruments as boiler-house analysis. However, because pulp-drier stacks are generally of large diameter and low profile, the gas-flow characteristics make reliable sampling difficult.

Brenton, Faist, et al. conducted an in-depth study of pulp-drier stack sampling and modified the measuring procedure to correct for the actual angle of gas flow in the stack. This method insures a reasonable accuracy of results (6). As in the case of the boiler-house, sampling equipment and techniques are reviewed by local agencies and stack studies are carried out with agency approval.

Pulp-drier stacks are generally of low profile and sample port location is a matter of local agency preference, but 1/5 the stack height down from the exit is basically a reasonable rule.

The sampling platform and ladder construction must conform to OSHA and local safety regulations.

Pulp Drier Emission Control

When gas or oil is burned as a primary fuel, pulp-drier stack clean-up is easier than with solid fuel.

The installation of cyclonic collectors in any drying system is considered good practice, to be followed by a secondary device. Three of the companies responding to the industry-survey report scrubbing to be an effective pulp-drier emission-control method.

1. Installing multiple cyclones of smaller diameter. For a given pressure drop, a smaller diameter cyclone has a better collection efficiency than a larger diameter unit.

2. Skimming a cyclone vent stack, removing much of the particulate from the stack, and reinjecting this exhaust gas and particulate matter load back into the fur-

nace as dilution air for temperature control. A diagram of this system is shown in Figure 21-1. This system has two major advantages which are: 1) A large portion of the particulate matter is removed from the exhaust, and 2) Increased thermal efficiency, up to 10%, can be realized because of the smaller heating load on the dilution air, since the recycle gas is already above 200 °F. A further refinement of this system would be the addition of a high efficiency collector in the recycle circuit, recovering the dust rather than burning it in the furnace.

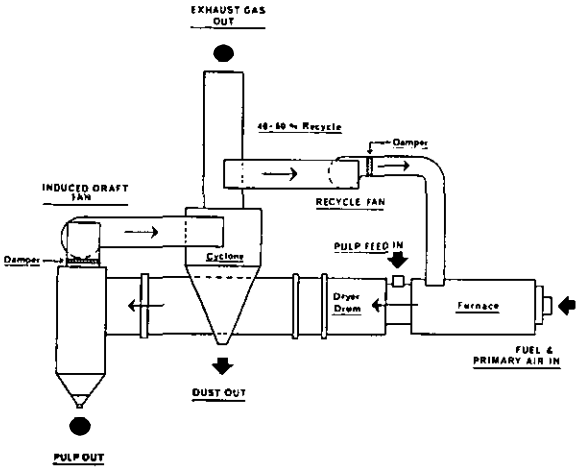

Figure 21-1. Pulp drier air pollution abatement system ***(Courtesy Stearns-Roger Corp.)***

The other source of air pollution in the pulp dryer is the dust created in the handling of dried pulp and pelleting equipment. This can be controlled with a well designed hood-pickup system and a high efficiency mechanical collector.

Miscellaneous Particulate Sources

1. *Sugar granulators.* A sugar granulator is a rotary drum drier equipped with lifters which cause the sugar to cascade through a moving volume of air (see pp 497-499). This operation causes a significant amount of fine sugar to become entrained in the air stream, which would become a particulate-type air pollutant if exhausted directly to the atmosphere. This problem is commonly solved by the use of wet scrubbing, using either water or a beet-end juice as the scrubbing fluid. This can be "once-through" flow or recycled bleed-off and makeup to control the Brix of the scrubbing fluid. The scrubbing juice is either sent to a melter, or returned to the beet end.

2. *Sugar conveying and sacking equipment.* The dust generated by conveying dry sugar is generally collected by means of a system of ducts and pick-up hoods at transfer point. The dust-laden air is cleaned by a "baghouse" or fabric-sock type of air filter. The collected dust is generally reprocessed through the melter.

3. *Lime dust.* The handling of rock and burned lime can be a dust problem. A hooded, dust-pickup system cleaned either by a "baghouse" or a wet scrubber is most commonly used.

4. *Lime kiln booster fan.* This is normally no problem insofar as air pollution is concerned. However, during the charging of the kiln the fan exhaust can

discharge enough particulate matter to be a problem. A wet scrubber can easily solve this, discharging the water through the carbon dixoide gas washer.

5. *Fugitive dust.* Many states are now enforcing regulations to control fugitive dust. Beside the above-mentioned sources, controls are being promulgated for road dust, coal handling, pulp loading and blowing dust, and debris from factory and agricultural operations.

Results of Air Clean-Up Programs. Because rules and regulations vary over a wide range of allowable emission, and because of the wide divergence of operating conditions, the cost-benefit ratios of the various industry programs vary. However, the industry members responding to the survey used in preparing this text report reasonable results with the types of equipment described herein.

The Future Outlook of the environmental clean-up program indicates more consideration will be given to the cost-benefit aspects of the program.

In 1977 Congress amended the Clean Air Act to control certain hazardous emissions such as asbestos, beryllium, mercury and vinyl chloride. Also adopted in the amended act was a provision for offsetting reduction trade-offs in non-attainment areas. This provision allows a factory to be built if existing sources are reduced enough to compensate for the new factory emission.

Odor*

Regulations

The odor problem is perhaps the most difficult to deal with, because odors offend the senses and often times cause emotional reactions rather than being health hazards. The EPA does not spell out specific rules and regulations, but rather alludes to odor as a nuisance. However, several states are setting up odor regulations, and the response to an industry survey indicates that most states are approaching odor as a nuisance. However, the state of Minnesota, among others, has enacted concise rules and regulations to deal with odor.

The general pattern for making up environmental regulation has been to follow basic principles established by the leaders in a particular area, and one can assume future odor regulations will adhere to the general definitions of Minnesota, but it is conceivable that population proximity to odor sources may dictate variable allowable emission limits to fit particular situations.

The following definitions are set forth in Minnesota odor regulations:

1. *Ambient air* shall mean that portion of the atmosphere external to buildings to which the general public has access.

2. *Odor concentration unit* shall mean the number of standard cubic feet of odor-free air needed to dilute each cubic foot of contaminated air, so that at least 50% of the odor concentration test panel does not detect any odor in the diluted mixture.

3. *Odor emission rate* shall mean the product of the number of standard cubic feet per minute of air or other gases emitted from a suspected odor pollution source, and the number of odor concentration units determined for that source.

4. *Odor source* shall be defined to include, but not be limited to, any stack, chimney, vent, window, opening, lagoon, basin, catch-basin, pond, open tank, storage pile, or any organic or inorganic discharge and/or application which emits odorous gas, gases or particulates.

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* by D.L. Stewart

Odor Emission Limits

The State of Minnesota has adopted the following odor emission limits:

1. Odor sources emitting from well-defined stacks of 50 feet or more above grade elevation, and with adequate dispersion characteristics as determined by the agency, shall not emit odors greater than 150 odor concentration units.

2. Odor sources of less than 50 feet above grade, or otherwise failing to create good dispersion conditions as determined by the agency, shall not emit more than 25 odor concentration units.

3. No odor source shall have an odor emission rate in excess of 1 000 000 odor concentration units per minute. (This limit has been criticized as being too restrictive and will be reviewed.)

4. No odor source shall emit air contaminants into the air which cause odor outside the alleged polluter's property line in excess of the following limitations:

a) One odor unit in areas zoned residential, recreational, institutional, retail sales, hotel or educational.

b) Two odor units in areas zoned light industrial.

c) Four odor units in areas zoned other than in subsections a and b.

Odor Testing

The State of Minnesota decrees that odor testing shall be conducted as follows:

1. Odor tests shall be conducted by the agency or under agency supervision and advisement.

2. Odor test panels shall be selected or approved by the agency.

3. Ambient air samples containing the alleged odorous air pollution obtained downwind and outside the property line of the alleged polluter, and samples of air contaminant from the odor source allegedly causing the odorous air pollution shall be obtained.

While the general practice is to treat odor as a nuisance, a question is posed as to the legal aspects of action by public or private citizens annoyed by odor. Several states address this by including a nuisance provision which states that compliance with the provisions of odor regulations shall not operate as a defense against action at law based upon a public and/or private nuisance.

Because of the wide differences in state values for allowable odor emissions, each individual case must be evaluated before a proper odor control program can be developed.

The basic determination of odor concentration is done by a panel selected and approved by the agency. The samples may be presented to the panel by several different methods, from a simple dilution method, (ASTM Dilution Method) (3) to extremely sophisticated equipment such as the Forced Choice Triangle Olfactometer. This system was designed by IIT Research Institute of Chicago under support by Fats and Protein Research Foundation, Des Plaines, Illinois (4,10).

The olfactometer utilizes the forced-choice triangle principle to determine how much an odorous gas sample must be diluted (with air) to make it indistinguishable from non-odorous air.

The system is programmed to deliver 6 dilution levels to sniffing ports, consisting of 3 glass ports each equipped with a choice button. Two of the test ports deliver room air (blanks) while the third carries the diluted odor sample. The

panelist is instructed to smell all three ports and select, by depressing the selection button, the port that contains the odorous sample. The samples are present in ascending odor concentration and the panelist must make a selection.

The selection is shown on the signal box and this choice is recorded by the panel leader (see Figure 21-2).

Figure 21-2. Technician demonstrates the Forced-Choice Triangle Olfactometer.

The forced-choice triangle method eliminates many of the factors that might influence a panel member's choice, and produces results that can be related to statistically significant confidence levels.

The method of sampling a source, such as a vent or stack, is essentially the same as stack sampling. In addition to the determination of the gas flow volume, a sample of the gas is captured in a Teflon sample envelope. From the captured sample the dilutions are made for panel presentation and evaluation.

The following survey was made while a factory was operating on storage beets and probably high odor concentrations were being emitted:

Table 21-4.
Source Point Odor Evaluation (27)

Test No.	Source	Odor Conc.	Odor Quality	Odor Emission Rate-Dry OU/Min**
1	Pulp Drier	700	Burnt Molasses	37×10^6
2	* Pulp Drier	560	Burnt Molasses	13.2×10^6
3	Diffuser Vent #6	1.6×10^5	Offensive–Cooked Cabbage	2.4×10^6
4	Vacuum Pan Vent	2600	Very Offensive Pig Pen	3.1×10^6
5	1st Carb Tank	1700	Offensive–Cooked Cabbage	3.4×10^6
6	Thin Juice Vent	1900	" " "	1.5×10^6
7	2nd Carb Tank	2.3×10^4	" " "	7.1×10^6
8	#2 Evap	1.7×10^4	" " "	No Flow Data
9	#4 "	2300	" " "	No Flow Data
10	Diffuser Ammonia	1×10^4	" " "	No Flow Data
11	Thin Juice Boiler	4200	Slightly Offensive	0.2×10^6
12	#2 White Pan	8×10^4	Offensive–Ammonia	0.2×10^6
13	#3 Boiler	60	Sulfur	1.9×10^6
14	Diffuser Vent ×3	2200	Offensive–Cooked Cabbage	0.17×10^6
15	Lime Kiln	17	Very Weak–Cement Type	0.036×10^6

*Sample drawn after scrubber

**OU/Min–odor concentration units/minute

Ambient air odor sampling can be accomplished with a portable unit such as the Barnebey-Cheney Scentometer, or captured samples may be presented to an odor panel using the techniques already described for source emissions.

An ambient air odor study was conducted in 1976 by American Crystal Sugar Company to identify and quantify odorous emissions and to assess the impact of the odor emission on the residential areas (17).

Because odors are a matter of concern when transport water is stored over winter months, an odor study was conducted with a federally-sponsored study of solids separation from transport water (9).

The analytical program consisted of trapping emitted gases from the lagoon in a floating tent, and these gases were drawn off and analyzed to determine odor intensity gas production rate, and the chemical composition of the gas.

Odor concentration and gas production for each pond was correlated with several other known variables; surface water temperature, COD, pH, and TDS (total dissolved solids). While there was lack of correlation between ponds, there was a positive correlation for all ponds between odor and pH, and odor and TDS.

This study was conducted during a winter operation and the observations carried on through the spring break-up period when the most serious odor problems occur (7).

Odor Control Systems

Several companies have reported success with stack scrubbing to control particulate and odor emission. However, there are complications when scrub waters are stored over an extended period. Then the sulfur compounds generate some very offensive odor compounds. pH is a critical factor in scrubber systems, not only to protect equipment but also to prevent the release of offensive gases.

There is a correlation between TDS and odor emission volume. A two-step biological treatment process has been developed to achieve purification in a closed anaerobic tank. (This system is described under Waste Water.) The method holds promise of control of odorous emissions in waste systems.

The odor problem is a very difficult issue to deal with because it tends to arouse emotional problems as well as technical requirements. However, better analytical methods and research aimed at identification of odor constituents makes control of odor more feasible than in the past. It is significant, however, that compliance with odor regulation does not necessarily protect against nuisance-type legal action.

Noise

Regulations

The Noise Control Act of 1972 spells out federal plans to keep noise at tolerable levels. The federal laws are directed to emissions, but provide technical assistance to states establishing performance laws. The situation is also complicated by an overlapping of authority of the Environmental Protection Agency and the Occupational Safety and Health Administration. As a battle developed over the acceptability of 85 or 90 dBA (decibel is a unit of sound pressure level based on a practical scale of 0 to 140 dBA) as the safe sound level for workers, it became evident that OSHA has a direct responsibility for worker safety, while EPA must protect the worker as a plant employee and also as a citizen.

The regulations developed require that industry exhaust engineering methods, then employ administrative procedures, and as a final step provide protective equip-

ment when the other methods were neither feasible nor practical. It soon became evident that in some factory areas it would be almost impossible to reduce noise to acceptable levels, so permissible exposure limits were established.

Exposure to continuous noise at two or more levels may not exceed a daily noise dose "D" of unity, where "D" is computed by formula:

$$D = \frac{C_1}{T_1} + \frac{C_2}{T_2} + \ldots\ldots \frac{C_n}{T_n} \qquad (1)$$

where C is the actual duration of exposure in hours at a given steady-state noise level, and T is the noise exposure limit (in hours) for the level present during the time C, computed by the formula:

$$T = \frac{16}{2}\left[0.2\ (L-85)\right]$$

where "L" is the workplace sound level in dBA on the slow scale of a standard sound level meter.

The permissible exposure to continuous noise shall not exceed an eight-hour time-weighted average of 90 dBA, with a doubling rate of 5dBA. For discreet permissible time exposure limits, refer to the following table.

Table 21-5

Sound Level dBA	Time Permitted, Hours
85	16
90	8
95	4
100	2
105	1
110	0.5
115	0.25

Worker exposure to steady-state continuous noise shall not exceed 115 dBA, and an impulse or impact noise of 140 dBA shall not exceed 100 such impulses per day, but a 10 dBA decrease in the peak pressure allows an exposure number increase by a factor of 10 impulses. Ambient or community noise is a problem for beet-sugar processing factories because the laws hold the operator responsible for noise from his device, vehicle, building, or property. Urban encroachment has placed many factories in a vulnerable situation as an ecologically minded public wants a better life quality. The industry survey indicates that there is a wide divergence in the present performance laws but some areas are facing very stringent community noise control. The State of Minnesota has established the following ambient noise level limits:

NOISE AREA	Day (0700-2200)		Night (2200-0700)	
	L50	L10	L50	L10
1. Residential	60 dBA	65 dBA	50 dBA	55 dBA
2. Light Industrial	65 dBA	70 dBA	65 dBA	70 dBA
3. Manufacturing	75 dBA	80 dBA	75 dBA	80 dBA

where L50 is the noise level, expressed in dBA, which is exactly 50% of the time for a one-hour survey at the noisiest time of the day or night. L10 is the same condition for 10% of the test hour. dBA is the weighted sound pressure by the use of A (concert pitch A 440 Hz) metering characteristics and weighting as specified in ANSI (American National Standards Institute) for sound level meters, S1.4 – 1971 (2).

The noise measurement is accomplished by the use of the following basic units (23): 1. The sound-level meter, which is used for making measurements. 2. The octave-band analyzer which analyzes noise. 3. The sound-level calibrator which helps keep the sound measuring instruments properly adjusted. Many models and variations of noise measuring instruments are available and agency people may suggest a list of vendors furnishing the equipment, if such information is needed.

Testing and Analysis of Noise

The testing of noise levels in a factory must be approached broadly so as to include plant conditions and community noise levels. As a practical matter, apparent high-level noise sources are studied for possible engineering or administrative action, and when these efforts prove to be ineffective, protective equipment use can be requested from the agency. A typical high level area is the centrifugal station. The following octave analysis of one such station is shown in Table 21-6.

Table 21-6.
An octave analysis.

Hz	31.5	63	125	250	500	1000	2000	4000
*Spl	92	90	90	90	104	92	85	82

8000	16000	31500	Overall level: 95-100 dBA
82	69	56	

*Sound pressure level (decibels).

Because the decibel is a logarithmic function, different values cannot be directly added or subracted. Figure 21-3 illustrates this function.

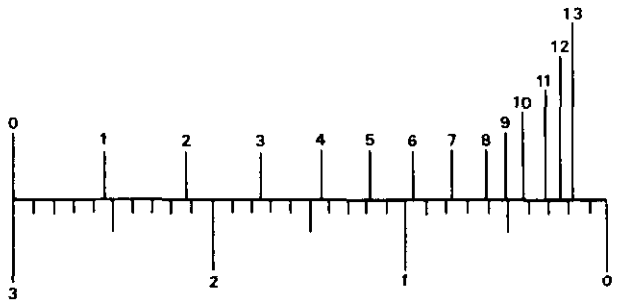

Figure 21-3. Graph for adding sound power and pressure levels.

Figure 21-3 is a chart which can be used to ad both sound power and pressure levels. To use it, subtract the lower from the higher level and find the difference on the top scale. Read the number directly below this difference on the bottom scale and add this number to the higher sound level to get total dB. For example, subtract readings of 95 dB and 90 dB. The numerical difference is 5. The number below 5 on the scale is 1.2. 1.2 added to 95 equals 96.2 dB total. As can be seen from the chart, once sound levels differ by more than 13 dB, they have little effect on one another and the total is, for all practical purposes, the higher reading (20).

On the basis of a survey of selected noise levels by plant areas, several stations were found to be in excess of the 90 dBA allowable level for workers. The following areas were involved:

1. Centrifugal station — 95-100 dBA

2. Centrifugal mixer station — 94-96 dBA
3. Granulator station — 92-96 dBA
4. Lime kiln area — 105-106 dBA
5. Vacuum pump area — 100-106 dBA
6. Air compressor area — 106 dBA
7. Sugar storage bin (scroll area) — 100-108 dBA
8. Boiler house (soot blower) — 120 dBA
9. Boiler house fans — 104 dBA
10. Pulp hammer mill — 96-98 dBA

Noise Control

The regulations dictate that engineering and administrative procedures (personnel shuffling) must be exhausted before authority to use protective equipment is given. There are numerous noise-control products available. Each of these control products has its own special properties just as each noise problem has its unique characteristics. For a successful noise control program, the properties of the noise control product must be properly matched to the special factory problem. Specification charts for noise-control products are published from time to time (11).

The question of high and low frequency noise levels is a matter of concern. Because the human ear can tolerate more low frequency sound than high frequency sound, most noise regulations are based on the "A" weighting rather than the dBB or dBC scale (A, B, C indicating musical pitch). Monitoring and audiometric testing program requirements vary according to state and local agency rules. Record keeping of worker hearing levels can be very involved, and OSHA requires employee audiograms be retained during the period of employment, plus 5 years. Normally a 30-day period is allowed to process new employees and this does reduce the necessary record keeping of seasonal employees, where turnover can be very high.

Liquid Waste Disposal*

The beet-sugar industry is essentially rural. Its factories were originally in rural areas or in districts which were not heavily populated. Many factories were located on or near streams which could furnish a water supply and also accept the waste discharge. Under these circumstances, waste disposal problems were minor until recent years. Now, however, modern civilization is demanding more exacting controls over factors affecting the environment. At this time, many beet-sugar factories which were originally in comparatively open-space areas now find themselves surrounded by residences. Thus, most of the industry is faced with the necessity of making extensive improvements in its factories, for treating and handling waste waters.

The overall problem is complicated by a number of factors. The factories in North America exhibit a wide range of variations in processes, capacities, and types of equipment. There is also extremely wide variation in the water-usage systems of the factories. Additional factors are the widening range of climatic conditions and periods of operation of the factories. At one time, sugar beets were processed primarily in the late summer, fall, and to some extent into the winter on stored beets. These processing plants were either in warm or cool climates. In recent years, through various means, operating periods have been extended to all months of the year under a variety of climatic conditions. These factors prevent the possibility of a

*by W.O. Weckel. Revised by D.L. Stewart

single best solution to the waste water problem applicable for all factories. While many improvements have general application, nearly every factory has its own specific problems requiring special consideration.

An understanding of the concept of water quality is essential to understanding of problems related to waste disposal and pollution prevention. In terms of pollution, water quality is a relative term. Normally water is considered polluted if it is degraded significantly from its original condition, or in relation to the water into which it is discharged. However, in the broader sense, this is not necessarily so. Water which contains nutrient materials which will stimulate undesirable growths in a pure stream or lake may be quite useful as an irrigation water. On the other hand, pure water of very low mineral content can be a pollutant to aquatic life in a saline environment.

A number of water quality factors are important in waste-water consideration. Of these one of the most important is, solids. This term can be subdivided into settleable solids, suspended solids, and dissolved solids. Each of these may be classified as organic or inorganic. The modern concept of "refractory compounds" is applied to those which are resistant to natural or engineered treatment processes. They may range from common inorganic to complex organic compounds, such as synthetic detergents and pesticides. Other water quality factors are: odor, pH, thermal pollution, and microbiological pollution. Many of these are interrelated; the degree of their individual importance varying widely depending upon local conditions.

Waste Water

Regulations

In October, 1972, Congress passed the federal water pollution control act amendments, designed to abate water pollution. One of the objectives of this law was to clean up the nation's waters by July 1, 1983.

This goal reflected deep public concern and also pointed the way for specific action by federal, state and local governments to formulate programs to control water pollution.

To facilitate the administration of the water-pollution control act it was proposed that a permit system for point-source categories be established. This system was labeled the National Pollution Discharge Elimination System (NPDES) and the beet-sugar industry was at the top of the listed categories.

On January 31, 1974, the Beet Sugar Processing Point Source Subcategory Effluent Guidelines and Standards were issued. These guidelines reflected comments by the beet-sugar processors on such factors as climate, age and size of plant. The original proposal of the EPA had been zero discharge, but industry commenters pointed out that zero discharge was not a practical goal because of water gained from beets.

The published rules allowed for several options for controlled discharge, where the sugar-beet processing capacity does not exceed 2300 tons per day of beets sliced, and the soil filtration rate is less than 1/16" per day. Because in many areas soil filtration rates are below this level, the regulations were reversed to make allowance for treated waste-water discharge, to avoid excessive lagoon construction in areas with such impermeable soil. Subsequent questions were raised on the question of requirements of state and local agencies concerned with the quality of ground water.

On January 31, 1974, the following effluent limitation guidelines were published in the Federal Register (14). The fact that these limitations are specifically

made for the beet-sugar industry does not prohibit a state or local agency from imposing more restrictive limits within their specific political realm.

"Effluent Limitations" (14).

(Barometric condenser water only)

English units (lbs/1000 lbs of product). Maximum BOD_5 (Biological Oxygen Demand, 5-days incubation); for any day, 3.3; average of daily values for 30 consecutive days, 2.2. Temperature not to exceed the temperature of cooled water acceptable for return to the heat-producing process and in no event greater than 90 °F.

When other beet processing waters are included the following limits are imposed: T.S.S. max for any day: 3.3 lbs/1000 lbs, and 2.2 lbs/1000 lbs average daily for 30 consecutive days.

Fecal coliform — not to exceed MPN (most probable number) of 400/100 ml at any time. New source standards are more restrictive and again may be superseded by local regulations.

The receiving stream is protected by water quality standards. These regulations establish minimum dissolved oxygen, pH, and temperature conditions in the stream. In colder climates the regulation prohibits discharge under ice. This requires lagooning of surplus water for eventual disposal or treatment.

When waste water is stored in ponds, the bottom and levees must be sealed to protect ground waters. The levees must be constructed according to good engineering practice using a 1 to 4 rise and run cross-sectional profile.

Composition of Waste Waters

Because of the variations described, "typical" values relating to volume and strength of BOD of the various beet-sugar factory waste waters have only an order-of-magnitude degree of reliability. One tabulation of these values is shown in Table 21-7.

Table 21-7. Wastes from Each Process in Beet Sugar Manufacture

	Waste Water Flow/ton beets Sliced (gal)	BOD (ppm)	BOD/ton Beets Sliced (lb)	Suspended Solids (ppm)	Suspended Solids/ton beets sliced (lb)
Flume water	2600	210	4.5	800-4300	17-93
Screen water: cont. diffuser	400	910	3.0	1020	3.4
Press water	180	1700	2.6	420	0.6
Lime cake slurry	90	8600	6.5	120 000	90.0
Condenser water	2000	40	0.67	---	---
Steffen water	120	10 500	10.4	100-700	0.1-0.7

Sampling and Analysis

The determination of biological oxygen demand is the basic tool in waste water analysis. The sampling and analytical methods may be found in "Standard Methods for the Examination of Water and Waste Water" (26), as well as methods for other parameters such as chemical oxygen demand, total soluble solids, and coliform bacteria.

Most agencies have some special modification or preference in methods, so it would be well to check on this matter with the head of the governing agency in the area in question.

Flume Water

As shown in Table 21-7, flume water is normally the waste stream accounting for the greatest volume, and in many cases the greatest BOD load. The flume water of a beet-sugar factory normally consists of the water used for sluicing, fluming, and washing the beets, and in some cases the water from the tare-laboratory operation. The source of flume water or its makeup, in the case of recirculation, varies. In some cases, the makeup consists of fresh cold water from wells or streams. In most other cases the source is condenser water, either from the seal tanks or after cooling in a tower or spray pond.

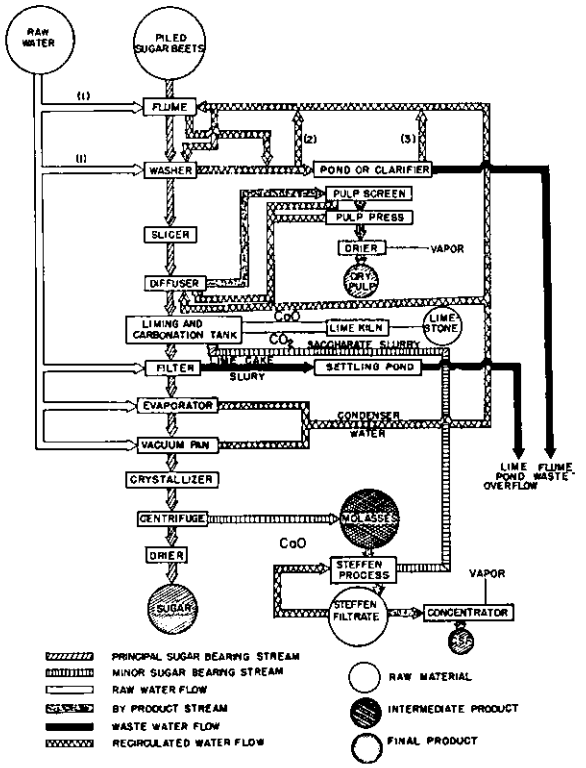

Figure 21-4. Materials flow in beet-sugar plant with "typical" water utilization and waste water disposal pattern.

Because of its large volume and, in many cases, its physical location, flume water generally also receives the miscellaneous small waste streams from the factory. These include excess condensate, cooling water, pump-gland leakage, leaks, and spills. As such, flume water is generally considered the main factory waste stream.

The composition of flume water can vary widely from other causes. In some cases, beets may be handled dry except for a flume which is so short that it is only an adjunct to the beet-washing equipment. In other systems, the flume system may be quite long. Most factories are equipped with wet hoppers for unloading. All of these differences create variations in the volumes and quality of flume water.

The composition of the flume water is greatly affected by the cleanness of the beets received. Since nearly all beets are now harvested mechanically, the amount of soil and trash delivered with the beets fluctuates considerably. Under good harvesting conditions, the amount of dirt tare is small; but, however, when harvesting in wet fields following a rain, the amounts of dirt may be extremely high. Again, after wet fields dry out, dirt in the form of clods is harvested and delivered with the beets. Under some conditions, flume water may reach solid contents approaching a slurry. The tops left on the beets and trash accompanying the beets can also add a large amount of solid organic matter to be handled.

When the beets are in relatively sound condition, the amount of sugar lost to the flume water is small. However, when frozen or deteriorated beets enter the fluming system, they contribute large amounts of soluble organic matter to the water. The leaching loss of sugar into the flume water is related in some degree to the temperature of the water. In some factories, an attempt is made to minimize this effect by using cold, fresh water for makeup. In some cases where condenser water is used for flume makeup, this relatively hot water is cooled over a cooling tower before use in the flumes. When frozen beets are being processed, hot condenser water is usually a requisite to thawing the beets sufficiently to permit handling and slicing.

To meet the goal of zero discharge and regulations regarding ground water, the system must provide secondary treatment with final disposal of liquid waste excess discharge to municipal treatment centers or to an approved irrigation system. Solid waste must be handled by agency-approved methods.

Longmont Project. In 1967 a project was initiated to study the recirculation and treatment of flume water at the Longmont, Colorado, factory of The Great Western Sugar Company, sponsored by the Beet Sugar Development Foundation with assistance from the Federal Water Pollution Control Administration.

Essentially, the project was intended to study a plant scale installation which would provide virtually full recycle of the flume water of this factory. Its design provided for: 1) lime addition to the system to keep the pH at levels high enough to retard bacteriological development, and to neutralize organic acids; 2) screening of flume water to remove large organic particles, subsequently fed to livestock; 3) primary and secondary settling to remove the settleable solids; 4) anaerobic treatment in a deep pond to reduce BOD prior to discharge; and 5) reduction of the discharge rate to a quantity equivalent to the inherent build-up in the system.

A schematic flow diagram is shown in Figure 21-5. The maximum recirculated rate at any one point in the system was 9 000 gpm. The total flow is screened through four Dorr-Oliver DSM, 3-mm screens, equivalent to a standard 10-mesh screen. The particles retained by the screen were mostly organic and were collected and fed to livestock along with the organic matter accumulated at the trash collectors and the tailing scroll. Other installations have been made using vibrating-type screens instead of the DSM screens, which have been found to be subject to substantial erosion by the sand particles in the water.

The flow of screened water was split; one-half returned to the fluming operation without treatment, and the remainder to the ponding system for treatment. The portion treated was mixed with milk of lime to maintain a pH of about 10-11. Following the lime addition, the water flowed to one of two primary settling ponds.

As shown in Figure 21-4, the two primary points were banana-shaped lagoons lying on each side of a secondary pond. Each primary pond was 400 ft long and at an initial, clean depth of 10 ft held approximately one million gallons. Water travel-

ed through only one of the primary ponds at a time, at a velocity of 3 ft/min. The objective of the two-pond system was to permit cleaning of one while the other was in operation.

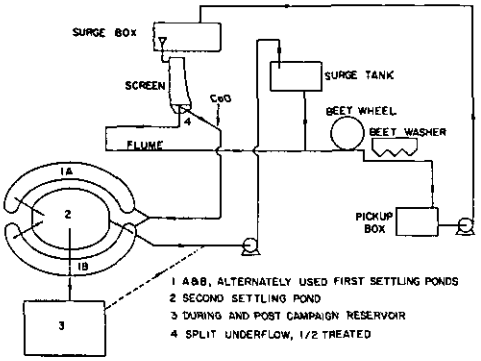

Figure 21-5. Flow diagram of Longmont Project for study of recirculation and treatment of flume water.

The secondary pond lay in the middle between the two primary ponds and at a depth of 9 ft contained about 2.3 million gallons. The maximum retention time in both the primary pond and the secondary pond was around five hours. After the lime-treated water flowed through the primary pond, it discharged into the secondary pond from which it was returned to the flumes.

The system was designed to operate at virtually complete recycle of the water, but provision was made to permit the discharge of approximately 100 gpm of the secondary pond effluent to a deep anaerobic pond. The design volume of this pond was in excess of 7 million gallons at a depth of 15 ft. At the design flow rate, the anaerobic pond had a retention time of 50 days, excluding losses by evaporation and percolation.

Since operation of anaerobic ponds is often subject to odors, attempts were made to avoid this by installation of mechanical surface aerators. Two electrically driven, floating, 5-hp mechanical surface aerators were installed. These were equipped with anti-erosion plates located in a horizontal plane below the aerator to minimize the drawing of other than surface water into the suction of the aerators. With this equipment, it was intended that an even stratum of oxygenated water across the surface would act as an efficient odor suppressor, while at the same time not interfering with anaerobic digestion below the surface layer.

Operation of the system was generally successful from the start, although several difficulties arose. These were mainly of a mechanical nature or related to problems in handling surges in water flows, — common in beet-fluming operations. Remedial changes were made before the second campaign, and the system operated essentially as designed.

Depending upon the cleanness of the beets received, the quantity of mud from the operation will vary. However, it is usually a large volume which can eventually become a problem. In most areas, it is not possible to freely dispose of the accumulated dirt because of the presence of weed seeds, nematodes, and other agricultural pests.

In addition to the pond system used at Longmont and the mechanical clarifier systems in use, several other variations have been suggested or employed. J. M. Henry (19) describes several, including a circular pond system divided into four

segments, and other pond configurations utilizing series flow through two or more ponds.

There is evidence that a significant reduction in the loss of sugar in the beet-fluming operation may result from a virtually complete recycle system. Blankenbach and Willison (5) described operation of a mechanical clarifier, flume-water recycle system used at Manitoba, Canada. High pH values were maintained with lime. The dissolved solids content of the flume water gradually increased through the first six weeks of operation to about 10 000 ppm. It remained at about this level to the end of the campaign. Since there is no fermentation in the water and a high pH of approximately 11.5, they reason that there is essentially an equalization of the osmotic pressures between the water and the beets. If this is correct, the loss of sugar from the beets into the flume water would be minimal. This could result in a significant saving in sugar normally attributed to this cause.

A recently developed method and apparatus for clarification and thickening (see pp 248) has been tested with flume water on a pilot scale). With the use of a polyelectrolyte settling aid, the new settler is estimated to require only about one-tenth of the area of a conventional clarifier for flume water. It appears that by virtue of the shorter retention time using the new settler, combined with the application of the polyelectrolyte, the amount of lime required for satisfactory flume water recycle may be greatly reduced, or virtually eliminated.

The closed loop system developed by the Manitoba Sugar Company suffered from a feature that is common to systems where highly loaded (BOD) waters are ponded; that being the emission of odors during the spring turnover. To correct this the Manitoba people installed an Enviro-Clear mini-clarifier to concentrate the main clarifier underflow to 35% solids. The Enviro-Clear underflow is then pumped with air assistance to a piling ground where the solids are stored in a semi-dry state. The stored solids are relatively odor free (24).

Waste-water systems are generally a source of odor which does not permit self-purification. To overcome this situation, A. B. Sorigona and SSA (Swedish Sugar Association) developed a process specifically for the treatment of sugar-factory waste waters.

The process utilizes the same natural processes that occur in storage lagoons, but under controlled conditions, and with much greater efficiency than the natural process. One of the appealing features of this Anamet® system is that the anaerobic stage is a closed system, and the gases are trapped and led to the process as an energy source. Thus odor emissions are reduced or eliminated.

The process separates the anaerobic and aerobic processes into two distinct steps, which allows optimization. The process starts with a high-efficiency anaerobic contact-reactor, where organic materials (mostly sugar) are converted into organic acids, and finally into methane and carbon dioxide, providing a 70-90% reduction of BOD_5. The remaining organic material, mostly acids which do not cause bulking sludge problems, is removed in the subsequent activated-sludge step, yielding an overall BOD_5 reduction of 99%.

A very important factor is an improvement in the nutrient balance in the system. This is attributed to the fact that the anaerobic treatment does not require a high nutrient ratio because the BOD_5 is converted into methane and carbon dioxide rather than bio-mass, without removing nitrogen and phosphorus thus increasing the ratio of BOD_5 : N P. The desirable ratio for an activated sludge process is BOD_5 = 100; N = 5; and P = 1 (21).

Control of temperature and pH is of major importance in the operation of the

Anamet system to assure the bio-activity necessary. The system is energy efficient in that waste heat is utilized to heat the feed to the system. Also, the system produces methane gas for process energy. This energy is reported to be equivalent to 4.3 tons of oil for 5000 tons of beets sliced/day. Fig. 6 shows the basic system.

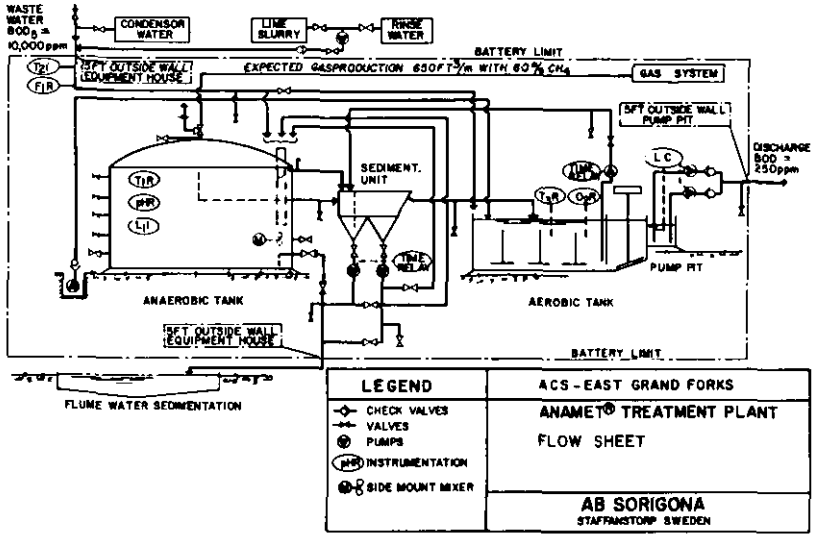

Fig. 21-6

The Braunschweigische Maschinenbauanstalt (BMA) offers a complete waste water system designed to meet the special requirements of the beet-sugar industry. The system consists of the BMA - Brukner classifier, the BMA - Brukner Sedimentation Basin and the BMA Biological Sewage Quick-Purification Procedure. The classifier and sedimentation systems, along with the Biological Sewage Procedure were installed in the Wahpeton and Hillsboro, North Dakota plants constructed by BMA.

These systems were also designed to separate transport water solids by vacuum filtration methods. This solids separation system was the subject of a study sponsored by EPA (9).

Preliminary evaluation of the data indicates the solids separation system requires more sophisticated controls to justify the cost-benefit ratio of the program. The Wahpeton and Hillsboro systems are operating satisfactorily on water treatment, but campaign length and beet quality, plus cold winter weather have caused problems in maintaining system capacity.

The final disposal of surplus waste water is a function of climate, geography and state and local regulations. The disposal options are as follows:

1. Evaporation — This option varies with temperature and rainfall and can cause great variations in the industry because of the geographic dispersion of factories.

2. Seepage — The matter of seepage was the subject of much comment during the promulgation of sub-category rules. It was amended to account for soil porosity and deliberate pond sealing to meet state and local ground water regulations.

3. Discharge to a municipal waste system is an option open to the industry, and according to the industry survey is used by some members but at a significant cost in pretreatment and discharge fees.

4. Irrigation as a disposal method has perhaps more variables than the other three options; because of allowable application rates and restrictions on soluble solid buildup. The method is quite acceptable in desert and semi-desert areas, but is a complicated and costly method in areas of dense soil and short growing seasons.

Lime Cake

Lime cake is almost always handled separately from the other factory wastes: normally by slurrying with water which is pumped to a pond or an area surrounded by levees.

The slurry consists largely of calcium carbonate precipitate plus sugar, other organic matter, and water. In the lime pond, the solids settle out, leaving a liquid phase on the surface. If the pond area is sufficiently large and dilution is kept at a minimum, percolation and evaporation may permit containment within the lime pond. However, it is desirable to keep the water depth shallow in the lime pond to avoid odors. Because of this, it is common to drain off excess water for separate land disposal or to send it to the excess flume water disposal system. The limecake supernatant liquid appears to furnish some beneficial nutrients, such as nitrogen, to the flume water.

Three factories pump the mud from the carbonation filters to the lime disposal area without dilution, thus eliminating the drainage problem. In one installation, limecake is fed into the suction of the pump by a vertical scroll. In the discharge line, two check valves are followed by the injection of compressed air at 10-15 psi to help maintain fluidity of the semi-liquefied mass. Using this system, limecake is transported about 1000 ft to the disposal area. The material flows out of the pipeline much like lava, without producing any liquid disposal problem. The moisture of the limecake is reported to be about 50%.

If limecake is recycled by reburning, conceivably the disposal problem could be decreased. However, neither of two factories now reburning lime has made special provisions for decreasing the limecake drainage resulting from the disposal of the discarded portion of the carbonation cake, but, reburning does reduce the space required by the pond lime.

Steffen Waste

Factories equipped with the Steffen process produce a liquid waste which has a high alkalinity and pH as well as a high BOD or organic content. The solids in the waste, in addition to the lime content from the Steffen treatment, consist of some sugar plus the nonsugars of the original molasses, including various inorganic, and a variety of organic and nitrogenous compounds.

When Steffen waste biologically degrades, it soon loses its alkaline nature and various malodorous compounds are formed. Where this waste is in ponds the odor problems have become acute. Also, when combined with the other wastes, the odor problem is transferred to the combined waste.

Because of the large variety of materials contained in Steffen waste, it has been given considerable study as a source of many products.

During World War I, a number of beet-sugar factories concentrated the Steffen waste and burned the concentrate to produce a crude potash salt for fertilizer.

Feeding and nutritional studies showed that CSF can supplement and partially replace molasses as a cattle feed. It can also act as a partial replacement for the molasses for the molasses dried with the beet pulp. When used as a dried-pulp additive, CSF is normally limited to a solids level, equivalent to something less than the molasses solids with which it is added.

Land spreading is another alternative method of disposal of Steffen waste. This can be accomplished with a minimum of odor production if managed properly. The dilute Steffen waste is spread in a thin layer over a land area which is quite level and divided into small parcels by low levees. This permits feeding the waste onto these parcels in sequence to allow for absorption and drying before further additions. It is beneficial to disc or till the soil between campaigns to enhance its absorptive capacity.

At the present time, there is insufficient experience to predict the length of time Steffen waste can be spread on land without destroying the capacity of the soil to further absorb, or whether soils can be successfully reclaimed after such treatment.

Two studies on biological treatment of Steffen waste were reported in 1968. In one (21), a four-pond system consisting of an aeration pond, a deep digestion pond, an algae pond, and a maturation pond, was studied. Carbonated Steffen waste was fed to the aeration pond, into which compressed air was distributed through perforated pipes. The digestion pond was seeded with sewage sludge from a municipal treatment plant. The algae and maturation ponds were provided with flow inducers to produce surface flow in these ponds as desired. These were operated either continuously or intermittently for a total time of two hours per day. Recirculation from the maturation pond back to the aeration pond was provided.

Different feed rates, the addition of phosphate ion, and various other variables were studied. From this work it appeared that such a system could be used to produce a stabilized waste without the production of significant odors. The cost to install and operate such a system would be fairly high, and unfortunately, under the conditions of the study, no usable product resulted from the treatment system to offset the costs.

In another study on a laboratory scale (8), it was demonstrated that Steffen waste can be treated with various yeasts, algae, and bacteria to produce a potential feedstuff in the process of stabilizing the waste. Successful separation of this material would then offer a potential revenue for the operation. Various specific organisms were studied. The addition of phosphate was also demonstrated to be very beneficial for the growth of the organisms. From this work, a basic scheme was outlined for such a treatment system.

Condenser Water

As shown in Table 21-7, condenser water is one of the largest components of the factory waste-water discharge. Large volumes of water are required in jet or barometric condensers to produce lowered pressures for the operation of evaporators and vacuum pans. Normally condenser water ranges in temperature between 50° and 65 °C, with a BOD of 50 ppm or less.

Where water supplies are plentiful, condenser water has been used on a once-through basis. Disposal has often been directly to a strream or other body of water. In some cases, detention ponds or other means of cooling have been used to prevent thermal pollution. Because of its only slightly degraded condition, condenser water is commonly used for both diffuser supply and make-up, and for flume water make-

up. This latter use is generally necessary in cold climates when working frozen beets. In such use, where flume-water recycle is employed, the clarified flume water is used to provide the condenser water supply. Thus, the passage through the condensers becomes a step of the flume water recycle system.

The practice of reuse of condenser water has increased in recent years (9a). Various means of cooling are employed, such as spray ponds, natural draft, and induced draft cooling towers. The latter are generally necessary in warmer and more humid climates. In most cases, it is not possible to provide recycled water at as low a temperature as the normal primary cold water source. Because of this, the recycle system has provision for increased water use through the condensers.

The use of cooling towers for condenser water recycle usually presents a problem in the growth of slime-producing organisms in the tower packing. In the presence of small amounts of sugar and other nutrients, and the warm temperatures, such growths are difficult to avoid. They are usually controllable by intermittent chlorination of the cooling-tower feed.

Complete recycle of condenser water is sometimes somewhat difficult to achieve in areas where the water is of poor or marginal quality. As recycle is increased, the scaling properties are increased by the concentration of solids through evaporation and increased pH from absorption of ammonia. Although most factories use some type of polyphosphate threshold treatment to prevent scaling, it may also be necessary to reduce the pH with acid. With some waters, a certain portion of fresh water make-up may be necessary.

Condenser water discharge is regulated under the Beet-Sugar Processing Point Source Subcategory (14).

Miscellaneous Wastes

In addition to the various wastes described, there are lesser additional ones. One of these is the gas scrubber water. A high quality water is not necessary but a sizeable volume of water is used. Crane of the British Sugar Corporation (8a) reports the reuse of clarified flume water in the gas washer, following which it is returned to the unclarified flume water portion of this system.

He also reports that selected cooling waters such as those used for cooling turbine oil are recirculated over a separate cooling tower. Many of the other cooling water streams are recycled back to the main cooling tower and reused. Where furnace ash is conveyed with water, a complete recirculation system is reported. A separate settling lagoon is provided where the water is decanted and recycled.

Facultative and Algae Pond Systems. It has been shown that stabilization ponds can be used in place of mechanized treatment processes in the biological degradation of organic waste waters. However, only recently these engineered ponds have been applied to beet-sugar factory wastes. Encouraged by the successful application of these principles in the treatment of other wastes, the Beet Sugar Development Foundation initiated a pilot plant study at the Tracy, California, factory of the Holly Sugar Corporation with W. J. Oswald, C. G. Golueke, and R. C. Cooper as consultants. (28)

In this pilot plant, screened, settled factory waste water was treated in a series of three ponds. These consisted of a 15 ft deep anaerobic pond, a facultative pond 7 ft deep, and an algae pond 3 ft deep, from which the effluent could be discharged and also recycled back to the anaerobic pond. The deep pond was designed with the intention of creating an intensely anaerobic environment at its bottom. The medium depth of the facultative pond was designed so that its surface could be aerobic and

its bottom anaerobic. The third or algae pond was designed to operate at a shallow depth, thus being potentially aerobic throughout its depth.

The three-pond system provided a cycle in which biodegradation was followed by organic growth in the form of algae. The basic reactions of the different phases in the cycle occurring in the three ponds is shown schematically in Figure 21-7.

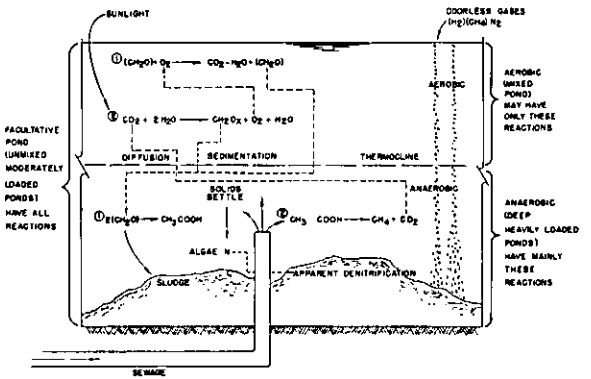

Figure 21-7. Interrelationships of conditions and reactions in ponds ***(from "Engineering Management of Water Quality," P. H. McGauhey, McGraw-Hill, 1968. Used with permission of McGraw-Hill Book Co.)***

In the operation of the ponds, the main series of tests were made with the ponds in the sequence: anaerobic, facultative, and aerobic. The two main factors tested were rates of loading and recirculation. Later, a floating mechanical aerator, similar to the type used in the Longmont pilot plant, was added to the anaerobic pond to provide surface aeration for odor prevention.

As a further experiment, the aerobic pond was provided with low-head, high-rate pumps and a set of baffles to provide a continuous internal recycle mixing. This assists growth of algae and oxygenation by providing for the commingling of the settleable bacterial sludge and the algae in the supernatant liquid. These mixing systems should have a velocity of at least 0.5 ft per second. For the greatest power economy, operation of the mixing pumps should be at night when the dissolved oxygen levels of the algae water are lowest, or if the pH exceeds 9.5, so that CO_2 may be replenished in the supernatant liquid by absorption from the atmosphere, if necessary.

When the pond system was altered to provide mixing in the aerobic pond, the effluent from the anaerobic pond was transferred directly to the aerobic pond with the facultative pond omitted.

Although mechanical and other disturbances resulted in less than perfect treatment operation, the system indicated that beet-sugar factory wastes can be thus successfully treated. BOD and COD were effectively removed in the pond systems tested with the highest removal rates in the anaerobic pond.

As long as algae were present in the aerobic pond, recycle from this pond back to the anaerobic pond was beneficial in the prevention of odors. Without either recirculation or the use of the aerator, there were odor problems in the anaerobic pond.

In the operation of the algae pond it was found that a luxurious growth of algae could be decimated overnight by an invasion of predators such as *daphnia* or *rotifers*. In turn, these can be successfully combated with degradable pesticides.

The influent to the pilot plant contained microorganisms of human origin from more than one source. It is significant that the reduction of coliforms and fecal streptococci was 99.99% in practically all cases.

In addition to the demonstration of a practical waste-water treatment method by the Tracy studies, a field for future investigation is revealed in nutrient removal from waste waters. As shown in Figure 21-5, the anaerobic digestion process provides a means of converting nitrogen to gaseous forms which escape to the atmosphere. This is also referred to by Eliassen and others (12). A further and more complete method of nutrient removal is offered in the production of algae (18). Not only does algae production offer the removal of nutrients, but the harvesting of this material for animal feed could provide an income (22).

In the operation of deep anaerobic ponds it has occasionally been noted that the water turns reddish, varying from purple to vivid pink. Studies conducted on two such ponds by Cooper (8) disclosed the presence of at least two types of purple sulfur bacteria. These bacteria apparently serve as biological deodorizers, using hydrogen sulfide photosynthetically and producing elemental sufur and sulfates. Where these bacteria are present in sufficient numbers, hydrogen sulfide odors are usually greatly diminished or eliminated.

Ion Exchange Wastes

Special processes, such as the Quentin and other ion exchange systems, are not yet in general use and the best disposal of these wastes is not yet known. The regenerant effluent from the Quentin treatment is mainly inorganic salts and may be best handled by the land spreading method described for Steffen waste. A recent installation of an ion exchange process has indicated the feasibility of concentrating the anion-regenerant material for use on dried pulp as for CSF, and the cation-regenerant material is combined with liquid ammonia to produce a liquid ammonium sulfate fertilizer. If this can be handled and marketed in dilute form, a problem waste material can be turned into a profit.

American Crystal Sugar Company at Moorhead, Minnesota, is pelletizing regenerants from an ion exchange system to produce a commercial fertilizer.

Ground Water

To safeguard public drinking water supplies, and to protect public health, Congress passed the 1974 Safe Drinking Water Act (25).

A 1975 survey found that the wide-spread use of high-nitrate fertilizer and extensive feed-lot operations contributed to a contamination of both surface and ground water. The survey resulted in state-set ground water rules. These regulations restrict irrigation and deep well disposal. They also set out construction standards for waste-water storage systems. Monitoring ground water is a general condition of waste-water-disposal permits, where ground quality is endangered.

Liquid Storage

An extension of ground- and surface-water protection is set forth in liquid storage regulations. This includes molasses, thick juice, and liquid sugar as well as fuel, acid, etc.

These regulations require sealing of revetment (ditch) systems to prevent seepage to ground water. The regulations also require revetment volume equal to the volume of the largest tank, plus one foot of freeboard. In areas near population centers or quake areas, the entire volume, plus freeboard, must be provided for in the diked area.

Solid Waste and Hazardous Waste

The Resource Conservation and Recovery Act (RCRA) is the first comprehensive federal regulation of solid waste and amends the Solid Waste Disposal Act of 1965. RCRA was enacted to achieve two basic objectives: (1) protection of public health and the environment and (2) the conservation of national resources.

RCRA provides three major programs to help reach these goals: 1) the establishment of a hazardous waste control program, to be administered by the states, or, where states so choose, by EPA; 2) the establishment of a land-disposal regulatory program in each state; and 3) the initiation and support of resource-conservation programs by state and local governments, to conserve resources and reduce the amount of solid waste requiring land disposal.

The regulations set forth two categories that are of major interest to the beet-sugar industry. These are:

(1) Regulations for the management of hazardous waste (Subtitle C);
(2) Land disposal of non-hazardous waste (Subtitle D) which will phase out open dumps.

Subtitle C sets forth a "cradle to grave" regulation for the disposal of hazardous waste, and compliance by a generator does not insulate the generator from liability in the event of negligence by a disposal contractor.

The selection of a site for a sanitary landfill, and the preparation of the site, must be approved by the proper federal or state agency. A permit for the operation of a sanitary landfill is also required, and in the case of hazardous waste will be limited to specially designated facilities where provision for bonding, closure cost, long-term care, and liability insurance are provided by the site operator.

When a permit for land disposal of non-hazardous waste is granted, a ramp-type system is considered to be most acceptable. Because of a daily sealing of the active or work surface, control of rodents and blowing trash is accomplished, and it is possible to quickly restore the filled area to normal appearance.

The progressive slope or ramp is suitable for a site that has a large depressed area. The trash truck unloads over the brink of the slope, where the trash is com-

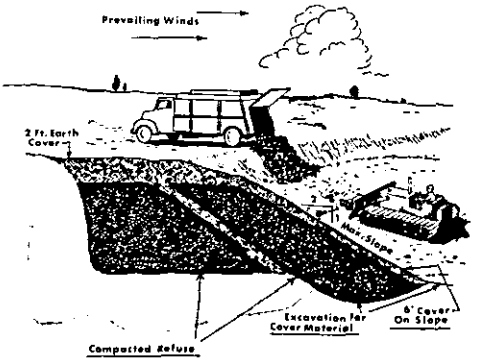

Figure 21-8. Progressive Slope Method of Land Fill Disposal.

pacted by crawler tractor, and then covered with earth previously excavated from the lower toe of the slope. Each day's deposit should be covered with 6 in. of dirt, then compacted, and when final elevation is reached, a 2 ft cover is added with due attention to compaction and drainage. This method is illustrated in Figure 21-8.

Toxic Substances

The toxic substances pollutant effluent standards (13) contained a list of nine substances that were considered dangerous to man and his environment.

Table 21-8. IV. The List of Toxic Pollutants

"Pursuant to section 307(a) of the Federal Water Pollution Control Act, as amended, effluent standards will be established for the pollutants listed below:

1. Aldrin (1,2,3,4,10.10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4,5,8 endo-exodimethanonnaphthalene). Dieldren (1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-endo,exo-5,8-dimethanonaphthalene).
2. Benzidine and its salts (para-para-diaminobiphenyl).
3. Cadmium and all cadmium compounds.
4. Cyanide and all cyanide compounds.
5. DDD. (TDE)1,1-dichloro-2,2-bis(parachlorophenyl)-ethane).
   DDE. (dichlorodiphenyldichloroethylene)1,1,1-trichloro-2,2-bis(para-chlorophenyl)ethylene.
   DDT. (dichlorodiphenyltrichloroethane)1,1,1-trichloro-2,2-bis(para-chlorophenyl)ethane.
6. Endrin. (1,2,3,4,10.10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,5-endo-endo-5,8-dimethanonaphthalene).
7. Mercury and all mercury compounds.
8. Toxaphene (chlorinated camphene).

(FR Doc 73-19018 Filed 9-6-73; 8:45 am)

In addition, the following list is under investigation and will probably be added to the restricted list in the near future.

Table 21-9 Compounds under Investigation

arsenic	ortho-chlorophenol
selenium	beta-naphthol
lead	alpha-naphthol
asbestos	beryllium
sevin	nickel
zinc	antimony
chlordane	heptachlor
lindane	camphor
acridine	methyl parathion
hydroquinone	parathion
	di-n-butyl phthalate

It is evident that several of the toxic compounds have been standard items in beet-factory operation.

Several hazardous substances are currently used through the beet-sugar industry, and are of major concern to those responsible for environmental programs. Among these compounds are PCB, used motor oil, lead acetate, asbestos, mercury, zinc, and nickel.

It would be prudent to review records, interview older plant personnel, and acquire maps and photographs to locate any contaminated rubble that may have resulted from factory operation over the years, so that precautionary measures may be taken.

Compounds which require special scrutiny are pesticides, rodenticides, process additives, herbicides, laboratory chemicals, and possible sources of radiation.

The problem of toxic substances is of great importance in the environmental control program of the beet-sugar industry, because of "third party" liability if injuries happen because of product contamination.

Summary

The environmental clean-up program was thrust upon us by crisis motivated legislation to satisfy a concerned public.

While the rules and regulations were well intended, many of them were not realistic in addressing available technology and the time table for accomplishing the established goals. There is now a campaign to correct these errors but the die is cast, and environmental clean-up must be considered as a significant part of the industry's technology and operating cost.

Glossary of Environmentally Related Abbreviations

ANSI	American National Standards Institute
BOD	Biological Oxygen Demand
BOD_5	Same, determined by 5-days' incubation.
COD	Chemical Oxygen Demand
D	Daily Noise Dose
E	Allowable emission, lbs/hr
EPA	Environmental Protection Agency
ESP	Electrostatic Precipitator
MPN	Most probable number
NPDES	National Pollution Discharge Elimination Administration
OSHA	Occupational Safety and Health Administration
OU	Odor concentration units
PCB	Poly-chlorinated Biphenyl
RCRA	Resource Conservation and Recovery Act
TDS	Total dissolved solids
TSS	Total suspended solids
SCF	Standard cubic feet
SCFM	Standard cubic feet per minute
SPL	Sound Pressure Level

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Chapter XXII
PROCESS CONTROL
The Process Control System*

The process control system consists of an assembly of electric, electronic, and electro-mechanical components designed to assist the economics of the process operation through: 1) improvement of product throughput and quality, 2) avoidance of production delays, 3) elimination of losses, 4) reduction of labor, and 5) elimination of safety hazards.

Control systems have three basic applications: for motors, for equipment and machinery, and for physical and chemical quantities or qualities.

Motor Control

The control for each motor generally consists of the following items: 1) the power supply disconnect and circuit protective device (circuit breaker or fused switch), 2) contactor and overload relays—referred to as a starter, and 3) a start-stop pushbutton station. The circuit protective device and starter form a control module which ususally is physically located in an individual enclosure or grouped with other control modules in a motor-control center (Figure 22-1). Motor-control centers are widely used in the beet-sugar and corn-syrup industries. They provide for the maximum utilization of floor space, minimal overall installed costs, and flexibility of arrangement — for both initial layout, and future changes resulting from expansion or modification of the process.

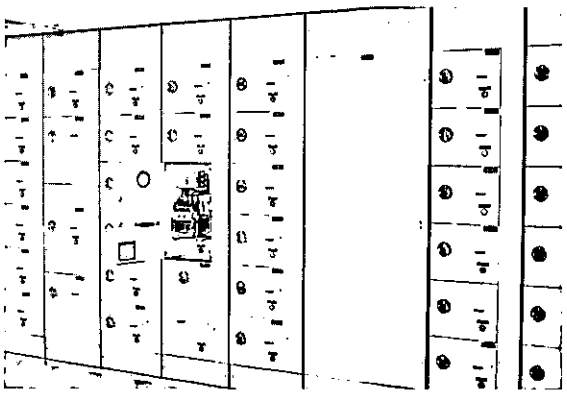


Figure 22-1. Grouped motor control center, at Manteca, California.

The most common motor control is the full-voltage, non-reversing **(FVNR)** motor controller used with a single-speed, squirrel-cage induction motor. The **FVNR** control is applied to pumps and mechanical drives having low mechanical inertias. Other types of motor controls are: **(FVR)** full-voltage, reversing; **(RVNR)** reduced-voltage, non-reversing; **(MSFV)** multi-speed, full-voltage; and **(WR)** wound-rotor induction motor. Multispeed motors and their controls are used where several

*by Arnold J. Daglia, revised by John M. Leavell.

fixed speeds are required, such as for cooling tower fans. Wound-rotor motors and their controls are usually used where speed control is required, such as for beet slicers.

Start-stop pushbutton stations usually are located at the motor, with additional stations located at either the motor control or at one or more operator panels.

Basic Concepts

The basic functions of the motor controller are: 1) the thermal overload protection of the motor, and 2) to control the motor upon command of the operator via the start-stop pushbutton station. But these functions do not protect the driven equipment or the process from abnormal or dangerous conditions, and also do not inform the operator that the abnormal or dangerous condition exists.

Therefore, a well-designed process control system incorporates the following design considerations:

1. *Process interlocking* with devices which function as a result of changes in flow, pressure, temperature, level, torque, or vibration, to name a few.

2. *Sequence interlocking* that ensures that various drives, which are all part of the same process, are either started and stopped in the proper sequence. Sequence interlocking is always associated with material-conveying systems.

3. Process and sequence *interlock bypasses* to permit unprotected operation, to facilitate maintenance or unusual operating conditions.

4. Centralized control of the process—for example a beet-receiving station.

5. The motor-control circuit should be standardized and flexible enough to be used in any application. Such standardization assists in organizing a process-control system, simplifies field installation, trouble-shooting, and specifications for the motor controllers.

Equipment and Machinery Control

The primary function of equipment and machinery controls is to reduce the drudgery and hazard of manual manipulation of multicomponent process equipment or machinery. These controls reduce labor and material costs, eliminate safety hazards, and most importantly, improve product throughput and quality. The almost countless uses for such controls include automatic beet sampling at a beet-receiving station, beet piling on a storage slab, program sequencing of filter valves and sugar centrifugals, and the sequential flame-ignition of carbon and lime-regenerating kilns.

These controls function by *monitoring* the status of process sensors and operator-control devices. The control then takes a preplanned action for each combination of device status. Typical examples of process sensors are pressure, temperature, flow, or level-sensing switches, travel limit or position switches, and timing devices. Operator-control devices consist of pushbuttons and selector switches which allow the operator to select the mode of operation and initiate control sequences. Once the control sequence has been initiated, the operator is free to concentrate his attention to that part of the process or to other processes which are not controlled.

In addition to responding to process and operator inputs, the control should provide the operator and maintenance personnel with *visual* and *audiable indicators* of the status of the control system and possibly the reason for system failure. Indicating lights, and status and alarm annunciators are samples of visual and

audiable indicators. Not all indications available from an equipment control system are needed by an operator. For example, in a filter-control system, a valve position or sequence failure alarm is important to the operator, but the actual position of each valve associated with the alarm is important to the maintenance personnel.

Basic Concepts

To correctly design an equipment control, the following basic concepts must be considered.

1. The desired *control sequences* which are required to control the equipment, and the *potential process problems* associated with each sequence. If the control is for an existing process, current operating and process problems must be corrected or accounted for in the design of the new control to achieve successful operation. For example, plugging chutes, undersized equipment, materials which permit corrosion or scaling, and other items requiring constant operator attention must be corrected. If the problem is a *'nature'* of the process and therefore cannot be easily corrected, then a process sensor must be found that will detect and notify the control that the problem exists so that the control may take a predefined action.

2. Operator and maintenance personnel responsibilities and their levels of experience and expertise must be defined so that the controls will permit both to function effectively.

3. The location of process equipment, operator stations, and control equipment must be known, to insure that operating and maintenance problems *are not* designed into the system.

4. Minimize causes of breakdowns. Most control failures are the result of wear or environment. The failure rate of a device can be estimated by comparing the operating rate of the device in the control system to its mechanical-life expectancy. For example, a device which operates once each minute for 24 hours/day, 7 days a week, would have to have a mechanical-life expectancy in excess of 500,000 operations to last a year. A device which is required to operate in a dusty or damp atmosphere should be sealed or protected in some manner.

The temperature of the air surrounding control devices greatly affects their life expectancy. The life expectancy of electrical-insulating materials is halved for each 10 °C increase in temperature of the material above its rated temperature. For example, an insulation rated 90 °C and operated at 100 °C will have a life expectancy of 50% and if operated at 110 °C only 25% of its normal life. Consideration must also be given to the physical location of the control equipment because of the long-term effects of steam, water, or acid leaks, and mechanical damage from such equipment as fork-lift trucks.

5. Provisions must be made to operate the equipment under abnormal or start-up conditions such as protective interlocking bypassed, or by direct control of each valve in the system.

Types Of Control Systems

Today the control designer or specifier has a wide variety of types of control components to select from. The age of electronics is truly upon us — not only from a technical standpoint, but from initial and long-term costs. Use of the previous 5 Basic Concepts will assist in the selection of one of the following types of control components.

1. *Relay Control Systems* consist of electro-magnetic devices which have one or more moving parts, usually exposed contacts, and usually require more floor

space than any other type of control. In addition they have from several to hundreds of individual wires connecting from contact to contact or terminal boards, and additional wires from the terminal boards to remote process or operator devices. 14 AWG wire is usually used for these types of controls because of their power requirements. A typical relay system is shown in Figure 22-2. The systems are usually easy to trouble-shoot, up to about 8 to 10 devices per system.

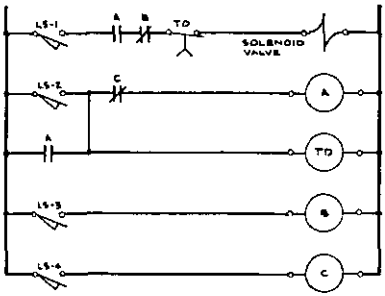


Figure 22-2. A typical relay control diagram including four limit switches, three control relays, and a time-delay relay to control a solenoid valve.

2. *Stepping Switch Control Systems* use a programmable-drum stepping switch plus standard control relays. This type of control is usually applied to processes which have one or more repeative cycles, such as a pressure filter, i.e. *drain, wash, precoat, filter, and repeat.* The stepping switch consists of a drum which rotates through 360 degrees in a predefined number of steps, has a number of separately actuated contacts (anywhere from 10 to 100), and an electro-mechanical drum drive which advances the drum one step at a time. The drum serves as the device to program the status *(open or closed)* of each contact for each drum position. Control relays are used for those functions which can not be easily accomplished by the drum. There is a reduction in the complexity, number of devices and wires, and cost over a relay system accomplishing the same control functions.

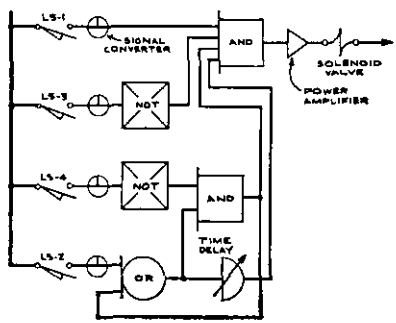


Figure 22-3. Basic logic concept (English logic) is introduced in this static control scheme. This includes the same equipment and machinery control function as shown in Figure 22-2.

3. *Static Logic Control Systems* consist of control elements which do not have moving parts, operate at low voltage and power levels, and require considerably less space than equivalent relay systems (Figure 22-3). They are usually used in systems requiring considerable sequence-type interlocking, or where the control logic is rather complicated, or where relays would not have sufficient mechanical life. This type of control system could be applied to a centrifugal station and has been used to completely automate several beet-piling systems.

4. *Programmable Controller Systems* are based upon the philosophies of their much larger brother – *the Process Computer.* The difference between the process

computer and programmable controller, as well as programmable controllers of various manufacturers, is the *size* and *complexity* of the control problem they are capable of handling. The *hardware* portion of the programmable controller is the electronic circuit boards, power supplies, and input/output circuit components — *process interfaces.* The *software* portion consists of logic statements which are programmed *(inserted)* into the logic-processing unit of the controller by its manufacturer. Inputs are *voltage* or *current* signals which are obtained from remote devices such as pushbuttons, process-sensing devices, or from process instruments. These inputs are monitored by the controller. Outputs are the equivalent to relay contacts in the relay control system, but are typically solid-state devices rather than electromechanical. The functional equivalents of time-delay relays and sometimes counters are incorporated in the logic to provide all of the basic functions of a relay control system.

The control problem is programmed into the controller's logic processing unit by an auxiliary device — *the Programmer.* To program a system, an elementary diagram for the control system is drawn in the controller manufacturer's format. Each process device is assigned an *input number* which corresponds to the physical connection point on the controller. Each process control device (solenoid, valve, starter, etc.) is assigned an *output number* which also corresponds to the physical connection point on the controller. The Programmer is then used to translate the functions shown on the elementary diagram into the controller's logic, and this logic is automatically inserted into the controller's logic processing unit. The act of programming the controller is equivalent to the laborious task of mounting and wiring the relays of a conventional, relay-control panel.

The Programmer is also used to trouble-shoot or modify the system. The Programmer is used to monitor the internal status of each of the controller's input and output functions. In addition, indicating lights are usually provided on each input and output module to assist the maintenance person in troubleshooting the system. Therefore, essentially the same procedure is used to troubleshoot a programmable controller system as would be used on a relay-control system, but the tools are different. Control modifications are accomplished by connecting or disconnecting process devices and reprogramming the controller.

Programmable controllers have almost completely replaced static logic-control systems because of their acceptance by maintenance personnel, cost advantages, and overall simplicity — minimal internal-panel wiring. In addition, programmable controllers are presently cost competitive with relay control systems having more than 10 to 15 relays.Their susceptibilities to electrical interference and component failure have been greatly reduced, and they provide almost infinite mechanical life.

5. *Microprocessor-Based Control Systems* are similar to programmable controllers but are not regimented in their approach to solving the control problem. The *Programmer* is replaced by a device called a *Compiler* which translates logical control statements into the language of the logic processing unit — now called a central-processing unit (CPU). The elementary diagram (Figure 22-4) is now used to define the process device connections to the controller. A **Listing** of logic-control statements and sometimes a logic flow chart and or truth table is used to define the control problem (Figure 22-5). Note that the logic-control statement is easier to interpret than the elementary diagram.

The microprocessor-based control system opens new vistas to the control designer, operator, and maintenance personnel. Very long and complicated process or sequence interlocking contact strings may be easily expressed in logical

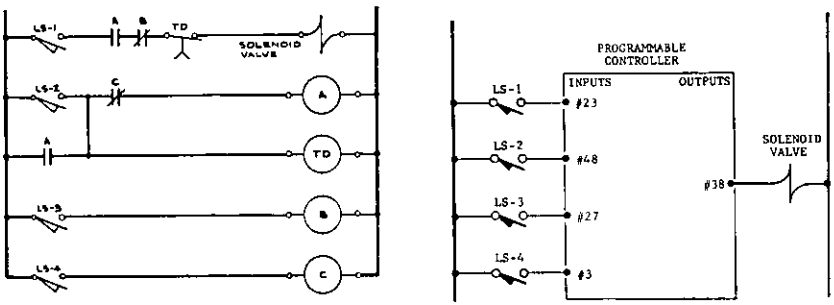


Figure 22-4. Equivalent-Relay and Programmable-Controller Elementary Diagrams.

Logic Control Statement: If Inputs (LS-1, and LS-2 for 5 seconds, and not LS-3, and not LS-4) then Output (Solenoid Valve).

Where:

LS-1 = Input No. 23
LS-2 = Input No. 48
LS-3 = Input No. 27
LS-4 = Input No. 3
Solenoid Valve = A.C. Output No. 38

Figure 22-5. Typical Logic-Control Statement.

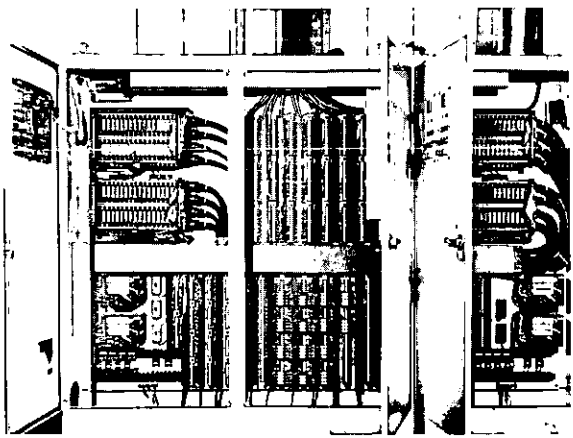


Figure 22-6. Four microprocessor control system.

statements or truth tables. Control actions may also be based upon mathematical computations. The number and type of control devices required by the operator to control and monitor the system can be reduced. For example, a single, multicharacter, alpha-numeric display may be used to display present sequence step number, time remaining on the step, the time setting for each timing function in the system, and the present value of calculated values. The operator would use a thumbwheel switch to select the desired function to be displayed. The same display could be used to inform the maintenance personnel of the reason for a system failure — the valve which failed to reach or stay in the required position.

Shown in Figure 22-6 is a microprocessor system consisting of 4 individual microprocessors. Each microprocessor is responsible for the actuation and continuous position checking of 30 valves through a 66-step, control sequence. In addition, 12 valves, common to the 4 microprocessors, are controlled by each microprocessor during several of the steps in the 66-step sequence. Digital interconnections between the microprocessors are provided to permit each microprocessor to determine that the 12 common valves are not in use prior to acquiring control of these valves. Only two conventional relays per microprocessor are used to provide a-c power sequencing and system-failure indication. Sequencing between steps is initiated by either operator devices, process interlocks, or software timers. The manual sequencing of steps, step time-value changes, system failure indication including which of two valves are out of position, and system status are available through the small operator's panels on the doors of the enclosure. Note that most of the panel area is occupied by terminal boards.

Control Consolidation and Centralization

Progress in the chemical and food processing industries lies in the consolidation and centralization of process controls. The sugarbeet process is well adapted to such consolidation, and the benefits to be gained include manpower savings, more efficient maintenance and breakdown repair, stabilization of product throughput and quality, and improvement in coordination between the unit beet-sugar processing areas.

Consolidation and centralization fall into three classifications: 1) the unit-process area, 2) the control application, and 3) the type of operator panel (Table 22-1).

Note that control consolidation techniques are not limited to a central control console, but the central console is just one of the arrangements allowing effective use of this approach. The central console does not eliminate the need for consolidation of motor-control centers, equipment controls, and instrument-control systems.

When consolidating into a centralized-control unit, careful study must be made of the operator duties. The distinguishing differences between *local, satellite,* and *central control consoles* are complexity, size, and cost; however a primary dif-

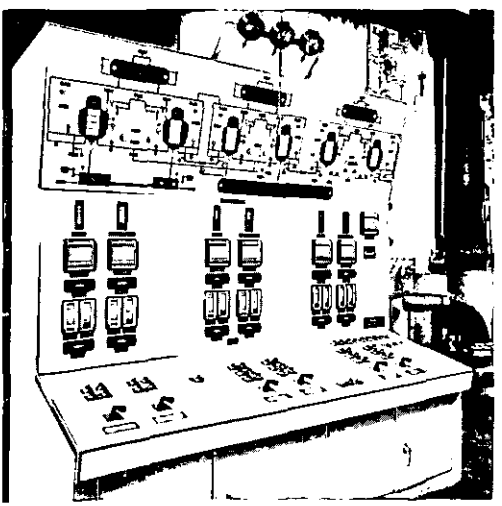


Figure 22-7. Vacuum pan satellite panel, Chandler, Arizona.

Table 22-1 How to Consolidate and Centralize the Process Control System

Consolidate and Centralize by	Designations of Unit Process Area, Control Application, or Type Operator Panel	General Remarks
Unit Process Area	1. Beet receiving 2. Beet storage 3. Beet handling and cleaning 4. Diffusion 5. Saturation 6. Lime kiln and Slaker 7. 1st Carb. Vac. Filtration 8. 2nd Carb. Filtration 9. Juice Evaporation 10. *Continuous Absorption Process 11. Std. Liquor Filtration 12. White & Intermediate Sugar Crystalization 14. Raw Sugar Crystalization 15. Product Granulation 16. Product Molasses 17. Pressing & Drying Pulp 18. Bulk Sugar Storage 19. Bulk Sugar Loading 20. *Sugar Packaging 21. *Liquid & Invert Sugar Production 22. *Steffen house 23. Utilities *May not be provided.	These 23 unit process areas provide natural dividing lines for systematic layout of motor, machinery and equipment, and instrumentation controls. The unit process areas provide a natural means for systematic layout of satellite operator panels and modular construction for the central control console. To determine the extent of contralization, the problem is one of concluding what unit process areas are going to be consolidated at the central console and to what degree. To arrive at these conclusions, the overall process must be extensively reviewed by unit process areas to establish consolidation alternates.
Control Application	1. Motor Control	To the extent practical, assign a motor control center for each unit process.
	2. Equipment and Machinery Control	House controls in individual cabinets or in common cabinet by process area.
	3. Physical and Chemical Quantities and Qualities Control (Instrumentation)	Same comment as above, but when combining pneumatic instruments and electric controls in common cabinet, provide barrier between them.
Type Operator Panel	1. Local	Locate near equipment being controlled or in immediate vicinity of its process unit area.
	2. Satellite	Use systematic layout; e.g., identify clearly relays for east pulp drier and west either with dividing barriers or other means.
	3. Central	Use building-block and modular layouts; e.g., process areas 3, 4, 7 locate in central Sec. 1; areas 5 and 6 in Sec. 2, etc. Provide at least one electric and pneumatic terminal box in each process area for interconnections between field and unit sections of central.

ference exists in the job descriptions of the operating personnel. *Local operator control stations* provide field operator control of individual equipment. The *satellite operator panel* (Figure 22-7) places coordination responsibility on its operator for only that portion of the process. Such panels may be used for the pulp drier, the vacuum pan, the centrifugal, and the sugar-product handling areas. The *central control console* (Figure 22-8) requires that its operator be responsible for the overall process coordination. He must make and carry out decisions and advise operators at satellite panels of changing process conditions.

To permit the central-control console operator to efficiently carry out his duties, the console should contain only those control devices and information displays needed by the operator to coordinate and control the overall process. The satellite-control panel then contains the control devices and information displays required by the local operator to control the unit process. Control devices may consist of motor start-up pushbuttons, motor and equipment operating-mode selector switches, and process-instrument controllers. Information displays consist of annunciators, motor-running indicators, and process-indicators and recorders. For example, central is only concerned with the supply of beets available for production, and thus only the *low* and *high* beet hopper levels need be provided. If the beet-hopper capacity is small, central may also need the status of the beet washing and conveying systems. Information from the vacuum pan may consist of pan feed-tank levels, vapor pressure, and pan status such as *charge, concentrate, boil, and strike.* From a process unit under automated operation, the central control console normally is provided with alarm indications of cycle failure, or status information. For a standard liquor filter installation, such items might be *off steam,* and *high differential pressure.*

The concept of consolidation and centralization is easy to implement during the design of new or major factory renovations. But even if a process area is to be

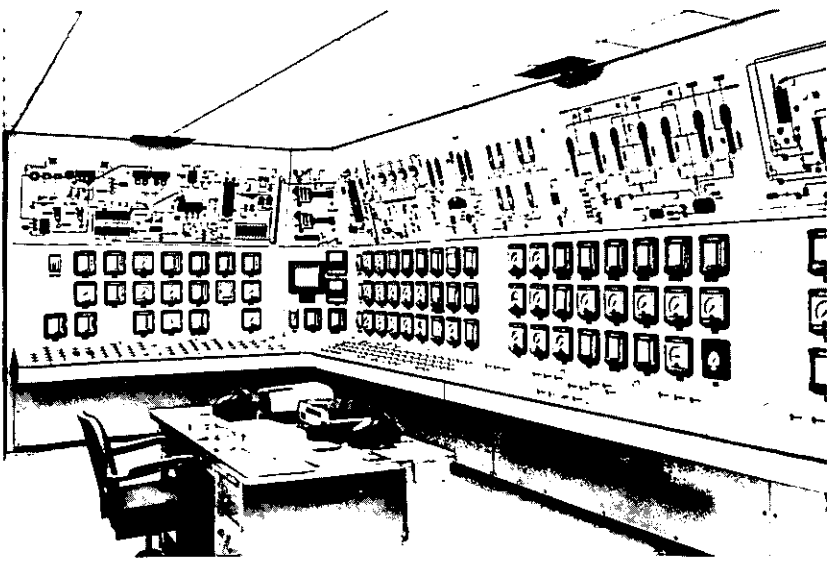


Figure 22-8. Central control center operator panel, Manteca, California.

modernized, these concepts should not be overlooked since they will make it easier to centralize in the future.

Basic Concepts

1. The control of motors and knowledge of their status is a primary function of the central-control concept. Status includes the conditions of *motor running, standby ready to run,* or *not ready to run.* The not ready to run status could result from any of the following conditions: a) motor-starter circuit-protective device open, b) open control-power fuse, c) tripped thermal-overload relays, and d) open motor, drive, or personnel safety interlocks. The functions of start-stop control and status indication can be referred to as **"Motor Monitor and Command Control."**

2. Field installation should be economical. This requirement complicates circuit design when motor start-stop and status functions are consolidated. For example, if conventional 110- or 440-volt control methods are used, a minimum of five 14-AWG control wires must be interconnected between the field-motor controller and the remote operator panel. Eight motors would require 40, 14-AWG wires in a 2 in. galvanized steel conduit.

Shown in Figure 22-9 is one of the many motor monitor and command control systems which not only meets the two basic concepts above but also meets the Motor Control-Basic Concepts. This circuit provides a number of advantages.

For inherent personnel safety, low voltages are used for the remote-control devices. The motor start-up control functions are provided with +24 V dc and −24 V dc; 12 V ac is used for the annunciator input control logic for motor status information. The low power levels required by these circuits allow the use of 18-AWG control wires, and this considerably reduces installation costs compared to conventional 110- and 440-volt usage. Ten motors would require a 50 conductor, 18-AWG cable in a 1.25 in. galvanized steel conduit.

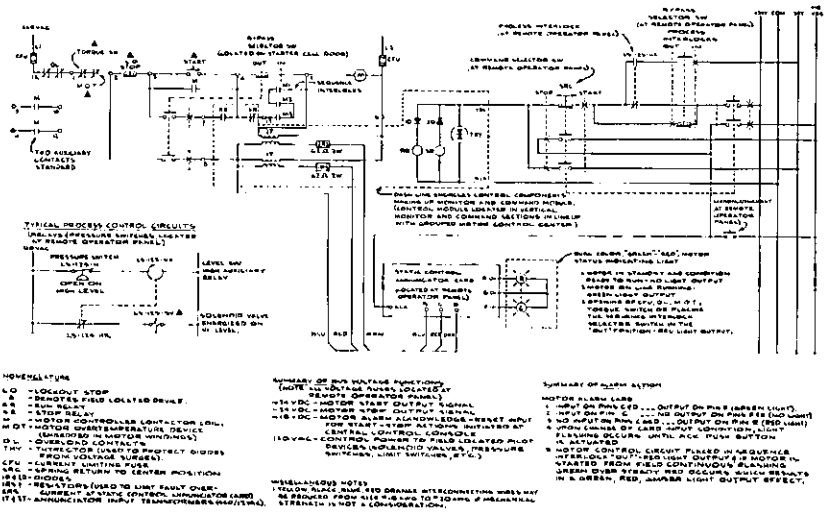


Figure 22-9. Monitor and command circuit for full-voltage nonreversing motor control application.

In addition, the circuit in Figure 22-9 is simple and reliable. The solid-state motor status annunciator card, diodes, and monitoring transformers have no moving parts. The circuit is standardized to the extent that FVNR starters can be requisitioned from fractional to several hundred horsepower capacities by computer code numbers. The circuit design is quite flexible in its process and sequence interlock capabilities. A pressure switch is used to provide the process interlock, and it senses the 3-15 psig output of the pneumatic field transmitter. Field installation is simple, since the individual 18-AWG wires in the 50-conductor cable are each color coded. This coding is readily available in this small-sized wire, but is difficult to secure in the 14-AWG wire size used for 110- and 440-volt circuits. Errors in field installation are greatly reduced, and installation time is shortened through the use of color coding. If information on motor status is not desired, transformers 1T and 2T may be eliminated, and also the associated blue, orange, and red interconnecting control wires between the field-grouped motor-control center and the remote operator panel.

3. All primary process instrument loops should be controlled, indicated, recorded, and alarmed at the central-control console. Instrumentation for unit or satellite processes which do not warrant a satellite-control panel may also be located at the central-control console.

4. The status of satellite processes or equipment controlled from a satellite-control panel should be indicated or alarmed to ensure that the central operator is aware of remote problems which may affect his future decisions.

5. The design should promote rapid failure diagnosis and repair. Standardization of control circuits and devices, color coding of interconnecting wiring to field mounted pilot devices, and color coding of interconnecting wiring between central and satellite panels all aid in this goal. A systematic layout of control components is essential. For instance, all relays applying to beet receiving should be installed in one cabinet or area and relays applying to beet storage in another cabinet. Each unit process or at least individual sections of central and satellite control panels should have their individual control power protection and disconnects to minimize the effect of trouble-isolating procedures. Labeling control devices with an abbreviation of its function instead of CR1 or TR1 assists the maintenance personnel in understanding and troubleshooting the control system.

6. Some type of a process graphic display should be provided to assist the operator in decision making. It is also an excellent training aid for new or junior operators.

Control System Startup

Process equipment will rarely immediately work in complete harmony with its control system because: the complexity of the equipment makes it difficult to anticipate the operating conditions; the control may be complex and engineering for complete perfection may be economically impractical; human errors enter both into the equipment and the control itself during design, manufacture, and installation phases; and a multitude of field adjustments may be required. For example, it is often necessary to prepare time-cycle charts which show the positions of various operating parts during the cycle. In other cases, as in instrumentation, the action under control may be connected with up-and-down process conditions. This requires assumed first-setting controller adjustments, as well as final tune-up adjustments in actual operation.

After the process equipment and its control facilities are installed, the checking out phase is reached. If this checkout is not done before operation, or if it is not done methodically, the results can be disastrous. Serious equipment damage can be prevented by the proper settings of temperature, pressure, torque, or of an overspeed switch. These settings should not be assumed correct, but should be physically checked for confirmation. Errors which have been overlooked in design, fabrication, or installation can often be caught during the checkout period. If these errors are located and corrected before operation, efforts during startup can be focused on meeting the major objectives of production capacity. If well thought out and organized simulated test runs are made in the checkout program, many basic system errors can be located prior to starting actual operation. Where possible the control system and equipment should be operated under design conditions. During the checkout, the controls and equipment are analyzed as an integrated and coordinated unit.

A control system checkout such as might be made in a new factory before operation, using a central control console, is detailed in the following section.

Summary of Checkout for New Processing Plant

A. Electrical

1. All pilot devices such as pressure switches, level switches, flow switches, and position switches are to be set in accordance with design data and operated to see if they work.

2. All process drive motors are to be started and stopped from both the central control console and the field. As this is done, each motor alarm indicating point is to be checked for proper action at the central control console graphic display. The correct direction of motor rotation shall be confirmed.

3. All process and motor sequence interlocks are to be tested to confirm that they work according to design.

4. All timing device settings are to be physically checked to confirm that they are in accordance with the recommended first-trial settings indicated on the design drawings.

5. All programming controls at the central control console, satellite panels, local operator stations, and motor control centers are to be physically run through their step sequences. Proper action is to be proved from the design programming charts.

6. Automatic start for stand-by equipment or diversion of material handling flows should be tested for proper operation.

7. All high- and low-speed limit settings for adjustable-speed motor drives are to be actually confirmed with the settings indicated on the design drawings and the motor name plates. In cases of automatic-speed control, the input reference signal is to be simulated and the motor output speed plotted at 0, 25, 50, 75, and 100% of its speed band. The latter will confirm that the speed regulating devices are correctly adjusted. If there are speed indicators, they are to be checked for proper calibration at this time.

8. All operator devices located at the central or satellite panels or at the local operator stations are to be actuated to prove that the proper control action occurs.

B. Instrumentation

1. Each control valve is to be stroked for proof of operation.

2. Each transmitter shall be given 0, 50, and 100% simulated inputs and the applicable controllers, indicators, and recorders checked for proper calibration. Where practical, all inputs are to be at the primary element. Modulating-type control valves are to be checked for proper travel positions at this time.

3. Ratio relays, high- and low-limit relays, high and low selectors, and damping functions in instruments shall be set in accordance with the design first-trial settings.

4. Cascade control loops are to be checked for satisfactory operation.

5. Thermocouple applications are to be checked for proper wiring connection and calibration.

6. Field adjustments should be made of special pieces of equipment such as densometers, magnetic flow meters, pH meters, and air-to-ground sonar equipment in strict accordance with manufacturers' instructions.

In insuring a successful control system checkout, the more significant problems requiring attention are scheduling, personnel training, and the provision of sufficient personnel for the checkout. The proper time to start a control system checkout is set by the completion dates of the mechanical, electrical, instrumental, and other projects. The amount of time available for the work is a function of the start-up date of the factory. The project control engineer must himself estimate the amount of time required for the checkout. His estimate is more realistic because of his familiarity with the control system.

It is always necessary to have a training program for factory electricians and instrument technicians. Not only are their services required during checkout, but also for future control facilities maintenance. The success of the process control facilities depends on the ability of these men to diagnose and repair. One or more seminars can provide familiarity with the control systems prior to the actual checkout.

In starting up a large process control system, the problem of obtaining sufficient technical personnel is important. Because this is closely related to the project and to the factory, it can best be solved by the responsible project and factory personnel. A team effort using control and mechanical design engineers, process operators, electricians, and instrument technicians can provide an excellent checkout, lead to a better understanding of the operation by all, and result in an expedited checkout and a more orderly process start-up.

The Process Computer

Increasing fuel costs will eventually force our industry to use process type computers to increase the thermal efficiency and economics of the various beet-sugar processes. The control of dilution, the management of heat, and the determination of incremental cost of production vs. product worth are all prime targets for computer control. The process computer is not intended to replace the pneumatic and electronic instruments which measure and control the individual process variables, but rather to manage these variables in such a way as to *maximize* the quality of these process variables.

The advantages of the process computer are:

1. **Consistency in the approach to the process problem.**
   Once the mathematical relationship *(algorithm)* of the significant process variables has been defined the computer will consistently solve the algorithm for the values of the process variables to be controlled.

Therefore, the operator is not required to have a detailed knowledge of the particular process to operate efficiently.

2. **Ability to solve complex process problems.**
   As the number of variables in a problem increases, man's ability to correctly solve for the optimum solution decreases rapidly, whereas the computer's ability is directly associated with the correctness and completeness of the control algorithm.
3. **Ability to remember and project the effects of process variable changes or upsets.**
   The effect from changes in process variables may not be recognized for seconds, minutes or possibly even hours. As the time between *change* and *result available* increases it becomes much more difficult, if not impossible, to use pneumatic or electronic instruments to control the process whereas the computer does not have a physical time delay limitation. Process variable *value* and *real time* become a data-set to be acted upon some time in the future depending upon process-delay time *(transport time)*. Unless unit process areas are amply buffered from one another, a process upset in a unit process migrates both upstream and downstream —affecting these unit processes also. But ample buffering is not always possible or practical, i.e. the effect of variable steam demand by the pans on the operation of evaporators and boilers.
4. **Ability to maintain data banks and produce reports, trends, variances, or rolling averages.**
   Our ability to project the future is directly related to the quality of historical data.
5. **Ability to optimize operating parameters based upon variable cost factors.**
   For example, as diffuser draft is increased sugar losses are decreased; but evaporation, chemical, and pumping loads are increased. The question is at what draft does the incremental cost of evaporation, chemicals, and pumping *exceed* the incramental cost of diffuser losses. The answer changes from day to day based upon changes in the cost of energy and chemicals, and value of the product.

The disadvantages of the process computer are:

1. **Equipment (hardware) and programming (software generation) costs.**
   Hardware costs are usually in addition to the instrumentation costs and therefore must be completely justified by reductions in operating costs and losses. Electronic instrumentation must also be used instead of the more familiar pneumatic instrumentation to provide compatible process variable signals. Software costs are somewhat different — in that process control knowledge must be either given to the operators or systems which control the process.
2. **Control system complexity.**
   After the initial supervisor, operator, and maintenance personnel training, the computer-controlled system is usually less demanding on the operators or supervisor but more demanding upon the maintenance personnel. Process instrumentation must be well maintained to permit the computer to effectively control the process.

Although there are several types of computers, we in the sugar industries are interested in the digital computer. The primary difference between digital computers used for *data processing* and *process control* is the additional hardware which permits the digital computer to monitor and control the process *(Process Input/output): Digital inputs, digital outputs, analog inputs, and analog outputs.* Once the process has been mathematically modeled, the algorithm may be programmed on either a data processing or process control computer. If a data-processing computer is used, the process variables used by the algorithm must be manually entered by the operator each time a solution is required. The operator is also responsible for making the resultant process setpoint changes. Note that although a data-processing computer may be used for process control, the benefits of computer control may be lost by operator errors in reading or inputing data, failure to accurately make the setpoint changes; and, most importantly, timely use of the algorithm to permit the algorithm to follow the progress of requested setpoint changes. The on-line process computer does not require the attention of the operator to control the process and therefore is more likely to achieve the predicted return-on-investment.

Two types of process control are the **Analog Supervisory Control (ASC)** and the **Direct Digital Control (DDC)** systems. The primary difference in the two systems is that the **ASC** system controls the setpoint of a typical analog controller which results in the error and dynamic response of the loop being controlled by the manual adjustments of gain, reset, and pre-act. In the **DDC** system the control algorithm is responsible for controlling the error and dynamic response of the loop which may be of considerable importance in some loops.

The size and cost of process computers are directly related to size of the task they are programmed to accomplish. A small micro-processor system with process I/O is presently being applied to a double-effect evaporator. Small to large process computer systems complete with standard control software packages are available from the major analog instrument suppliers.

FUTURE TRENDS

In the years since the last edition, the greatest advancement has been the change in the Price-Performance ratio of electronic controls and instrumentation. The use of Large Scale Integration (LSI) techniques has greatly increased the reliability, serviceability, and applicability of electronic-control systems while decreasing their relative costs. Other industries have pioneered and justified their use, so we may also approach these systems with confidence. The prime targets are pump drives, combustion, dilution, the management of heat, and process optimization. The application of these systems requires not only a detailed knowledge of the operation of the process control system but process knowledge. The control system cannot be superimposed on the process but must be coordinated and integrated into the process.

Instruments*

[The scope of this section is such as to familiarize the reader with some of the many instruments and types of equipment used today in the beet-sugar industry in North America. A complete description of all of them would fill a large book; therefore only a few have been chosen, and their functions are shown. The text is

*by G. C. Kunkel, revised by C. P. Chua

also presented in simple language, and professional terminology has been sparingly used. Process descriptions are short and limited. This section is designed for the novice to the industry, and not as reference for the expert.]

Basic Measuring Instruments

A. Thermometers

1. Metal Expansion Thermometer. Its operation is based on physical expansion of the metal with temperature increase. This expansion is indicated by a dial needle, through mechanical linkage.

2. Bimetallic Thermometer. This operates on the same principle. Two dissimilar metals are welded together to form a "sandwich," and since each metal has a different expansion coefficient, greater mechanical movement results. These are widely used.

3. Electrical Thermometers. a) the *resistance thermometer* depends for its operation on the change of electrical resistance of a metal with temperature change, the change being proportional to the temperature change. Figure 22-10 is a diagram of a single-arm unit.

b) *Thermistor thermometer.* Thermistors are semiconductors of ceramic materials, made by sintering mixtures of metallic oxides. Depending on the mixture and type of metallic oxide, the resistance can be made from a few up to 100 megohms. All initial resistances are measured at 25 °C. The resistance increases as the temperature lowers.

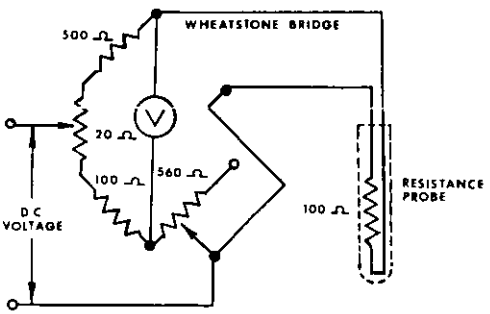


Figure 22-10. Wheatstone bridge type resistance thermometer.

c) *Thermocouples.* A measuring junction of two different metals is exposed to the temperature to be measured. The other junction of the two metals is kept at a constant temperature. The resultant voltage through the circuit is measured, which is proportional to the temperature difference between the two junctions.

d) *Thermal elements.* These are liquid-filled capillary tubes or bulb probes. The expansion of the liquid activates a diaphragm, bellows, or spring within a mechanical indicator.

B. Measuring Pressure

1. Spring Gauges use the principle of the pressure of a spring to oppose the force of pressure to be measured in a sensor.

2. Capsule Gauge. This has as primary element a capsule attached to a capillary tube, and a pressure transmitting device, such as a diaphragm, leading to an indicator.

3. Bourdon Gauge. The primary element is a semiflat, circular tube. One end is fixed. The free end moves as the internal pressure changes. The free end is linked to an indicating needle. Such actuators are frequently the essential elements of pressure-actuated switches. This type of gauge is the most widely used.

4. Diaphragm Gauge. This is a combination of a Bourdon tube and a diaphragm assembly, and it is used to measure pressure in corrosive media. The lower side of the diaphragm, made of corrosion-resistant metal, is exposed to the liquid or gas medium to be measured. The upper side and the connecting tube is filled with suitable oil. The pressure change of the oil actuates the Bourdon tube.

5. Spiral Tube Gauge. This is very similar in principle to the Bourdon gauge. A flat, ribbon-like tube is wound in a helix. The helix is mechanically connected to the meter movement.

C. Level Gauges

1. Float Gauge. A float is attached to a wire rope, which actuates an indicator.

2. Weight. The weight of the material in the tank may be used as measure of tank level, if the density of the liquid is sufficiently constant. There are also level-measuring devices which use the weight of liquid column above the sensing element.

3. Nuclear Radiation. A method derived from nuclear technology depends on the interference of the medium with a beam of radiation from some radioactive isotope; frequently of cobalt, or cesium.

4. Capacitance Probes. The action of this electronic device is indicated in Figure 22-11.

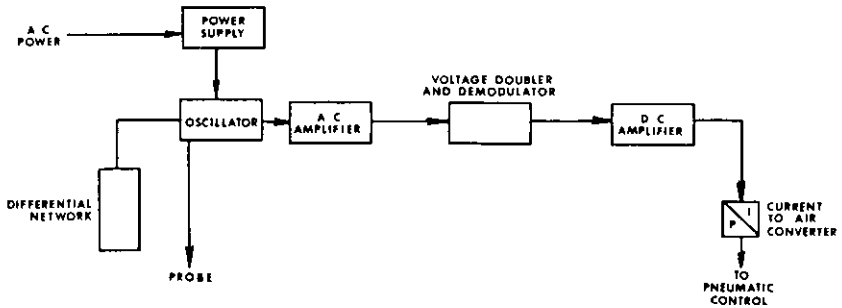


Figure 22-11. Block diagram of electronic circuitry of a capacitance probe.

The power supply furnishes the electronics circuits 24 V dc. The oscillator excites the capacitance probe, which is insulated from the medium in which it is placed. The insulation plus the dielectric constant of the medium makes up the total dielectric constant. The metal tank makes up the other plate of the capacitor. This capacitor is in a feedback loop to the oscillator. The frequency of the feedback will

vary depending on the dielectric properties of the medium. This variation is fed into an a-c amplifier, where it is amplified so that a voltage doubler is driven, which doubles its amplitude and frequency. The demodulator disposes of all ac components, and the remaining dc current is amplified in the dc amplifier which drives a current-to-air converter. The differential network establishes a bias, and also stabilizes the oscillator so that its amplitude and frequency will not change with the temperature.

5. **Air-bubbler System.** A tube inserted into a liquid that measures the hydrostatic head by back pressure created in the tube, with the air entering from the top and bubbling out from the bottom.

6. **Pressure Capsule.** The principle described in B. 2 is frequently used as a measure of tank level.

7. **Delta Cells.** "Delta" indicates differential, in this case differential pressure. These cells are used in connection with bubble tubes. They can be direct sensing elements, when the pressure side is exposed to the medium, and the other side is vented.

8. **Diaphragm.** A metal diaphragm with force-balance of hydrostatic head acting on one side and air pressure on the other side, with a nozzle-bleed system.

9. **Ultrasonic Detector.** Depends on travel time of ultrasonic energy between the electronic transmitter-receiver and the surface of the liquid or solids. The electronic system converts the impulses into measurement of level.

D. Density Measurement

All of the devices in section C can be used to measure the juice density, if the level in the tank is held constant. In addition:

1. **Hydrometers.** This is a float with calibrated graduation marks, indicating its depth of immersion in a liquid, which is inversely proportional to the density of the liquid.

E. Flowmeters

1. **Magnetic Flowmeters.** These convert the rate of flow of the liquid being metered, into an ac voltage of the supply line frequency. A conductive fluid flowing through a measuring pipe section, which is surrounded by a magnetic field, and which is excited by 120 V ac 60-cycle current, develops an ac current (eddy current) in the fluid. This is then picked up by a pair of electrodes, mounted 180° apart in the wall of the flowmeter. The impulse is transmitted to an electrical measuring instrument.

2. **Turbine (Rotary Vane) Flowmeter.** This uses a magnetic pickup coil. The meter will accurately measure fluid-flow rates, whether or not electrically conductive. The edges of the metal turbine or vane, passing through the magnetic field of the pickup coil, induce an electrical pulse; the rate of pulsation is proportional to the rate of flow.

3. **Orifice-Plate Flowmeter.** This consists of a flange with a pressure tap upstream from the orifice plate, the orifice plate, and a flange with a pressure tap downstream from the orifice plate. The pressure difference between the taps is a function of the flow rate.

4. **Pitot Tube Flowmeter.** The operation of a Pitot tube flowmeter is basically dependent on differential pressure. Two tubes of small diameter protrude into the pipe at some distance from each other, usually to about the center of the flow. One is slightly curved toward the direction of flow, while the other is curved the opposite

way. These small tubes do clog up very easily, and a small amount of dirt will change the differential pressure. Maintenance is costly and reliability is not good.

F. pH Measurement

pH measuring systems are analytical systems which measure the hydrogen-ion activity with considerable precision and accuracy. Such systems consist of a) a glass or antimony electrode, b) a reference electrode, and c) a thermo-compensator, to correct for the effect of changing temperature.

G. Electrical Conductivity Measurements

Such measurements are made using a balanced Wheatstone electrical conductivity bridge. The bridge consists of three known resistances, and one unknown, "the sensor resistance," which is the resistance of the fluid and probe to be measured. The bridge principle is shown in Figure 22-10. There is also a source of ac current to excite the bridge, and electronic circuitry to measure the potential difference.

H. Measuring Moisture in Gases

1. Hair Hygrometer. This measures the moisture content of a gas through the variation in length of an animal hair, when it has reached moisture equilibrium with the gas.

2. Lithium Chloride Detector. This is a standard item in air-drying equipment. A small purge air stream is diverted through a cell containing this chemical. A change in color indicates a change in the wetness of the air. Dry air is essential, *when used in modern pneumatic control equipment.*

3. Semiconductor Detectors. These relatively new devices involve the diffusion of a semiconductor material into a substrate in a grid pattern, similar to a strain gauge. Since the humidity-sensitive portion of the sensor is restricted to the surface, moisture is sorbed by ADsorption instead of ABsorption. The result is a very fast response to change in humidity. The grid resistances vary from 30 ohms to 100 megohms.

I. Measurement of Speed of Rotation

1. Magnetic Tachometers. A magnetic pickup is a cylindrical permanent magnet behind a pole piece of iron, around which a coil has been wound. The frequency of the voltage developed is a function of the rotational speed. The pickup is gear driven. These may also be adapted by attaching a small generator to the shaft of the machine to be measured.

CONTROL DEVICES

A. Valves

1. Butterfly Valves. These are essentially damper-type valves. This valve is used in all control loops for which its characteristics are suitable. See Figure 22-12.

2. Control Valves. a) *Ported valve plug.* The valve plug or inner valve, has a port feature designed especially for specific applications. See Figure 22-13.

b) *Balanced Valve Plug.* This type is mostly used in piston-balanced valves for service involving high pressure drops across the valve. As shown in Figure 22-14, it is basically a single-seated valve in a double-seated body.

c) *Flat Valve Plug.* As shown in Figure 22-15, this type of valve plug is a com-

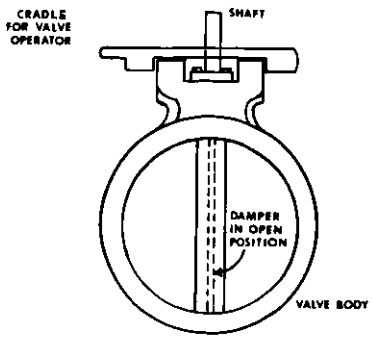


Figure 22-12. Butterfly valve.

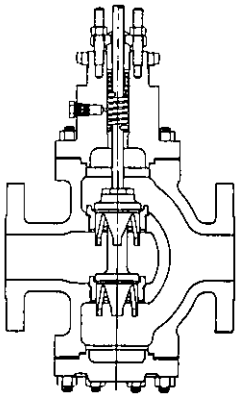


Figure 22-13. Ported valve plug. *(Courtesy, Fisher Controls Co.)*

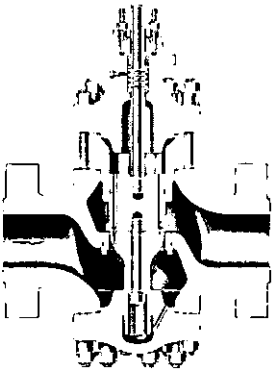


Figure 22-14. Balanced valve plug. *(Courtesy, Fisher Controls Co.)*

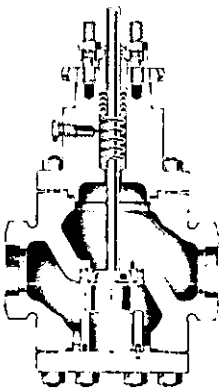


Figure 22-15. Flat valve plug. *(Courtesy, Fisher Controls Co.)*

mon configuration for general-purpose use. These valves are not suitable for very critical applications.

The flow characteristics of a valve depend on a number of criteria, such as the inner plug configuration, the pressure drop across the valve, and the size of the piping leading to and from the valve. To determine the size of valve needed, these factors must be considered, as well as the precise function of the valve.

3. Solenoid Valves. These are electrically operated (see Figure 22-16). The basic types are:

a) *Two-way,* either normally open or closed, as specified.

b) *Three-way,* with normally open, or normally closed vent.

c) *Four-way,* general purpose valves, frequently used with piston cylinder operators.

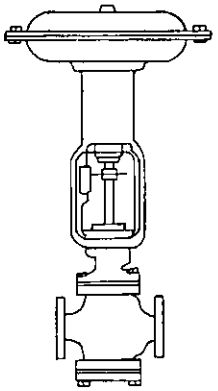


Figure 22-16. Control valve. *(Courtesy, Fisher Controls Co.)*

B. Rotational Speed Control

1. Governors. These centrifugal controllers are still in use in many forms.

2. Variable Speed Controllers. Many types are built. A widely used type in the United States is either controlled electrically or pneumatically, varying the pitch and ratios of the drive pulleys.

CONTROL LOOPS IN THE BEET SUGAR FACTORY

In this section are described typical control loops, or configurations of control devices used for the individual unit processes in a beet-sugar factory. The particular illustrations chosen are only typical, and details will vary widely, as do the individual factory equipment installations.

Following is a table of the abbreviations and symbols used in this section. (pp 714,715).

1. Control of Beet Flow

For a description of this part of the factory, see Chapter V. Figure 22-17 shows an arrangement of equipment which uses a beet pump to raise the beets to the washer level. Here there is only one feed control, on the beet-feed wheel, driven by a variable drive-speed motor, which is controlled electrically from a remote station or panel.

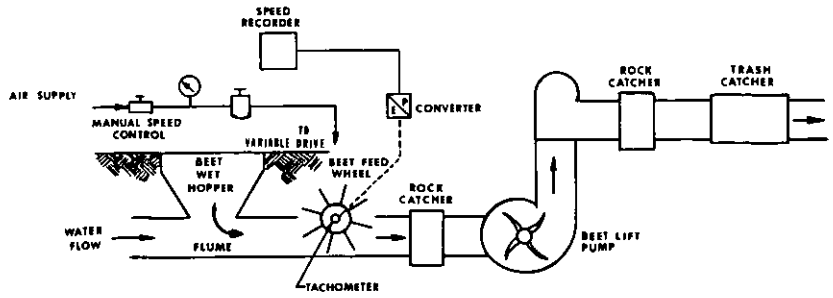
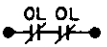
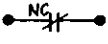
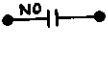
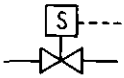
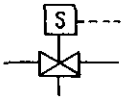
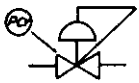
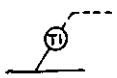


Figure 22-17. Typical equipment moving beets into factory.

Table 22-2 Abbreviations and Symbols

I.S.A.* Symbols	Symbols used in text.	
None	120 v, a-c, 60 Hz	120 volt, alternating current, 60 Hertz (cycles per second).
None	d-c	Direct current.
L	LS	Level switch or electrical level sensor.
None	H	"Hot wire", of control supply current.
None	N	Neutral wire, or common wire, of control supply current.
Same	P	Pressure.
dP	ΔP	Differential pressure.
	I	(In box) current transmitter.
None		Pump.
PI		Pressure gauge.
None		Voltmeter.
None		Ammeter.
Same		Speed indicator (tachometer).
		Transducer, or converter (converts one type of signal to another).
None		Electrical resistor, fixed value.
None		Variable resistor, such as a rheostat or potentiometer element.
None		Overload contacts on motor starter. Normally closed, but open by heat from high current flow.
None		Contact, normally closed, when relay or controller is de-energized.
None		Contact, normally open. Also, a latching or interlock contact. Placed across a momentary-contact, push-button starter to keep the circuit closed after the button is released, or across other primary control switches which have the same function as a push-button.

*Instrument Society of America

I.S.A.* Symbols	Symbols used in text.	
		Symbol of any recorder or controller. Abbreviated function indicated inside box.
		Valve, manually operated.
		Butterfly, damper or louver valve.
		Control valve, without valve positioner.
None		Control valve, with positioner.
		Solenoid valve, electrical, normally open or closed, as required.
		Solenoid valve, electrical, with exhaust port for 3-way operation. Exhaust is normally open.
PCV, Not Specified		Pressure reducing valve, manually adjusted.
		Pressure reducing valve, automatic.
		Shutoff cock, rotary plug, or ball valve.
Not Specified		Shutoff cock valve, with internal latching or interlock contact. The contact is wired in series with other safety contacts in the whole electrical control circuit.
Not Specified		Manually reset valve. This has internal solenoid to hold valve open after reset, and this quickly shuts, on a power failure.
Same		Pneumatic (air-operated) control line.
Same		Pneumatic; or other pipeline if symbols are used in mixed electrical and pneumatic control loops.
Same		Electrical control lines.
		Electrical temperature sensor: thermocouple, resistance, or thermistor element.

2. Diffusion

For a description of this part of the process, see Chapter VI. There are many possible diffusion control loops, depending on the amount of automation desired. To name a few: 1) speed control, 2) cossette-feed control, 3) diffuser supply water flow control, 4) pulp discharge, and 5) all controls needed for the motor-generator (MG) set, exciter motor, voltage regulators, internal as well as external. A simplified version of the speed control is shown in Figures 22-18 and 22-19. The sketches are general, and can apply to almost any type of continuous diffuser. Most use alternating current, converted to direct current by a motor generator set.

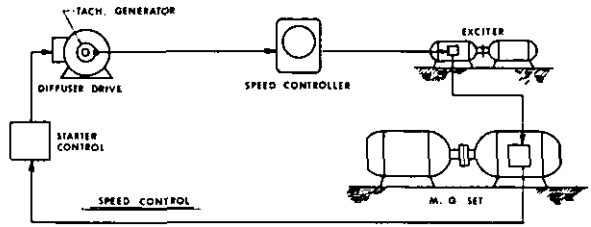


Figure 22-18. Block diagram of diffuser drive control system.

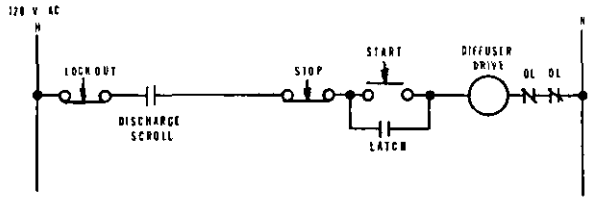


Figure 22-19. Interlock system of diffuser control.

The procedure for start-up of the diffuser drive is: 1) the MG set and the exciter are started manually from the control board; 2) a rheostat in the speed controller unit is slowly advanced, which activates a field winding in the dc generator, which fixes a stationary South-and-North-pole, or magnetic field of opposite polarity; 3) after the scrolls and slicer are started manually, the start button of the diffuser control is depressed momentarily. The latching contacts close and hold the starter relay closed; the diffuser drive starts to turn slowly.

A tachometer attached to the diffuser drive senses the speed, or rpm, and feeds a proportional signal to the speed controller. A set point in the controller is adjusted at the desired speed, and the controller establishes an equilibrium and controls the exciter current flow to the MG set, to the set point, within a tolerance depending on sensitivity and other bias functions of the controller. Normally the controller also serves as a recorder to record the rpm of the diffuser.

The first element of the control system is a lockout located at the diffuser drive, so that the diffuser cannot be started in case of repairs, and the complete control is de-energized.

3. First Carbonation

See Chapter VIII for background on this station. Figure 22-20 shows a typical control loop for the pH in first carbonation, which is accomplished through control of the rate of addition of the carbonating gas, or, in some cases, the liming agent.

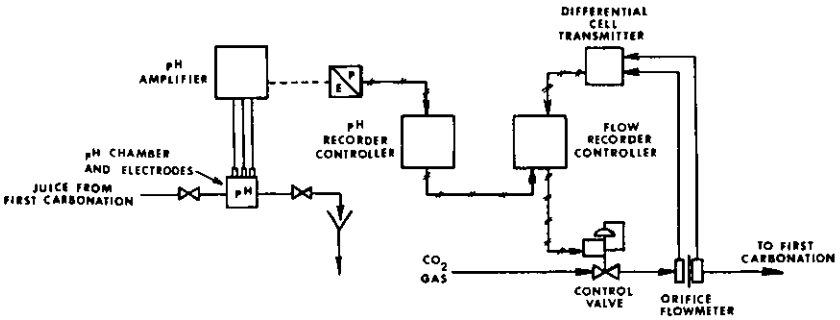


Figure 22-20. Typical control loop for first carbonation, for control of pH.

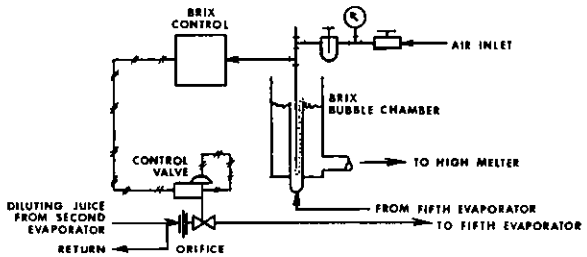


Figure 22-21. Circuit of equipment controlling density of evaporator thick juice, by dilution with second effect effluent.

4. Control of Thick Juice Density

Such a control is commonly applied to the thick juice emerging from the last evaporator effect. Please see Figure 22-21.

5. Evaporators

Operation of this station is described in Chapter XI. Figure 22-22 is a plan of a control system for steam and vapors.

6. Juice Flow Control

The diagram in Figure 22-23 shows an automatic juice flow control loop.

7. Vacuum Pan Control

Please see Chapter XII for background. The automation of a vacuum pan represents one of the more difficult tasks of control. A typical system is shown in Figure 22-24.

In this particular control system, the measurement of four variables is required; the pressure within the vacuum pan, the syrup supersaturation, the massecuite level, and the massecuite consistency, (1,2). The pan pressure is controlled by the flow of condenser water. It is necessary that the pressure be held quite constant during the boiling of the strike. The syrup supersaturation is measured indirectly through a very sensitive capillary temperature bulb which is installed in the vapor space between the pan dome and the condenser. By measurement of small changes in the temperature of the superheated vapor as it leaves the surface of the boiling magma, a good indication is obtained of the true massecuite temperature

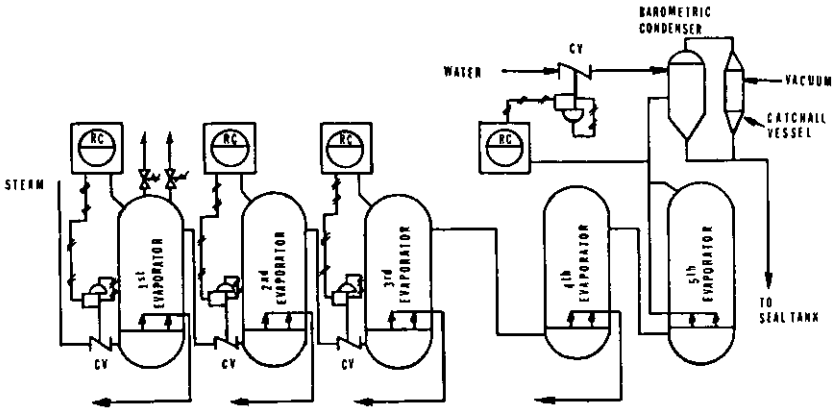


Figure 22-22. Juice evaporator steam and vapor control system.

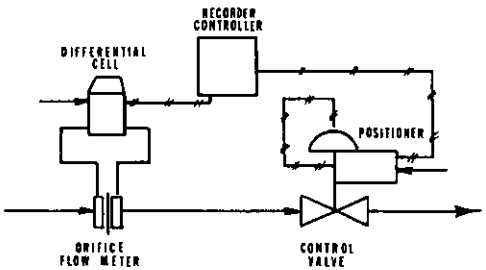


Figure 22-23. Juice flow controller.

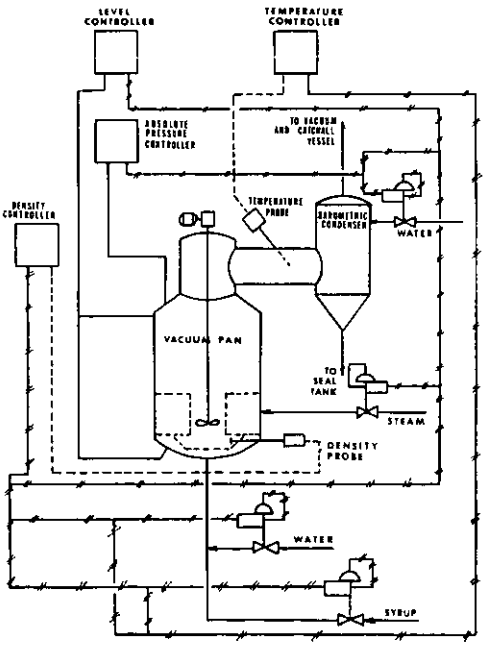


Figure 22-24. A vacuum pan control system. *(Courtesy, The Amalgamated Sugar Company)*

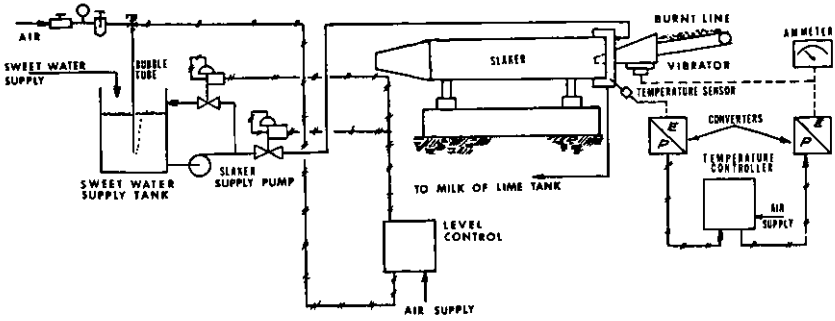


Figure 22-25. Control system of a lime slaking and milk of lime station.

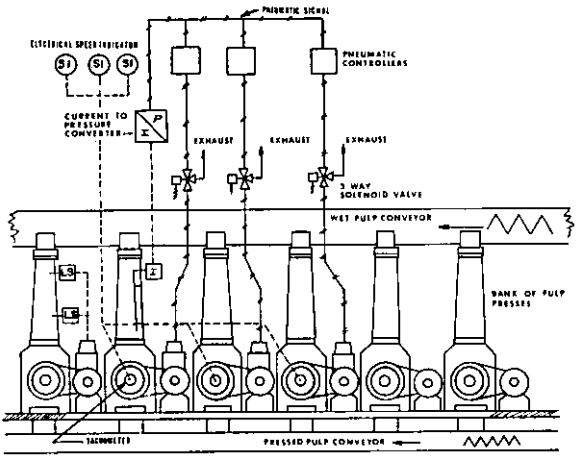


Figure 22-26. Control system for a battery of horizontal pulp presses.

and its supersaturation, throughout the entire course of the boiling. The level is controlled at the time of the original feed syrup charge, and at the maximum allowable pan level. A small, motor-driven mechanical rotor is used to measure changes in the consistency of the massecuite, and to control the steam, syrup feed, and water flow to obtain the optimum tightness and final consistency.

8. Milk of Lime Station

The operation of this station is described on pp. 589-592. Figure 22-25 shows a typical control system.

9. Pulp Presses

The requirements and operation of this station are discussed on pp. 626-27, 629-30. In Figure 22-26 is shown a control loop applied to an installation of horizontal pulp presses.

The principal purpose of the speed control of the pulp presses is to keep the rate of wet pulp supply in balance with the pulp being pressed. Usually a fixed

number of presses operate at a fixed rate and speed. In Figure 22-26 it will be noticed that the first press has two level sensors, one in the top and one in the lower part of the supply chute. If the wet pulp supply is greater than the press demand of the fixed speed presses, the wet pulp tends to rise and reaches the upper sensor, closing an electronic circuit and activating a relay with this particular press starting circuit, and this starts the press. This press runs until the pulp level drops below the lower sensor, and it is then shut off until the supply rises again. The other type of control, shown on the second press, uses the latest development in level sensors. A capacitance probe varies the speed of the press rotor, and consequently the pressed pulp volume. See section C. 6, above. The output of the probe drives a current-to-air converter, with the resulting air signal operating the pneumatic speed controller of the press variable drive. The 3-way solenoid valves are parts of the speed control, and are used for safety to allow the variable drive to coast down to its lowest speed in case of a power failure or discontinuance. If this were not done, and the press stopped, the drive belts of the variable drive would tear or break at a new start-up. Associated with these valves are electrical run-down timers.

10. Pulp Driver Drum Furnace

See pp. 626-633 for description of this operation. Figures 22-27 and 22-28 show a typical control loop.

Figure 22-27 shows the latching or interlock, which has the purpose of preventing the starting of the furnace, if any set of contact is open. The interlocks are all arranged in series, and this is an important safety feature.

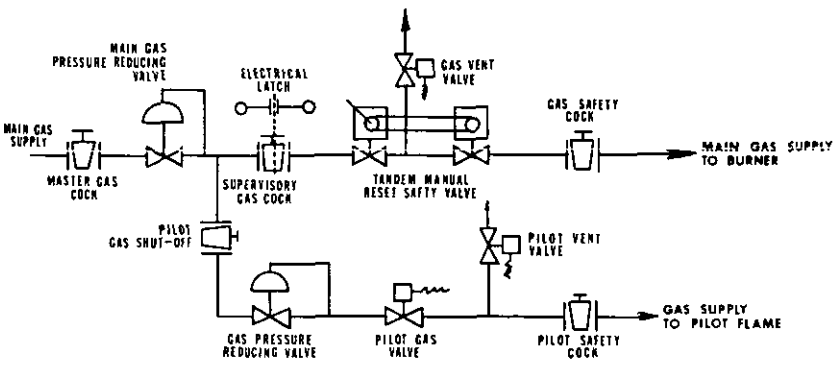


Figure 22-27. Latching or interlock system for pulp drier furnace.

In Figure 22-28, the large rectangle represents a so-called F S or "Flame Safety System," which is very complex electronically. If all the auxiliary control contacts are closed, the furnace can be started as soon as the ultraviolet flame detector senses the pilot flame, and a small Flame Safe relay is closed, which permits the electronic circuitry to be activated and the main gas valve to be opened. If the gas flame should fail, the Flame Safe relay opens, and all safety valves close, and the control circuit opens. Thereafter, to start-up, the whole procedure has to be followed through again. The various electrical control loops going in and out of the large rectangle are the various externally located control contacts, and the items of equipment needed for the complete system.

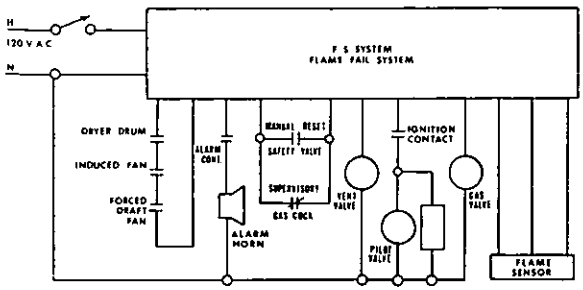


Figure 22-28. Block diagram of Flame Fail system of pulp drier drum furnace.

The normally open contacts for the Flame Safe circuit are located in the various motor starters of the equipment, and are called auxilliary locking contacts. They are normally open (relay de-energized), and all are in series; i.e., the drier drum will be started first, then the induced draft, and finally the forced-draft fan. This sequence will close the respective auxilliary contats and ready the furnace control for start-up.

Recommended for additional reading

Artwick, B.A., "Microcomputer Interfacing," Prentice-Hall, N.J., 1980

Bird, "Programs and Machines – An Introduction to the Theory of Computation, John Wiley and Sons, Inc., New York, N.Y., 1974

Considine, "Process Instruments and Controls Handbook," 2nd Ed., McGraw-Hill, New York, N.Y., 1976

"Control Valve Handbook," Fisher Controls Co., Marshalltown, Iowa, 1979.

Sheingold, D.H., "Transducer Interfacing Handbook," Analog Devices, Inc., Norwood, Mass., 1980

Skrokov, M.R., "Mini- and Microcomputer Control in Industrial Processes," Van Nostrand - Reinhold, New York, N.Y., 1980

Soissen, "Instrumentation in Industry," John Wiley and Sons, Inc., New York, N.Y., 1975

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(H.P. Gildersleeve)

Chapter XXIII
MICROBIOLOGY AND PLANT SANITATION*

The perishable nature of sweet liquids had been known to man for thousands of years, but it was not until the middle of the nineteenth century that the cause was traced to the "animalcules" first seen through the crude microscopes of Anthon Van Leeuwenhoek in the seventeenth century. This burgher of the city of Delft was far ahead of his time, and while he was convinced that his little creatures were the products of pre-existing life cells, his ideas were not shared by other scientists for about two hundred years.

In 1864 and the following years the work of Louis Pasteur finally convinced the scientific world that the organisms causing fermentation and food spoilage as well as infectious diseases in man and other living creatures were not created by spontaneous generation or chemical processes, but were present in the air, soil, and water.

Souring juices, slime deposits and gas formation were common occurrences in the early days of the beet-sugar industry, but were considered inevitable and only feeble attempts were made to combat them. Even in the latter part of the nineteenth century and the beginning of the twentieth, when the causes were generally known, the losses from inadequate sanitation were larger than realized. The respected Dr. H. Claassen (9) stated that large amounts of sugar may be decomposed in the diffusion battery when conditions are favorable for bacterial action, but he also concluded that with ordinary precautions such as maintaining the temperature at 75° to 80 °C and rapid juice flow, the loss of sugar was not perceptible.

While Claassen's recommendations were sound, the actual losses were only "not perceptible" due to inadequate chemical analyses and lack of accurate measurements.

When sanitizing agents were used, it was to cure rather than prevent. Unlike the sugar losses, the costs of sanitizing agents were known, which restricted their use.

Prior to World War II there was little in the literature dealing with bacterial infection and means of control. Among those early recognizing the problems and describing the use of various disinfectants were Saillard (35), and Reindel, Frey, and Mandl (34). In recent years there has been increasing awareness of the economic importance of these problems.

Microbiology in the Process

Harvesting, Transportation, and Storage

The organisms which cause loss of sugar in the factory originate mainly in the beet fields. Populations of 50 to 100 million per gram are not uncommon in the top layer of soil, falling off rather rapidly with increasing depth. Highest counts are found in the spring and in rich, damp, well-cultivated soils, when the pH values are about neutral. In general, soil conditions favorable to plant growth are also favorable to bacterial growth.

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*by H.E. Halden, Revised by M. Bolinder

When unbroken, the beet skin is an effective barrier against the invasion of microorganisms, in which action it is probably aided by the high concentration of saponins located just under the skin, which are highly toxic to lower forms of life (see p. 43). Skin lesions are caused by rodents, certain soil fungi, and nematodes and permit the entrance of soil bacteria which are carried with the beets to the process. Mechanical harvesting has added to the problems. More beets are fractured than in hand harvesting and there is an increase in the dirt and beet leaves transported to the factory. Such extraneous matter carries a high population of microorganisms. Recent studies reported by Bugbee, Cole, and Nielsen (5a) indicated even healthy internal beet tissue contains over 10 000 bacteria per gram.

During transportation and storage of beets several factors affecting microbial decomposition are important. There is greater activity in dirty beets and in drying, warm weather. The removal of adhering soil and trash before storage will minimize hot spots where the microbial activity is high. The cooling effect of natural or forced ventilation of the storage pile greatly aids in reducing damage.

The Microbial Flora

The flora of microorganisms entering the sugar factory is essentially that of the fields harvested, where it varies with the season and locality in the relative numbers rather than in the species. Schone (37) made a thorough study of the microorganisms found in beet juices and traced them all to the soil brought into the factory with the beets.

The organisms may be grouped according to growth temperatures, as shown in Table 23-1.

Table 23-1 Growth Ranges of Bacteria.

	Temperature °C		
	Minimum	**Optimum**	**Maximum**
Mesophilic	5-25	25-40	40-50
Thermophilic	25-45	50-55	60-73

The mesophilic organisms make up the largest group by far. The ratio of mesophiles to thermophiles in soils may range from 100:1 to 1000:1, varying according to the season, but in general the prevailing temperatures favor the growth of the mesophiles.

The temperature ranges are approximate and overlap, so that some form of active microbial life is present over the whole range, 5 ° to 73 °C.

While most organisms show active growth in neutral media, there is an optimum pH for each species. The range of pH in which the organisms will grow is variable and is influenced by the composition of the environment. The waste products of their metabolism may be either acidic or basic and the pH changes accordingly. Organisms which use carbohydrates usually form organic acids, while those ingesting proteins tend to produce basic products. Depending on the available nutrients, some bacteria may form either acidic or basic products. The chief types appearing in beet juices are given below:

Yeasts, principally of the genus *Saccharomyces,* which are generally strongly fermentive and hence destroy more sugar.

Molds, of which there is a great variety in the soil. They are the only organisms which are easily visible, since their colonies can be seen in accumulations

of soil under beet-storage bins and on damaged beets in piles. They generally destroy less sugar than yeasts.

Slime bacteria, of which the most frequently mentioned is *Leuconostoc mesenteroides.* This organism forms slime from sucrose and acid from dextrose. While its optimum activity range is between 21 ° and 25 °C the slime deposit of dextran protects the organism so that it can tolerate much higher temperatures. It has been known to cause serious obstructions in diffuser screens and to clog juice filter cloths. The organism is facultatively anaerobic and will thrive in closed systems at favorable temperatures.

Lactobacillus. This genus covers a great number of bacteria, differing in the temperature ranges of optimum activity, which may be between 28 ° and 62 °C, or higher. The end product is principally lactic acid, but some also produce alcohol, carbon dioxide and acetic acid.

Bacillus subtilis is widely distributed in soil and has an optimum activity range of 28 ° to 40 °C. It produces acid from sucrose, and nitrites from nitrates.

Bacillus stearothermophilus. This organism is also widely distributed in soils. The optimum growth temperatures are between 50 ° and 65 °C, with growth variable at 37 ° and 70 °C. It is a strong acid former and nitrites are usually produced from nitrates.

Clostridium thermosaccharolyticum is found in soil. The optimum activity range is between 55 ° and 62 °C. It produces acid and gas from sucrose. Nitrites are not produced from nitrates.

Clostridium thermohydrosulfuricum. An aerobic, hydrogen sulfide forming thermophile found in soil. Optimum activity is at 68 ° to 70 °C. It was studied and classified by Klaushofer and Parkkinen (17), who also suggested the name. This organism and *C. thermosaccharolyticum* have less ability than *B. stearothermophilus* to destroy sugar.

The beet and the extracted juice contain an abundance of nutrients for the different organisms. In addition to the sugars which supply carbon, there is an ample supply of proteins and inorganic trace elements. The latter constitute the "ash" and at least some are essential for the metabolism of the organisms.

The sugar-destroying actions of microorganisms with the resulting end products are termed *fermentation.* While specifically used by Pasteur to indicate the conversion of sugar into ethyl alcohol by yeasts, it has since been in general use to designate processes of carbohydrate decomposition by different organisms.

Fermentation

1. Alcoholic Fermentation. The formation of ethyl alcohol which, through oxidation, is further converted to acetic acid is principally caused by the yeasts at an optimum pH of about 5, and to a lesser extent by a few molds and certain bacteria. As the responsible organisms are mainly mesophilic the alcoholic fermentation is only possible before the high temperature section in the diffuser. The possibility of reinfection of cooled juices will be considered later.

2. Lactic Acid Fermentation. While lactic acid may be formed by many bacteria and yeasts, the lactic acid bacteria are the main cause of this type of fermentation. These bacteria are grouped as homofermentative; i.e., fermenting carbohydrates to produce essentially only lactic acid, and heterofermentative; producing lactic acid, and also acetic, propionic, and butyric acids. Both groups can produce alcohol and carbon dioxide.

Table 23-2 End Products of Fermentation of Carbohydrates by Bacteria.

Lactate	Succinate	Propanol
Acetate	Malate	Butyrate
Formate	Oxaloacetate	Butanol
Hydrogen	Diacetyl	Acetone
Carbon dioxide	Acetoin	Isopropanol
Water	Butylene glycol	Trimethylene glycol
Ethyl alcohol	Propionate	

3. Other Fermentations. Lamanna and Mallette (18) list a number of compounds which are the end products of fermentations of carbohydrates by bacteria.

The varying tolerance to heat and pH make the control of organism growth and the resulting sugar destruction a very difficult task. It is obvious that the regulation of heat and pH only are insufficient, and other measures must be taken.

The total destruction of all microorganisms, usually by heat, is termed *sterilization*. The term *disinfection* refers to the destruction of organisms by toxic chemical substances. Both sterilization and disinfection are limited in use by undesirable side effects. For example, in the diffuser, heat alone may make pulp pressing less efficient, and may also aid in the activity of high temperature thermophiles. The selection of chemicals and the amounts used must be governed not only by their effectiveness, but also by the residuals which may be carried into the process and indirectly cause losses in sugar, process difficulties, or have toxicity implications.

Beet Flumes and Washing

The application of disinfectants cannot begin effectively until the beets are removed from storage. The flume water, made up from fresh water or cooled condensates, is generally recirculated for conservation. It is screened to remove leaves, beet tails, and fragments, and fine silt is usually removed in a settling system. The water temperature may be from 30° to 40 °C and, when frozen beets are worked, even higher.

Soil bacteria find excellent conditions for growth in this water. The temperatures will generally be favorable, and sugar washed from the fractured beets furnishes available nutrients.

Where recirculation is practiced, many bacteria remain in the water for long enough to become thoroughly adapted to the environment.

The separation of the beets from the flume water is done by dragging them over screens, rolls or by other means, after which they are subjected to jets of fresh water. Even so, a large number of microorganisms are retained in the natural crevices of the beets and the exposed interiors. McDill (25) described the use of calcium hypochlorite at the rate of 5 ppm Cl_2 added to the wash water. The primary purpose was to suppress odors near the beet washer, and it was concluded that its value as a disinfectant within the system was relatively insignificant.

Andersen (1) believes that on the basis of experiments conducted in Denmark and Sweden, chlorination of the wash water has a beneficial effect in reducing the mesophilic activity in the diffuser.

The population of microorganisms in the flume water will vary with the season and degree of recirculation, but it is obvious that it will increase steadily unless measures of sterilization are practiced. While the sugar washed from the

beets would be lost in any case with the flume water discard, the constant activity of microorganisms on the exposed surfaces increases the loss. The number of organisms carried into the factory is also increased. Based on tests in Sweden, Weman (42) considered chlorination with an excess of chlorine in the flume water to be of extreme importance. Maudru (24) obtained a kill of 68% of the total bacteria by spraying the whole beets with a 1200 ppm solution of Roccal, a quarternary compound, apparently using about 5.5 gallons per 100 tons of beets. Norman and Rorabaugh (29) treated whole beets experimentally with solutions of quaternary amine and phenolic compounds. They found the compound appeared quite effective in lowering the fermentation potential. Here, as at later points in the process, the cost of sanitizing agents must be weighed against the potential loss of sugar from microbial activity.

When the beets are sliced, it can be assumed that the microorganisms will be uniformly distributed through the mass of cossettes and will continue into the diffuser. The beet slicers should be cleaned frequently with steam to remove any accumulations of cossette particles.

Diffusers

The diffusers in use in the beet sugar industry can be placed in three classes: closed, batch-type battery diffusers; open continuous diffusers; and closed continuous diffusers.

In all three classes of diffusers the cossettes are immediately brought into contact with the outgoing hot juice. The sudden elevation of temperature retards the activity of mesophiles and further contact with heat causes a sharp decrease in the number of living organisms. The activity of the thermophiles will also be retarded, but it may resume with renewed vigor after a short time. When cossette scalding is used, these retardations occur earlier in the diffuser.

If it were possible to operate a diffuser so that uninterrupted countercurrent flows would always occur, the microbial loss of sugar would be at a minimum, and could nearly be eliminated by temperature control and minor additions of disinfectants. Unfortunately such ideal conditions have not been realized, not only due to equipment design but also to fluctuating quality of the cossettes. Cossettes from beets damaged by soil rot, frost, storage deterioration or other causes, and trash carried in with the cossettes create more or less impervious pockets or nests in the diffuser which obstruct normal flow. Such obstructions furnish ideal spots for activity of microorganisms which increases rapidly.

In the closed, battery-type diffuser, each cell takes its place in turn so that once in each cycle it is emptied, cleaned, and refilled. This procedure reduces the retention of a trouble spot to the maximum time of one cycle. In closed continuous diffusers which are not completely filled, the interiors cannot be cleaned during operation, and many trouble spots may remain for long times. The interior surfaces of the transfer equipment or open continuous diffusers, which are exposed to air, may be steamed, but air darkens the juices through oxidation and encourages the growth of certain bacteria. Closed diffusers, which are completely filled in normal operation, are free from this disadvantage.

If not killed or retarded by disinfectants, the majority of the bacteria capable of activity at elevated temperatures pass right through before the full potential of their destructive ability is realized. Some are retained in the pockets and become fully adapted to the environment. Some are returned with the water pressed from the exhausted pulp, and these are thoroughly adapted to the diffuser conditions.

Controlling Diffuser Infection

Thus the places within the diffusion system which require the greatest attention are the trouble spots and the pulp press water system. Adequate application of heat or disinfectants to these spots benefits the whole system and retards the activity of the "transient" organisms.

The locations of the trouble spots vary with equipment construction and juice temperature. Possibly the fastest means of detecting increased bacterial activity is by changes in the pH pattern through the diffuser.

The pH of the diffuser supply water may vary in different factories. It is generally governed by the preference of the management, based on operating conditions. The pH pattern through the diffuser is influenced by the diffuser water; the buffering action of the juice, which increases with its solids content; and the products produced by bacteria. If bacterial action is absent, the pH pattern should follow smooth curves similar to those shown in Figure 23-1. Bacterial action resulting in acid formation will cause irregularities in the pattern and indicate the locations of trouble spots.

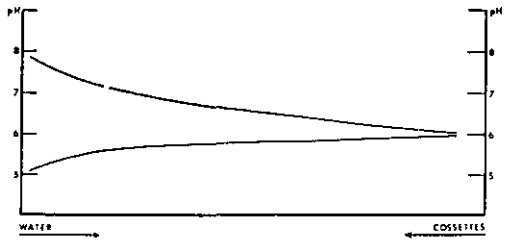


Figure 23-1. Normal pH patterns in a diffuser. Curves represent the normal patterns when using acidified or alkaline water, respectively, for the pH values shown and assuming a diffusion juice pH of 6.

Diffuser Disinfectants. The use of various disinfectants, such as formalin, sulfur dioxide, chlorine, and others is mentioned in the literature.

The most popular disinfectant appears to be formalin, a 40% solution of formaldehyde. Because of lower cost, formaldehyde is often prepared in the sugar factory by depolymerization of paraformaldehyde in which case the solution obtained is generally of low strength. Hallbeck (14) described a simple method for obtaining a 10% solution of formaldehyde by using water adjusted to 10.5 pH and steam injection to raise the temperature to 40 °C. By increasing the pH to 12 he found that a solution containing about 23% formaldehyde could be obtained. Formalin is a powerful bactericide and is effective in dilute solution.

Sulfur dioxide is sometimes used in the diffuser and added to the supply water. It is a good disinfectant, and in lowering the pH improves the pressing qualities of the pulp, but excessive use will lower the natural alkalinity of the juices, increase thin-juice lime salts, and may reduce the effectiveness of formaldehyde.

Chlorine is an extremely efficient bactericide when in a free state and in sufficient concentration. However, it reacts very quickly with organic constituents of the juice and becomes ineffective.

Control by Heat. The application of heat to the diffuser is partly through the supply water, but mostly by diffuser steam jackets or direct steam injection. While it would be beneficial for the control of bacteria to raise the temperature of the supply water to nearly 80 °C, the high temperature would cause the exhausted pulp to be mushy and difficult to work in the presses, as well as result in a loss of pulp substance with a diminished recovery of dried pulp and juice purification dif-

ficulties. In addition to recovery of pulp substance the return of press water would have additional benefits, such as to minimize waste water disposal, improvement of heat economy, and sugar recovery. The latter depends on the number of active bacteria returned to the diffuser, and unless the pulp press water is properly treated by heat sterilization or chemical disinfection, an overall loss rather than a gain may result.

Most of the mesophiles are killed in the diffuser, but a few survive in an inactive state and pass through with the diffusion juice. The thermophilic bacteria mostly enter the diffuser in an inactive state. The normal life cycle of bacteria is shown in Figure 23-2.

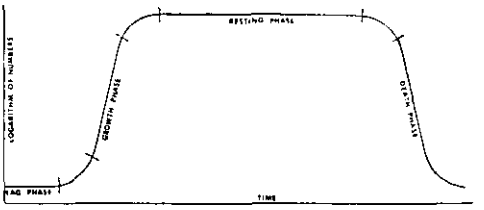


Figure 23-2. Life cycle of bacteria.

The duration of the inactive or lag phase depends on the ability of the organism to adjust to the environment. This varies with the species, but the normal retention time in a diffuser is probably not sufficient to bring the organism into the growth phase, when there are uniform material flows and complete scavenging of material from equipment surfaces. In pockets formed by retained cossettes and on coated surfaces the bacteria are held long enough to enter the growth phase and they increase rapidly. The loss of sugar is in proportion to their activity. In addition to those retained, a large number of well-adjusted bacteria may be returned with the press water.

Frequent observations should be made of the temperature and pH patterns through the diffuser, and probably supplemented by tests for activity from several places in the diffuser and the pres water return system. These observations should be used as guides for the application of disinfectants and heat. The lasting effectiveness of a disinfectant is only limited by the quantity applied, but the use of heat has its limitations.

Lamanna and Mallette (18) state that the maximum temperature for growth and metabolism of any known organism is 73 °C. This statement is based on tests conducted on organisms isolated from hot springs. Tests to show uptake of radioactive phosphorus as an index of metabolism above 73 °C gave negative results. Some bacteria form spores, which are minute, thick-walled, resistant bodies within the bacterial cells. The spores from practically all spore-forming thermophilic organisms will survive boiling temperatures and a few can withstand steam under pressure at 120 °C for short periods. Upon reexposure to temperatures of 73 °C or lower in a favorable medium, these spores can germinate and develop into vegetative cells. It has also been reported by Weman (43) and Lawson (19) that fermentation in diffusers can occur at 80 °C. Such observations are probably correct when only the temperature of the liquid phase is considered. The temperature at the actual place of fermentation, such as a pocket, may be lower. Carruthers and Oldfield (6) prepared cultures of organisms derived from soil which were exposed to a temperature of 75 °C for 96 hours. They grew readily when subcultured into a medium at 70 °C. Schone (38) found that the majority of the organisms would be killed between 75 ° and 80 °C.

As shown in Table 23-1, the temperature from the water end of the diffuser should be raised to a minimum of 73 °C as quickly as is practical. Higher temperatures are desirable further along in the juice flow as indicated from the preceding paragraph. (See Chapter VI.)

The temperature of the diffuser supply water or the first section of the diffuser, in factories in North Ameria and Europe, varies from about 50 ° to 80 °C.

Use of Formalin. If for some reasons high temperatures cannot be maintained and equipment design permits the formation of pockets or trouble spots, it is necessary to rely on disinfectants.

In the case of formalin it has for many years been the practice to add it in rather large doses, assuming that it takes a high concentration to kill the bacteria in a trouble spot. When once killed it may not be necessary to repeat the performance for as long as twelve hours. The places of addition are mainly determined by the pH pattern, but the formation of acid, as indicated by the drop in pH, does not necessarily indicate the exact spot of activity. An increase in activity occurs before a drop in pH can be measured. Therefore the disinfectant should be added upstream of the juice flow from the point indicated by the pH lowering.

The practice of adding formalin in large doses is common in batteries and in open continuous diffusers where the location for addition can be freely chosen. The procedure is difficult or impractical with closed continuous diffusers such as towers. Fermentation in this type of diffuser, according to Carruthers and Oldfield (7), tends to be localized towards the juice end. By the addition of formalin to the water feed or to the returning press water, the inhibitive effect can be transmitted to relatively inaccessible parts of the diffusion system. The major portion of the formalin is not decomposed by juice constituents.

Carruthers and Oldfield (7) found that the greater part of the formalin added to a diffuser is carried by the juice into the process as free formaldehyde. It is finally completely decomposed during liming, the degradation products consisting of closely related isomeric acids. Vavra (41) found that a continuous addition of formalin at the rate of 0.010-0.015% on beets would prevent microbiological action in the diffuser, and remain effective until first carbonation. He supplemented this with 5-10 liter additions to the press water at 1 to 2 hour intervals. The total addition to the system amounted to 0.018-0.023% on beets.

As the method of formalin addition depends largely on the diffuser type, the most beneficial procedure must be worked out by the individual factory. Lilienskiold and Becker (21) recommend irregular applications to avoid developing resistant strains of bacteria. Other disinfectants recommended are chlorine and chlorine-amine compounds as variants or in combination with formalin.

Return of Press Water to Diffuser. When pulp press water is returned to the diffuser a large number of active bacteria can be added if treatment is not used. Andersen (1) has found that untreated press water may contain 10 million thermophilic bacteria per ml, or 10 000 times more than is carried to the system by the cossettes. He does not infer that the danger of activity in the diffusers is increased in the same proportions, but he has shown that thermophilic fermentation is more probable with pulp press water of high activity.

As in the diffuser, means of controlling bacterial activity are heat and disinfectants. Pipes, and in particular elbows and valves, may be coated with a deposit favoring bacterial growth. Heat is not effective unless the temperature is kept over 73 °C and preferably as high as 80 °C.

In diffusion systems with cossette scalding, the raw juice is reheated when leaving the diffuser, but it is quickly cooled again by the cossettes passing through it. The heating will, therefore, affect the mesophiles in the cossettes rather than the thermophiles in the juice.

Diffusion Juice Screens and Heaters. Before entering the factory juice system the juice is usually screened. This unit operation is particularly vulnerable to bacterial activity due to fine pulp particle deposits on the surfaces, and to favorable temperatures. In addition, to increase in thermophilic activity, there is also the possibility of mesophilic activity from bacteria or spores which have passed through the diffuser in a dormant state or have entered by reinfection from the surroundings.

In a well designed factory the transfer lines from the diffuser to the juice heaters are kept as short as possible. Means for steaming out the lines are provided and are used whenever there is a temporary shutdown of the diffuser, for even a short period.

Juice Purification

During juice purification, most if not all of the mesophiles are eliminated. The spores of high temperature thermophiles may pass through, but most will be caught in the thick juice filters. Reinfection may occur from such sources as remelt return, and when granular carbon treatment is used, the retention time and temperature are favorable for developing spores into active sugar-destroying organisms.

Vacuum Pans and Mixers

After concentration in the vacuum pans, the massecuite is exposed to the action of airborne organisms. Destructive activity will occur where the moist material accumulates, is cooled off, and is exposed to the air. It is therefore of the greatest importance to keep accumulations at a minimum, particularly where rapid cooling may occur.

Centrifugals, Scrolls, and Elevators

The sugars leaving the centrifugals are moist and do not flow freely. Accumulations in centrifugal chutes, scrolls, and elevators must be removed frequently, and surfaces steamed off. Fungi in the air find readily available nutrients in this material and cause sugar losses. Even the slightest odor of fermentation must be considered a danger sign and indicate the necessity for sanitary measures.

Storage Tanks

Condensation on cool metal surfaces in tank head space will provide favorable growth conditions for fungi if not cleaned frequently. If tanks are used for intercampaign storage of liquors, disinfectants must be added.

Even with the best equipment design, it is practically impossible to avoid spills. While such spills are cleaned up and supposedly recovered, the gesture becomes one of "housekeeping" rather than "sanitation" unless disinfection and sterilization are practiced. Each factory will present a different problem, but the potential dangers should be realized and dealt with in the best manner possible.

Sanitation Tests

The best way to determine sanitation efficiency is by culturing samples taken throughout the factory. In addition to the intermediate and finished products, gutters, sumps, and accumulations should be sampled.

The counts of mesophilic bacteria, yeasts, and molds are of particular interest in determining infection and growth of airborne organisms and the need for sanitation. Counts of thermophilic organisms indicate the filtration efficiency and also serve to determine the possible introduction of thermophilic organisms in the material flow.

Methods of Analysis

The counts obtained by plating methods for determining the viable organisms in a fluid sample taken from the fluming, diffusion or juice systems are generally not satisfactory for immediate control purposes, for two reasons: the plating methods require two days or longer before results are available, and the change from the factory material environment may influence the metabolism of the organisms and the results may be misleading. Such platings can be used as "before" and "after" measures of changes in the use of disinfectants or heat.

Determination of pH

Changes in the pH pattern are the fastest and most convenient means for detecting major changes in microbiological activity, but the pH values cannot show if the highest practical degree of disinfection is reached, since the buffering action of the juice may mask minor increases in acid formation.

Determination of Lactic Acid

Various methods for estimating sucrose loss by determining the acid formation in the diffusion system have been proposed and used. From experiments conducted by Carruthers and Oldfield (7) it was concluded that a very high proportion of the total acid produced in the diffuser by bacteria is lactic acid. In continuous cultures at pH 6.1 and 65 °C they found that lactic acid averaged 55% of the sugar metabolized and that the sucrose loss was equal to about twice the weight of lactic acid produced. Norman and Rorabaugh (29) found ratios of sugar to lactic acid ranging from 0.81 to 4.44.

Of lactic acid methods specifically designed for use with beet juices, Stark, Goodban and Owens (39) developed a procedure for separating lactic acid by ion exchange and employed a colorimetric method of Barker and Summerson (4) for determination. Olsen (31) published a determination of lactic acid by paper chromatography. Kubadinow and Rosner (17a) described an enzymatic determination of lactic acid. The method is rapid and simple enough to be used as a routine analysis.

Oldfield and Shore (30a) and Oldfield, Parslow and Shore (30b) developed methods for the determination of lactic acid, principally in molasses, but also applicable to raw diffusion juice. They combine an ion exchange separation and a gas-liquid chromatographic method for the determination of lactic acid in raw juice. An estimate of lactic acid, of relatively high precision, can be obtained within about one hour and 40 minutes.

Determination of Nitrites

Certain of the bacteria thriving at higher temperatures reduce nitrates to nitrites. As only nitrates and not nitrites are found in soil, the reducing power of the bacteria to nitrites in the diffuser can be used as a measure of their activity.

Carruthers, Gallagher and Oldfield (8) used a photometric method developed by Buckett, Duffield and Milton (5). Cox and Tveit (10) used another photometric method and found good agreement between the two. Shinn (38a) describes another photometric method for nitrite which Lew (20a) has used successfully with

sugarbeet process materials. The nitrite determination is a more reliable measure of activity than the pH, as buffering action of the juice does not interfere.

Mauch and Bourzutschky (23) recommended an indicator paper, "Nitur Test" (30), for detection of nitrites in solution by the formation of red coloring, and concentrations as low as 1-2 mg NO_2-/l can be detected. It has not been available in the United States.

Determination of Thermophilic Activity

If a sample of juice is taken from the diffuser and incubated at the temperature of interest, the rate of formation of acid, as indicated by a drop in pH, may be used as a measure of the bacterial activity of the organism thriving at that temperature. In order to minimize the buffering action of the juice, a small measured quantity of juice may be placed in a larger volume of an artificial medium or a sterile juice. Methods described by Andersen (2) and Carruthers and Oldfield (7) are similar. Andersen used an artificial medium and incubated at 55 °C, while Carruthers and Oldfield used sterile juice and incubated at 65 °C.

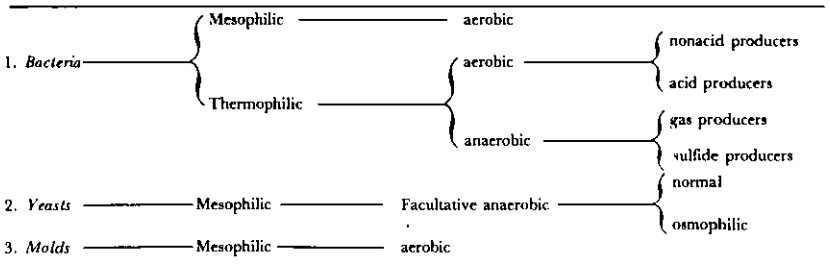
When testing light colored samples such as press water it is possible to use indicators and to use the time required for decolorization as a measure of activity.

Andersen (2) described a method for press water using a 0.1% thionine (3,7-diaminophenothiazonium chloride) solution in water as an indicator. The sample, with addition of three to four drops of the indicator, is incubated in stoppered test tubes at 55 °C.

Microbiology in the Product

The microorganisms which may be present in finished sugar products are classified by the sugar industry as shown in Table 23-3.

Table 23-3 Classification of Organisms in Finished Sugar Products.



Except for those tolerating high heat and surviving factory processing, the majority of the organisms present in finished sugar products are airborne from locations favorable to their growth. In addition to the sources within the factory mentioned in the previous section, outside locations can contribute a large share through airborne dust.

Dry Sugars

Mesophiles. Dry sugars do not offer favorable growth conditions for bacteria and yeasts, and their numbers are gradually reduced in storage due to dehydration. The molds may survive when only traces of moisture are present and spores may germinate in a cold solution of the sugar.

Table 23-4 Microbiological Standards for "Bottlers" Granulated Sugar. National Soft Drink Association (28).

	Maximum Per 10 grams
Mesophilic bacteria	200
Yeasts	10
Molds	10

The standards set for "Bottlers" granulated sugar by the National Soft Drink Association are shown in Table 23-4.

Thermophiles. Thermophilic organisms are of great importance to canners as they may cause spoilage in some products. The bacteria recognized as spoilage organisms in canned foods are shown in Table 23-5.

The standards set by the National Food Processors Association are generally recognized throughout the world (Table 23-6).

Table 23-5 Thermophilic Spoilage Organisms in Canned Food Products.

1. Aerobic	a. non "flat sours"	Acid not produced (Various species of genus *Bacillus*)
	b. "flat sours"	Produce acid, no gas (*Bacillus stearothermophilus*)
2. Anaerobic	a. Gas formers	Produce acid, hydrogen and carbon dioxide (*Clostridium thermosaccharolyticum*)
	b. Sulfide producers	Produce little acid, mainly hydrogen sulfide (*Clostridium nigrificans*)

Table 23-6 Standards for Thermophilic Organisms in Granulated Sugar. National Food Processors Association (27).

Samples taken from each of five bags or barrels of a shipment or lot.

1. *Total Thermophilic Spore Count.* For the 5 samples examined, there shall be a maximum of not more than 150 spores and an average of not more than 125 spores per 10 grams of sugar.
2. *Flat Sour Spores.* For the 5 samples examined there shall be a maximum of not more than 75 spores and an average of not more than 50 spores per 10 grams of sugar.
3. *Thermophilic Anaerobic Spores (Gas formers).* There shall be present in not more than 3 (60%) of the 5 samples and in any 1 sample to the extent of not more than 4 (65+%) of the 6 tubes inoculated.
4. *Sulfide Spoilage Spores.* There shall be present in not more than 2 (40%) of the 5 samples and in any 1 sample to the extent of not more than 5 spores per 10 grams. This would be equivalent to 2 colonies in the 6 inoculated tubes.

Liquid Sugars

The production of liquid sugars has increased since World War II from a very modest beginning to a major product in certain areas (See Chapter XV).

The perishable nature of the products has caused the sugar industry to realize the importance of microbiological control through sanitary measures, which in turn have contributed improvement to the overall sanitation of the factory.

Liquid sugars are made by dissolving granulated sugar in water at a temperature of about 80 °C. Two general types are produced: sucrose syrup with solids content of about 66.5%; and partially inverted syrups with solids contents from 68 to 77%, and invert contents from 10 to 90% on solids.

According to Owen (32) the mesophilic bacteria normally occurring in sugar rarely, if ever, cause deterioration. Their numbers may indicate the degree of contamination, which is generally caused by airborne dust.

The yeasts and molds are the predominating spoilage organisms in syrups and cold-process products made therefrom. In severe infections the yeasts may produce a fermented odor and taste, and the molds may give an unsightly appearance to products and produce objectionable odors and tastes.

The standards set for "Bottlers" liquid sugars by the National Soft Drink Association are generally accepted, at least in the United States. These are shown in Table 23-7.

Table 23-7 Microbiological Standards for "Bottlers" Liquid Sugars. National Soft Drink Association (28).

A. Mesophilic Bacteria	1. Last 20 samples average 100 organisms or less per 10 grams D.S.E.—dry sugar equivalent. 2. Ninety-five percent of last 20 counts show 200 organisms or less per 10 grams D.S.E. 3. Of the last 20 samples, only one sample showing a count of more than 200 bacteria per 10 grams D.S.E. may be excluded in calculating the running average, provided counts of other samples tested the same day are within the tolerances as stated in A-1 and A-2.
B. Yeast	1. Last 20 samples average 10 organisms or less per 10 grams D.S.E. 2. Ninety-five percent of last 20 counts show 18 organisms or less per 10 grams D.S.E. 3. Of the last 20 samples only one sample showing a count of more than 18 yeast per 10 grams D.S.E. may be excluded in calculating the running average, provided counts of other samples tested the same day are within the tolerances as stated in B-1 and B-2.
C. Mold	Same standards as for "Yeast."

Liquid Sugar Installation. After being cooled the syrups are pumped to storage tanks to await shipment to customers. The syrups must be protected against invasion of microorganisms from the time of leaving the cooler until pumped to a carrier. If the manufacturer's tank trucks or railroad tank cars, for the cleaning of which he is responsible, are used, the responsibility for quality usually extends until the syrup is pumped into the customer's plant.

A simplified installation for the manufacture and storage of liquid sugars is shown in Figure 23-3.

An airtight system, or one to which only filtered air could be admitted, would be ideal. This is unfortunately difficult to bring to reality. Any leaking valve or pump in the system may be an opening to the atmosphere and admit the ever-present spoilage organisms. Leaking pipe connections fall into the same category. Possibly the most susceptible places for infection are the storage tanks. Air entering the tanks from the ventilating system may be carrying organisms if the filters are defective. Organisms may be introduced through leaks around ultraviolet lamp housings. Loading hose outlets are also potential entrances for organisms and should be covered with plastic bags when not in use to prevent entrance of insects.

The most efficient installation from an engineering standpoint is frequently unsatisfactory from a sanitation standpoint. The multiple-purpose use of pumps and pipelines causes cross infection. Such a system also may involve stagnant sections in

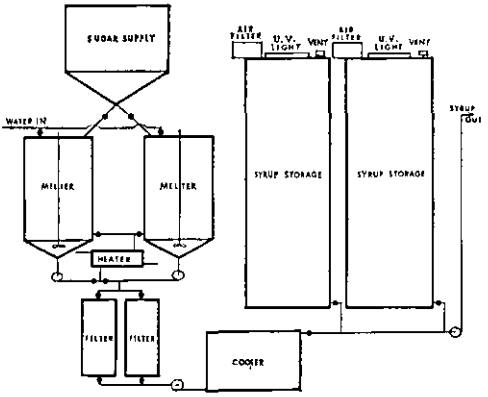


Figure 23-3. Flow diagram of manufacture and storage of liquid sugar, showing aspects of microbiological importance.

pipelines, which provide places for incubation. In these sections the organisms adhere to the walls and infect subsequent batches of syrup, even if such syrup is sterile when entering.

A similar condition may also build up on the storge tank walls above the liquid level. The infection may have started in the supply lines to the tank, or may have entered through the ventilating system. Condensation causes a layer of dilute syrup to be formed on the surface which furnishes excellent growth conditions for fungi. As the level of the syrup drops a film is deposited on the wall and the organisms multiply rapidly. If a fresh batch of syrup is pumped into the tank the turbulence of the syrup and the rise in level wash the organisms off the wall and they are mixed with the syrup. Such a condition usually can be avoided if a cleaned storage tank is filled to capacity before any withdrawals are made, and if fresh syrup is not added until the tank has been emptied and again cleaned.

Mesophilic Organisms in Liquid Sugar

Bacteria. The species of mesophilic bacteria which may be found in syrups are widely distributed in nature. They can be traced to the soil, plants, occasionally water, and the intestinal tracts of man and other animals. According to Owen (32) "The only group of mesophilic bacteria occurring in sugars are the spores of the mesenteric group of levan-forming species."

Yeast. Most commonly encountered in syrups are species of *Saccharomyces* and, in particular, *S. cerevisiae,* which is a strong fermenter of sucrose. Less frequent are weak fermenters, such as *Cryptococcus (Torulopsis), Hansenula,* and *Rhodotorula.* The colonies of *Rhodotorula* develop a carotenoid pigment. This yeast is nonfermentive.

While most yeasts grow best in low-density sugar solutions, some may develop a tolerance to higher osmotic pressure and others will only grow in high density syrups. Examples of such yeasts are:

Saccharomyces cerevisiae: low density sugar tolerance;
Saccharomyces rouxii: low and high density sugar tolerance;
Saccharomyces mellis: high density sugar tolerance.

Yeasts tend to be more of a problem when sucrose is blended with certain other sweeteners. Blends of sucrose and regular corn syrups are known to be very susceptible to yeast spoilage. Experiments have indicated that regular corn syrup contributes certain nitrogenous micronutrients necessary for yeast growth (4a).

Highly refined syrups will normally not undergo yeast fermentation because of a lack of micronutrients.

Two important factors in predicting the propensity of a syrup to undergo yeast spoilage are: a) the presence of nitrogenous micronutrients and b) the syrup water activity (A_w). Water activity may be estimated by the following calculation (4b):

$$A_w = \frac{\text{Moles of water}}{\text{Moles of water} + \text{Moles of solute}}$$

Laboratory experiments have indicated a syrup with less than about 25 ppm nitrogenous substances or a syrup with an A_w less than 0.80, is reasonably safe from spoilage (4a). As the minimum concentration of micronutrients or the minimum A_w is approached, syrup spoilage from yeasts is highly time retarded. Even syrup inoculated with yeasts and maintained at optimum growing temperatures will remain unspoiled for long periods of time.

Molds. The molds encountered in sugar syrups originate in the soil, from which well over 250 species have been isolated. Henrici (15) lists the most common genera as *Aspergillus, Penicillium, Zygorrhynchus, Trichoderma, Fusarium, Mucor, Rhizopus, Alternaria,* and *Cladosporium.*

Another organism of importance is the so-called "black yeast." The organism has, over the years, been given a number of different classifications. It is now classified as a mold of the genus *Aureobasidium.* The organism is nonfermentative and strictly aerobic. A typical colony on agar media appears first cream colored and mucoid, then after a few days darkens and eventually becomes black and leathery. Initial stages appear yeast-like both when observed macroscopically and microscopically.

The organism is troublesome in syrup lines and may be found on the sides of syrup storage tanks usually above the liquid level. The organism has even been found growing near ultraviolet lamps.

Liquid sterilization or the application of steam have generally little effect as the leathery coating protects the active cells underneath. Mechanical brushing, followed by liquid sterilization is generally necessary for elimination.

Sanitation. The preliminary step in the cleaning of equipment used for dry and liquid sugar products is thorough washing with hot water. If heavy deposits of sugar are not completely removed, the subsequent use of sanitizing agents may not be effective. If an infection of organisms has occurred which produces deposits, it may be necessary to supplement the hot water washing with mechanical cleaning.

This cleaning should be followed by rinsing with a sanitizing solution. Closed sections of the system, such as pumps, pipelines and fittings, should be completely filled with solution and allowed to stand for a sufficiently long period.

The possibility of undesirable residuals must be considered when using sanitizing agents. A chlorine compound is probably the most satisfactory known at present, and a number are on the market. Hypochlorous acid, HClO, is the active sanitizing agent in most chlorine compounds. The maximum concentration or dissociation of hypochlorous acid occurs between pH 4 and 6.

At the favorable temperature the optimum growth of most yeasts occurs near pH 4.8. Leethem and Eis (20) showed that the population of yeasts in infected syrup samples decreases in storage with increasing pH values. It was found that syrups

with known yeast counts and adjusted to pH 8.0 or higher were free from viable yeasts after a short period of storage. Application of the tests to practice, by simply increasing the syrup pH from about 7 to about 8, effectively reduced problems with yeasts. The pH change may be accomplished by adjusting the hydroxyl alkalinity of the water used for dissolving the sugar.

The radiation of syrups with ultraviolet light has been practiced for many years. Such installations generally consist of unshielded ultraviolet tubes in the top sections of storage tanks. As the effectiveness of the lights decreases inversely with the square of the distance, the benefits of sterilization of the syrup surface are doubtful unless the surface is close to the lights. Where ventilation through circulation of air is practiced, some sterilization of the air may be accomplished.

Due to the limited penetrating power of ultraviolet light, complete sterilization of syrup cannot be accomplished unless the syrup is treated in a thin layer, and in close contact with the light for some time.

Equipment which meets these requirements is on the market under different trade names. The ultraviolet tube is enclosed in a quartz tube which permits free penetration of ultraviolet light. The assembly is placed in the center of a stainless steel pipe, through which a turbulent flow of syrup passes. The sterilizing units presently marketed contain from 4 to 24 tubes.

Halden (11) made tests with a unit containing 24 tubes. It was designed for a maximum input pressure of 80 psi, which limits the flow rate to about 20-25 gpm for a syrup with a solids content of 77.1%.

Sterile syrups with densities varying from 50.2° to 75.0° Brix were inoculated with strains of *Saccharomyces cerevisiae*, *Rhodotorula* and *Aureobasidium*, respectively. Complete kills were obtained of *Saccharomyces cerevisiae*, and *Rhodotorula* after one pass through the sterilizer even when the initial concentration of the organisms was TNTC (too numerous to count) on 10 g des. *Aureobasidium* could not be killed by one pass through the sterilizer, even in low concentration.

Sterilizing units containing two sets of four tubes each, in series, have been used successfully in circulating systems to storage tanks to eliminate minor infections.

Microbiological Control. Frequent sampling and testing are essential factors in the successful operation of a liquid sugar station. Due to the random distribution of organisms it becomes very difficult to obtain a represesntative sample. This is particularly true with respect to yeasts and molds, for which the limit is set by the National Soft Drink Association (28) at 10 organisms per 10 grams of dry sugar equivalent. The Association recognizes this difficulty by basing the average count on the last 20 samples taken from a particular source. The chances for a more representative sample are increased if a continuous sample can be taken from a flowing stream in a pipeline or pump. Halden, Leethem and Eis (12) describe a continuous liquid sugar sampler which may be used with aseptic techniques.

The microbiological method for yeasts and molds specified by the National Soft Drink Association states that not less than five grams of dry sugar equivalent must be taken for each sample and distributed on four culture plates. Due to the random distribution of the organisms it is clear that a more reliable count will be obtained if a larger sample and more plates are used. When a number of samples are tested, the total number of plates is limited for practical reasons.

Table 23-8 is based on tables of "Poisson's Exponential Binomial Limit" (26). It shows the expected counts of organisms at various levels in 90% of trials. In 5%

Table 28-8 Expected Count per 10 g dse at Various Actual Organism Levels (not more than 5% under minimum or 5% over maximum value listed).

Actual Product Average Org./10 g dse	g. dse Plates	5 4	10 8	15 12	20 16	25 20	50 40
1	Min	0	0	0	0	0	0
	Max	6	4	3	3	2	2
5	Min	0	2	2	3	3	3
	Max	12	10	8	8	7	6
10	Min	4	5	6	6	7	8
	Max	20	16	15	15	14	12
20	Min	10	13	14	15	16	17
	Max	32	29	28	26	25	23
50	Min	36	39	40	42	—	—
	Max	66	63	61	59	—	—
100	Min	78	84	—	—	—	—
	Max	126	118	—	—	—	—

of trials the count may be expected to be less than the minimum value shown, and in 5% of trials greater than the maximum value.

The use of molecular membrane filters and fluid media as alternatives for culture plates and agar media was proposed by the ICUMSA Committee on Microbiological Tests (16). The membrane method allows the use of a larger sample than is generally used in the pour-plate methods. Among those describing the application of the membrane method to sugar products are Perkins (33), Halden (13), Schaufus (36), Lorenz (22), and Attenborough (3).

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Chapter XXIV

TECHNICAL ACCOUNTING AND PROCESS CONTROL*†

The principal functions of the beet-sugar factory control laboratory are 1) to account for sugar entering and for factory yields and losses, 2) to account for the usage and losses of other process materials, 3) to make analyses for process control, and to measure and record the many process variables, and 4) to analyze the products and by-products for quality control. Of course many measurements and analyses serve more than one of these functions.

In this chapter analytical methods will not be described, as they are adequately covered in other works (2,4-6).

Sugar Accounting

The Material Balance

For the proper guidance of management and factory operators, the best estimate of sugar entering and sugar leaving the process at points shown in Table 24-1 must be monitored on a regular basis. A material balance detailing the sugar introduced and the sugar leaving in the various streams is drawn up by the chief chemist each month or each week, showing the results of the current period operation, and the operation to date over the entire campaign. For close control the chemist may be required to make a daily balance for each day of operation.

The most general material balance equation (20) for any process is:

$$\textit{total input} = \textit{total output} + \textit{accumulation} \qquad (1)$$

If the accumulation is negative, it is called the depletion. Applied to the sugar factory, the equation may be written as:

$$\textit{sugar entering} = \textit{sugar produced} + \textit{sugar losses} + \textit{sugar accumulation} \qquad (2)$$

But for a given accounting period,

$$\textit{sugar accumulation} = \textit{sugar in process at end of period} - \textit{sugar in process at beginning of period} \qquad (3)$$

Then (2) becomes,

$$\textit{sugar entering} = \textit{sugar produced} + \textit{sugar in process at end} - \textit{sugar in process at beginning} + \textit{losses.} \qquad (4)$$

It is clear that this is not a complete statement, since some of the sugar in process will be produced as molasses. Beet molasses is about 60 purity, or 60% of the weight of molasses solids is sugar and 40% is nonsugars.

The procedure of estimating the weight of sugar and nonsugars in the factory at the beginning and end of an accounting period is called "stocktaking." It consists in weighing, or otherwise measuring the amounts of juices and syrups in the various

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*1st edn by S. Moore. 2nd edn. revision by R.A. McGinnis, R.E. Hallbeck, J.R. Johnson. This edn revision by R.E. Hallbeck

† The use of the words "sugar" and "nonsugar" is avoided in the previous chapters, unless reference is intended to all sugars, or to every nonsucrose substance.

However, in the field of technical accounting the usage of these words as the equivalents of "sucrose" and "nonsucrose" is so firmly established, that application to the text of this chapter is postponed for a future edition.

Table 24-1 Routes by Which Sugar May Enter or Leave the Factory

Sugar Entering	Sugar Leaving
1. In beets purchased	1. Sugar produced
2. In foreign molasses worked	(a) Granulated
3. In house molasses worked	(b) Specialty grades
4. In sugar and sirup carried over from a previous campaign	(c) Brown sugars
5. In damaged or raw sugar purchased or returned for refining or reprocessing	(d) Edible sirups and liquid sugars
6. Sugar received, but not purchased, in polarizable tare material	2. In molasses produced
7. Minor Sources	3. Losses outside the factory proper
(a) Warehouse rejects	(a) False polarization of tare laboratory samples. (not an actual loss)
(b) Sugar for vacuum pan seeding	(b) Sugar lost in transit, yard storage, bin storage, and pile storage by beet respiration and microbiological action
(c) Sugar in tare laboratory samples analyzed for another factory	(c) Loss of tails and polarizable material in trash
	(d) Loss by diffusion to beet flumes and washer water
	4. Losses inside the factory readily accountable.
	(a) In pulp
	(b) In pressed pulp water
	(c) In lime flume
	(d) In Steffen hot filtrate
	5. Losses inside the factory not readily accountable
	(a) Factory waste, leaks, spills and entrainment
	(b) By inversion in process
	(c) By microbiological action
	(d) Entrainment to boiler feed and a portion of that entrained to diffusion supply water

tanks and pieces of equipment, and determining their dry substance contents and purities. The dry substance is expressed as °Brix or as % rds, and the purity as %.

The quantities of juices, syrups, and massecuites are usually expressed in volumetric units determined from direct measurements of calibrated tanks. Weights per cubic foot and weights per gallon are available in tabulated form for pure sucrose solutions (15), and are generally assumed to be valid for the impure process syrups. It is convenient to correct the measured volumes to the standard temperature of 20 °C (6), in which the following relations are true:

$$\textit{lbs dry substance} = \textit{(cu ft juice)} \times \textit{(lbs/cu ft)} \times \textit{(rds/100)} \quad (5)$$

$$\textit{lbs total sugar} = \textit{lbs dry substance} \times \textit{(purity/100)} \quad (6)$$

$$\textit{lbs nonsugars} = \textit{lbs dry substance} - \textit{lbs total sugar.} \quad (7)$$

And from the above,

$$\textit{lbs sugar in (potential) molasses} = 1.5\ \textit{(lbs nonsugar)} \quad (8)$$

and by difference,

$$\textit{lbs. sugar in (potential) production} = \textit{lbs total sugar} - \textit{lbs sugar in (potential) molasses.} \quad (9)$$

A hypothetical stock in process statement is shown in Table 24-2.

Separating the sugar in the potential granulated from that in the potential molasses, the material balance equation (4) becomes,

Sugar entering = sugar produced + potential sugar production in process at end of period − potential sugar production

in process at beginning of period + sugar in molasses produced + sugar in potential molasses in process at end of period − sugar in potential molasses in process at beginning of period + sugar losses. (10)

Table 24-2 Statement of Stock in Process. Inventory of Sugar in Process of Manufacture

Product	rds	Apparent Purity	Cubic Feet	*Lbs Dry Substance Per Cu Ft	Weight Total Dry Substance Lbs	Weight of Sugar, lbs
	(1)	(2)	(3)	(4)	(5)	(6)
Steffen	—	88.0	—	—	12 500	11 000
Beet end	14.0	88.5	15 092	9.21	139 000	123 000
Evaporators	28.4	88.9	681	19.82	13 500	12 000
Thick juice	55.8	88.5	296	43.93	13 000	11 500
Std. liquor	60.9	92.3	1 712	49.07	84 000	77 500
White massecuite	91.3	92.6	1 203	84.82	102 000	94 500
Intermediate massecuite	89.8	87.1	1 539	82.82	127 500	111 000
Raw massecuite	91.4	79.0	7 192	84.95	611 000	483 000
White mch. syrup	78.5	87.8	838	68.64	57 500	50 500
Inter. mch. syrup	77.1	76.5	508	66.98	34 000	26 000
Molasses**	85.2	61.8	221	76.89	17 000	10 500
Low melter	69.5	95.0	172	58.27	10 000	9 500
Total					1 221 000	1 020 000
(7) Weight total nonsugars = (5) — (6)					201 000	
(8) Weight sugar in molasses at 60 purity = 1.5 × (7)					301 500	
(9) Weight total granulated equivalent = (6) — (8)					718 500	

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Time

$$* \left(\text{Density} \times \frac{\text{rds}}{100} \right)$$

**If molasses as measured is over 60 purity, the amount of sugar involved in the higher purity is credited to potential granulated.

This is the basis for the complete material balance, or yields and losses statement, in Table 24-3.

The statement for a Steffen house is a little more complicated because of recirculation of molasses, and will be considered in connection with molasses accounting. The terms *"sugar produced + potential sugar production in process at end − potential sugar production in process at beginning"* are the net, or corrected, sugar production.

"Extraction" is defined as the ratio of the net sugar produced to sugar entered

$$\text{Percent extraction} = \frac{\text{net sugar produced} \times 100}{\text{sugar entered}} \tag{11}$$

This ratio is such an important measure of the efficiency of the factory in recovering sugar, that the factory sugar balance is often called the "extraction statement." The alternative designation "statement of sugar yields and losses" is a better description, since the meticulous accounting of losses is the first requisite to achieving and maintaining high extraction.

Statement of Sugar Yields and Losses

The yields and losses statement in Table 24-3 is based on sugar in beets slices, and excludes sugar losses outside the factory. Such a statement, starting at the

Table 24-3 Hypothetical Extraction for Non-Steffen Factory

Statement of Factory Yields and Losses

Factory

Beets Introduced — Period From Oct. 1 to Nov 1 Beets

Based On: BEETS SLICED — 62,000 Tons — 84.6 Purity — 16.50% Sugar

Weight ..... 124,000,000 pounds
Sugar % 16.50
Purity 84.6

	Pounds	% On Beets	% On Sugar in Beets
SUGAR ENTERED			
In beets sliced	20,460,000	16.50	100.00
Total	20,460,000	16.50	100.00
GRANULATED SUGAR PRODUCTION			
Reported produced	16,891,000	13.62	82.56
Available in process, end of period	638,000	.52	3.12
Total	17,529,000	14.14	85.68
Available in process, beg. of period	650,000	.53	3.18
Total net production	16,879,000	13.61	82.50
SUGAR IN MOLASSES			
Reported produced	2,881,000	2.32	14.08
Available in process, end of period	114,000	.10	.56
Total	2,995,000	2.42	14.64
Available in process, beg. of period	106,000	.09	.52
Total net production	2,889,000	2.33	14.12
SUGAR LOSSES			
Known pulp	542,000	.44	2.65
Known lime cake	41,000	.03	.20
Known inversion	26,000	.02	.13
Known total waste	45,000	.04	.22
Total accounted	654,000	.53	3.20
Undetermined	38,000	.03	.18
Total	692,000	.56	3.38

.....Chief Chemist November 19

point (the slicers) where the beets actually enter the factory, is a convenient one for the factory superintendent, since it considers only those losses which are under his direct control. It is sometimes called the *technical statement.*

Sugar Balance for a Continuous Diffuser

Let W_B, W_J, W_P, and W_W be the weights of beets as cossettes, raw juice, wet pulp, and diffuser supply water, respectively, and let P_B, P_J, and P_P be the corresponding sugar contents, expressed as decimal fractions. Also let "$S_B = P_B W_B$," etc. be the weight of sugar in the cossettes, etc. The basic material balance equations are:

$$W_B + W_W = W_J + W_P \tag{12}$$

$$S_B = S_J + S_P \tag{13}$$

$$P_B W_B = P_J W_J + P_P W_P \tag{14}$$

If W_B, W_W, and W_J are all determined, one can estimate the loss in the diffuser. If only one or two of these variables are determined, the sugar balance can be calculated using (12) and (14). There are seven possible cases, with solutions as indicated.

Case 1. The Weights of Cossettes and Diffusion Juice are Measured.

$$W_P = \frac{P_B W_B - P_J W_J}{P_P} \tag{15}$$

W_W, if desired, is then found by substituting numerically in (12).

Case 2. The Weights of Cossettes and Diffusion Supply Water are Measured.

$$W_P = -\left[\frac{(P_B - P_J)\,W_B - P_J W_W}{P_J - P_P}\right] \tag{16}$$

W_J, if desired, is found by numerical substitution in (12).

Case 3. The Weights of Diffusion Juice and Diffusion Supply Water are Measured.

$$W_B = \frac{(P_J - P_P)W_J + P_P W_W}{P_B - P_P} \tag{17}$$

$$W_P = \frac{P_B W_W - (P_B - P_J)W_J}{P_B - P_P} \tag{18}$$

Case 4. The Weight of Cossettes Only is Measured. We must assume a value for W_P, say,

$$r = W_P/W_B$$

Then

$$W_J = \frac{(P_B - rP_P)W_B}{P_J} \tag{19}$$

and

$$W_W = \frac{[P_B - P_J + r(P_J - P_P)]\,W_B}{P_J} \tag{20}$$

The assumption is made that $r = 1$, then,

$$W_W = W_J = \frac{(P_B - P_P)\,W_B}{P_J} \tag{21}$$

Dividing (21) by $W_B/100$, the formula for draft by sugar content is obtained for the continuous diffuser:

$$D = \frac{W_J \times 100}{W_B} = \frac{P_B - P_P}{P_J} \times 100 \tag{22}$$

This ratio is independent of any weight determinations, and is calculated from the analytical data only.

Case 5. The Weight of the Diffusion Juice Only is Measured. Again, $r = W_P/W_B$.

$$W_B = \frac{P_J W_J}{P_B - rP_P} \tag{23}$$

Again, assuming $r = 1$, then,

$$W_B = \frac{P_J W_J}{P_B - P_P} \tag{24}$$

Case 6. The Weight of the Diffuser Supply Water Only is Measured. Setting $r = W_P/W_B$,

$$W_B = \frac{P_J W_W}{P_B - P_J + r(P_J - P_P)}, \tag{25}$$

$$W_J = \frac{(P_B - r(P_P)W_W}{P_B - P_J + r(P_J - P_P)} \tag{26}$$

Assuming r = 1,

$$W_B = \frac{P_J W_W}{P_B - P_P} \tag{27}$$

and again,

$$W_J = W_W. \tag{28}$$

Case 7. The Pulp Press Water is Returned to the Diffuser. Two general methods are offered for this case, the *Schneider formulas* (7), and the *Tasco formulas* (1).

The Schneider Formulas. These involve the following new symbols: D_B and D_J, the dry substance contents of the cossettes and diffusion juice, respectively; P_{pp} and D_{pp}, the percent sugar and dry substance of the pressed pulp expressed as decimal fractions; L, the pulp loss, expressed as a decimal fraction, percent on beets, and D, the percent draft.

For these calculations, determinations must be made of the moisture contents of the cossettes, and the pressed pulp; of the percent sugar of the cossettes, diffusion juice, and pressed pulp; and of the purity of the diffusion juice, which yields D_J [= P_J/(juice purity)].

$$L = \frac{P_{pp}(P_B D_J - D_B P_J)}{P_{pp} D_J - D_{pp} P_J} \tag{29}$$

$$D = \frac{100(B_D P_{pp} - P_B D_{pp})}{P_{pp} D_J - D_{pp} P_J} \tag{30}$$

The Tasco Formulas. For these computations, the percent sugar in the pressed pulp water and the moisture in the pressed pulp must be determined, in addition to sufficient determinations of the percent marc of the cossettes. The purity of the pulp press water is assumed to be 50.0%.

The following additional symbols are used; P_{PW}, the percent sugar of the pulp press water (determined hourly by Amalgamated); Mc_B, the percent marc of the cossettes (determined for early, mid-, and late campaign); M_{pp}, the percent moisture in pressed pulp (determined on shift composites and average for the day); Q_{PW}, the purity of the pulp press water, or 50.0%; Mc_{pp}, the marc in pressed pulp, calculated by (32); and U_{pp}, the weight of pressed pulp on beets. R, the ratio of the percent solids to the percent moisture in the pulp press water, is obtained by:

$$R = \frac{P_{PW}/Q_{PW}}{1 - (P_{PW}/Q_{PW})} \tag{31}$$

$$R = \frac{2\,P_{PW}}{1 - 2\,P_{PW}} \tag{31a}$$

It is assumed that the ratio, R, holds for the liquid phase of the pressed pulp from which it has been separated.

$$Mc_{pp} = 1 - M_{pp} - RM_{pp} \tag{32}$$

$$P_{pp} = RM_{pp}/2 \tag{33}$$

$$U_{pp} = Mc_B/Mc_{pp} \tag{34}$$

$$L = P_{pp}\, U_{pp} \tag{35}$$

Following is an example of the calculation: If Mc_B is 4.7, P_{PW} is 1.5, M_{pp} is 78.5, and the assumed pulp press water purity is 50,
from (31), $R = 2(0.015)/[1\text{-}2(0.015)] = 0.0309$
from (32), $Mc_{pp} = 1 - (0.0309 \times 0.785) = 0.1907$
from (33), $P_{pp} = 0.0309(0.785)/2 = 0.0121$
from (34), $U_{pp} = 0.047/0.1907 = 0.2465$
from (35), $L = 0.0121\ (0.2465) = 0.0030 = 0.30\%$

Obtaining the Diffuser Data. The losses at the diffuser are determined by the use of the available weighing and metering equipment, the laboratory analyses for percent sugar in cossettes, pulp, and diffusion juice, the percent moisture in the cossettes and pulp, the percent marc in the cossettes, and the appropriate statement of the material balance from the above compilation. However, the situation may be complicated by many factors.

If the beets are weighed before the slicers, the sample of cossettes will be contaminated with condensate. In this case, it is a frequent practice to shut off the steam just before and during the period the cossette sample is taken. The quantity of steam used by the slicers may be estimated from the temperature increase of the cossettes, but this involves inaccurate measurements and assumptions, and is of little value for purposes of sugar accounting.

As a rule, the continuous diffusers in this country employ continuous scales for weighing the cossettes, and a diffuser-supply water meter. Some use is made of steam flow meters, to measure the water condensed on the cossettes.

Known Losses, Except Molasses

The sugar lost in the lime flume is based on the weight of lime rock charged to the kiln, and a "CaO balance." There is no CaO, as such, in the rock, and not all the lime in the lime flume is in that form; the term "percent CaO" as here used means the total lime content, calculated as CaO. The normal practice is to weigh the skips of rock used in loading the kiln, and to determine, usually on a weekly composite sample, the percent CaO equivalent in the rock. An estimate may be made of the spalls discarded from the slaker, and deducted from the weight of lime loaded into the kiln to obtain a net CaO produced. The percent CaO in the lime flume is determined by acid-base titration, and the percent sugar by polarization.

Then the following relations are valid:

$$\textit{Weight of CaO in rock} = \textit{Weight of rock} \times \textit{percent CaO in rock}/100, \tag{36}$$

where the weight of rock is corrected for spalls.

$$\text{Weight of lime flume} = \frac{\text{Weight of CaO in rock} \times 100}{\text{percent CaO in lime flume}}. \tag{37}$$

$$\textit{Weight of sugar lost to lime Flume} = \textit{weight of lime Flume} \times \textit{percent sugar in lime Flume}/100. \tag{38}$$

In a Steffen house some lime is lost to hot filtrate, which is determined by weighing, or measuring the Steffen filtrate, and measuring the CaO content by titration with standard acid. Then these relations are valid:

Wt. CaO in Steffen hot filtrate = Wt. Steffen hot filtrate × percent CaO in Steffen hot filtrate/100, (39)

Wt. net CaO to carbonation = Wt. CaO in rock − Wt. CaO in Steffen hot filtrate, (40)

Wt. sugar lost in lime flume = Wt. net CaO to carbonation × percent sugar in lime flume/percent CaO in lime flume. (41)

In the continuous Steffen process, the starting point for the CaO balance is the weight of lime powder on the belt feeder to the vortex pot, since the powder is easier to sample than the rock, and the lime addition to the Steffen process is calculated more accurately on the basis of the powder weight. A friction clutch driven by the belt feeder turns a counter which is calibrated in feet of belt traveled, and the weight of lime powder per lineal foot of belt is determined periodically. Then the weight of CaO to the Steffen process is,

$$\frac{\text{feet of belt} \times \text{lbs lime per foot} \times \text{percent CaO in powder}}{100} \quad (42)$$

In general, the CaO to carbonation calculated from the limerock charge to the kiln is higher than that calculated from the measurement of lime powder to the process. This difference is usually rationalized in the CaO balance as "CaO direct to carbonation." When milk of lime is added to the second carbonation, or to the saccharate milk, or during periods when the Steffen process is shut down, this designation is correct. Otherwise, it represents loss of lime in dust or leaks, and may also reveal analytical and measuring inaccuracies.

Sugar lost in the Steffen hot filtrate is calculated readily from the percent sugar by polarization, and the weight of the filtrate. This weight may be determined by a tank scale, measuring tank, or weir, and sometimes with a weir meter. Volumetric measurements are of course combined with density determinations and recorded temperatures to give the weight. Displacement and orifice-type meters are not suitable for Steffen hot filtrate, because of its rapid scaling tendencies.

If the Steffen hot filtrate is not measured, the quantity may be determined from the analytical data and the amount of molasses worked.

The material balance for the cold and hot process is conveniently calculated by the method of Osborn and Davis (8) of The Great Western Sugar Company and is given in Table 24-4.

The Molasses Balance

In the straight house, the beets sliced represent all the sugar entering except for the minor sources, such as sugar from the previous campaign, pan-graining sugar, and tare laboratory samples. The main "loss," in addition to the diffuser loss and the lime flume loss is the sugar in molasses produced. Of course this "loss" may be recovered if the molasses is subsequently subjected to the Steffen process at another factory.

Molasses produced may be weighed in a tank scale, and sampled for polarization at the same point. If the molasses is pumped directly into tank cars for shipment, it is weighed in the cars on railroad scales. If pumped into a storage tank, the molasses is best gauged with an open-tube mercury manometer. To keep molasses from flowing into the manometer, the mercury is first blown up with air pressure to a head higher than the head of molasses. The valve to the air line is closed, and the valve to the molasses tank opened. When pressure equilibrium is reached, the difference in mercury level is measured in inches. For best accuracy, the temperature of

Table 24-4 Material Balance for Steffen Cold and Hot Process

Symbol	Item	Example
a	Tons sugar to coolers	100.00
a'	Percent sugar in solution for coolers	6.00
b'	Tons solution for cooler, 100 a/a'	1667
b'	Tons lime powder	135
c	Tons cold wash water	500
d	Tons total, $b + b' + c$	2302
e	Percent sugar in cold sacch. cake	13.50
f	Percent sugar in cold water	.57
g	Difference, $e - f$	12.93
h	df	1312
h'	100 $a - h$	8688
i	Tons cold saccharate cake*, h'/g	672
j	Tons cold filtrate, $d - i$	1630
k	Tons sugar in cold filtrate, 0.01 fi	9.29
m	Temp, °C, cold filtrate lvg. heat exchanger†	20
m'	Temp, °C, hot filtrate	87
n	Condensate factor,‡ $\frac{n' - m}{n' - m'}$	1.12
o	Tons cold filtrate and condensate, jn	1826
p	Tons hot wash water	40
q	Tons total, $o + p$	1866
r	Percent sugar in hot sacch. cake	11.00
s	Percent sugar in total filtrate	.15
t	Difference, $r - s$	10.85
u	qs	280
v	100 $k - u$	649
w	Tons hot sacch. cake, v/t	60
x	Tons total filtrate, $q - w$	1806
y	Tons sugar in total filtrate, 0.01 sx	2.71
z	Tons sugar recovered, whole process, $a - y$	97.29
z'	Alkalinity total filtrate	.34
z''	CaO lost in total filtrate, 0.01 $z'x$	6.1

*$ei + f(d-i) = 100\ a$, or $i = (100\ a - df)/(e-f)$

†Correct if necessary for extra heating by steam injection. If the filtrate water or finished cooler solution is heated by steam or *vapor* injection at some other point than in the hot saccharate heating tank, this addition of condensate should be taken into consideration by subtracting from item *(m)* the number of degrees of temperature rise produced by such heating, and using the corrected figure in the calculation of item *(n)*. Such a correction does not apply if the finished cooler solution is heated indirectly in a heater without the addition of condensate. The use of the value *(n')*, see item *(n)*, which does not exactly correspond to the kind of vapor used in such supplementary heating is of no consequence for the few degrees of temperature rise involved.

††The values given in this footnote represent the enthalpy of various vapors. The value that represents the particular vapor used for direct heating should be employed. n' for exhaust = 645, for first vapor = 640, and for third vapor = 636.

the mercury should also be taken. Then,

Weight of molasses in tank in pounds = area of tank in sq in × inches mercury × psi per inch mercury at 0 °C × density of mercury, t °C/density of mercury, 0 °C. (43)

where t °C is the temperature of the mercury. This measurement represents only the molasses above the point of connection of the manometer to the tank. Volumetric measurement of molasses is not satisfactory, because of the wide variations in quantity of foam and temperature, which both vary the apparent density.

Table 24-5 Daily Molasses calculation for a Steffen Factory

	Tons Molasses
CASE 1. *Molasses Worked is Weighed*	
On Hand—yesterday in storage tanks	700.000
On Hand—today in storage tanks	710.000
Difference	+10.000
Molasses worked	150.000
Apparent produced	160.000
Minus foreign molasses received	70.000
Difference	90.000
Plus molasses shipped	80.000
Net produced	170.000
CASE 2. *Molasses Produced is Weighed*	
On Hand—yesterday in storage tanks	700.000
On Hand—today in storage tanks	710.000
Difference	—10.000
Molasses produced	170.000
Apparent worked	160.000
Plus foreign molasses received	70.000
Sum	230.000
Minus molasses shipped	80.000
Net molasses worked	150.000

The Steffen House Balance

For a balance in a Steffen factory, the following quantities must be known: 1) the weight of foreign molasses received for processing; 2) the weight of discard molasses shipped; 3) the storage tanks inventory, by gauging; 4) either the weight of molasses worked (entered to Steffen process) or molasses produced.

Table 24-5 shows a hypothetical daily calculation for the two possible cases. In the calculation of this table, dilution of molasses with steam has not been considered. This dilution can be calculated by a material balance, if both the molasses worked and molasses produced are weighed.

In a Steffen house there is a net loss of sugar to molasses only if there is more sugar in the molasses produced than in the molasses worked. If there is more worked than produced, due to processing of foreign molasses, this additional sugar worked is added to the sugar introduced into the factory. Even in this case, however, there may be a real molasses sugar loss in the discard molasses. Table 24-6 is a hypothetical extraction statement for a Steffen factory working additional molasses, and Table 24-8 is a molasses balance based on the same data, which clearly distinguishes between foreign and house molasses, and shows the net molasses produced as discard.

So far, the following losses have been considered: pulp and pulp press water, lime flume, Steffen hot filtrate, and molasses. All these are fairly amenable to direct measurement, and are considered as "known" or "accountable" losses. However, an extraction statement based on beets sliced which considers only these losses, will usually fail to balance by one or two percent, and this additional loss is usually considered "unknown" or "unaccountable."

Evaluation of Unaccountable Losses, Inside the Factory

These can arise from the following causes: 1) polarizable substances, other than sucrose, in the beets sliced, referred to as "false polarization;" 2) factory leaks and spills; 3) entrainment into evaporator condensate, and into pan and evaporator condensers; 4) inversion during processing; and 5) the destruction of sucrose by microorganisms or chemicals in the process.

Table 24-6 Hypothetical Extraction Statement For a Steffen Factory Producing Excess Molasses

Statement of Factory Yields and Losses

Factory

Beets Introduced — Period From Oct. 1 to Nov. 1

Beets

Based On: BEETS SLICED — 66,000 Tons — 84.2 Purity — 15.91% Sugar

Weight ..... 132,000,000 pounds
Sugar % 15.91
Purity 84.2

	Pounds	% On Beets	% On Sugar in Beets	% On Total Sugar Introduced
SUGAR ENTERED				
In beets sliced	21,000,000	15.91	100.00	100.00
Total	21,000,000	15.91	100.00	100.00
GRANULATED SUGAR PRODUCTION				
Reported produced	19,290,000	14.61	91.86	91.86
Avail in process. end of period	726,500	.55	3.46	3.46
Total	20,016,500	15.16	95.32	95.32
Avail in process. beg. of period	716,500	.54	3.41	3.41
Total net production	19,300,000	14.62	91.90	91.90
SUGAR IN MOLASSES				
Reported produced	4,335,000	3.28	20.64	20.64
Avail. in process. end of period	307,500	.24	1.46	1.46
Total	4,642,500	3.52	22.10	22.10
Avail. in process. beg. of period	302,500	.23	1.44	1.44
Total net production	4,340,000	3.29	20.66	20.66
Total worked in Steffen	3,846,000	2.91	18.31	18.31
Difference (Molasses loss)	−494,000	—.38	−2.35	—2.35
SUGAR LOSSES				
Diffuser	719,000	.54	3.42	3.42
Lime cake	52,000	.04	.25	.25
Steffen filtrate	175,000	.13	.83	.83
Inversion	76,000	.06	.36	.36
Waste water	79,000	.06	.38	.38
Boiler feed				
Total accounted	1,101,000	.83	5.24	5.24
Unaccounted	105,000	.08	.50	.50
Total	1,206,000	.91	5.74	5.74

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False Polarization. "Destructible" polarization is due to dextrorotatory substances of unknown composition, which are destroyed under the strongly alkaline conditions of carbonation, but not in the relatively mild conditions of the lead acetate digestion. The laevo-rotatory fructose, which may be present in the beets, is removed in the lead acetate digestion preceding polarization. It is usually determined by the method of Stanek and Vondrak, who found about 0.13% with juices from Czechoslovakian beets (18). About the same amounts were found in Colorado practice (13). McCann (10) states, however, that little was found in California juices. If determined, false polarization can be deducted from the sugar charged to the factory as it does not represent a real loss of sugar.

Factory Leaks and Spills. These, and most entrainments eventually pass to the factory waste water. The polarization of the waste is ordinarily determined hourly, and the quantity of sugar lost may be estimated with fair accuracy if the volume of waste water is known. This may be measured directly by a weir, or by calibrating

and recording the time of operation of the waste water pumps. An indirect measurement of the waste water volume may be made by metering the water supply to the factory and deducting from this volume, the volume of water lost by other routes, such as Steffen hot filtrate.

Entrainments. Sugar entrained to the boiler feed is, of course, destroyed immediately. The harmful effect on the boilers is of considerably greater importance than the loss of sugar. However, the amount may be estimated with a low order of accuracy by comparing the routine α-naphthol tests of the boiler feed water with a color chart made up from tests on solutions of known sugar content. The anthrone test (12) may also be used for this purpose and is better suited for quantitative estimation.

Table 24-7 Hypothetical Extraction Statement for a Steffen Factory Working Excess Molasses

Statement of Factory Yields and Losses

Factory

Beets Introduced — Period From Nov. 1 to Dec. 1

Based On: BEETS SLICED — 62,000 Tons — 84.2 Beets Purity — 15.31% Sugar

Weight 124,000,000
Sugar % 15.31
Purity 84.2

	Pounds	% On Beets Sliced	% On Sugar in Beets Sliced	% On Total Sugar Entered
SUGAR ENTERED				
In beets sliced	18,987,000	15.31	100.00	99.19
In additional molasses worked	156,000	.13	.82	.81
Other				
Total	19,143,000	15.44	100.82	100.00
GRANULATED SUGAR PRODUCTION				
Reported produced	17,903,000	14.44	94.29	93.52
Avail. in process, end of period	718,500	.58	3.78	3.75
Total	18,621,500	15.02	98.07	97.28
Avail. in process, beg. of period	726,500	.59	3.82	3.80
Total net production	17,895,000	14.43	94.25	93.48
SUGAR IN MOLASSES				
Reported produced	4,222,000	3.40	22.24	22.06
Avail. in process end of period	301,500	.25	1.58	1.57
Total	4,523,500	3.65	23.82	23.63
Avail. in process beg. of period	307,500	.25	1.62	1.61
Total net production	4,216,000	3.40	22.20	22.02
Total worked in Steffen	4,372,000	3.53	23.02	22.83
Difference	156,000	.13	.82	.81
SUGAR LOSSES				
Diffuser	777,000	.63	4.09	4.06
Lime cake	50,000	.04	.26	.26
Steffen filtrate	188,000	.15	.99	.98
Inversion	78,000	.06	.41	.41
Waste water	82,000	.07	.43	.43
Boiler feed	1,000	.00	.01	.00
Total accounted	1,176,000	.95	6.19	6.14
Unaccounted	72,000	.06	.38	.38
Total	1,248,000	1.01	6.57	6.52

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Table 24-8 Hypothetical Monthly Beet Molasses Balance Based on Data of Table 24-7

	Pounds Sugar	Tons Molasses
Home Molasses		
On hand Nov. 1		
Actual	837,000	824.000
In process	307,500	307.500
Total	1,144,500	1,131.500
On hand Dec. 1		
Actual	1,320,000	1,293.000
In process	301,500	301.500
Total	1,621,500	1,594.500
Sold	386,000	380.000
Net production	863,0000	843.000
Steffenized	3,353,000	3,331.000
Gross production	4,216,000	4,174,000
Foreign Molasses		
On hand Nov. 1	236,000	232.000
Receipts	877,000	860.000
On hand Dec. 1	94,000	92.000
Steffenized	1,019,000	1,000.000
Total Molasses		
Net production		
Workable	412,000	403.000
Discard	451,000	440.000
Total	863,000	843.000
Foreign molasses Steffenized	1,019,000	1,000.000
Difference + or —	—156,000	—157.000
Steffenized total	4,372,000	4,331.000
Gross production	4,216,000	4,174.000

Considerable success has been found with the use of continuous ion detectors for this purpose. Miller, *et al*, (11) reported on the use of the Marius potassium flame photometer (19,22). This satisfactorily measured the concentration of the potassium ion in boiler feedwater, which is closely correlated with the sugar content. All of the factories of the Spreckels Sugar Company have since been equipped with Marius flame detectors.

Inversion in Process. Inversion in the process causes a significant loss of sugar—up to 2 or 3% of sugar introduced in some cases. Since most of the reducing sugars present in the diffusion juice are destroyed in first carbonation, most of the invert found in the molasses produced is assumed to have been caused by subsequent inversion. If invert sugar is simultaneously produced and destroyed in the evaporators and the sugar end of the factory, an estimate of sugar lost by inversion based on this assumption is too low.

Loss Through Microorganisms. Destruction of sugar by bacteria and other microorganisms is difficult to measure directly. If it is large, it may be determined by difference, after all the other losses above are accounted for. Generally, however, this loss is less than the error of the other measurements. In some cases, significant bacterial action may be demonstrated by secondary effects; for example, the drop in pH in the diffuser (21).

The Beets Purchased Statement

An extraction statement based on beets purchased is illustrated in Table 24-9. This is for a factory where beets are purchased on a clean beets basis. Thus, sugar in crowns is added to sugar purchased. The total outside loss is the difference between

Table 24-9. Extraction Statement Based on the Data of Table 24-7, with the Addition of Data on Beets Purchased and Outside Losses

Statement of Factory Yields and Losses

_____ Factory
Period from Nov. 1 to Dec. 1

	Tons	%Sugar	%On Beets Received
Beets purchased	58,740	16.20	97.01
Crowns	1,810	11.06	2.99
Total beets received	60,550	16.05	100.00
Beets sliced	62,000	15.31	102.39

	Pounds	%On Sugar in Beets Received	%On Sugar in Beets Purchased	%On Net Sugar Entering Factory
Sugar In				
Beets Purchased	19,032,000	97.94	100.00	
Crowns	400,000	2.06	2.10	
Beets Received	19,432,000	100.00	102.10	
Sugar in Beets Sliced	18,987,000	97.71	99.76	
Sugar Lost Outside Factory				
Rail Transit	80,000	.41	.42	
Piling	197,000	1.01	1.03	
Tailings	81,000	.42	.43	
Loss in Flume	101,000	.52	.53	
Total Accounted	459,000	2.36	2.41	
Unaccounted	−14,000	−.07	−.07	
Total	445,000	2.29	2.34	
Net Sugar Entering Factory				
In Beets Sliced	18,987,000	97.71	99.76	99.19
In Additional Molasses worked	156,000	.80	.82	.81
Total	19,143,000	98.51	100.58	100.00
Net Granulated Sugar Produced	17,895,000	92.09	94.03	93.48
Sugar in Molasses				
Net Production	4,216,000	21.70	22.15	22.02
Worked in Steffen	4,372,000	22.50	22.97	22.84
Difference	156,000	.80	.82	.81
Sugar Lost in Factory				
Diffuser	777,000	4.00	4.08	4.06
Lime Cake	50,000	.26	.26	.26
Steffen Filtrate	188,000	.97	.99	.98
Inversion	78,000	.40	.41	.41
Factory Waste Water	82,000	.42	.43	.43
Boiler Feed Water	1,000	.00	.01	.00
Total Accounted	1,176,000	6.05	6.18	6.14
Unaccounted	72,000	.37	.37	.38
Total	1,248,000	6.42	6.55	6.52
Total Sugar Losses				
Accounted	1,635,000	8.41	8.59	8.54
Unaccounted	58,000	.30	.30	.30
Total	1,695,000	8.71	8.89	8.84

total sugar entering and sugar in beets sliced, and is broken down into the quantities estimated by the method in the next section. The accuracy of these estimates is low. The losses inside the factory are the same as described.

Losses Outside the Factory*

A yields and losses statement based on sugar in beets purchased is constructed on the same principles as the beets sliced statement. Sugar entering is calculated from the delivered weight of beets, and the tare laboratory analysis (see Chapter III). In addition to the data in the beets-sliced statement, sugar losses occurring outside the factory are involved.

These may be 1) sugar loss in beets during transit from remote receiving stations to the factory, 2) loss in bins and yard storage, 3) loss in beet piles, 4) loss of beet fragments and tails in trash catchers and the like, and 5) loss of sugar to the fluming water.

Losses During Transportation. Transit, storage, and piling losses have been the subject of numerous investigations (3,17). Transit losses can become appreciable when beets are shipped long distances in a hot climate, as in California. These losses can be estimated by weighing and sampling beets into and out of rail cars, although this is expensive. A better method may be that of Fort and Stout (9). Samples are prepared according to their directions, and may be transported in cars with the beets and recovered for analysis. The losses found in the samples may then be extrapolated to estimate the loss for the cars represented. Spreckels Sugar Company (17) has found a relationship between the rate of sugar loss in beets in transit or storage, and the mean ambient air temperature. For California beets in 1966-67, the following relation was found valid:

$$\ln Y_c = 0.0415X - 1.9445 \quad (44)$$

where $\ln Y_c$ is the natural logarithm of the sugar loss in beets, expressed as pounds of sugar per ton-day storage, and X is the mean ambient air temperature during the storage period in °F.

Barr, Mervine and Bice (3) reported the sugar loss rate of beets in storage to be

$$\ln Y_c = 0.0418X - 3.1567 \quad (45)$$

Piling and Normal Storage Losses. Losses in piles are the most significant outside-factory loss of sugar. See also Chapter IV. Equation (44) has been found to apply to beets in static as well as transit storage, in California. The Spreckels Sugar Company has found that replicate samples, made up from a truck load out of one beet field, is the best for studying sugar losses in piles under practical conditions. Each replicate is contained in an open-mesh, plastic-fiber bag. The number of replications must be very large for statistically significant results.

Losses in Tails and Flume Water. Loss of sugar in tails in the trash catcher is determined by occasional sampling and sugar analysis, and an estimate of the weight of trash. The latter weight may be determined directly by weighing the trash trucks, if this method of disposal is used.

Loss of sugar by diffusion to the beet flume water is calculated from occasional polarizations of flume water, and an estimate of its quantity.

*By R.E. Hallbeck

Accounting for Other Process Materials

Lime and coke are important process supplies. Their purchased weights are known, and they are weighed again into the kiln skip. The distribution of lime in the factory is detailed in the above-described CaO balance for lime flume calculations.

Other important process materials for which records are kept and usage reported on the factory technical reports are soda ash, sulfur or sulfur dioxide, filter aid, filter cloth, carbon, sodium aluminate, boiler treatment chemicals, settling aids, evaporator treatment chemicals, boil-out chemicals (muriatic acid, caustic soda, and inibitors) and foam breakers.

Fuel to the boilers and steam generated are considered in Chapter XX. The steam balance is detailed in Chapter XI.

Process Control

Any physical measurement or chemical analysis which indicates the magnitude of a process variable may be considered process control. In general, instrumentation and chemical analysis are outside the scope of this chapter. There are, however, a number of derived functions which are frequently useful in sugar factory operations.

Elimination

Assume that materials, such as cossettes and juices enter a process at a given percent purity, p_E, and leave at a higher percent purity, p_L. Then the percent nonsugar elimination is given by

$$\text{percent elimination} = \frac{10\,000\,(P_L - P_E)}{p_L\,(100 - P_E)} \tag{46}$$

This formula assumes only that nothing other than nonsugars is lost in the process. It is applicable to the diffuser, where p_E is the purity of the cossettes and p_L is the purity of the diffusion juice.

Crystallization

If the massecuite purity, p_F, and the green syrup purity, p_G, are known, the percent crystallization on total sugar (sucrose) is given by

$$\text{percent gross crystallization} = \frac{10\,000\,(p_F - p_G)}{p_F\,(100 - p_G)}, \tag{47}$$

The percent crystallization on total dry substances is given by

$$\text{percent gross crystallization} = \frac{100\,(p_F - p_G)}{100 - p_G} \tag{48}$$

The net crystallization, or net yield, takes into consideration the purity of the raw sugar.

$$\text{percent net crystallization} = \frac{100\,(p_F - p_M)\,p_S}{p_F\,(p_S - p_M)}. \tag{49}$$

Percent net crystallization on total dry substance, likewise, is,

$$\text{percent net crystallization} = \frac{100\,(p_F - p_M)}{p_S - p_M}, \tag{50}$$

where p_F is the massecuite purity, p_M the machine syrup purity, and p_S the purity of the washed sugar.

The percentage of crystals in the massecuite is given by,

$$\text{percent crystals} = \frac{(p_F - p_G)\, D_F}{100 - p_G}, \tag{51}$$

where D_F is the percentage of dry substance in the massecuite.

Carbon Dioxide Absorption in Carbonation

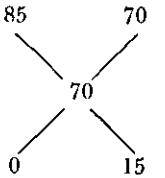
If the amount of carbon dioxide is determined in the gas going to carbonation with the Orsat apparatus, and in the carbonation vent stack, the percent absorption is given by

$$\text{percent absorption} = \frac{10\,000\,(p_E - p_L)}{p_E\,(100 - p_L)} \tag{52}$$

where p_E and P_L are the percentages of carbon dioxide in the gas entering and leaving.

Rectangular, or Spider Diagram

Suppose it is necessary to mix 85% rds syrup and water to make 100 pounds of 70% rds syrup. Considering water as containing 0% solids, a rectangle is set up by placing the desired percent rds in the center, the percent rds values of the original solutions on the side, and then completing the rectangle by subtracting the smaller numbers from the larger ones, along the diagonals, as shown below.

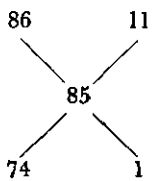


In this example, 70 pounds of 85% rds syrup and 15 pounds of water (0% rds) were required to make 85 pounds of syrup. Since 100 pounds were desired, we multiply these quantities by 100/85.

$$\begin{aligned} 70 \times 100/85 &= 82.35 \text{ lbs 85\% rds syrup} \\ 15 \times 100/85 &= \underline{17.65} \text{ lbs water} \\ 100 &\quad \text{lbs 70\% rds syrup.} \end{aligned}$$

This method is obviously applicable to the dilution of acids, bases and any other solution where the compositions are known.

It may be used to estimate pan purities. For example, suppose the high raw pan contains 1000 cu ft of massecuite at 86 purity. It is desired to lower the purity to 85 by boiling back 74-purity low machine syrup. What is the required final volume of the pan? Set up the spider diagram and subtract along the diagonals.



Then 11 parts of 86 purity material mixed with 1 part of 74-purity material, gives 12 parts of the finished massecuite. 1000 cu ft is 11 parts, then 1 part is

$$1000 \times 1/11 = 91 \text{ cu ft.}$$

Thus, low machine syrup should be added to make a massecuite volume of 1000 + 91 = 1091 cu ft before "Brixing up." Since the measurement of syrup added is made after boiling in the pan, the syrup density is unimportant.

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APPENDIX A

Glossary of Technical Terms in Common Use in the Beet-Sugar Factory, and Obscure Terms in this Text

Here are listed terms which are not defined, or not explicitly defined in the text. Reference should be made to the index when seeking the meaning of a technical term. In general, terms are not included which have obvious meanings, which are commonly used in other industries, or which are in use in only one factory, or in one company's factories. Terms followed by an asterisk (*) are as defined in "Technical Vocabulary for American Beet Sugar Processors," compiled by E.M. Hartmann, Journal American Society Sugar Beet Technologists. 19: (4) 345-364 1977.

Abbreviations: n. = noun; adj. = adjective; v. = verb
agr. = agricultural; biochem. = biochemical; biol. = biological;
bot. = botanical; chem. = chemical;
coll. = colloquial in the industry; geol. = geological;
instr. = instrumental; math = mathematical;
micr. = microbiological; phys. = physics;
phys. chem. = physical-chemical; zool. = zoological

accounted losses.* Quantities of sucrose determined or estimated by measurements and analyses to be escaping the process through known channels of loss.

activity. *chem.* The effective concentration of a substance.

abscissa. *math.* The horizontal axis in a two-coordinate graph.

absorption. *phys. chem.* The assimilation of molecules of gases or of dissolved substances, within solid bodies with which they are in contact.

acrylic. *chem.* Refers to a resin made by polymerizing (q.v.) esters of acrylic acid.

adsorbate. *chem.* Material adsorbed.

adsorption. *chem.* The adhesion of molecules of gases or of dissolved substances to the surfaces of solid bodies.

affination.* *coll.* Process for purifying an impure crystalline sugar by mixing it with syrup to form a magma, then spinning the magma with or without washing.

affination magma.* *coll.* Magma produced by mechanically mixing crystals with syrup (see *affination).*

after product. *coll.* Describes any material obtained after the white-sugar boiling, including remelt sugars, molasses, etc.

agglomerate* To intergrow mutually so as to form a conglomerate.

aldol condensation. *chem.* A chemical reaction involving two aldehydes or ketones, resulting in a longer carbon chain (e.g., $C_3 + C_3 = C_6$).

aldose. *chem.* Relating to aldehydes; i.e., monosaccharides are *aldose* when they are dehydroalcohols.

alkalinity.* *chem.* In the product streams of a beet-sugar factory, the result of a titration with standardized acid solution to a phenolphthalein endpoint or

equivalent pH, expressed as g CaO per 100 ml. In water analysis the endpoint must be specified and the result is usually expressed as ppm or gpg $CaCO_3$.

alluvial. *geol.* Composed of sand or gravel, deposited by running water.

alphanumeric. Pertains to characters consisting of letters, numbers, punctuation marks, and such signs as $, @, and #.

ammonia lines. Pipe vent lines for removal of noncondensable gases from various calandrias.

amorphous. *phys.* Not having crystalline shape.

analog. A physical variable which remains similar to another variable insofar as the proportional relationships are the same over some specified range. For example, a temperature may be represented by a voltage which is its analog.

angle of repose. *phys.* The angle formed by the surface of a pile of granular or particulate material with the horizontal surface on which it rests. A greater angle results in flow of the material.

anaerobic. *micr.* Living or active in the absence of free oxygen.

anion. *chem.* A negatively-charged ion (q.v.) in solution.

apc, apparent purity. *coll.* The percentage proportion of sugar determined by direct polarization on solids, the solids being determined by Brix hydrometer or by refractometer. When the solids are so determined, the results differ slightly from each other and from dry substance determined by drying, and purities based on them are specifically designated (see true purity).

ash. *chem.* The solid residue left after incineration in the presence of oxygen. In analysis of sugar products, sulfuric acid is added to the sample, and this residue as "sulfated ash" heated to 800 °C is taken to be a measure of the inorganic constituents. Sometimes determined indirectly by measure of the electrical conductivity of solutions of the products.

asymmetric. Not symmetrical.

asymptotic. *math.* Gradually approaching but never reaching a limit.

back-mixed crystallizer. *coll.* A crystallizer (or pan) having a continuous feed, a mixing of the contents within the crystallizer, and a continuous effluent product removal which represents the full range of residence times of the continuously nucleated or seeded crystals in the crystallizer. The crystals in the product stream withdrawn will have a corresponding full range of sizes.

bag filter. A filter made of a series of fabric bags hung vertically to spigots in a tube sheet contained in a filter tank; e.g., Taylor filter, being filled with the liquid to be filtered.

bar. *phys.* A pressure of 14.504 lbs/sq. in., or one megadyne per square centimeter at 0 °C and standard conditions of gravity.

barometric leg.* A column of pipe beneath a condenser operating at less-than atmospheric pressure, permitting the removal of condensate or condenser water by gravity.

bar screen.* Self-defining. A more descriptive term than "grizzly." Often used for separating beets from flume water.

base. *chem.* An oxide or hydroxide that will neutralize acidic material; i.e., produces hydroxyl ions.

batch cooler.* The classical reactor in the Steffen process, consisting of a vertical, cylindrical vessel equipped with a calandria heat exchanger (for cooling) and a propeller stirrer.

beet (pl: beets).* The sugarbeet root.

beet bins.* A-frame structures built over beet flumes for the short-term storage of beets at the factory site.

beet end.* The section of the factory encompassing the process from the beet flumes through the juice purification. May also include the evaporators, especially in older factories not originally designed for the extensive use of evaporator vapors in heating the vacuum pans.

beet feeder.* An apparatus which regulates the flow of beets in a beet flume; usually a variable-speed, rimless wheel whose spokes project downward into the flume.

beet flume.* A concrete-lined ditch or metal trough designed for the hydraulic transport of beets.

beet knife.* A rectangular piece of steel rolled or milled into a serrated shape for slicing beets into cossettes.

beet pile.* A stack of beets usually destined for long-term storage; i.e., more than a day or two.

beet pump.* A specially designed pump for elevating beets and flume water.

beet rasp.* A device designed to obtain representative samples of beet roots in the form of fine particles.

beet screen.* A mechanical device for cleaning the beets at the receiving station; usually a specially designed roller table.

beet slab.* A paved area, usually with embedded beet flumes, for the short-term storage of beets at the factory site.

beet washer.* An apparatus for wet cleaning of the beets.

beet wheel.* A bucket wheel that elevates the beets and, in some cases, water from the beet flume.

biennial. *bot.* A plant requiring two years for full life cycle.

biomass. *bot.* Total weight of plant material; root, leaves, petioles.

biotin. *chem.* A compound which is a member of the vitamin-B group.

Bird centrifugal. A continuous chemical centrifugal with a horizontal axis. Not used for sugar.

blowdown. *coll.* A discharge from a system, designed to prevent a buildup of some material, as in a boiler to control dissolved solids.

BOD. *biochem.* Biochemical oxygen demand is the amount of oxygen required to biologically oxidize the organic matter in a sample over a period of time, usually five days.

body. The sensory impression of density in a fluid.

boiling scheme.* The overall plan of crystallization-separation stages in the sugar end, chiefly designated by the number of stages or "boilings" used.

boiling time.* In batch sugar boiling, the elapsed time from the admission to the cutoff of the heating steam or vapor.

bolters.* Beets that have grown a flower or seed stalk.

bolting silk. Silk cloth of various mesh sizes used for screening pulverized or granular materials.

bone char. Bone charcoal, bone coal, bone black, charcoal prepared from selected bones of cattle. The material functions as an activated adsorber for color

and ash, particularly in the sugar industry. It was widely used in beet-sugar factories in the 19th century. It is in current use in cane-sugar refineries.

Boltzman function. *phys. chem.* Molecules making up a chemical system will have various, quantitized levels of energy. The Boltzman principle is a mathematical, probability description of the energy level of a molecule in that system.

bottlers sugar.* Sugar meeting the standards of the National Soft Drink Association. Can be either granulated or liquid sugar.

bottom blow. *coll.* Boiler blowdown from the bottom or mud drum of the boiler.

brei.* Fine beet particles—the product of a beet rasp, beet saw, or similar device.

Brix.* The percent dry substance by hydrometry, using an instrument (Brix hydrometer) or table calibrated in terms of percent sucrose by weight in water solution.

brown sugar.* A marketable product consisting of fine sugar crystals lightly coated with a yellow- or brown-colored syrup that contains a relatively high percentage of invert sugar, which keeps the product slightly moist.

buffer. A substance in a solution which acts to maintain an existing, usually hydrogen ion, concentration. *v.* To react in the same fashion.

bulk bin.* A facility for storing granulated sugar in bulk form.

cake. *coll.* The solid material separated from sugar syrups or juices by filters. See also lime cake and saccharate cake.

calandria.* A heating element used in certain types of evaporator bodies and vacuum pans, consisting of a drum- or lens-shaped body traversed by vertical tubes with the steam or heating vapor in the space surrounding the tubes.

calcination. *chem.* The roasting or burning of any substance to bring about physical or chemical changes; e.g., the conversion of lime rock to quicklime.

cambia. *bot.* The region in the plant where cell division occurs.

campaign.* The period in which the beets are processed. May also include factory test-out and lay-by periods, periods in which the beets are harvested but not processed, and periods in which stored juice, partially refined sugar, molasses, or other substances are processed or reprocessed.

capillary. *phys.* A tube having a very small bore.

carmelize. To form products from sugar by burning sugar or similar carbohydrates.

carbamate. *chem.* A salt of carbamic acid. H-N-COOH.

carbonate.* To introduce a gas rich in CO_2 into the juice.

carbonation.* The phase of the process in which a gas that is rich in CO_2 is added to the limed juice to precipitate excess lime as $CaCO_3$. Thus, *first carbonation, second carbonation.*

carbonation-filter feed.* The feed slurry for the (usually) vacuum filters that desweeten the precipitates from the carbonation. Often also embodies cakes from other filtering stages which require desweetening.

carbonator.* 1. Apparatus in which carbonation is accomplished. Preferable to "carbonation tank." 2. Person operating such apparatus.

cascade control loop. *instr.* A closed-loop control in which the set point of a

controller of one variable is adjusted or modified by means of another measured variable.

caster sugar. *coll.* A fine-grained granulated sugar, so-called because it is used in table casters with perforated tops.

catalyst. *phys. chem.* A substance which accelerates a reaction without itself being changed.

catch-all.* A device for separating entrained matter from the vapor leaving a vacuum pan or evaporator body.

cation. *chem.* A positively charged ion (q.v.) in solution.

caustic. *adj.* or *n.* Capable of destroying or eating away by chemical action. Applied to strong bases.

cavitation. *phys.* The formation of a cavity, caused by the vaporization of a liquid resulting from a partial vacuum created by a rapidly revolving propeller; also the cavity itself.

cell fill. *coll.* The weight of cossettes per unit volume in the individual cell or diffusing space of a diffuser.

center well.* Communicating tube of relatively large diameter traversing the axis of a calandria which permits a convective circulation of the liquid being heated; i.e., upward through the tubes and downward through the center well.

centrifugal* In the sugar end, an apparatus which separates the crystals from the syrup in a magma by filtration through a screen, enhanced by centrifugal force.

chain-grate stoker. A stoker system which moves the coal in a continuous bed from the bottom of a feed hopper into the furnace by means of a moving grate, consisting of a continuous belt constructed of many individual cast-iron chain links so assembled as to allow air to pass through.

chelate. *chem.* To form a soluble complex with a dissolved substance, effectively reducing its activity.

chemical symbols used in text.

Al = aluminum	H = hydrogen	P = phosphorus
Ba = barium	I = iodine	K =potassium
B = boron	Fe = iron	Na = sodium
Ca = calcium	Mg = magnesium	Sr = strontium
C = carbon	Hg = mercury	S = sulfur
Cl = chlorine	N = nitrogen	Sn = tin
Cu = copper	O = oxygen	Zn = zinc

chips. *coll.* Cossettes.

chloromethylation. *chem.* Introduction of the chloromethyl group, CH_2Cl^-, into a compound.

chloroplast. *bot.* Tiny chlorophyll-containing organelle in leaf cells where light energy is harvested, carbon dioxide fixation occurs and sugars are synthesized.

chromatography. *chem.* Separation of the constituents of a mixture of chemicals by passage through a column or layer of absorbent material. The individual materials are absorbed at different rates, and layers or bands of pure substances build up.

clarification. *coll.* The process of removing undissolved materials from a liquid. Specifically, removal of solids either by settling or filtration.

clarifier.* Apparatus for the elimination by sedimentation of suspended solids from a turbid liquid; thus *flume-water clarifier, main-waste clarifier, first-carbonation clarifier.* Note: When the main objective is to recover the solid phase, the term *thickener* is preferred; thus *hot saccharate thickener* (q.v.).

clarifier overflow.* The supernatant liquid leaving the clarifier.

clarifier underflow.* The concentrated suspension of separated solids leaving the clarifier.

clean-beets weight.* The purchase weight of the delivered beets; i.e., first-net or screened weight adjusted for percent tare or percent clean beets.

clinker. To fuse together by heat. *n.* The result of such fusion.

COD. *biochem.* Chemical oxygen demand. Its determination provides a measure of the oxygen equivalent of that portion of the organic matter in a sample which is susceptible to oxidation by a strong chemical oxidant.

cold filtrate.* The liquid effluent from the filters that separate the cold saccharate (*cold-saccharate filters).*

cold wash.* The liquid effluent from the washing section of a cold-saccharate filter, if separated from the balance of the filtrate.

colloid. *chem.* A substance in finely divided state, ranging from 0.2 to 0.005 microns in diameter.

concentrator.* An evaporator body for precisely adjusting the concentration of thick juice or standard liquor (q.v.) destined for long-term storage, or of CSF (q.v.); or for preparing graining charges (q.v.).

condensate.* Water obtained by condensation of steam or vapor in surface condensers, or in the steam or vapor chests of heating or evaporating vessels.

condenser.* Apparatus for the condensation of steam or vapor, generally using water as the cooling medium.

condenser water.* A mixture of condensate and cooling water produced by a direct-contact condenser.

confectioners' sugar. *Powdered sugar.*

conglomerate.* A cluster of mutually intergrown crystals.

cossettes.* Slender strips of beets cut by the slicers.

cross-linking. *chem.* Of a resinous polymer, bonding between widely separated portions of polymeric structures.

crown.* *n.* The part of the beet to which the petioles are attached; *v.* To remove the crown from a beet.

crystallization.* The total weight of sucrose in the solid product of the centrifugals, including the dissolved sucrose in any syrup adhering to the crystals, expressed as a percentage of the total weight of sucrose, both crystallized and dissolved, in the massecuite spun.

crystallizer.* Apparatus for continuing the crystallization of sugar in a massecuite after its discharge from a vacuum pan, by providing retention time, stirring, and cooling at a controlled rate.

CSF* Concentrated Steffen filtrate—a syrup-like liquid produced by deliming the Steffen filtrate and then concentrating it to about 60% dry substance. Valuable as an animal feed or feed additive.

cultivar. *bot.* A cultural variety.

curing.* *coll.* The crystallization of sugar in a massecuite by means of a crystallizer.

cut. *coll.* Used in the sense "to separate." To cut a pan; to split or divide the contents between two pans. To cut sugar; the act of removing or plowing centrifuged sugar from the centrifugal basket.

CV. Coefficient of variation of the screen opening through which sugar crystals will just pass. See *MA*.

cycle time.* In batch sugar boiling, the elapsed time from the admission of one graining charge to the admission of the succeeding graining charge.

cyclone. A mechanical classifying device normally used to remove solid particles from a gas stream by means of centrifugal force. Tangential entry of the particulate laden gas stream creates a vortex action within the cyclone, forcing the solid particles to the periphery of a cylindrical container, and allowing the clarified gas to be removed from the "eye" of the vortex.

cyst form. The sugarbeet nematode. They are observed in the field as females, which appear as small white cysts (lemon-shaped bodies) clinging to the fine beetroot hair. Each cyst contains 100 to 600 eggs from which the wormlike larvae hatch and feed on the sugarbeet roots.

daphnia. *zool.* A tiny crustacean, the "water flea." *daphnia puler.*

decrepitate. *phys.* To calcine (q.v.) so as to cause crackling or cracking.

defecate. *coll.* To purge; specifically, to purify juices by adding massive doses of calcium oxide or hydroxide.

defeco-carbonation.* Simultaneous liming and carbonation.

dehydrated beets.* Beets that are at least partly desiccated. The term "wilted beets" should be phased out since, technically speaking, wilting is characteristic of leaves rather than roots.

Δg. Delta energy or delta f. Free energy.

denaturation.* Alteration of the beet-cell protoplasm, usually through the effect of temperature, with the result that the cell-wall permeability and the coagulation of the cell-protein matter are increased. The term "plasmolysis" should not be used here.

dendritic. *min.* Nonperfect, fault-filled crystals, normally characterized by an open scattering of crystal branches, often as lace-like or fern-leaf-like crystalline branches. Most often found in thin films of mother liquor.

desweeten.* *v.* To reduce the sucrose content of a filter cake, resin bed, carbon bed, etc. to a practical minimum by displacing the sucrose-bearing liquid phase with water.

diatomaceous earth. A type of siliceous material naturally occurring, and processed for use as filter aid. Made of the siliceous skeletons of marine unicellular or colonial algae.

dielectric constant. *phys.* The force, F, between two electric charges, e, separated by distance, r, $F = e^2/Dr^2$. In a vacuum, D = 1. In any other medium, D = the dielectric constant.

diffuser.* Apparatus for obtaining sugar-bearing juice from the cossettes by a combination of leaching and cell-membrane dialysis.

diffusion.* The phase of the process in which sugar-bearing juice is obtained from the cossettes by a combination of leaching and cell-membrane dialysis.

diffusion loss.* Sucrose leaving the process in the pressed pulp, when the press water is being returned to the diffuser; otherwise, sucrose leaving the process in the wet pulp. Usually expressed as percent on beets or percent on sucrose entered.

digital readout. *instr.* An instrumental reading, in actual digits.

dipole moment. *phys.* The moment of a molecule due to physical distance separating individual positive and negative centers.

direct polarization.* Content of sucrose as estimated after simple clarification, if necessary, followed by measurement of the polarization.

divinyl benzene. *chem.* DVB, or vinyl styrene. $C_6H_4(CH=CH_2)_2$, usually in the para form.

draft.* 100 • weight of raw juice produced/weight of cossettes introduces into the diffuser.

dress. *coll.* Of a filter, to install new filter cloth.

drill. *agr.* To sow seeds.

drink. *v. coll.* The addition or act of adding liquid to the vacuum pan in the process of boiling massecuite.

drips. *coll.* Condensates from the steam chests or calandrias of heaters, vacuum pans, or evaporators.

drop. *coll.* To drop a strike, to discharge the contents of a vacuum pan.

dropping the strike.* Discharging the batch of finished massecuite from the vacuum pan.

dry bottom furnace. The firebox of a pulverized coal-fired boiler in which gas velocities and flow patterns are such that all the ash from the coal is swept out of the boiler through the passes and into the flue-gas system.

dry substance. The water-soluble, moisture-free matter present.

dust box. A device to remove sugar dust from air, usually employing water sprays; a dust collector.

dynamic simulation. *instr.* A simulation in which one or more variables are manipulated simultaneously to study their effect on the system and the response of the system to a real time environment.

einwurf. A "crystal within a crystal" or crystalline overgrowth of an earlier crystal produced when a low purity, fine grain is used as a seed for a subsequent higher purity boiling. Einwurf boiling is often identifiable by the impurity occlusions within the small crystal (grown in a low purity strike or boiling) and which are very visibly evident within the subsequent growth of those same crystals used as the seed in a following higher purity boiling; i.e., a dark, internal structure surrounded by a more clear structure. The use of a preformed seed for a subsequent boiling is the basis of many boiling systems. In this instance it is a lower purity seed for a final, higher purity boiling.

electrodialysis.* *phys chem.* Ultrafiltration (q.v.) accelerated by the influence of an electric potential.

electrostatic precipitator. A gas cleaning device using the principle of placing an electrical charge on a solid particle which is then attracted to an oppositely charged collector plate. The device uses a d-c potential approaching 40,000 volts to ionize and collect the particulate matter. The collector plates are intermittently rapped to discharge the collected dust into a hopper below.

elimination.* The fraction of the nonsucrose that is separated from the main-

product stream in a given phase of the process; thus "diffuser elimination," "carbonation elimination." *Percent elimination* = elimination × 100.

enthalpy.* *phys. chem.* The sum of the internal and external energies of a fluid system.

entropy.* *phys. chem.* A measure of the unavailable energy in a thermodynamic system.

epoxy. *chem.* A prefix indicating the presence of an -O- bridge in a molecule. Also indicates a type of adhesive or protective coating.

epuration.* *coll.* An older term for purification.

equivalent. *chem.* Having the same combining or reactive value.

evaporator body.* A single evaporator vessel.

evaporator effect.* An evaporator body or group of bodies working as one of the series-connected units in a multiple-effect evaporator.

evaporator station.* The aggregate of the various evaporator bodies and their auxiliary equipment.

exothermic. Exothermal, giving off heat.

extraction.* The net sucrose produced/sucrose entered. *Percent extraction* = 100 × extraction.

facultative pond. *coll.* A combination aerobic-anaerobic pond divided by loading and thermal stratification into aerobic surface, and anaerobic bottom strata.

false grain.* Undesirably small crystals found among the crystal population of a massecuite—the result of unwanted spontaneous nucleation during the sugar-boiling operation.

fanged beets.* Beets with multiple tap roots. A more descriptive term than "sprangled beets."

fatty acids. *chem.* Monobasic acids containing only the elements carbon, hydrogen, and oxygen, and consisting of an alkyl radical attached to the carbonyl group.

feedback instrument. *instr.* An automatic controller which takes a measurement signal from an output of a process, compares it with the set point, and feeds it back into a process input through a final element such as a control valve. Thus, an automatic controller is commonly referred to as a feedback or closed-loop instrument.

Fehling's solution. *chem.* A reagent used in a test for sugars; comprising a mixture of cupric sulfate and alkaline tartrate solutions.

fillmass. (from German füllmasse) See massecuite.

filter aid. A material which is either precoated on a filter membrane, or slurred in the liquid to be filtered, which, because of its porous nature, permits more solids to be filtered out before the filter cake becomes impermeable, or improves the clarity of the filtered liquid. Examples: diatomaceous earth, perlite, asbestos.

filter cake. Solid material deposited on the filter septum during filtration.

filter medium. A layer of cloth, wire, paper, etc., providing actual initial filter surface, used interchangeably with "filter septum."

filtrate. *chem.* Liquid after passing through a filter.

filtration. Removal of solid particles from liquid or particles from an air or gas stream by passing the liquid or gas stream through a permeable membrane.

first-carbonation clarifier.* Apparatus for the separation by sedimentation of the suspended matter from the first-carbonation slurry. Preferable to the term "thickener" in this instance because the primary objective here is to clarify the purified juice.

first-carbonation slurry.* The product of the first carbonation, consisting of precipitated nonsucrose and $CaCO_3$ suspended in purified juice.

first net weight.* The weight of beets in a delivery after removal of the screened dirt or screenings.

fixed-bed. *chem.* Referring specifically to adsorbents or ion-exchangers, using a nonmoving bed in a column of the active material.

flame photometer. *chem.* A photometer which measures the presence of an ion of substance by the color of a flame in which the substance is injected.

flash. *coll.* Of a liquid, to lower the pressure so that the liquid vaporizes to dispose of its excess heat.

flight. A projecting baffle in a piece of equipment.

Flora. *bot.* Referring to plants or plant life.

fluidic. *instr.* A general array of devices or systems for controlling liquid or gas flow to perform sensing, logic amplification, computation, or other control functions.

flume.* *v.* To transport beets by means of a beet flume.

flume effluent.* The water withdrawn or discarded from the beet-flume system to maintain water levels or to control the solids content of the flume water.

flume makeup.* The water supplied to the beet-flume system to replace the flume effluent.

flume water.* The water used to transport beets in a beet flume.

flux. *v.* To fuse; *n.* A material which assists fusion.

footing. *coll.* In pan boiling, a quantity of high-purity massecuite or syrup which is used to furnish crystal surface for crystallization from lower purity liquors.

foot valve. *coll.* The valve which discharges the massecuite from a vacuum pan.

fraiser. A file used in sharpening the edge of the splitters in a beet knife. There are two types, right- and left-hand.

frozen beets.* Beets in which at least a portion of the cellular juice has been frozen.

Gamma rays. *phys.* One of the several kinds of electromagnetic emissions given off by radioactive decay of substances such as cesium 137. These rays are absorbed in varying amounts by materials such as lead, steel, liquids, and even organic substances such as sugar massecuites. Measurement of the degree of absorption in a fixed-dimension path through an intervening massecuite is a measure of the concentration of the mother liquor and crystals. Gamma ray instrumentation also is applied to level measurement in sealed vessels, limestone level in kilns, milk of lime slurry density, etc.

gas washer. Apparatus used to remove entrained solids and tarry substances from carbon dioxide gas from a lime kiln.

Gibbs' function. A mathematical definition of the energy requirements in the formulation of a solid particle (nucleus) from the liquid phase. Free energy of formation, as related to crystal size, further defines the crystal nucleus size beyond which

a stable crystal lattice will continue to grow.

grain.* *n.* The agregate of the crystals in a massecuite; *v.* to introduce or initiate a crop of crystal nuclei in a graining charge.

graining charge.* In batch sugar boiling, the batch or charge of syrup drawn into the vacuum pan at the beginning of the batch cycle.

granulated sugar.* Crystalline sugar of market quality. May be characterized by such modifiers as *fine, extra fine, manufacturers, bakers,* relating to the average crystal size and size distribution.

green sugar. *coll.* Sugar which has not been properly purged in the centrifugals.

green syrup.* The filtrate leaving a centrifugal before initiation of the wash, consisting of mother liquor and whatever fine crystals pass the screen.

grist. Improperly used to describe the overall distribution of particle sizes in a granular material.

grizzly. *coll.* A grating with elongated parallel slots or openings, used in separating water and trash from beets.

growing the grain.* The phase of batch sugar boiling following graining, in which the crystals are grown to the desired size and the volume of the massecuite increased by simultaneously adding syrup and evaporating water.

guide bars.* Removable steel bars attached to a knife block just ahead of the cutting edges of the beet knives.

hammering. *coll.* Pounding on equipment to dislodge adhering materials or material buildup obstructing free flow.

hardness. *chem.* The content of dissolved substances which prevent the lathering of soap; i.e., calcium and magnesium salts.

hardpan. *agr.* A cement-like layer under the surface of soil.

high green syrup, high wash water, high machine syrup.* The respective liquid products of the centrifugals in the white boiling *(white centrifugals).*

high melter.* A melter for redissolving sugar produced by the intermediate boiling *(intermediate sugar).* Also used for redissolving sugar products which require re-refining.

homogeneous. *phys. chem.* Describes a reaction taking place in a single phase, such as liquid or gaseous. *coll.* Completely mixed so that none of the components are physically identifiable.

hot filtrate.* Liquid effluent from the filters, if any, that separate the hot saccharate per se.

Howard feeder. A liquid feeder, involving a wheel with constant-capacity buckets or cups on its periphery, which dip into the liquid to be flow-controlled, and which discharge into an outlet. Flow variation is provided by change of the speed of rotation, and exact metering is obtained.

humectancy. *chem.* Degree of sensory moistness.

humus. *agr.* Partially decomposed vegetable matter.

hydrate. *chem.* A compound formed by union with water.

hydrocyclone. A device in which solids are separated from a liquid by centrifugal force. The liquid rotates within the conical equipment, which has no moving parts.

hydrolysis. *chem.* A chemical change involving addition of the elements of water.

hydrometer. *phys.* An instrument by which the specific gravity or density of a liquid may be determined through the depth of immersion of its stem, when it floats in the liquid.

hydrophilic. *chem.* Water loving.

hygroscopicity. *chem. The ability to readily absorb and retain moisture.*

hypertonic. *phys. chem.* Having greater osmotic pressure than isotonic (q.v.).

ideal solution. *phys.chem.* A solution which thermodynamically shows no change in internal energy on mixing, has no attractive forces between components, and follows Raoult's law (an expression of changes in partial pressure of the solution due to dissolved solids) over all ranges of temperature and concentration. These ideal conditions are more often found in very dilute solutions.

inorganic. *chem.* A compound or substance of mineral origin; not organic (q.v.).

integrated circuit. *instr.* A complete electronic circuit which has been fabricated as an inseparable assembly of circuit elements in a single small structure.

interlock. *instr.* Mechanical or electrical devices for the purpose of preventing independent operation of inter-connected equipment, and to assure proper sequencing of equipment and machinery controls.

intermediate boiling.* The middle-purity stage of the crystallization-separation process. It uses as feed the separated mother liquor from the white boiling, sometimes mixed with thick juice. Its non-marketable crystalline product is redissolved for recrystallization in the white boiling. In a four-boiling scheme there would be two intermediate boilings, designated as *first intermediate* and *second intermediate.*

intermediate green syrup, etc.* The respective liquid products of the centrifugals in the intermediate boiling *(intermediate centrifugals).*

invertase. *chem.* An enzyme which catalyzes the splitting of sucrose into glucose and fructose.

invert sugar.* A mixture of glucose and fructose, formed in equal quantities by the hydrolysis of sucrose.

invert-sugar content.* Content of an equal-part mixture of glucose and fructose. If it is established that invert sugar is the only reducing substance significantly present, then the results of an analysis for reducing substance may be reported as "% invert sugar"; however, the term "reducing substance as % invert sugar" should be used in the case of unknown mixtures.

ion. *chem.* An electrically charged atom or group of atoms, in solution. Thus common salt, NaCl, dissociates in solution into the cation Na^+ and the anion Cl^-.

isomer. *chem.* A substance containing the same weight percentage of elements, but differing in properties, due to different arrangement of the elements in the molecule.

isotonic. Having equal osmotic pressure.

juice. *coll.* Almost any sugar-containing liquid in the main line of the beet-sugar manufacturing process, up to the melter station.

Kelvin equation. One of the four basic equations of surface chemistry. It relates the effect of mechanical pressure (surface tension of the liquid) to the vapor pressure of the liquid and to the radius of the liquid drop being formed. This rela-

tion is crucial to the condensation nucleation of droplets big enough for continued drop growth. With a similar analysis, it provides the background for the Gibbs equation which defines the energetics of critical-sized nuclei for crystal formation from solution.

ketose. *chem.* Relating to ketones; i.e., monosaccharates are called *ketose* when they are ketone-alcohols.

kilovar. *phys.* A unit of reactive or wattless electrical power flow for which the current is displaced 90 electrical degrees lagging from the driving voltage.

kinetics. The study of chemical phenomena dealing with the rate of change from the initial to final states, under non-equilibrium conditions, for example, crystallization rates.

Kirk key lock. A mechanical locking arrangement used on multiple electrical operating devices and with a common keying system to make mandatory the proper operating sequence of the devices.

knife blocks.* Steel frames for supporting the beet knives in the slicers.

K_{sp}. *chem.* Solubility produce (q.v.).

labile. *phys.* Readily undergoing or susceptible to change; i.e., crystallization. Unstable.

labile zone.* The region of supersaturation in which nucleation will readily occur.

laminar. In thin, parallel, plate-like layers.

leg line. The vertical pipe comprising the barometric column in an apparatus under vacuum.

levitation. Of a bed of adsorbing material, fluidizing or carrying the particles up from the bed by high flow rates.

lime cake.* Filter cake from the filters that desweeten the carbonation-filter feed.

lime mud. *coll.* The washed calcium carbonate filter cakes, including the sludges from first and second carbonation, and perhaps Steffen filtrate carbonation.

lime pond.* The basin for the storage and dewatering of waste-lime slurry.

lime-salts content.* The content of dissolved calcium salts, expressed as percent CaO on dry substance.

lime sewer. Lime sludge mixed with sufficient water for pumping. Modern term, lime flume.

lime sucrose.* A chemical compound of Ca or CaO with sucrose; thus *monolime-sucrose, dilime-sucrose, trilime-sucrose.*

lime waste. See waste-lime slurry.

linear variable differential transformer. *instr.* An electro-mechanical transducer which produces a voltage output directly proportional to the displacement of a movable armature or magnetic core.

liquid invert sugar.* Liquid sugar containing a significant proportion of invert sugar; usually characterized by the invert-sugar content expressed as a percentage of the dry-substance content, e.g., "40% invert syrup."

liquid sucrose.* See sucrose sugar.

liquid sugar.* Marketable syrup, usually prepared by dissolving granulated sugar in water. May consist of a mixture of dissolved sucrose and invert sugar—the latter usually being produced by hydrolysis of some of the dissolved sucrose.

lister. *agr.* A lister drill. A machine in which a plow is combined with a drill which sows seed as the furrow is opened.

logic. *instr.* The process of decision making, equipment which makes logical decisions, or the intelligence (programming) of decision-making equipment.

low melter.* The melter for redissolving sugar produced by the raw boiling *(raw sugar).*

low wash syrup.* The wash syrup from the centrifugals in the raw-boiling stage *(raw centrifugals).*

machine syrup.* The total liquid product of a centrifugal when the green and wash syrups are allowed to commingle.

MA. Mean aperture. The average dimension of a screen opening, through which granulated sugar will just pass. Used to characterize grain size.

macroscopic. Observable to the naked eye, as opposed to microscopic.

magma.* A mixture of crystals and syrup.

main liming.* The treatment of the juice with relatively large amounts of lime, e.g., 1% CaO or more, to precipitate certain nonsucroses and to promote the decomposition reactions between lime and certain nonsucroses.

main waste.* The principal liquid-waste effluent from the factory.

marc.* The water-insoluble constituent of the sugarbeet root.

massecuite. Magma produced by crystallization—a suspension of crystals in their mother liquor.

mass transfer. *phys. chem.* The law of physical chemistry which states that the product of the concentrations (or activities) of the substances produced at equilibrium in a chemical reaction, divided by the product of the concentrations of the reacting substances, each concentration raised to that power which is the coefficient of the substance in the chemical equation, is a constant, at a given temperature.

maturation. The process of coming to full development.

McCabe's ΔL law. The generalization of the ΔL law is that the total growth of all crystals in a magma, over the same time interval and as measured by the length, L, of similar axes, is the same for all of the crystals in the magma. This law assumes: (1) the crystals have similar geometric shapes and remain invariant during growth and (2) the supersaturation is the same for all the crystals in all locations in the magma.

melanoidin. *chem.* Coloring material formed by complex reactions of reducing sugars with amino compounds, especially with amino acids and peptides.

melassigenic. *coll.* Tends to increase amount of molasses produced.

melt. *coll.* In the sugar industry, to dissolve.

melter.* The apparatus for dissolving crystalline sugar in juice, syrup, or water.

mesophyll cells. *bot.* The green parenchyma (q.v.) between the epidermal layers of a foliage leaf.

metabolite. *biol.* Any product of plant metabolism.

metastable zone.* The region of supersaturation in which crystals already present will grow, but new-crystal formation (nucleation) will not readily occur.

micelle. A molecular structure built up from complex molecules in colloids (q.v.).

mill. To mechanically crush, or pulverize.

milled seed.* Finely ground sugar crystals used for seeding, usually produced by wet grinding in isopropanol.

Miller indices. A precise method for identifying each of the faces of a crystal. Simple multiples of unit cell distances measured along the three basic crystallographic axes provide three axial intercepts, which, in turn, define the orientation of the plane corresponding to a crystal face.

mingler.* Apparatus for receiving the massecuite discharged from a crystallizer, adjusting or controlling its temperature, and distributing it among a set of centrifugals—usually a trough or tube equipped with a rotating coil of pipe through which water at a controlled temperature is circulated. *Stevens coil.*

mitochondrial. *bot.* Associated with the mitochondria. The cellular organelle where respiration occurs.

mixer.* A surge vessel for receiving massecuite batchwise from a vacuum pan and discharging it more or less continuously to a centrifugal station or continuous crystallizer.

modulating-type valve. Any control valve with a continuously variable orifice area in its body, the orifice area being a proportional function of the valve-operator input signal.

molality. *chem.* An expression for solution concentration in which the amount of solute is stated in terms of moles and the amount of solvent per kilogram of solvent. The unit of molality is mole of solute. (Do not confuse with molarity which is mole %).

molasses.* The green or machine syrup from the raw centrifugals—the sugar-bearing product of the sugar end whose purity has been reduced to the point that further crystallization of sugar is uneconomical.

molasses, discard. *coll.* Molasses which will not be sent to a process to extract more sucrose, but is available for sale, or for addition to cattle feeds.

mole. *chem.* The molecular weight of a substance, expressed in grams.

monitor.* In handling beets, a swiveling hydraulic nozzle used to flush the stored beets into the flumes of a beet slab.

monoclinic. *phys.* A crystal having one oblique intersection of its axes.

monte jus. *coll.* A juice-pumping device without moving parts except for valves, which operates by steam or air pressure.

mother liquor. The solution from which crystals are formed.

mucoid. Mucous-like.

mud. *coll.* First or second carbonation precipitates.

mud pond.* A basin for the storage and dewatering (usually by evaporation and percolation) of the slurry of solids separated by a flume-effluent or main-waste clarifier.

multiple-effect evaporator.* A series of evaporator effects operating at successively reduced vapor pressure, and so connected that the first effect is heated by steam, the second by vapor generated by the first effect, and so on.

mycelia. *micr.* A mass of interwoven, thread-like filaments.

neutrality. *chem.* The condition of a water solution when it is neither acidic nor basic; i.e., at pH 7.

nonsaccharide. See nonsugar.

nonsucrose.* Any water-soluble matter present which is not sucrose.

nonsugar, nonsaccharide.* *chem.* Any water-soluble matter present which is not a saccharide. The more precise term *nonsaccharide* is preferable.

nucleation. *phys. chem.* To form small nuclei, such as seed crystals.

nucleotide. *chem.* Sugar-phosphate esters of the nucleoside bases, adenosine, uridine, etc.

old settlers. *coll.* In pan boiling, crystals remaining from a former strike, which grow to very large size.

organic. *chem.* A compound containing carbon.

osmose process. An obsolete process used in separating sugar from molasses by means of dialysis through parchment membranes.

overfire air. A system used in stoker-fired coal boilers consisting of a series of air jets blowing a high velocity stream of air into the furnace above the grate. The purpose is to achieve complete combustion by creating turbulence, thereby obtaining a more intimate mixture of combustion air with the unburned volatile combustibles evaporated from the coal by the heat.

oxalogenic. *chem.* Substances forming oxalate on decomposition or reaction.

oxidize. *chem.* To be reacted with oxygen, or the equivalent.

parenchyma. *bot.* Plant tissues composed of living cells, which make up the bulk of the tissues.

pCa^{++}. *chem.* The negative logarithm of the reciprocal of the calcium ion concentration.

pelleted seed.* Beet seed brought to uniform size by coating.

peptize. *phys. chem.* To bring into colloidal solution.

percent clean beets. 100 minus percent tare.

percent crystallization.* The weight of crystalline sucrose formed in a massecuite expressed as a percentage of the total weight of sucrose, both crystallized and dissolved, in the massecuite.

percent tare.* The unwanted matter in the first-net or screened beets; i.e., leaves, crown portions, earth, and other foreign matter, expressed as a percentage of the first-net or screened weight.

perfectly washed cake. *coll.* A theoretical material which cannot be practically obtained. A Steffen saccharate cake, which, when further washed, will not increase in purity.

perfect-wash purity.* The purity of the syrup that remains after deliming a sample of saccharate cake that has been given an extra washing in the laboratory. Purpose of making this determination is to evaluate the effectiveness of the washing on the factory filters.

petiole.* A beet-leaf stalk.

picking table.* A roller table or broad belt conveyor designed to spread out the flow of beets for the manual removal of foreign objects.

plasmalemma. The membrane surrounding the cytoplasm of cells.

polarization.* The amount of rotation given by a substance to the plane of plane-polarized light under standard conditions (ICUMSA), expressed as equivalent percent sucrose.

powdered sugar.* Finely divided sugar produced by pulverizing granulated sugar, usually containing an anti-caking additive.

power incorporation. *agr.* The use of an implement driven by the power take-off on a farm tractor to incorporate into the soil an approved chemical weedicide or insecticide, or a combination of both chemicals, to protect the sugarbeet crop.

precoat. A layer of solid particles, calcium carbonate, filter aid, etc., deposited on a filter septum to establish initial clarity of the filtrate.

predefeco. *coll.* A prefix meaning "in advance of mainliming."

preexponential. The Arrhenius expression for crystallization rates consists of the product of two terms:

$$k = A \cdot e^{-E/RT}$$

The term A is a proportionality factor, sometimes identified as a frequency factor, and by others it is called the preexponential factor, presumably because it precedes the second or exponential term. A can be temperature and supersaturation dependent.

preliming.* Treatment of the raw juice with relatively small amounts of lime, e.g., 0.2.—0.3% CaO, over a period of about 20 minutes, to precipitate proteinaceous matter in a relatively stable form, such that its repeptization is minimal in subsequent main liming and carbonation, or defeco-carbonation.

pressed juice.* The cellular juice obtained from beets by pressing.

pressed pulp.* The pulp leaving the pulp presses.

press water.* The liquid effluent from the pulp presses.

prime mover. An engine or machine which receives and modifies force and motion as supplied by some material source, thereby driving machinery.

probability paper. *math.* A graph paper, so printed that if the standard distribution curve (Maxwell-Boltzmann) is plotted, a straight line is obtained.

processed seed.* Beet seed of uniform size, prepared by mechanical treatment and screening. Usually also treated with a fungicide.

protoplasm. *bot.* The essential substance of both the body and nucleus of vegetable cells. Viscous, translucent material holding fine granules in suspension.

puddle. To make fluid.

pulp.* Cossettes that have been exhausted by diffusion.

pulpefanger. (German, pulp collector.) A dewatering screen, usually with mechanical wipers or carriers, over which beet pulp passes, usually from a diffuser to pulp press or wet silo.

purge.* To remove the (bulk of the) syrup from the crystals of a magma.

purity.* 100 × sucrose/dry substance.

racemic mixture. *chem.* An optically inactive mixture of dissolved substances made of dextro- and laevo-rotatory forms.

radical initiation. *phys. chem.* One way in which chain polymerization may be started. It requires the generation of a reactive species known as a free radical. These are uncharged molecular fragments having an unpaired electron. These radicals, generated from a variety of compounds, and by a number of different reactions, start ("initiate") the polymerization chain depicted by reacting with a neutral monomer molecule thereby forming a new radical which, in turn, repeats the process.

radioactive isotope. *phys.* A form of an element which emits particles or rays from the disintegration of the nuclei of its atoms.

Raman spectrocopy. *chem.* When light of a given frequency, v_0, is scattered by the molecules of a solid, liquid or gaseous substance, certain lines of the frequency, v, of the scattered light are changed, as $v = v_0 \pm v_1$. The method is useful in determining molecular structure.

ratio relay. *instr.* An indicating or nonindicating instrument designed to multiply a signal input by a factor, and to bias the resulting output.

raw boiling.* The final, lowest-purity stage of crystallization-separation. It uses as feed the mother liquor separated from the last intermediate boiling and yields a non-marketable crystalline product that is redissolved for recrystallization in the white or intermediate boiling.

raw juice.* The sugar-bearing juice obtained from the beets in the diffuser. The term "diffusion juice" is not sufficiently more descriptive to offset its greater length.

raw sugar.* 1. A partially refined sugar; 2. In a beet-sugar factory, the crystalline product of the raw boiling (*q.v.*).

rds.* The percent dry substance by refractometry, using an instrument or table calibrated in terms of percent sucrose by weight in water solution.

receiving station.* The station at which the growers' beets are weighed, unloaded, usually dry cleaned, and sampled.

reduce. *chem.* To deoxidize, or the equivalent.

reducing substance.* In the sugar industry usually refers to matter, chiefly reducing saccharides, which is able to reduce alkaline-copper reagents such as Fehling's solution.

reed switch. *instr.* A switch containing small reed-like arms which form the contacts. The contacts are sealed in glass in an inert atmostphere. The reed contacts are actuated either by magnets or by energizing a coil surrounding the glass which sets up a magnetic field.

refractometer. *phys.* An instrument for measuring refractive indices. Because the refractive index of a solution is a measure of the total solids in solution, the instrument is useful in the sugar laboratory for giving an approximate measure of total dry substance.

relaxation methods. *phys. chem.* The equilibrium of a chemical reaction is rapidly upset by changing some important condition, such as temperature or pressure. The change in the concentrations of reactants or products is monitored while the reaction reequilibrates (relaxes).

remelt.* *v.* To redissolve crystallized sugar for the purpose of recrystallizing it; *adj.* characterizes collectively the intermediate and raw boilings, their equipment, and their products; e.g., *remelt boilings, remelt centrifugals, remelt sugar.*

remelt syrup.* The syrup made by redissolving crystallized sugar—the product of a melter.

ribbon pan. A vacuum pan with a spiral ribbon-form or plate-type heating element.

rock catcher.* Apparatus that removes stones and other dense matter from the beet flume, usually by differential flotation.

rocker bars.* Guide bars whose angle with respect to the knife block is adjustable.

rock wheel.* A bucket wheel that elevates the stones and other matter separated by the rock catcher.

roller table. *coll.* A conveyor made of rollers used for dewatering, and cleaning beets of leaves and trash. It usually follows the beet washer.

rotation. *phys.* Refers to the optical activity possessed by certain substances, of rotating the plane of polarized light, as in a polariscope. Used analytically to determine the concentration of sugars in solution.

rotifer. *zool.* A minute, many-celled aquatic animal having the anterior end modified into a retractile disc bearing circles of strong cilia, which, in rotation, look like rapidly revolving wheels.

saccharate.* A precipitate formed in the Steffen process, consisting of lime-sucrose, lime, and some nonsucrose from the molasses; characterized as *cold saccharate* or *hot saccharate* depending upon the stage of the Steffen process in which it originates.

saccharate cake.* Filter cake from the washing filters in the Steffen process, consisting of saccharate, some adhering filtrate, and moisture.

saccharate milk. A slurry of saccharate cake in sweetwater (q.v.), used for preliming, main liming, or defeco-carbonation in Steffen factories.

saccharate purity.* The purity of the syrup that remains after the saccharate cake is delimed.

saccharate thickener.* Apparatus for separating the hot saccharate from the bulk of its mother liquor by sedimentation. Preferable to "clarifier" in this instance because the solid matter is the most-desired product.

saccharimeter. *phys.* A polariscope, with a scale reading percentage of sucrose. Sometimes applied to any instrument for measuring the amount of sucrose in solution, as a hydrometer, or refractometer.

salt-out. Decreased solubility of a liquid system due to the addition of another solute (or solvent), which, in turn, causes precipitation, coagulation, or crystallization of solute from the solution.

sand catcher. Apparatus which separates sand and earth from a liquid stream, such as raw juice, press water, or flume water.

saponify. To hydrolyze, or react with water. Specifically, to react a fatty acid with an alkali to form a soap.

saturated. *chem.* In organic chemistry, compounds containing no carbon atoms with double or triple bonds.

saturation. *coll.* The treatment of sugar liquors with gases, either carbon dioxide or sulfur dioxide. First and second saturation describes first and second carbonation. Third saturation describes sulfitation. Inaccurate, because the juices are not saturated with the gases.

scalder.* The apparatus in which scalding is achieved.

scalding.* Preheating of the cossettes before diffusion by mixing them with hot raw juice.

screened dirt, screenings.* The earth, tops, weeds and other matter separated from the beets by the beet screen.

screened weight. See first net weight.

scroll. A screw-type material conveyor. Normally a long screw operating in a closely-fitting enclosure.

seal tank. *coll.* The seal on the bottom of a barometric leg pipe.

sedimentation. *phys. chem.* The falling of solid particles in a liquid, as a sediment.

seeding.* Supplying crystal nuclei to the graining charge, after the latter has been concentrated to the metastable zone (q.v.) of supersaturation, in numbers equal to the desired crystal population of the finished massecuite.

seed slurry.* A liquid suspension of finely divided sugar used for seeding—usually milled seed in its isopropanol suspension.

selector, high or low. *instr.* A device used to set either or both maximum and minimum limits of instrument signal, and between the limits the signal is transmitted without change.

sequence memory bank. *instr.* A shift register made up of electric relays which follows a process through its various steps. In a tare laboratory, it may consist of a panel of indicator lights, which visually shows the presence of a sample in any of the steps.

servo-control. *instr.* An automatically controlled system in which the control variable is a mechanical position.

settling basin.* A basin or pond for the elimination by sedimentation, and also the storage of suspended solids from flume effluent or main waste.

shroud. *coll.* The hydraulic surface of the fluid pumping system that encloses the tips of the blades or vanes of the axial-flow impeller; i.e., a pan circulator impeller and its surrounding center-well surfaces. The space or clearance between the impeller and the shroud is normally quite small. As this clearance is enlarged, the leakage around the impeller tips increases and thereby reduces the head, capacity, hydraulic efficiency and horsepower requirement.

side bars.* Removable wear bars attached to the curved sides of certain types of knife blocks.

sigmoid. *math.* Curved in two directions, like the letter S.

silica. *chem.* Silicon dioxide.

silo. A pit, vat, or circular structure of wood, concrete, etc., for packing away fodder to convert it into silage by fermentation. Frequently applied colloquially to storage bins for granulated sugar.

skimmer blow. *coll.* Boiler water blowdown from the upper boiler drums at approximately the water level.

slicer.* Apparatus which cuts the beets into slender strips.

sludge. *coll.* The settled mud from the clarifier, before filtration and washing. Also, in the Steffen process, the vacuum-filter tray bottoms returned to the process as wet lime for preliming the diluted molasses. Generally, almost any flocculated, settled mass.

smear.* *v.* To produce false grain in a massecuite to the extent that it becomes difficult to purge; *n.* The mixed-grain condition resulting from smearing.

soft sugar.* A term preferred by the cane-sugar industry to *brown sugar.*

solubility product. *phys. chem.* The product of the concentrations (or activities) of the ionized parts of a substance in solution. A constant, at constant temperature.

solute. *chem.* A dissolved substance.

solvent. *chem.* A liquid capable of dissolving substances.

spin.* *v.* To treat a magma in a centrifugal.

spindle. *coll.* A hydrometer. Also, the connecting shaft between the centrifugal drive, or pulley and basket.

sprangled. *agr. coll.* Having many roots.

spreader stoker. A stoker system which throws the coal into the firebox by means of a rotary impeller or spreader.

squirrel-cage motor. An a-c machine of the induction type, consisting essentially of the stator and rotor. The rotor assembly resembles a squirrel cage treadmill, and has no electrical connections to a power source.

standard liquor.* The principal feed for the white boiling (q.v.). In a conventional three-boiling scheme it consists of thick juice plus remelt syrup. In a conventional four-boiling scheme it consists of remelt syrup containing only water as the solvent.

static control. *instr.* A system controlling machines or processes electrically, with no moving parts.

steady-state simulation. *instr.* A simulation performed with constant inputs representing the system under static or steady-state conditions.

Steffen factory.* A factory that incorporates a version of the Steffen process. Conversely, *non-Steffen factory.*

Steffen filtrate.* The liquid effluent from the Steffen process, consisting of the decantate (*overflow*) from the saccharate thickener plus the hot filtrate, if any. If the hot-saccharate stage is not used, the cold filtrate becomes the Steffen filtrate.

Steffen house.* The section of the factory that encompasses the Steffen process.

Steffen loss.* The sucrose leaving the process in the Steffen filtrate; usually expressed as a percentage of the sucrose in the molasses worked, of the weight of beets sliced, or of the total sucrose entering the factory.

Steffen process.* A process for the extraction of sucrose from molasses by combination with CaO and Ca $(OH)_2$. This term should be retained as a memorial to the inventors, Steffen *pere et fils,* in preference to the terms "saccharate process" and "calcium saccharate process." "Steffens" and "Steffens process" are incorrect.

stereoisomerism. *phys.* The condition of having isomers, depending on the arrangement of the elements in space.

stoichiometric. *chem.* Pertaining to the combining weights of the elements, and to the relations between the properties of substances and their compositions. Frequently applied to chemical computations in general.

strike.* Batch of finished massecuite from a batch sugar-boiling process.

string proof. *coll.* In pan boiling, a condition of the mother liquor, determined by placing a quantity between the thumb and first finger, and pulling the fingers apart rapidly. A thread will form if the desired consistency has been reached.

stripping. *chem.* The freeing of an adsorbing material of adsorbed materials by eluting or washing.

stroke. *coll.* Of a control valve, applying minimum and maximum input signals so that the operator stem experiences full travel. Prior to physical start-up this is normally done to assure that the stem travel assembly is not damaged.

styrene. *chem.* An unsaturated hydrocarbon, $C_6H_5CH{:}CH_2$.

sucrose sugar. Liquid sugar containing essentially ***liquid sucrose.**** only dissolved sucrose.

sugar.* 1. A product of the sugar industry which essentially consists of, or is derived from, sucrose. 2. (esp. when accompanied by an article) any of the saccharides. When referring specifically to sucrose, it is better to use the word *sucrose.*.

sugar boiling* The transformation of syrup into a mixture of crystals and mother liquor (see *massecuite),* by simultaneous evaporation and crystallization.

sugar drier.* Apparatus for drying the damp-sugar product of the white centrifugals.

sugar end.* The section of the factory that includes the processes for separating the final sugar product from the purified juice by crystallization and recrystallization. Many include the evaporator station(s), especially in factories which make extensive use of vapors for heating the vacuum pans.

sugar factory.* A processing facility which extracts and refines sugar from sugarbeets or sugar cane.

sugar mill.* A processing facility which extracts sugar from sugarbeets or sugarcane to produce raw sugar or partially refined syrup.

sugar refinery.* A processing facility which produces refined sugar from raw sugar or syrup.

sulfitation* The addition of SO_2 to the juice.

sulfonation. *chem.* Introduction of the sulfonic acid group, HSO_3^-, into a compound. A common sulfonating agent is concentrated sulfuric acid.

supernatant. *chem.* Floating on the surface.

supersaturation.* The quotient of the sucrose/water ratio in the given solution divided by the sucrose/water ratio of a solution that is saturated with sucrose under given conditions.

surface tension. *phys. chem.* A force in a liquid, acting in a direction parallel to the boundary surface, which tends to reduce that surface to a minimum. A liquid with low surface tension will form stable foams.

swaged. Shaped by hammering to fit a form.

sweeten. *coll.* Introducing a sugar-containing liquid to a substance, replacing the liquid phase previously present, if any.

sweetwater.* The liquid effluent from the filters that desweeten the carbonation-filter feed, consisting of the displaced juice and some of the wash water.

symmetrical component of fault. *instr.* That portion of total fault current flowing at any one time that is symmetrical about the zero axis; i.e., without d-c component or offset.

syrup.* The general term for sugar solutions of relatively high concentration.

tail. The lower, slender part of the beet root.

tailings separator. An apparatus that recovers beet tails and pieces from the separated beet tops, weeds, and other unwanted matter; usually utilizes the differential flotation or bouncing property of the beet-root matter.

tare. Material which must be discarded. *Also,* the empty weight of a container used for weighing or transporting material.

tare laboratory.* The station at which the beet samples taken at the receiving station are analyzed. The analytical results of the tare laboratory are used in deter-

mining the payments for the delivered beets.

thermodynamics. *chem.* The relationship of equivalence of heat, work, and other forms of energy are more particularly of energy equilibrium in chemical reactions and in changes of state (crystallization).

thermolabile. Unstable to heat.

thermophilic. Heat-loving organisms.

thermostable. Stable to heat.

thick juice.* Concentrated purified juice—the sugar-bearing product of the juice evaporator system.

thin juice.* The purified juice before evaporation.

third presses. *coll.* Filter presses used to filter thin juice following sulfitation.

third saturation. *coll.* Refers to sulfitation of the thin juice, although this is by no means carried out to the point of saturation.

tightening the strike.* The final phase of batch sugar boiling in which the syrup feed is stopped and evaporation continued to further exhaust the mother liquor of sucrose and to adjust the massecuite to the desired final density.

tops.* The beet leaves and petioles, which may or may not be accompanied by crowns or pieces of crown.

total solids.* The moisture-free matter present.

total-sugars content.* Content of saccharides as defined by the method(s) of analysis used and the basis for reporting; e.g., "total sugars by acid hydrolysis and Lane-Eynon titration, as % invert."

tramp air.* *coll.* The air that enters a boiler in such a way that it does not participate in the combustion process, thereby lowering efficiency.

trash catcher.* Apparatus that removes beet tops and weeds from the beet flume; usually a mechanism of traveling, self-cleaning rakes or combs.

trash rolls. Specially designed roller table, often similar to a beet screen, for separating beets from flume water and simultaneously separating beet tops and weeds from the beets.

trash screen.* Apparatus that removes the coarser suspended solids (mostly vegetable matter) from the flume water or flume effluent; often a vibrating screen.

twin.* A pair of mutually intergrown crystals.

true purity. *coll.* The percentage proportion of true sucrose to total dry substance. Usually sucrose is determined by an inversion method, and total dry substance by drying.

twin. *phys.* A compound crystal composed of two or more crystals, in reverse position to each other.

UDP. *chem.* Uridine diphosphate.

Ulbricht sphere. *phys.* A hollow sphere, used for optical integration of the light flux from the source of illumination. In the *sphere photometer*, it integrates the light scattering by the sample solution.

ultrafiltration. *phys. chem.* Filtration in which particles of colloidal size are separated from molecular and ionic substances by drawing the liquid through a membrane, in which the capillaries are very small. Dialysis.

unaccounted loss.* The deficit in the sucrose balance of the process after the production and accounted losses are subtracted from the quantity of sucrose entered.

underfeed stoker. A stoker system consisting essentially of a fixed grate to which coal is fed by means of a reciprocating ram or screw feeder, which forces the coal from below to the surface of the grate by mechanical force.

vacuole. *bot.* A cavity or vesicle in the protoplasm of a cell, containing watery fluid.

vapor.* Steam derived from boiling juice or syrup, as differentiated from steam originating in the boiler house.

vernalization. *agr.* To produce premature flowering or fruiting of a plant.

wash syrup.* The filtrate leaving a centrifugal after the initiation of the wash, consisting of displaced green syrup plus the solution of sugar crystals dissolved by the wash.

waste lime.* The solid matter remaining after the waste-lime slurry is dewatered.

waste-lime loss.* Sucrose leaving the process via the waste-lime slurry, usually expressed as percent on beets or percent on sucrose entered.

wet hopper.* The rectangular hopper constructed over a section of beet flume, for the transport of beets to the factory process immediately upon their unloading from a railroad car, truck, or other carrier.

wet pulp.* Pulp leaving the diffuser.

wet scrubbing. *coll.* A gas cleaning system using water or some suitable liquid to entrap particulate matter, fumes, and absorbable gasses. The collected substances are then withdrawn along with the scrubbing liquid.

white boiling.* The crystallization-separation scheme that operates at the highest spurity, using chiefly standard liquor (q.v.) as its feed and yielding a crystalline product that becomes marketable sugar upon washing, drying, and (usually) screening.

zeolite. *geol.* A hydrous silica ion-exchange material.

APPENDIX B

A Tabular, Chronological History of all Beet-Sugar Manufacturing Enterprises in the United States

Abbreviations: cap. = capacity, in tons beets sliced per day; pres. = present; des. = designed; mol. wkd. = tons molasses worked in Steffen process per day; Great Western = The Great Western Sugar Company; Spreckels = Spreckels Sugar Company; Utah-Idaho = Utah-Idaho Sugar Company; Amalgamated = The Amalgamated Sugar Company; American Crystal = American Crystal Sugar Company; and Holly = Holly Sugar Corporation.

Year Built	Location	Original Construction and Equipment	Remarks
1830	Philadelphia, Pa.	No factory constructed.	Seed distributed and beets raised.
1838	Northampton, Mass.	A small factory built.	Operations ceased after 3 years.
	White Pigeon, Mich.	A small factory built.	Operations ceased after 2 years.
1853	Salt Lake City, Utah	Built by Mormon Church.	Failed, 1855.
1856	San Francisco, (Ocean View), Calif.	Built by Bepler, coppersmith.	Abandoned, 1858.
1868	Chatsworth, Ill.	Built by Germania Beet Sugar Co. Cap., 50 tons.	Sponsors included Gennert Bros., N. Y., Joseph Bunn, capitalist, Springfield, Ill. Failed, 1871, and moved to Freeport by Bunn.
	Fond Du Lac, Wis.	Built by Otto and Bonesteel. Cap., 10 tons. Cost, $12,000.	Four acres harvested, 1868; 120 acres, 1869. Then abandoned, after fair success.
1870	Black Hawk, Wis.	Built by cooperative. Some machinery brought from Fond Du Lac and Freeport.	Hampered by bad shortage of water. Failed, 1875.
	Alvarado, Calif.	Built by Calif. Beet Sugar Mfg. Co. Steffen process.	First successful beet-sugar factory in Western Hemisphere. Closed after Spring, 1967. Equipment moved to other factories. Manufacturers liquid sugars. Serves as bulk storage and distribution point. Buildings intact.
1871	Brighton, Calif.	Built by Sacramento Valley Sugar Co. Cap., 70 tons. First factory to use diffusion battery in U.S.A.	Made sugar, 1871-3. Made molasses, 1874-6. In 1879, machinery moved to Alvarado, Calif.
	Freeport, Ill.	J. Bunn installed machinery from Chatsworth, Ill. in new buildings.	Failed after first campaign, making 100 tons sugar. Machinery moved to Black Hawk, Wis.
1874	Soquel, Calif.	Bonesteel and Otto moved machinery here from Alvarado.	Ceased operations, 1877. Reopened, 1879 or 1880 and made 150 tons of sugar. Not run again.
1876	Portland, Me.	Build as adjunct to Forest City Sugar Refining Works. Imported German machinery.	Moderately successful for 5 years. Abandoned, 1881, account shortage of beets.

Year Built	Location	Original Construction and Equipment	Remarks
1877	Isleton, Calif.		Built to obtain sugar from watermelons, and failing, converted to beets. Abandoned, 1878. Operated for one year, 1880.
	Edgemoor, Dela.	Built by Delaware Beet Sugar Co. Cap., 60 tons.	Failed after first campaign due to poor cultivation, short crops, no profits.
	Franklin, Mass.	Built by Franklin Sugar Refining Co.	Large crop of beets, but they were worth more for cattle food than the factory could afford to pay. Closed after first campaign.
1888	Watsonville, Calif.	Built by Western Beet Sugar Co.	Dismantled after Spreckels, Calif. factory built in 1899.
1890	Grand Island, Neb.	Built for Oxnard Beet Sugar Co. by Carion-Delmotte of Lille, France. Cap., 350 tons. Steffen process, pulp drier.	Enlarged by Dyer, 1916, to 500 tons. Purchased by American Crystal. Closed, 1964. Equipment and building sold. Used for contractors headquarters.
1891	Lehi, Utah	Built by Utah Sugar Co. Cap., 300 tons. Steffen process, bonechar process, never used.	Enlarged by Dyer, 1900, to 1200 tons. For many years had 3 auxiliary slicing stations at Springville, Bingham Junction, and Provo, pumping the juice to the factory. Extracted juice was limed, and then pumped. System abandoned because of pipe corrosion. Factory dismantled, 1937.
	Norfolk, Neb.	Built for Norfolk Beet Sugar Co. by Carion-Delmotte of Lille, France. Cap., 350 tons.	Moved to Lamar, Colo., 1905. First Steffen process in U.S.A.
	Chino, Calif.	Built by Chino Valley Beet Sugar Co. German machinery from Grevenbroich Maschinenfabrik. Cap., 400 tons. Steffen process, pulp drier.	Ceased operations after campaign of 1917, and moved to East Grand Forks, Minn., for 1926 campaign.
1892	Staunton, Va.	O. K. Lapham of N. Y. installed beet-sugar machinery in some tannery buildings.	Operated 1892-3. Burned, 1904.
1896	Eddy (Carlsbad), N.M.	Erected by Pecos Valley Beet Sugar Co., (Milwaukee brewing interests) French machinery from Berthierville, Canada, Cap., 200 tons.	Operated 1896-8. Burned, 1903.
1897	Los Alamitos, Calif.	Built by Dyer for Los Alamitos Sugar Co. Cap. 350 tons. Steffen process.	Enlarged by Dyer, 1898, to 900 tons. Dismantled in 1926.
	Rome, N. Y.	Built by First New York Beet Sugar Co.; German (Hallesche Machinenfabrik) machinery from West Farnham, Que. Cap., 200 tons.	Operated 1897-99. Machinery moved to Visalia, Calif., 1906, by G. S. Dyer.

Year Built	Location	Original Construction and Equipment	Remarks
1897	Menominee Falls, Wis.	Built for Wisconsin Sugar Co. by Dr. Korn. Cap., 500 tons. Pulp drier.	Operated 1897; closed until rebuilt by Henry Hinze, for the Co. in 1901, to 600 tons cap. Dismantled in 1932.
1898	Ogden, Utah	Built by Dyer for Ogden Sugar Co. Cap., 350 tons. *Steffen process.*	Enlarged by Dyer, 1912, to 1000 tons. Machinery moved to Nampa, Ida., 1942.
	Oxnard, Calif.	Built by Oxnard Construction Co. for Pacific Beet Sugar Co. Cap. 2000 tons. Steffen process, pulp drier.	Acq. by American Crystal. Incr. to 3000 tons. Closed 1958. Sold as junk.
	Crockett, Calif.	Built by California Beet Sugar and Refining Co.; Kilby machinery, in old Starr Flour Mill. Cap., 500 tons.	Also refined cane sugar. Closed, 1903. Beet processing machinery moved to Corcoran, Calif., 1908.
	Bay City, Mich.	Built for Michigan Sugar Co. by Fred W. Wolf. Cap., 500 tons.	Operated, 1898-1905. Machinery moved to Waverly, Iowa, 1907.
	St. Louis Park, Minn.	Built by Minnesota Sugar Co.; Dyer and Kilby Construction. Cap., 350 tons.	Operated 1898-1904. Burned, 1905. Salvaged machinery, moved to Visalia, Calif., 1906.
	Binghamton, N. Y.	Built by Binghamton Beet Sugar Co.; French (Fives-Lille) machinery. Cap., 350 tons.	Operated 1898-1902. Machinery moved to Blackfoot, Ida., 1904.
	La Grande, Ore.	Built by Dyer for Oregon Sugar Co. Cap., 350 tons.	Operated 1898-1911. Machinery moved to Burley, Ida., 1912.
1899	Betteravia, Calif.	Built by Union Sugar Co. J. C. H. Stut, engr.; Marschutz and Cantrell machinery. Cap., 500 tons. Steffen process, pulp drier.	Steffen process discontinued. Pres. cap. 5600 tons. R. T. diffuser.
	Spreckels (Salinas),	Built by Spreckels. Cap., 3000 tons. Steffen process, pulp drier. Claus Spreckels imported machinery from Germany; then, world's largest factory.	Pres. cap., 6000 tons, mol. wkd., 400 tons. Gran. carbon.
	Grand Junction, Colo.	Built by Dyer for Colorado Sugar Mfg. Co. Cap., 350 tons.	Enlarged to 900 tons. Acquired by Holly Sugar Corp., 1922. Dismantled, 1943.
	Holland, Mich.	Built by Dyer for Holland Sugar Co. Cap., 350 tons.	Closed 1929-32, purch. by Lake Shore Sugar Co., restarted 1933. Closed 1942, farmers growing war-useful crops. 1942 leased to Doughnut Corp. making dextrose from wheat flour. 1946 made gluten for IMCC Detroit MSG plant, until 1948, after which dismantled.
	Waverly, Wash.	Built by Emil Salich for D. C. Corbin. Cap., 350 tons.	Operated 1899-1910. Machinery moved to Centerfield, Utah, 1918.

Year Built	Location	Original Construction and Equipment	Remarks
1899	Kalamazoo, Mich.	Built by Kalamazoo Beet Sugar Co., Fred W. Wolf machinery. Cap., 500 tons.	Closed, 1903. Machinery moved to Chippewa Falls, Wis., 1904.
	Pekin, Ill.	Built by Fuehrman and Hapke for Illinois Sugar Refining Co., machinery of Grevenbroich design. Cap., 700 tons.	After one year's operation, converted to glucose factory. Machinery moved to Glendale, Ariz., 1905.
	Rochester, Minn.	Built by Fred W. Wolf for Detroit Sugar Co. Cap., 500 tons.	Operated 1899-1903. Machinery moved to Madison, Wis., 1905.
	Benton Harbor, Mich.	Built by Dyer for Wolverine Sugar Co. Cap., 350 tons.	Operated 1899-1900. Machinery moved to Berlin, Ont., 1902.
	Leavitt, Neb.	Built by Oxnard for Standard Beet Sugar Co. Cap., 500 tons.	Enlarged to 1,100 tons. Oper. 1899-1906. Machinery moved to Scottsbluff, Nebr. 1910.
	Bay City, Mich.	Built by Kirby for Bay City Sugar Co. Cap., 600 tons. Steffen process, pulp drier.	Enlarged to 1,400 tons. Dismantled 1941.
	Alma, Mich.	Built by Kilby, for Alma Sugar Co. Cap., 600 tons. Steffen process, pulp drier.	Acquired by Michigan Sugar Co. Closed after 1952 campaign. All buildings dismantled except sugar warehouse and boiler house.
	West Bay City, Mich.	Built by Bartlett-Hayward Co., for West Bay City Sugar Co. Cap., 500 tons. Pulp drier.	Enlarged to 900 tons. Dismantled in 1943.
	Caro, Mich.	Built by A. Wernicke Maschinenbau Anstalt of Halle for Peninsular Sugar Refining Co. Cap., 600 tons. Steffen process, pulp drier.	Present owner, Michigan Sugar Co. Present cap., 2400 tons. Steffen process removed, makes liquid sugars.
1900	Rocky Ford, Colo.	Built by Oxnard for American Beet Sugar Co., Cap., 1000 tons.	Cap. 3350 tons, 110 tons molasses wkd. Closed after 1978-79 campaign.
	Sugar City, Colo.	Built by Bartlett-Hayward Co. for National Beet Sugar Co. Cap., 500 tons.	Incr. to 1200 tons. Acquired by Utah-Idaho. Closed after 1942 campaign. Part of equipment moved to other factories. Building dismantled, 1949. Warehouse sold.
	Fremont, Ohio	Built by Dyer for Continental Sugar Co. Cap., 350 tons.	Acquired by Great Western in 1955. Now operated by Northern Ohio Sugar Co., Great Western subsidiary. Present cap., 3200 tons. Pulp drier. Refines cane sugar during off season, 450 tons melt (cane conversion April, 1978).
	Marine City, Mich.	Built by A. W. Colwell for Marine Sugar Co. Cap., 350 tons. Bone char process.	Enlarged by Kilby to 600 tons. Dismantled, 1928.
	Lyons, N. Y.	Built by Fuehrman and Hapke, machinery of Grevenbroich design, for Empire	Operated 1900-9. Machinery moved to Anaheim, Calif., 1911.

Year Built	Location	Original Construction and Equipment	Remarks
1900		State Sugar Co. Cap., 600 tons.	
1901	Bay City (Salzburg), Mich.	Built by German-American Sugar Co., Meinhausen machinery. Cap., 400 tons. Steffen process (not used at present), pulp drier.	Present owner, Monitor Sugar Co. Present cap., 4300 tons.
	Lansing, Mich.	Built by Kilby for Lansing Sugar Co. Cap., 800 tons. Pulp drier.	Acquired by Michigan Sugar Co., incr. to 1100 tons. Closed after 1950 campaign. Main building used as hardware distn. center. Rest dismantled 1954.
	Loveland, Colo.	Built by Kilby for Great Western Sugar Co. Cap., 600 tons. Steffen process. (Great Western's first factory.)	Present cap., 3400 tons. 185 tons mol. wkd. Pulp drier added.
	Logan, Utah	Built by Dyer for Logan Sugar Co. Cap., 350 tons.	One of the first to use the Osmose process. Dismantled in 1936.
1902			Ogden Sugar Co. (Ogden factory), Logan Sugar Co. (Logan factory) and Oregon Sugar Co. (La Grande factory) combined to form The Amalgamated Sugar Co. Lewiston Sugar Co. (Lewiston factory) joined Amalgamated in 1914.
	Saginaw, Mich.	Built by Kilby for Saginaw Sugar Co. Cap., 600 tons.	Operated 1901-3. Moved to Sterling, Colo., 1905.
	Greeley, Colo.	Built by Dyer for Greeley Sugar Co. Cap., 600 tons.	Merged with Great Western in 1905. Present cap., 2100 tons. Pulp drier added.
	Eaton, Colo.	Built by Kilby for Eaton Sugar Co. Cap., 600 tons.	Merged with Great Western in 1905. Pulp drier added. Closed following 1975-76 campaign.
	Carrollton (Saginaw), Mich.	Built by Kilby for Valley Sugar Co. Cap., 600 tons. Steffen process, pulp drier.	Present owner Michigan Sugar Co. Present cap. 2400 tons; Steffen process not used.
	Mt. Clemens, Mich.	Built by Kilby for Macomb Sugar Co. Cap., 600 tons. Pulp drier.	Acq. by Franklin Sugar Co., Incr. to 1250 tons. Opd. 1902-28, 1932-42, 1944, 1946-47, 1950. In 1946-47 also made dextrose for Doughnut Corp. as well as sliced beets. Dismantled 1951 centrifugals to Fremont, Ohio, Kelly filters to Croswell, Misc. equipment to Menominee, Mich. Balance of equipment junked and ground cleared, 1955.
	Croswell, Mich.	Built by Oxnard for Sanilac Sugar Refining Co. Cap., 600 tons. Steffen process (not used at present), pulp drier.	Acquired by Michigan Sugar Co. Present cap., 1900 tons.

Year Built	Location	Original Construction and Equipment	Remarks
1902	Sebewaing, Mich.	Built by Fuehrman and Hapke, for Sebewaing Sugar Refining Co. Cap., 600 tons.	Present owner, Michigan Sugar Co. Present cap., 2600 tons.
1903	Garland, Utah	Built by Dyer for Utah Sugar Co. Cap., 600 tons. Steffen process.	Acq. by U & I. Cap. 2700 tons. 140 tons molasses wkd. Closed after 1978-79 campaign.
	Idaho Falls, Idaho	Built by Dyer for Idaho Sugar Co. Cap., 600 tons. Pulp drier.	Acq. by U & I. Cap. 4400 tons. 140 tons mols. wkd. Closed after 1978-79 campaign.
	St. Louis, Mich.	Built by Dyer for St. Louis Sugar Co. Cap., 500 tons. Steffen process, pulp drier.	Operated continuously until 1954. Acq. by Lake Shore Sugar Co. at some time. 1954 dismantled; pilers and unloaders sold to Monitor. All bldgs. dismantled except sugar whse.
	Menominee, Mich.	Built by Kilby for Menominee River Sugar Co. Cap., 1000 tons. Pulp drier.	At some time acq. by Superior Sugar Refining Co. Incr. to 1300 tons. Opd. 1903-26, 1933-53. Machinery scrapped, 1954. Plant dismantled, 1956.
	Owosso, Mich.	Built by Kilby for Owosso Sugar Co. Cap., 1000 tons. Pulp drier.	Enlarged to 1600 tons. Inoperative since 1933. Dismantled, 1948.
	Ft. Collins, Colo.	Built by Kilby for Fort Collins Sugar Co. Cap., 1200 tons. Steffen process.	Merged with Great Western in 1905. Closed in 1955. Abandoned in 1965. Dismantled in 1967.
	Windsor, Colo.	Built by Kilby for Windsor Sugar Co. Cap., 600 tons.	Merged with Great Western in 1905. Closed in 1968. Some equipment to Goodland, Kansas. Plant dismantled in 1968.
	Longmont, Colo.	Built by Kilby for Longmont Sugar Co. Cap., 600 tons. Steffen process.	Merged with Great Western in 1905. Pulp drier added. Closed following 1976-77 campaign.
	East Tawas, Mich.	Built by Kilby for Tawas Sugar Co. Cap., 600 tons.	Operated 1903-4. Machinery moved to Chaska, Minn., 1906.
1904	Chippewa Falls, Wis.	Built by Chippewa Sugar Refining Co.; Wolf machinery moved from Kalamazoo. Cap., 600 tons. Pulp drier.	Dismantled, 1934.
	Janesville, Wis.	Built by Theodore Hapke for Rock County Sugar Co. Cap., 600 tons. Pulp drier.	Machinery originally set up at Dresden, Ont. by Fuehrman and Hapke. At one time used the Osmose process. Moved to St. Hilaire, Que., 1940.
	Blackfoot, Ida.	Built by Snake River Valley Sugar Co. French (Fives-Lille) machinery moved from Binghamton, N. Y. Cap., 600 tons.	Acq. by Utah-Idaho. Closed season of 1948. Part of equip. moved to other factories. Bldg. and whse. still in use for dried pulp and sugar storage.

Year Built	Location	Original Construction and Equipment	Remarks
1904	Sugar City, Ida.	Built by Dyer for Fremont County Sugar Co. Cap., 350 tons. Steffen process.	Acq. by Utah-Idaho. Closed season of 1942. Part of equip. moved to other factories. Bldg. dismantled 1949. Whse. sold.
1905	Lewiston, Utah	Built by Dyer for Lewiston Sugar Co. Cap., 600 tons. Steffen process.	Acq. by Amalgamated. Cap. 1900 tons; 105 mol. wkd. Closed 1972. Dismantled.
	Blissfield, Mich.	Built by Dyer for Continental Sugar Co. Cap., 600 tons. Steffen process, pulp drier.	Opd. 1905-42, 1945-46, 1948-50. At some time acq. by Great Lakes Sugar Co. Closed after 1954 campaign. Centrifugals sold to Fremont, Ohio. Factory sold to Great Western 1955. Never operated since.
	Riverdale, Ill.	Built by Chas. Pope; Pope Engineer's machinery. Cap., 350 tons. Bone char, Steffen process, pulp drier.	Last operated in 1925. Dismantled in 1927.
	Lamar, Colo.	Built by American Beet Sugar Co.; French machinery from Norfolk, Neb. Cap., 400 tons. Steffen process.	Enlarged to 500 tons. Last operated, 1912, and dismantled, 1927.
	Sterling, Colo.	Built by Sterling Sugar Co. Machinery moved from Saginaw, Mich. Cap., 600 tons. Steffen process (Steffen process later abandoned).	Merged with Great Western in 1905. Present cap., 2450 tons. Pulp drier added.
	Madison, Wis.	Built by United States Sugar Co., machinery moved from Rochester, Minn. Steffen process, pulp drier. Cap., 600 tons.	Dismantled in 1924.
	Glendale, Ariz.	Built by Fuehrman and Hapke for Eastern Sugar Co. Cap., 800 tons.	Operated on beets to 1915; then ground cane for 2 short campaigns. Beet processing machinery moved to Delta, Colo., 1920.
	Holly, Colo.	Built by Holly Sugar Co. Cap., 600 tons.	Machinery moved to Sheridan, Wyo., 1915.
1906	Hamilton City, Calif.	Built by Oxnard for Alta California Beet Sugar Co. Cap., 600 tons. Steffen process (not used).	Acq. by Holly, 1935. Ion exch. used on int. green several years in 60's, early 70's. Cap. 2800 tons.
	Brush, Colo.	Built by Dyer, for Morgan County Construction Co. Cap., 600 tons.	Acq. by Great Western. Incr. to 1600 tons. Closed, 1955. Abandoned, 1965, dismantled.
	Fort Morgan, Colo.	Built by Morgan County Construction Co.; Riter-Conley machinery. Cap., 600 tons. Steffen process.	Present owner, Great Western. Present cap., 3250 tons. 185 tons mol. wkd. Added pulp drier.
	Swink, Colo.	Built by Holly Sugar Co. Cap. 1200 tons. Steffen process.	Closed, 1959. Sold to American Crystal. Equip. moved to other plants or junked. Bldgs. and property sold.

Year Built	Location	Original Construction and Equipment	Remarks
1906	Billings, Mont.	Built by Kilby for Billings Sugar Co. Cap., 1200 tons. Steffen process, pulp drier.	Present owner, Great Western. Present cap., 4050 tons. 230 tons mol. wkd. Enzyme process.
	Garden City, Kan.	Built by Stearns-Roger for U. S. Sugar and Land Co. Steffen process, pulp drier.	Acq. by the Garden City Co. Last campaign 1955. Purch. by Holly and some machinery removed. 1959 purch. by American Crystal, removed some machinery. 1960, sold and scrapped. Whse. intact, rented for commercial use.
	Chaska, Minn.	Built by Carver County Sugar Co. Cap., 600 tons. Machinery moved from East.	Acq. by American Crystal. Cap., 2200 tons. Closed 1971. Retained for distribution center and liquid sugar mfg.
	Nampa, Ida.	Built by Dyer for Western Idaho Sugar Co. Cap., 750 tons.	Closed, 1911, due to ravages of the "white fly." Machinery moved to Spanish Fork, Utah, 1916, by Utah-Idaho.
	Visalia, Calif.	Built by C. S. Dyer for Pacific Sugar Corp. Machinery moved from Rome, N. Y. and St. Louis Park, Minn. Cap., 350 tons.	Machinery moved to Hooper, Utah 1919.
	Charlevoix, Mich.	Built by Walburn-Swenson for West Michigan Sugar Co. Cap., 600 tons.	Machinery moved to Ottawa, Ohio, 1912.
1907	Las Animas, Colo.	Built by Dyer for American Beet Sugar Co. Cap., 700 tons.	Last operated, 1920; dismantled, 1942.
	Waverly, Iowa	Built by Iowa Sugar Co. Machinery from Bay City, Mich. Cap., 400 tons. Pulp drier.	Incr. 700 tons. Operated 1907-13, 1920-1, 1942. Made corn sugar, 1945-6. Not operated since.
1908	Corcoran, Calif.	Built by Pacific Sugar Corp. Machinery from Crockett, Calif. Cap., 600 tons.	Machinery moved to Whitney, Ida., 1921-2.
	Santa Ana (New Delhi), Calif.	Built by Southern California Sugar Co., Walburn-Swenson machinery moved from Wiarton, Canada, by Hinze. Cap., 600 tons. Steffen process, pulp drier.	Operated by Holly Sugar Co., 1916-20. Dismantled. 1924.
1910	Scottsbluff, Neb.	Built by Scottsbluff Sugar Co., Oxnard machinery from Leavitt, Neb. Cap., 1200 tons. Steffen process.	Present owner, Great Western. Present cap., 3300 tons. 190 tons mol. wkd. Pulp drier added.
	Paulding, Ohio	Built by Larrowe for German-American Sugar Co. Cap., 700 tons. Steffen process, pulp drier.	Acq. by Paulding Sugar Co. Consolidated with Great Lakes Sugar Co. 1948. Did not operate after that date. Dismantled, 1953.
1911	Findlay, Ohio	Built by Dyer for Continental Sugar Co. Cap., 100 tons. Pulp drier.	Acq. by Great Western in 1955. Oper. by Northern Ohio Sugar Co., Great

Year Built	Location	Original Construction and Equipment	Remarks
			Western subsidiary. Closed following 1977-78 campaign. Sugar terminal remains.
	Monte Vista, Colo.	Built by Dyer for San Luis Valley Beet Sugar Co. Cap., 600 tons.	Closed, 1915. Machinery moved to Lovell, Wyo., 1916.
	Elsinore, Utah	Built by Dyer for Utah-Idaho. Cap., 500 tons.	Enlarged to 1100 tons. Dismantled, 1942.
	Fallon, Nev.	Built by Nevada Sugar Co. Machinery from Watsonville, Calif., installed by Hinze. Cap. 500 tons.	Dismantled, 1933.
	Anaheim, Calif.	Built by Anaheim Sugar Co. Machinery from Lyons, N. Y. Pulp drier.	Machinery moved to Sidney, Mont., 1925.
	Huntington Beach, Calif.	Built by Holly Sugar Co., Steffen process, pulp drier. Cap., 750 tons. Also had potash plant.	Machinery moved to Torrington, Wyo., 1926.
1912	Dyer (Santa Ana), Calif.	Built by Dyer for Santa Ana Cooperative Sugar Co. Cap., 600 tons. Steffen process, pulp drier.	Acq. by Holly. Cap., 1800 tons. 120 tons mol. wkd. Converted to cane refinery, 1979.
	Burley, Ida.	Built by Dyer for Amalgamated Machinery from La Grande, Ore. Cap., 400 tons.	Enlarged to 1275 tons. Abandoned, 1948.
	Toledo, Ohio	Built by Larrowe for Toledo Sugar Co. Cap., 1000 tons. Steffen process, pulp drier.	Enlarged to 1500 tons. Dismantled, 1941.
	Ottawa, Ohio	Built by National Construction Co. for Ohio Sugar Co. Machinery from Charlevoix, Mich. Cap., 600 tons. Pulp drier.	Present owner, Buckeye Sugars, Inc. Cap., 1600 tons. Closed, 1979.
	Decatur, Ind.	Built by Larrowe for Holland-St. Louis Sugar Co. Cap., 700 tons. Steffen process, pulp drier.	Dismantled, 1944.
1913	Payson, Utah	Built by Dyer for Utah-Idaho Sugar Co. Cap., 500 tons.	Enlarged to 750 tons. Dismantled, 1940.
1915	Layton, Utah	Built by Dyer for Layton Sugar Co. Cap., 500 tons.	Operated for several years with full ion exchange. Acq. by Utah-Idaho. Closed, 1958. Part of equip. moved to other factories. Whse. now used for sugar storage. Factory bldg. dismantled, 1969.
	Sheridan, Wyo.	Built by Holly. Machinery from Holly, Colo. Steffen process, pulp drier.	Closed in 1947. Equip. moved to other factories or junked. Whses. used for sugar storage. Factory bldg. rented.
1915 (or 1916)	Maxwell, N. M.	In defunct plant of Rocky Mountain (Meat) Packing Co. With too small boilers, used locomotive boiler to assist. Two "Scotchmen."	Closed after 6 months opn. Not enough irrign. water.

Year Built	Location	Original Construction and Equipment	Remarks
1916	Lovell, Wyo.	Built by Dyer for Great Western. Machinery from Monte Vista, Colo. Cap., 600 tons.	Present cap., 2100 tons. Pulp drier added.
	Twin Falls, Ida.	Built by Larrow for Amalgamated. Cap., 600 tons. Steffen process.	Present cap., 4600 tons, 200 tons mol. wkd. Added Mag-Ox process, CSF.
	Gering, Neb.	Built by Great Western. Cap., 1200 tons. Steffen process, pulp drier.	Present cap., 2100 tons, 130 tons mol. wkd.
	Spanish Fork, Utah	Built by Dyer for Utah-Idaho. Machinery from Nampa, Ida. Cap., 750 tons. Steffen process.	Enlarged to 1750 tons. Abandoned, 1942. Dismantled, 1948.
	West Jordan, Utah	Built by Dyer for Utah-Idaho. Cap., 500 tons. Pulp drier.	Enlarged to 1700 tons. Closed in 1971. Bldgs. conv. to bulk sugar stge., pkg. and distr. center.
	Brigham City, Utah	Built by Dyer for Utah-Idaho. Cap., 500 tons.	Enlarged to 1300 tons. Dismantled, 1941.
	Grant's Pass, Ore.	Built by Dyer for Oregon-Utah Sugar Co.	Operated 1916-17. Machinery moved to Toppenish, Wash. 1919.
1917	Tracy, Calif.	Built by Dyer for Pacific Sugar Corp. Cap., 500 tons. Steffen process, pulp drier.	Acq. by Holly, 1927. Present cap., 4300 tons. 150 tons mol. wkd. Corn HFCS unit added 1979.
	Mini-Cassia (Paul), Idaho	Built by Larrowe for Amalgamated. Cap., 600 tons.	Present cap., 6450 tons. Quentin process.
	Smithfield (Amalga), Utah	Built by Dyer for Amalgamated. Cap., 700 tons.	Machinery moved to Clarksburg, Calif., 1935. Sold to American Crystal, 1936.
	Bayard, Neb.	Built by Great Western. Cap., 1300 tons. Pulp drier.	Present cap., 2200 tons.
	Brighton, Colo.	Built by Larrowe for Great Western. Cap., 1300 tons. Pulp drier.	Closed following 1976-77 campaign. Sugar terminal remains.
	Moroni, Utah	Built by James Stewart & Co., for the Peoples Sugar Co. Cap., 400 tons.	Moved to Toppenish, Wash., 1937.
	Manteca, Calif.	Built by Dyer for Spreckels. Cap., 1200 tons.	Present cap., 4200 tons. Multi-hearth kiln pond lime reburner. Gran. carbon.
	Missoula, Mont.	Built by Dyer for Great Western. Cap., 1300 tons.	Operated 1 yr. Machinery moved to Mitchell, Neb., 1920.
	Delta, Utah	Built by Stearns-Roger for Great Basin Sugar Co. Cap., 1000 tons.	Acq. by U & I, 1920. Closed in 1924 due mainly to curley top. Machinery moved to Belle Fourche, S. D., 1927.
	Shelley, Ida.	Built by Dyer for Utah-Idaho. Cap., 750 tons. Pulp drier.	Closed, 1942. Part of equip. moved to other factories, remainder junked. Building and whse. now used for dried pulp and sugar storage.

Year Built	Location	Original Construction and Equipment	Remarks
	North Yakima, Wash.	Built by Dyer for Utah-Idaho Sugar Co. Cap., 750 tons. Pulp drier.	Machinery moved to Chinook, Mont., 1925.
	Cornish, Utah	Constructed by Cannon and Lynch for West Cache Sugar Co. Dyer equipment moved from Raymond, Alta. Cap., 600 tons.	Purchased by Amalgamated, 1920. Moved to Missoula, Mont., 1927. Sold to American Crystal, 1936.
	Worland, Wyo.	Built by Larrowe for Wyoming Sugar Co. Cap., 600 tons. Steffen process.	Present owner, Holly. Present cap., 2200 tons, 75-90 tons mol. wkd. Thick jce. stge.
	Mason City, Iowa	Built by Larrowe for Northern Sugar Corp. Cap., 1200 tons. Steffen process, pulp drier.	Acq. by American Crystal. Steffen process abandoned, 1964. Cap., 2400 tons. Closed, 1973. Retained as distribution center.
1918	Springville, Utah	Built by Dyer for Springville, Mapleton Sugar Co. Cap., 350 tons.	Dismantled, 1940.
	Whitehall, Mont.	Built by Larrowe for The Amalgamated Sugar Co. Cap., 600 tons.	Never operated. Machinery moved to Honeyville, Utah, 1920, which was never completed.
	Centerfield, Utah	Built by Gunnison Valley Sugar Co. Machinery moved from Waverly, Wash. Cap., 500 tons.	Incr. to 1500 tons. Acq. by Utah-Idaho. Closed 1960. Equip. and bldg. sold, salvaged for junk.
1919	Rigby, Ida.	Construction started by A. P. Cooper—completed by Henry Schwartz. Cap., 800 tons.	Went into bankruptcy, 1922. Acq. by U & I, 1924. Discontinued opn. in 1930. Dismantled in 1939. Converted to sugar warehouse.
	Hooper, Utah	Constructed by Dan. Gutleben for Hooper Sugar Co. Cap., 600 tons.	Machinery originally erected in France; moved to West Farnham, Que.; Rome, N. Y., 1897; Visalia, Calif., 1905. Dismantled, 1936.
	Sunnyside, Wash.	Built by Larrowe for Utah-Idaho. Cap., 750 tons.	Moved to Raymond, Alta, Sold to B. C. Sug. Ref. Co., 1921.
	Toppenish, Wash.	Built by Dyer for the Utah-Idaho Sugar Co. Machinery from Grant's Pass, Ore. Cap., 750 tons. Steffen process.	Machinery moved to Bellingham, Wash., 1925.
1920	Belmond, Iowa	Built by Dyer for Iowa Valley Sugar Co., Cap., 600 tons. Pulp drier.	Operated by American Crystal 1928-30. Equip. moved to other plants 1933-42. Bldgs. sold, 1942.
	Mount Pleasant, Mich.	Built by Columbia Sugar Co., des. by H. A. Vallez. Cap., 1200 tons. Pulp drier.	Acq. by Michigan Sugar Co. Used first ion exchange process. Incr. to 1400 tons. Closed after 1947 campaign. Sugar whse. used for sugar storage. Other bldgs. dismantled, 1967.

Year Built	Location	Original Construction and Equipment	Remarks
1920	Mitchell, Neb.	Built by Great Western Machinery moved from Missoula, Mont. Pulp drier.	Present cap., 2150 tons.
	Green Bay, Wis.	Built by A. Bentley & Son, Schwartz Engineering Co., for Green Bay Sugar Co. Cap., 600 tons.	Acq. by Menominee Sugar Co. Incr. to 900 tons. Closed after 1954 campaign.
	Delta, Colo.	Built by Holly. Machinery moved from Glendale, Ariz. Cap., 600 tons. Steffen process.	Cap., 1640 tons; 55-60 tons mol. wkd. Closed, after 1976-77 campaign. Dismantled.
	Fort Lupton, Colo.	Built by James Stewart & Co. for Industrial Sugar Co. Cap., 600 tons.	Acq. by Great Western. Incr. to 1200 tons. Closed 1948; abandoned 1952; dismantled 1966.
1921	Preston, Ida.	Built by Preston Sugar Co., Dan Gutleben, engr. Kilby machinery. Cap., 900 tons.	Machinery originally erected at Crockett, Calif., 1898; moved to Corcoran, Calif., 1908. Acq. by Franklin County Sugar Co. Acq. by Amalgamated, 1960. Last campaign, 1962-63. Dismantled, 1964. Factory bldg. and whse. intact.
1925	Chinook, Mont.	Built by Utah-Idaho, machinery from Yakima, Wash. Cap., 900 tons. Pulp drier.	Incr. to 1500 tons. Closed 1951. part of equip., main bldg., and pulp drier moved to Moses Lake, Wash. Main bldg. redesigned for Steffen house. Remainder was junked.
	Sidney, Mont.	Built by Holly. Machinery moved from Anaheim, Calif. Cap., 1800 tons.	Present cap., 4000 tons. Thick jce. stge.
	Bellingham, Wash.	Built by Utah-Idaho. Machinery moved from Toppenish, Wash. Cap., 1150 tons.	Machinery moved to South America, 1941.
1926	Johnstown, Colo.	Built by Great Western.	Special plant for extraction of sugar from mol. by barium process. Refinery closed, 1977—liquid sugar terminal remains. Great Western built MSG plant in 1954. Closed 1977. Converted to HFCS plant 1980.
	Ovid, Colo.	Built by Great Western. Pulp dryer. Cap., 1900 tons. Const. new Steffen house, 1927—Steffen process since abandoned.	Present cap., 2650 tons.
	East Grand Forks, Minn.	Built by Honolulu Iron Works for American Beet Sugar Co. Machinery moved from Chino, Calif. Pulp drier.	Acq. by American Crystal. 1976, additional equip. added from Montezuma, N. Y., combined with another side, B.M.A., making a double factory. 7200 tons.
	Minatare, Neb.	Built by Great Western. Pulp drier. Cap., 1800 tons.	Dismantled, 1948.

Year Built	Location	Original Construction and Equipment	Remarks
1926	Torrington, Wyo.	Built by Holly. Machinery moved from Huntington Beach, Calif. Steffen process with enzyme process.	Present cap., 3200 tons.
1927	Missoula, Mont.	Built by Amalgamated. Machinery moved from Cornish, Utah. Pulp drier.	Acq. by American Crystal. Closed, 1965. Plant and equip. sold for junk.
	Lyman, Neb.	Built by Great Western. Cap., 1900 tons.	Closed, 1943. Operated 1947-48. Closed 1949. Bldg. steel to Goodland, 1966. Equip. to various factories. Dismantled.
	Belle Fourche, S. D.	Built by Utah-Idaho. Machinery moved from Delta, Utah. Cap., 1700 tons.	Closed, 1964. Part of equip. to other factories, remaining junked. Bldg. and whse. sold.
1930	Wheatland, Wyo.	Built by Great Western. Cap., 1500 tons.	Closed, 1940. Equip. to various factories, sold 1957.
	Clarksburg, Calif.	Built by Amalgamated. Machinery moved from Smithfield, Utah.	Present owner, American Crystal. Present cap., 3000 tons.
1937	Woodland, Calif.	Built by Spreckels. Cap., 1800 tons. Steffen process.	Present cap., 3300 tons, gran. carbon used.
	Hardin, Mont.	Built by Holly Corp., 1800 tons.	Cap., 1800 tons. Closed after 1970-71 campaign. Dismantled.
	Toppenish, Wash.	Built by Utah-Idaho. Machinery moved from Moroni, Utah. Cap., 1800 tons. Pulp drier.	Present cap., 4400 tons. Closed, 1979.
1938	Nyssa, Ore.	Built by Amalgamated. Cap., 2300 tons. Steffen process, pulp drier.	Present cap., 6350 tons. 200 tons mol. wkd. Quentin, CSF.
1942	Nampa, Ida.	Built by Amalgamated. Cap., 2000 tons. Machinery moved from Ogden, Utah. Added Quentin process and thick juice storage.	Present cap., 9000 tons.
1947	Brawley, Calif. (Carlton factory)	Built by Holly. Cap., 2700 tons. Pulp drier, and Silver chain-type continuous diffuser.	1960, added Silver slope diffuser and thick juice storage. Pres. cap., 6500 tons.
1948	Moorhead, Minn.	Built by American Crystal. Cap., 2500 tons. Silver chain-type continuous diffuser.	Replaced diffuser with Silver slope: Added ion exchange and thick juice storage. Cap., 4600 tons.
1953	Moses Lake, Wash. (Columbia Basin)	Designed for 4000 tons with juice storage. Steffen process, Silver scroll and slope diffusers.	Final cap., 10,500 tons. closed after 1978-79 campaign.
1954	Crookston, Minn.	Built by American Crystal. Cap., 3400 tons. Silver chain-type diffuser.	Diffuser replaced with Silver slope. Present cap., 4350 tons.
1963	Mendota, Calif.	Built by Spreckels. Cap., 3800 tons.	Steffen process. Lime from burning pond lime in rotary kiln. Slope diffuser. Cap., 4000 tons, gran. carbon.

Year Built	Location	Original Construction and Equipment	Remarks
1964	Hereford, Texas (Merrill E. Shoup)	Built by Holly.	Present cap., 6500 tons. RT diffuser, thick juice storage. Quentin process, 1978.
1965	Drayton, N. D.	Built by American Crystal. Cap., 5000 tons. D.D.S. slope diffuser.	Cap., 5400 tons.
	Montezuma, N. Y.	Built by Pepsico. Cap., 4000 tons. Also raw cane processing equip.	Operated campaigns 1966-67, 1967-68. Acq. by Maine Sugar Industries of Montezuma. Did not operate, 1968-69. Equip. to East Grand Forks, Minn., 1975.
1966	Easton, Maine	Built by Maine Sugar Ind. Also raw cane processing equip. BMA equip.	Operated by Maine Sugar Industries. First campaign, 1966-67. Cap., 3000 tons. Opd. 1 short campaign, 1976. Closed. Purch. by Quebec Sugar Refinery, 1979. Being dismanted.
1967	Chandler, Ariz.	Built by Spreckels. Cap., 4250 tons.	Oliver-Morton diffuser. Cap., 3750, gran. carbon.
1968	Goodland, Kan. (Frank Kemp)	Built by Great Western. Cap., 3300 tons.	Present cap., 3400 tons. Direct filtration, 1st carb effluent, served only by beet trucks. 660,000 cwt. sugar bin cap.
1974	Wahpeton, N. D.	Built by BMA for Minn-Dak Farmers Cooperative. Cap., 4500 tns.	1977, added lignite-fired boilers, thick juice stage. 1980, added Anamet Waste Water treatment system.
	Hillsboro, N.D.	Built by B.M.A., for Red River Valley Farmers Cooperative. Cap., 5000 tons.	Merged with American Crystal July, 1975. Present cap., 4700 tons.
1975	Renville, Minn.	Built by Ferguson Engineering Co. for Southern Minn. Sugar Cooperative. Cap., 6500 tons. Thick juice stge. RT diffuser.	Opd. 1975, 1976-77, by American Crystal; since 1978 opd. by owners. 1978 added BMA washer.

APPENDIX C

Year Built	Location	Original Construction and Equipment	Remarks
1881	Farnham, Que.	Built by La Compagnie de sucre de betterave de Quebec. Machinery from Hallesche Maschinenfabrik, Germany. Cap., 200 tons.	Operated 1881-3, 1890-2. Machinery moved to Rome, N. Y., 1897.
	Coaticook, Que.	Built by Pioneer Sugar Co., with German machinery. Cap., 200 tons	Operated 1881-3.
	Berthier, Que.	Built by Five-Lille, France, for L'Union franco-canadienne. Cap., 200 tons.	Factory closed, 1896. Machinery moved to Eddy, N. M.

Year Built	Location	Original Construction and Equipment	Remarks
1902	Berlin, Ont. (later, Kitchener, Ont.)	Built by Dyer for Ontario Sugar Co., Ltd. Machinery from Benton Harbor, Mich. Cap., 800 tons.	Ceased operations, 1920.
	Dresden, Ont.	Built by American Construction and Supply Co. for Dresden Sugar Co., Ltd. Cap., 600 tons.	Closed, 1904; machinery moved to Janesville, Wis.
	Wallaceburg, Ont.	Built by Kilby for Wallaceburg Sugar Co., Ltd. Pulp drier. Cap., 600 tons.	Has used Barium process on beet molasses. Plant upgraded to 2500 tons and cane raw sugar refining. Plant closed and liquidated 1960 because of lack of beets. Owned by C&D, now Redpath Sugars.
	Wiarton, Ont.	Built by Walbum-Swenson for Wiarton Beet Sugar Mfg. Co., Ltd. Cap., 350 tons.	Closed, 1904. Machinery moved to Santa Ana (New Delhi), 1908.
1903	Raymond, Alta.	Built by Dyer for Knight Sugar Co. Cap., 350 tons.	Closed, 1914. Machinery moved to Cornish, Utah, 1917.
1916	Chatham, Ont.	Built by Dominion Sugar Co., mostly Kilby construction. Cap., 1500 tons.	Plant upgraded to 3500 tons but closed permanently after last crop in 1967 and liquidated. Owned by Redpath Sugar.
1925	Raymond, Alta.	Built by Utah-Idaho Sugar Co.; machinery from Sunnyside, Wash.	Ceased operations in 1963. Factory dismantled. Bulk storage for refined sugar retained for storage of Taber sugar which is later packed at Taber.
1936	Picture Butte, Alta.	Built by Canadian Sugar Factories, Ltd. Cap., 1450 tons.	Ceased operations Dec., 1977. Bulk storage for refined sugar retained. Sugar moved to Taber factory for packing. Factory to be dismantled.
1940	Fort Garry (Winnipeg, Man.)	Built by Manitoba Sugar Co. Ltd. Name changed to Manitoba Sugar Company - a division of B C Sugar, Vancouver, B.C.	Present cap., 2720 tonnes (3000 short tons)
1944	Mont St. Hilaire, Que.	From Janesville, Wis. Cap., 1200 tons.	Built by Quebec Provincial Government, for La Rafinerie de Sucre de Quebec. Pres. cap., 1600 tons.
1950	Taber, Alta.	Built by Canadian Sugar Factories ltd. Name changed to Alberta Sugar Company - a division of B C Sugar, Vancouver, B. C.	Pres. cap., 4000 tonnes (4400 short tons)

APPENDIX D

Beet-Sugar Factories in Operation, North America, 1981-1982 Campaigns

Location (Name)	Operating Firm	First Year	Stand. Slice	Steffen	Other
Taber, Alberta	Alberta Sugar Co.	1950	4000 (metric)		
Twin Falls, Idaho	The Amalgamated Sugar Co.	1916	4500	X	Mag-Ox, CSF, Quentin, T.J. Stge.
Nampa, Idaho	The Amalgamated Sugar Co.	1942	9500		Quentin, T.J. Stge., Mag-Ox, pre-purge
Nyssa, Oregon	The Amalgamated Sugar Co.	1938	6350	X	Quentin, CSF, Mag-Ox
Mini-Cassia (Paul, Idaho)	The Amalgamated Sugar Co.	1917	6800		Quentin, Mag-Ox, pre-purge
East Grand Forks, Mn.	American Crystal Sugar Company	1926	6900		Anamet process
Clarksburg, California	American Crystal Sugar Company	1935	3000		
Moorhead, Minnesota	American Crystal Sugar Company	1948	4400		Ion Exch., T.J. Stge. Anamet process
Crookston, Minnesota	American Crystal Sugar Company	1954	4350		
Drayton, North Dakota	American Crystal Sugar Company	1965	5400		
Hillsboro, North Dakota	American Crystal Sugar Company	1974	4800		
Loveland, Colorado	The Great Western Sugar Company	1901	3300	X	
Greeley, Colorado	The Great Western Sugar Company	1902	2150		
Sterling, Colorado	The Great Western Sugar Company	1905	2450		
Fort Morgan, Colorado	The Great Western Sugar Company	1906	3250	X	
Billings, Montana	The Great Western Sugar Company	1906	4050	X	Enzyme
Scottsbluff, Nebraska	The Great Western Sugar Company	1910	3300	X	
Lovell, Wyoming	The Great Western Sugar Company	1916	2120		
Gering, Nebraska	The Great Western Sugar Company	1916	2100	X	
Bayard, Nebraska	The Great Western Sugar Company	1917	2200		
Mitchell, Nebraska	The Great Western Sugar Company	1920	2150		
Ovid, Colorado	The Great Western Sugar Company	1926	2650		

Goodland, Kansas (Kemp)	The Great Western Sugar Company	1968	3450		
Hamilton City, California	Holly Sugar Corp.	1906	2800		
Worland, Wyoming	Holly Sugar Corp.	1917	2200	X	Thick jce. stge.
Tracy, California	Holly Sugar Corp.	1917	4300		HFCS
Sidney, Montana	Holly Sugar Corp.	1925	4000		Thick jcs. stge.
Torrington, Wyoming	Holly Sugar Corp.	1926	3200	X	Enzyme
Brawley, California	Holly Sugar Corp.	1947	6500		
Hereford, Texas (Shoup)	Holly Sugar Corp.	1964	6500		Quentin
Winnipeg, Manitoba	Manitoba Sugar Co.	1940	3000 (metric)		
Caro, Michigan	Michigan Sugar Co.	1899	2700		
Croswell, Michigan	Michigan Sugar Co.	1902	1750		
Carrollton, Michigan	Michigan Sugar Co.	1902	2500		
Sebawaing, Michigan	Michigan Sugar Co.	1902	3200		
Wahpeton, North Dakota	Minn-Dak Farmer's Cooperative	1974	4500		Th.J. Stge, Anamet process
Bay City, Michigan	Monitor Sugar Company	1901	4300		
Fremont, Ohio	Northern Ohio Sugar Co. (Great Western subs.)	1900	2900		Refines cane sugar in inter-season.
Mont-St-Hilaire, Quebec	Quebec Sugar Refining Co.	1944	3630		Makes raw sugar. Ion Exch.
Renville, Minnesota	Southern Minnesota Beet Sugar Cooperative	1975	6500		Th. J. Stge.
Spreckels, California	Spreckels Sugar Div., Amstar Corp.	1899	6000	X	Gran. Carbon. tr. thin jce.
Manteca, California	Spreckels Sugar Div., Amstar Corp.	1917	4000		Gran. Carbon. tr. thin jce.
Woodland, California	Spreckels Sugar Div., Amstar Corp.	1937	3300	X	Gran. Carbon. tr. thin jce.
Mendota, California	Spreckels Sugar Div., Amstar Corp.	1963	4000	X	Gran. Carbon. tr. thin jce.
Chandler, Arizona	Spreckels Sugar Div., Amstar Corp.	1967	3750		Gran. Carbon. tr. thin jce.
Betteravia, California	Union Sugar Company (subs. Consolidated Foods Corp.)	1899	5200		

APPENDIX E

GRUTS TABLE

TEMP. IN °C.	100	99	98	97	96	95	94	93	92	91	90	89	88	87	86	85	84	83	82	81
30	218	217	216	215	214.5	214	213.5	213	212.6	212.3	212	211.8	211.7	211.7	211.8	212	212.3	212.6	213	213.5
31	220	219	218	217	216.5	216	215.3	214.7	214.2	213.8	213.5	213.3	213.2	213.2	213.3	213.5	213.8	214.1	214.5	215
32	223	221	220	219	218.2	217.7	217	216.2	215.7	215.3	215	214.8	214.7	214.7	214.8	215	215.3	215.6	216	216.5
33	224	223	222	221	220	219.5	219	218.5	218.1	217.8	217.5	217.3	217.2	217.2	217.3	217.5	217.8	218.1	218.5	219
34	226	225	224	223	222	221.5	221	220.4	219.8	219.3	219	218.8	218.7	218.7	218.8	219	219.3	219.6	220	220.5
35	228	227	226	225	224	223.5	223	222.3	221.7	221.3	221	220.8	220.7	220.7	220.8	221	221.2	221.4	221.7	222.1
36	230	229	228	227	226	225.2	224.6	224	223.5	223	222.7	222.5	222.4	222.4	222.5	222.7	222.9	223.1	223.4	223.7
37	232	231	230	229	228	227.2	226.5	225.7	225	224.5	224.2	224	223.9	223.9	224	224.2	224.5	224.8	225.1	225.5
38	234	233	232	231	230	229	228.2	227.5	227	226.5	226.2	226	225.9	225.9	226	226.2	226.4	226.7	227	227.5
39	236	235	234	233	232	231	230.4	229.7	229	228.5	228	227.8	227.7	227.7	227.8	228	228.2	228.4	228.8	229.3
40	238	237	236	235	234	233	232.3	231.6	231	230.5	230	229.8	229.7	229.7	229.8	230	230.2	230.5	231	231.5
41	241	239	238	237	236	235	234.2	233.8	233.3	232.8	232.5	231.8	231.7	231.7	231.8	232	232.2	232.5	232.8	233.2
42	243	241	240	239	238	237	236	235.5	235	234.5	234	233.8	233.7	233.7	233.8	234	234.2	234.5	235	235.7
43	245	243	242	241	240	239	238	237.5	237	236.5	236	235.8	235.6	235.6	235.8	236	236.2	236.5	237	237.7
44	248	245	244	243	242	241	240.2	239.5	239	238.5	238	237.9	237.8	237.9	238	238	238.5	239	239.5	240
45	250	248	247	246	245	244	243	242	241.5	241.1	240.7	240.6	240.5	240.3	240.4	240.5	241	241.5	242	243
46	252	250.2	249	248	247.2	246.5	245.7	245	244.5	244	243.5	243.3	243.1	242.9	243	243	243.5	244	244.7	245.5
47	255	253	252	251	250	249	248	247.2	246.8	246.4	246	245.8	245.6	245.3	245.4	245.5	246	246.5	247	248
48	257	255.5	254	253	252	251.2	250.5	250	249.5	249	248.5	248.3	248.1	247.9	248	248	248.5	249	250	251
49	260	258	257	256	255	254	253.2	252.5	252	251.5	251	250.8	250.6	250.4	250.4	250.5	251	251.5	252.5	253.5
50	262	261	260	259	258	257	256	255.5	255	254.5	254	253.6	253.2	252.9	253	253	253.5	254.3	255.5	256.8
51	265	264	263	261.7	260.5	259.7	259	258.2	257.8	257.4	257	256.7	256.4	256.1	256	256	256.5	257	257.7	258.7
52	268	266.5	265.2	264	263	262.5	262	261.1	260.7	260.3	260	259.6	259.2	258.8	259	259	259.5	260	260.5	261.6
53	271	269.5	268	267	266	265.5	265	264.1	263.7	263.3	263	262.6	262.2	261.9	262	262	262.5	263	263.7	264.5
54	274	272.5	271	270	269	268.2	267.7	267.1	266.5	266	265.5	265.3	265.1	264.9	265	265	265.5	266	266.5	267.5
55	277	275.5	274.5	273.5	272.7	272.1	271	270.2	269.5	269	268.5	268.3	268.1	267.9	268	268	268.5	269	270	271
56	280	278.5	277	276.2	275.5	274.8	274	273.2	272.7	272.1	271.6	271.2	271	270.9	271	271	271.5	272	273	274
57	283	281.5	280	279	278.3	277.7	277	276.2	275.8	275.4	275	274.4	274.2	274.1	274	274	274.5	275	276	277.2
58	286	284.5	283.5	282.5	281.7	280.8	280	279.2	278.7	278.3	278	277.7	277.4	277.1	277	277	277.5	278	279	280.5
59	289	287.5	286.5	285.5	284.7	284	283.2	282.7	282	281.5	281	280.7	280.4	280.1	280	280	280.5	281.2	282.2	283.5
60	293	291.5	290	289	288	287	286.2	285.7	285	284.5	284	283.8	283.5	283.2	283	283	283.5	284	285.2	286.5
61	296	295	293.5	292.5	291.5	290.5	289.7	289	288.5	288	287.5	287	286.8	286.6	286.4	286.5	287	288	289	290.5
62	300	298.5	297	296	295	294	293.2	292.7	292	291.5	291	290.8	290.5	290.2	290	290	291	292	293	294
63	303	302	301	300	299	298	297	296.5	296	295.5	295	294.7	294.4	294.2	294	294	294.7	295.5	296.5	297.7
64	307	305.5	304.5	303.5	302.5	301.5	300.7	300	299	298.5	298	297.8	297.5	297.2	297.2	297.5	298	299	300	301.5
65	311	310	309	307.8	306.5	305.5	304.7	304	303.2	302.5	302	301.8	301.5	301.2	301.2	301.5	302	303	304	305
66	315	314	313	312	311	310	309	308	307	306.5	306	305.8	305.5	305.2	305.2	305.5	306	307	308	309
67	319	318	317	316	315	314	313	312	311	310.5	310	309.8	309.5	309.5	309.8	310	310.5	311	312	313.5
68	323	322	320.8	319.5	318.5	317.5	316.7	316	315.2	314.5	314	313.8	313.5	313.5	313.8	314	314.5	315	316	317.5
69	327	326	325	324	323	322	321	320	319	318.5	318	317.8	317.5	317.5	317.8	318	318.4	319	320	321.5
70	331	329.5	328.5	327.7	327	326	325	324	323	322.5	322	321.8	321.5	321.3	321.7	322	322.8	323.5	324.5	325.8
71	335	334	332.8	331.5	330.5	329.5	328.7	328	327.3	326.7	326.2	326	325.8	325.8	326	326.2	327	328	329	330
72	339	338.5	337.2	336	335	334	333	332	331.7	331.3	331	330.8	330.4	330.6	330.9	331.2	331.5	332	333.5	335
73	344	343	342	341	340	339	338	337	336.3	335.7	335	334.9	334.6	334.9	335	335.2	336	336.8	338	340
74	349	347.5	346.5	345.5	344.5	343.5	342.5	341.5	341	340.5	340	339.8	339.6	340	340.2	340.5	341	342	343.4	345
75	354	352	351	350	349	348	347	346	345.7	345.3	345	344.8	344.8	345	345.2	345.5	346	347	348.4	350
76	358	357	356	355	354	353	352	351	350.7	350.3	350	349.8	349.8	350	350.2	350.5	351	352.2	353.5	355
77	363	362.5	361	360	359	358	357	356.3	355.8	355.4	355	354.8	355	355.2	355.5	356	356.5	357.5	359	361
78	369	368	366.8	365.5	364.2	363.3	362.5	362	361.4	361	360.7	360.5	360.3	360.7	361	361.5	362.2	363.5	365	367
79	374	373	372	371	370	369	368	367	366.7	366.3	366	365.9	366	366.2	366.5	367	368	369.5	371	373
80	380	378.5	377	376	375	374	373.5	373	372.5	372.2	372	371.9	372	372.2	372.5	373	374	376	378	380

GRUTS TABLE (CONT.)

80	79	78	77	76	75	74	73	72	71	70	69	68	67	66	65	64	63	62	61	60	TEMP. IN °C.
214	214.5	215	215.7	216.5	217.3	218.1	218.9	219.7	220.3	221	221.7	222.5	223.5	224.5	225.5	226.5	227.5	228.5	229.7	231	30
215.5	216	216.7	217.5	218.5	219.3	220.1	220.9	221.7	222.5	223.5	224.2	225	226	227.2	228.5	229.5	230.5	231.7	233.2	235	31
217	217.7	218.5	219.2	220	221	222	223	224	225	226	227	228	229	230.2	231.2	232.5	233.7	235	237	239	32
219.5	220	220.5	221	221.7	222.5	223.5	224.7	226	227.2	228.5	229.7	231	232.2	233.5	234.8	236.2	237.7	239.2	241	243	33
221	221.5	222	222.7	223.5	224.5	225.5	226.8	228.1	229.5	230.9	232.2	233.6	235	236.5	238	239.6	241.3	243	244.7	246.5	34
222.5	223	223.5	224.2	225.2	226.3	227.5	229	230.5	232	233.6	235	236.6	238.2	239.8	241.4	243	244.7	246.4	248.2	250	35
224	224.7	225.5	226.5	227.5	228.5	230	231.5	233	234.7	236.5	238.2	240	241.7	243.3	245	246.7	248.5	250.2	252	254	36
226	226.7	227.5	228.5	229.7	231	232.5	234	235.5	237	239	241	242.7	244.5	246.2	248	250	252	254	256	258	37
228	229	230	231	232	233.2	234.7	236.5	238.2	240	242	244	246	248	249.8	251.8	253.7	255.7	257.8	260	262	38
230	230.7	231.5	232.7	234	235.5	237	239	241	243	245	247	249	251	253	255	257	259	261.2	263.5	266	39
232	233	234	235.2	236.5	238.2	240	242	244	246	248	250	252	254	256	258	260.5	262.8	265.2	267.5	270	40
234	235	236	237.1	238.5	240	242	244	246	248	250.2	252.5	254.9	257.2	259.5	262	264.2	266.5	269	271.5	274	41
236.5	237.2	238.2	239.5	241	242.5	244.5	246.5	248.5	250.7	253	255.5	258	260.5	263	265.5	268	270.5	273	275.7	278.5	42
238.5	239.5	240.5	242	243.5	245	247	249	251	253.5	256	258.5	261	263.7	266.4	269	271.7	274.5	277.2	280	283	43
241	242	243	244.5	246.3	248	250	252	254	256.5	259	261.5	264	266.5	269	272	275	278	281	284.2	287.5	44
244	245	246	247.5	249	250.5	252.5	254.7	257	259.5	262	264.5	267	270	273	276	279	282	285	288.5	292	45
246.5	247.5	249	250.5	252	253.5	255.5	257.7	260	262.2	265	267.5	270	273	276	279.5	282.5	286	289.5	293	296.5	46
249	250	251.5	253	254.5	256.3	258	260.5	263	265	267.7	270.5	273.5	276.5	279.5	283	286.5	290	293.7	297.5	301	47
252	253	254.5	256	257.5	259	261	263.5	266	268	271	274	277	280	283.5	287	290.5	294	297.5	301.2	305	48
254.7	256	257.2	258.5	260	262	264	266.5	269	271.5	274.5	277.5	280.5	284	287.5	291	294.5	298	302	306	309.5	49
258	259.2	260.4	261.7	263.2	265	267.5	270	272.5	275	278	281.3	284.5	288	291.5	295	298.5	302.2	306	310	314	50
259.8	261	262.5	264	266	268	270.5	273	275.5	278.5	281.5	284.5	288	291.5	295	299	303	307	311	315	319	51
262.7	264	265.5	267	269	271.2	273.5	276.2	279	282	285	288.6	292	295.5	299	303	307	311	315	319.5	324	52
265.7	267	268.5	270	272	274.5	277	279.5	282.5	285.5	288.5	292	295.5	299	303	307.5	311.5	315.5	320	324.5	329	53
269	270.5	272	273.5	275.5	278	280.5	283	286	289	292.5	296	299.5	303.5	307.5	311.8	316	320.5	325	329.5	334	54
272	273.5	275	277	279	281	283.5	286.5	289.5	293	296.5	300	304	308	312	316	320.5	325	330	334.5	339	55
275.5	277	278.5	280	282	284	287	290	293	296.5	300	304	308	312	316.2	320.5	325	330	335	340	345	56
278.5	280	281.5	283	285	287.5	290	293	296.5	300	304	308	312	316.5	321	325.5	330.5	335.5	340.7	346	351	57
282	283.5	285	287	289	291	294	297	300	304	308	312	316.5	321	325.5	330.5	335.5	340.7	346	352	357	58
285	286.5	288	290	292	294	297	300	304	308	312	316	321	325.5	330.5	335.5	340.7	346	352	358	364	59
288	289.7	291.5	293.5	295.7	298	301	304	308	312	316	320.5	325	330	335	340	345.5	351	357	363	370	60
292	293.5	295	297	299	301.5	304	307.5	311.5	315.5	320	325	330	335	340	345	351	357.5	364	370.5	377	61
295.5	297	299	301	303	305	308	311.5	315	319.5	324	328.5	333.5	338.5	344	350	356.5	363	370	377.5	385	62
299	300.5	302	304	306	309	312	315.5	319	323.5	328	333	338	343.5	348.5	356	363	370	377.5	385	393	63
303	304.5	306	308	310	313	316	319	323	327.5	332.5	337.5	342.5	348.5	355	361.5	369	376.5	384.5	392.5	400.5	64
306.5	308	310	312	314.5	317	320	323.5	327.5	332	337	342	347	353	360	367	375	383	391	400	409	65
310.5	312.2	314	316.5	319	321.5	324.5	328	332	336.5	341.5	346.5	352	358.5	365.5	373	381	390	399	408	418	66
315	317	319	321	323	326	329	332	336.5	341	346	351.5	357.5	363.5	371	379	388	397	407	417	427	67
319	321	323	325	327.5	330	333.5	337	341	346	351	357	363	370	378	386	395	405	416	426	437	68
323	324.5	327	329	332	335	338.5	342	346	350.5	356	362	368	376	384	393	403	413	424	435	447	69
327	329	331	333.8	337	340	343	347	351.5	357	362	368	375	382	391	400	410	421	432	444	457	70
332	334	336.5	339.5	343	346.2	350	354	358	363	368.5	375	382	390	399	409	420	431	443.5	456	469	71
337	339	342	345	348.5	352	356	360	364	369	376	382	389.5	398	408	418	429	441	454	468	481	72
342	344.5	347.5	351	354	358	362	366.5	371.5	377	383	390	398.5	407.5	418	429	441	454	468	481		73
347	350	353	357	361	365	369	374	379	385	392	399.5	409	419	430	443	457	471	487			74
352	355	358.5	362.2	366.5	371	376	381	387	393	400	409	419	431	444	458	472	488				75
357.5	361	364.5	368.5	373	378	383	388.5	394.5	401	409	419	431	444	458	473	490					76
363.5	367	371	375	380	385	391	397	403	411	419	430										77
370	373	377	382	387	392	398	405	412	420	429	440										78
376	380	384	389	394	400	406.5	413	421	429	439											79
383	387	392	397	402	408	415	422	430	439	450											80

APPENDIX F

S.I. SYSTEM OF UNITS

The International System of Units (S.I.) which is a modern version of the MKSA (meter, kilogram, second, ampere) system is published and controlled by the *General Conference on Weights and Measures,* and other associated international organizations.

The data in this Appendix are based on information approved by the above, and the U.S. Bureau of Standards, which form the "I.E.E.E. Standard Metric Practice," sponsored by The Institute of Electrical and Electronics Engineers, Inc., second printing, January, 1980, and published with their permission.

There are seven base S.I. units: length, the meter; mass, the kilogram; time, the second; electric current, the ampere; thermodynamic temperature, the kelvin; amount of substance, the mole; and luminous intensity, the candela. In addition there are two supplementary units which may be approved as base units: the plane angle, radian, and the solid angle, the steradian. There are 59 units, formed by combining base units with supplementary or derived units. They are distinguished also for use with S.I. in special fields, 5 units, and 6 units for temporary use with S.I. units.

In the Table, "Classified List of Units," conversion data are given using three vertical columns: "To convert from," "To," and "Multiply by." In the last column, the conversion factors are each followed by the letter E (for exponent), a plus or minus sign and two digits which indicate the power of 10 by which the number must be multiplied to obtain the correct value. For example, 3.523 907 E-02 is $3.523\ 907 \times 10^{-2}$, or 0.035 239 07. Similarly, 3.386 389 E+03 is $3.386\ 389 \times 10^{3}$, or 3 386.389. An asterisk (*) after the sixth decimal place indicates that the conversion factor is exact, and that all subsequent digits are zeroes. Note that $E+00 = 10^{0} = 1$, and $E+01 = 10^{01} = 10^{1} = 10$.

The individual conversions are grouped under general fields, such as "ACCELERATION" and the S.I. units in that field are described by, for example, "meters per second^2; m/s^2; S.I. derived unit," or "name; symbol; and type of S.I. unit."

A second table follows, giving S.I. multiples and submultiples of the S.I. units, except for the kilogram.

To convert from	*To*	*Multiply by*
ACCELERATION		
meter per second^2; m/s^2; S.I. derived unit		
ft/s^2	meter per second^2 (m/s^2)	3.048 000*E-01
free fall, standard (g)	meter per second^2 (m/s^2)	9.806 650*E+00
gal	meter per second^2 (m/s^2)	1.000 000*E-02
in/s^2	meter per second^2 (m/s^2)	2.540 000*E-02
radian per square second; rad/s^2; angular acceleration; S.I. derived unit		
ANGLE		
radian; rad; °; In use with S.I.		
degree (angle)	radian (rad)	1.745 329 E-02
grad	degree (angular)	9.000 000*E-01
grad	radian (rad)	1.570 796 E-02
minute (angle)	radian (rad)	2.908 882 E-04
second (angle)	radian (rad)	4 848 137 E-06
steradian; sr; solid angle; S.I. supplementary unit.		

To convert from	*To*	*Multiply by*
AREA		
square meter; m²; S.I. derived unit		
acre	meter² (m²)	4.046 873 E+03
are	meter² (m²)	1.000 000*E+02
barn	meter² (m²)	1.000 000*E−28
circular mil	meter² (m²)	5.067 075 E−10
darcy	meter² (m²)	9.869 233 E−13
ft²	meter² (m²)	9.290 304*E−02
hectare	meter² (m²)	1.000 000*E+04
in²	meter² (m²)	6.451 600*E−04
mi² (international)	meter² (m²)	2.589 988 E+06
mi² (US statute)	meter² (m²)	2.589 998 E+06
yd²	meter² (m²)	8.361 274 E−01
BENDING MOMENT OR TORQUE. MOMENT OF FORCE		
newton meter; N•m; S.I. derived unit		
dyne•cm	newton meter (N•m)	1.000 000*E−07
kgf•m	newton meter (N•m)	9.806 650*E+00
ozf•in	newton meter (N•m)	7.061 552 E−03
lbf•in	newton meter (N•m)	1.129 848 E−01
lbf•ft	newton meter (N•m)	1.355 818 E+00
BENDING MOMENT OR TORQUE PER UNIT LENGTH		
newton meter per meter; N•m/m.		
lbf•ft/in	newton meter per meter (N•m/m)	5.337 866 E+01
lbs•in/in	newton meter per meter (N•m/m)	4.448 222 E+00
CAPACITY (SEE VOLUME)		
DENSITY (SEE MASS PER UNIT VOLUME)		
ELECTRICITY AND MAGNETISM		
ampere; A; electric current. S.I. base unit		
abampere	ampere (A)	1.000 000*E+01
EMU of current	ampere (A)	1.000 000*E+01
ESU of current	ampere (A)	3.335 6 E−10
gilbert	ampere (A	7.957 747 E−01
statampere	ampere (A)	3.335 640 E−10
ampere per meter; A/m; magnetic field strength. S.I. derived unit.		
oersted	ampere per meter	7.957 747 E+01
ampere per square meter; A/m²; current density; S.I. derived unit.		
coulomb; C; A•s; quantity of electricity, electric charge; S.I. derived unit.		
coulomb per cubic meter; C/m³; electric charge density; S.I. derived unit.		
coulomb per square meter; C/m²; electric flux density; S.I. derived unit.		
1abcoulomb	coulomb (C)	1.000 000*E+01
ampere hour	coulomb (C)	3.600 000*E+03
faraday (based on C-12)	coulomb (C)	9.648 70 E+04
faraday (chemical)	coulomb (C)	9.649 57 E+04
faraday (physical)	coulomb (C)	9.652 19 E+04
statcoulomb	coulomb (C)	3.335 640 E−10
farad; F; C/V; electric capacitance; S.I. derived unit.		
abfarad	farad (F)	1.000 000*E+09
EMU of capacitance	farad (F)	1.000 000*E+09
ESU of capacitance	farad (F)	1.112 650 E−12
statfarad	farad (F)	1.112 650 E−12
farad per meter; F/m; permittivity; S.I. derived unit.		
henry; H; Wb/A; inductance; S.I. derived unit.		
abhenry	henry (H)	1.000 000*E−09
EMU of inductance	henry (H)	1.000 000 E−09
ESU of inductance	henry (H)	8.987 554 E+11
stathenry	henry (H)	8.987 554 E+11
hertz; Hz; 1/s; frequency; S.I. derived unit.		
ohm; Ω ; V/A; electric resistance. S.I. derived unit.		
abohm	ohm (Ω)	1.000 000*E−09
EMU of resistance	ohm (Ω)	1.000 000 E−09
ESU of resistance	ohm (Ω)	8.987 554 E+11
statohm	ohm (Ω)	8.987 554 E+11
ohm meter; Ω• m;		

To convert from	*To*	*Multiply by*
ohm centimeter	ohm meter (Ω•m)	1.000 000*E−02
ohm circular mil per foot	ohm meter (Ω•m)	1.662 426 E−09
henry per meter; H/m; permeability (magnetic); S.I. derived unit.		
siemens; S; A/V; Conductance. S.I. derived unit.		
abmho	siemens (S)	1.000 000*E+09
mho	siemens (S)	1.000 000*E+00
statmho	siemens (S)	1.112 650 E−12
tesla; T; Wb/m²; magnetic flux density. S.I. derived unit.		
gamma	tesla (T)	1.000 000*E−09
gauss	tesla (T)	1.000 000*E−04
weber; Wb; V•s; magnetic flux; S.I. derived unit.		
maxwell	weber (Wb)	1.000 000*E−08
unit pole	weber (Wb)	1.256 637 E−07
volt; V; W/A; electric potential, potential difference, electromotive force. S.I. derived unit.		
abvolt	volt (V)	1.000 000*E−08
EMU of electric potential	volt (V)	1.000 000*E−08
ESU of electric potential	volt (V)	2.997 9 E+02
statvolt	volt (V)	2.997 925 E+02
volt per meter; V/m; electric field strength; S.I. derived unit.		
ENERGY (INCLUDES WORK)		
joule, J; N•m; energy, work, quantity of heat. S.I. derived unit.		
British thermal unit (International Table)	joule (J)	1.055 056 E+03
British thermal unit (mean)	joule (J)	1.055 87 E+03
British thermal unit (thermochemical)	joule (J)	1.054 350 E+03
British thermal unit (39 °F)	joule (J)	1.059 67 E+03
British thermal unit (59 °F)	joule (J)	1.054 80 E+03
British thermal unit (60 °F)	joule (J)	1.054 68 E+03
calorie (International Table)	joule (J)	4.186 800*E+00
calorie (mean)	joule (J)	4.190 02 E+00
calorie (thermochemical)	joule (J)	4.184 000*E+00
calorie (15 °C)	joule (J)	4.185 80 E+00
calorie (20 °C)	joule (J)	4.181 90 E+00
calorie (kilogram, International Table)	joule (J)	4.186 800*E+03
calorie (kilogram, mean)	joule (J)	4.190 02 E+03
calorie (kilogram, thermochemical)	joule (J)	4.184 000*E+03
electronvolt	joule (J)	1.602 19 E−19
erg	joule (J)	1.000 000*E−07
ft•lbf	joule (J)	1.355 818 E+00
ft•poundal	joule (J)	4.214 011 E−02
kilocalorie (International Table)	joule (J)	4.186 800*E+03
kilocalorie (mean)	joule (J)	4.190 02 E+03
kilocalorie (thermochemical)	joule (J)	4.184 000*E+03
kW•h	joule (J)	3.600 000*E+06
therm	joule (J)	1.055 056 E+08
ton (nuclear equivalent of TNT)	joule (J)	4.184 E+09
W•h	joule (J)	3.600 000*E+03
W•s	joule (J)	1.000 000*E+00
joule per kelvin; J/K; heat capacity and entropy; S.I. derived unit.		
joule per kilogram; J/kg; specific energy; S.I. derived unit.		
joule per kilogram kelvin; J/(kg•K); specific heat capacity, specific entropy; S.I. derived unit.		
joule per cubic meter; J/m³; energy density; derived S.I. unit.		
ENERGY PER UNIT AREA TIME		
watt per meter²; W/m².		
Btu (thermochemical)/(ft²• s)	watt per meter² (W/m²)	1.134 893 E+04
Btu (thermochemical)/(ft²•min)	watt per meter² (W/m²)	1.891 489 E+02
Btu (thermochemical)/(ft²•h)	watt per meter² (W/m²)	3.152 481 E+00
Btu (thermochemical)/(in²• s)	watt per meter² (W/m²)	1.634 246 E+06
cal (thermochemical)/(cm²•min)	watt per meter²(W/m²)	6.973 333 E+02
erg/(cm²•s)	watt per meter² (W/m²)	1.000 000*E−03
W/cm²	watt per meter² (W/m²)	1.000 000*E+04
W/in²	watt per meter² (W/m²)	1.550 003 E+03
watt per meter kelvin; W/(m•K); thermal conductivity; S.I. derived unit.		

To convert from	*To*	*Multiply by*
FLOW (SEE MASS PER UNIT TIME or VOLUME PER UNIT TIME		
FORCE		
newton; N; kg•m/s². S.I. derived unit.		
dyne	newton (N)	1.000 000*E−05
kilogram-force	newton (N)	9.806 650*E+00
kilopond	newton (N)	9.806 650*E+00
kip (1000 lbf)	newton (N)	4.448 222 E+03
ounce-force	newton (N)	2.780 139 E−01
pound-force (lbf)	newton (N)	4.448 222 E+00
lbf/lb (thrust/weight [mass] ratio)	newton per kilogram (N/kg)	9.806 650 E+00
poundal	newton (N)	1.382 550 E−01
ton-force (2000 lbf)	newton (N)	8.896 444 E+03
FORCE PER UNIT AREA (SEE PRESSURE)		
FORCE PER UNIT LENGTH		
newton per meter; N/m; surface tension; S.I. derived unit.		
lbf/ft	newton per meter (N/m)	1.459 390 E+01
lbf/in	newton per meter (N/m)	1.751 268 E+02
HEAT		
watt; W; J/s; power, radiant flux; S.I. derived unit.		
watt per meter kelvin; W/(m•K); thermal conductivity; S.I. derived unit.		
Btu (International Table) • ft/(h•ft²•°F) (*k*, thermal conductivity)	watt per meter kelvin [W/(m•K)]	1.730 735 E+00
Btu (thermochemical) • ft/(h•ft²•°F) (*k*, thermal conductivity)	watt per meter kelvin [W/(m•K)]	1.729 577 E+00
Btu (International Table) • in/(h•ft²•°F) (*k*, thermal conductivity)	watt per meter kelvin [W/(m•K)]	1.442 279 E−01
Btu (thermochemical) • in/(h•ft²•°F) (*k*, thermal conductivity)	watt per meter kelvin [W/(m•K)]	1.441 314 E−01
Btu (International Table) • in/(s•ft²•°F) (*k*, thermal conductivity)	watt per meter kelvin [W/(m•K)]	5.192 204 E+02
Btu (thermochemical) • in/(s•ft²•°F) (*k*, thermal conductivity)	watt per meter kelvin [W/(m•K)]	5.188 732 E+02
cal (thermochemical)/(cm•s•°C)	watt per meter kelvin	4.184 000*E+02
watt per steradian; W/sr; radiant intensity; S.I. derived unit.		
joule per meter²; J/m².		
Btu (International table)/ft²	joule per meter² (J/m²)	1.135 653 E+04
Btu (thermochemical)/ft²	joule per meter² (J/m²)	1.134 893 E+04
cal (thermochemical)/cm²	joule per meter² (J/m²)	4.184 000*E+04
watt per meter²kelvin; W(m²•K)		
Btu (International Table)/(h•ft²•°F) (*C*, thermal conductance)	watt per meter² kelvin [W/(m²•K)]	5.678 263 E+00
Btu (thermochemical)/(h•ft²•°F (*C*, thermal conductance)	watt per meter² kelvin [W/(m²•K)]	5.674 466 E+00
Btu (International Tale)/(s•ft²•°F)	watt per meter² kelvin [W/(m²•K)]	2.044 175 E+04
Btu (thermochemical)/(s•ft²•°F)	watt per meter² kelvin [W/(m²•K)]	2.042 808 E+04
joule per kilogram; J/kg; heat capacity, specific energy; S.I. derived unit.		
Btu (International Table)/lb	joule per kilogram (J/kg)	2.326 000*E+03
Btu (thermochemical)/lb	joule per kilogram (J/kg)	2.324 444 E+03
cal (International Table)/g	joule per kilogram (J/kg)	4.186 800*E+03
cal (thermochemical)/g	joule per kilogram (J/kg)	4.184 000*E+03
joule per kilogram kelvin; specific heat capacity; J/kg•K)		

To convert from	*To*	*Multiply by*
Btu (International Table)/(lb. °F)		
(*c*, heat capacity)	joule per kilogram kelvin (J/kg•K)	4.186 800*E+03
cal (Int. Table)/g •°C)	joule per kilogram kelvin (J/kg•K)	4.186 800*E+03
cal (thermochemical)/g •°C)	joule per kilogram kelvin (J/kg•K)	5.184 000*E+03
cal (thermochemical)/cm²•min)	watt per meter² w/m²	6.973 333 E+02
cal (thermochemical)/(cm²•s)	watt per meter² w/m²	4.184 000*E+04
joule per meter³; J/m³.		
Btu (Int. Table)/ft³	joule/meter³ (J/m³)	3.725 895 E+04
Btu (thermochemical)/ft³	joule/meter³ (J/m³)	3.723 402 E+04
joule per mole; J/mol; molar energy; S.I. derived unit.		
joule per mole kelvin; J/(mol•K); molar entropy; molar heat capacity; S.I. derived unit.		
joule per kelvin; J/K; entropy and heat capacity; S.I. derived unit.		
watt; W.		
cal (thermochemical)/min	watt	6.973 333 E-02
cal (thermochemical)/s	watt	4.184 000*E+00
watt per square meter radian; W•m⁻²sr⁻¹; radiance; S.I. derived unit.		
watt per meter²; W/m²; heat flux density, irradiance; S.I. derived unit.		
kelvin per watt; K•m/W.		
°F•h/ft²/[Btu (Int. Table)•in]		
(thermal resistivity)	kelvin meter per watt	6.933 471 E+00
°F•h•ft²[Btu (thermochemical)•in]		
(thermal resistivity)	kelvin meter per watt	6.938 113 E+00
kelvin meter² per watt; K•m²/W		
clo	kelvin meter² per watt	2.003 712 E-01
°F•h•ft²/Btu (Int. Table)		
(*R*, thermal resistance)	kelvin meter² per watt	1.761 102 E-01
°F•h•ft²/Btu (thermochemical)		
(*R*, thermal resistance)	kelvin meter² per watt	1.762 280 E-01

LENGTH

meter; m; length; S.I. basic unit.

angstrom	meter (m)	1.000 000*E-10
astronomical unit	meter (m)	1.495 979 E+11
chain	meter (m)	2.011 684 E+01
fathom	meter (m)	1.828 804 E+00
fermi (femtometer)	meter (m)	1.000 000*E-15
foot	meter (m)	3.048 000*E-01
foot (US survey)	meter (m)	3.048 006 E-01
inch	meter (m)	2.540 000*E-02
light year	meter (m)	9.460 55 E+15
microinch	meter (m)	2.540 000*E-08
micron	meter (m)	1.000 000*E-06
mil	meter (m)	2.540 000*E-05
mile (international nautical)	meter (m)	1.852 000*E+03
mile (US nautical)	meter (m)	1.852 000*E+03
mile (international)	meter (m)	1.609 344*E+03
mile (US statute)	meter (m)	1.609 347 E+03
parsec	meter (m)	3.085 678 E+16
pica (printer's)	meter (m)	4.217 518 E-03
point (printer's)	meter (m)	3.514 598*E-04
rod	meter (m)	5.029 210 E+00
yard	meter (m)	9.144 000*E-01

LIGHT

candela; cd; luminous intensity; S.I. basic unit.

candela per square meter; cd/m²; luminance; S.I. derived unit.

cd/in²	candela per meter² (cd/m²)	1.550 003 E+03
footlambert	candela per meter² (cd/m²)	3.426 259 E+00

To convert from	To	Multiply by
lambert	candela per meter² (cd/m²)	3.183 099 E+03
stilb	candela per meter² (cd/m²)	1.000 000*E+04
footcandle	lux (lx)	1.076 391 E+01

*lumen; lm; luminous flux; cd*sr; S.I. derived unit.*
lumen per square meter; lm/m²; derived S.I. unit.

lux; lx; lm/m²; illuminance; S.I. derived unit.

phot	lumen per meter²	1.000 000*E+04

MASS
kilogram; kg. S.I. basic unit.

carat (metric)	kilogram (kg)	2.000 000*E−04
grain	kilogram (kg)	6.479 891*E−05
gram	kilogram (kg)	1.000 000*E−03
hundredweight (long)	kilogram (kg)	5.080 235 E+01
hundredweight (short)	kilogram (kg)	4.535 924 E+01
kgf s²/m (mass)	kilogram (kg)	9.806 650*E+00
ounce (avoirdupois)	kilogram (kg)	2.834 952 E−02
ounce (troy or apothecary)	kilogram (kg)	3.110 348 E−02
pennyweight	kilogram (kg)	1.555 174 E−03
pound (avoirdupois)	kilogram (kg)	4.535 924 E−01
pound (troy or apothecary)	kilogram (kg)	3.732 417 E−01
slug	kilogram (kg)	1.459 390 E+01
ton (assay)	kilogram (kg)	2.916 667 E−02
ton (long, 2240 lb)	kilogram (kg)	1.016 047 E+03

MASS PER UNIT AREA
kilogram per meter²; kg/m.²

oz/ft²	kilogram per meter² (kg/m²)	3.051 517 E−01
oz/yd²	kilogram per meter² (kg/m²)	3.390 575 E−02
lb/ft²	kilogram per meter² (kg/m²)	4.882 428 E+00

MASS PER UNIT CAPACITY (See MASS PER UNIT VOLUME)

MASS PER UNIT LENGTH
Kilogram per meter; kg/m.

denier	kilogram per meter (kg/m)	1.111 111 E−07
lb/ft	kilogram per meter (kg/m)	1.488 164 E+00
lb/in	kilogram per meter (kg/m)	1.785 797 E+01
tex	kilogram per meter (kg/m)	1.000 000*E−06

MASS PER UNIT TIME (Includes FLOW)
*kilogram per pascal second meter²; (kg/Pa*s*m²)*

perm (0 °C)	kilogram per pascal second meter² [kg/(Pa*s*m²)]	5.721 35 E−11
perm (23 °C)	kilogram per pascal second meter² [kg/(Pa*s*m²)]	5.745 25 E−11

*kilogram per pascal second meter; kg/(Pa*s*m).*

perm*in (0 °C)	kilogram per pascal second meter [kg/(Pa*s*m)]	1.453 22 E−12
perm*in (23 °C)	kilogram per pascal second meter [kg/(Pa*s*m)]	1.459 29 E−12

kilogram per second; kg/s.

lb/h	kilogram per second (kg/s)	1.259 979 E−04
lb/min	kilogram per second (kg/s)	7.559 873 E−03
lb/s	kilogram per second (kg/s)	4.535 924 E−01
ton (short)/h	kilogram per second (kg/s)	2.519 958 E−01

To convert from	*To*	*Multiply by*
kilogram per joule; kg/J.		
lb/hp•h (SFC, specific fuel consumption)	kilogram per joule (kg/J)	1.689 659 E−07

MASS PER UNIT VOLUME (Includes DENSITY and MASS CAPACITY)

kilogram per cubic meter; kg/m³; mass density, density; S.I. derived unit.

kilogram per pascal second square meter; kg/(Pa•s•m²)

grain/gal (US liquid)	kilogram per meter³ (kg/m³)	1.711 806 E−02
g/cm³	kilogram per meter³ (kg/m³)	1.000 000*E+03
oz (avoirdupois)/gal (UK liquid)	kilogram per meter³ (kg/m³)	6.236 021 E+00
oz (avoirdupois)/gal (US liquid)	kilogram per meter³ (kg/m³)	7.489 152 E+00
oz (avoirdupois)/in³	kilogram per meter³ (kg/m³)	1.729 994 E+03
lb/ft³	kilogram per meter³ (kg/m³)	1.601 846 E+01
lb/in³	kilogram per meter³ (kg/m³)	2.767 990 E+04
lb/gal (UK liquid)	kilogram per meter³ (kg/m³)	9.977 633 E+01
lb/gal (US liquid)	kilogram per meter³ (kg/m³)	1.198 264 E+02
lb/yd³	kilogram per meter³ (kg/m³)	5.932 764 E−01
slug/ft³	kilogram per meter³ (kg/m³)	5.153 788 E+02
ton (long)/yd³	kilogram per meter³ (kg/m³)	1.328 939 E+03
ton (short)/yd³	kilogram per meter³ (kg/m³)	1.186 553 E+03

POWER

watt; W; J/s; power, radiant flux; S.I. derived unit.

Btu (International Table)/h	watt (W)	2.930 711 E−01
Btu (International Table)/s	watt (W)	1.055 056 E+03
Btu (thermochemical)/h	watt (W)	2.928 751 E+01
Btu (thermochemical)/min	watt (W)	1.757 250 E+01
Btu (thermochemical)/s	watt (W)	1.054 350 E+03
cal (thermochemical)/min	watt (W)	6.973 333 E−02
cal (thermochemical)/s	watt (W)	4.184 000*E+00
erg/s	watt (W)	1.000 000*E−07
ft•lbf/h	watt (W)	3.766 161 E−04
ft•lbf/min	watt (W)	2.259 697 E−02
ft•lbf/s	watt (W)	1.355 818 E+00
horsepower (550 ft • lbf/s)	watt (W)	7.456 999 E+02
horsepower (boiler)	watt (W)	9.809 50 E+03
horsepower (electric)	watt (W)	7.460 000*E+02
horsepower (metric)	watt (W)	7.354 99 E+02
horsepower (water)	watt (W)	7.460 43 E+02
horsepower (UK)	watt (W)	7.457 0 E+02
kilocalorie (thermochemical)/min	watt (W)	6.973 333 E+01
kilocalorie (thermochemical)/s	watt (W)	4.184 000*E+03
ton (refrigeration)	watt (W)	3.516 800 E+03

watt per square meter; W/m²; power density; S.I. derived unit.
watt per square meter steradian; W/(m²•sr); radiance; S.I. derived unit.
watt per steradian; W/sr; radiant intensity; S.I. derived unit.

PRESSURE or STRESS (FORCE PER UNIT AREA).

pascal; Pa; N/M²; pressure, stress; S.I. derived unit.

atmosphere (standard)	pascal (Pa)	1.013 250*E+05
atmosphere (technical = 1 kgf/cm²)	pascal (Pa)	9.806 650*E+04
bar	pascal (Pa)	1.000 000*E+05
centimeter of mercury (0 °C)	pascal (Pa)	1.333 22 E+03
centimeter of water (4 °C)	pascal (Pa)	9.806 38 E+01
dyne/cm²	pascal (Pa)	1.000 000*E−01
foot of water (39.2 °F)	pascal (Pa)	2.988 98 E+03
gram-force/cm²	pascal (Pa)	9.806 650*E+01
inch of mercury (32 °F)	pascal (Pa)	3.386 38 E+03
inch of mercury (60 °F)	pascal (Pa)	3.376 85 E+03
inch of water (39.2 °F)	pascal (Pa)	2.490 82 E+02
inch of water (60 °F)	pascal (Pa)	2.488 4 E+02

To convert from	*To*	*Multiply by*
kgf/cm²	pascal (Pa)	9.806 650*E+04
kgf/m²	pascal (Pa)	9.806 650*E+00
kgf/mm²	pascal (Pa)	9.806 650*E+06
kip/in² (ksi)	pascal (Pa)	6.894 757 E+06
millibar	pascal (Pa)	1.000 000*E+02
millimeter of mercury (0 °C)	pascal (Pa)	1.333 22 E+02
poundal/ft²	pascal (Pa)	1.488 164 E+00
lbf/ft²	pascal (Pa)	4.788 026 E+01
lbf/in² (psi)	pascal (Pa)	6.894 757 E+03
psi	pascal (Pa)	6.894 757 E+03
torr (mmHg, 0 °C)	pascal (Pa)	1.333 22 E+02

newton meter; N•m; moment of force; S.I. derived unit.

bar; bar = 10^5Pa; S.I. limited use.

RADIOLOGY

curie	becquerel (Bq)	3.700 000*E+10
rad (absorbed dose)	gray (Gy)	1.000 000*E−02
rem (dose equivalent)	sievert (Sv)	1.000 000*E−02
roentgen	coulomb per kilogram (C/kg)	2.58 E−04

gray per second; Gy/s; absorbed dose rate; S.I. derived unit.
1 per second; s^{-1}; activity (radioactive); S.I. derived unit.

SPEED (See VELOCITY)

STRESS (See PRESSURE)

SUBSTANCE, AMOUNT OF

mole; mol; amount of substance; S.I. basic unit.

mole per cubic meter; mol/m^3; concentration (of amount of substance; S.I. derived unit.)

TEMPERATURE

kelvin; K; thermodynamic temperature; Basic S.I. unit.

degree Celsius	kelvin (K)	$T_K = t_{°C} + 273.15$
degree Fahrenheit	kelvin (K)	$T_K = (t_{°F} + 459.67)/1.8$
degree Rankine	kelvin (K)	$T_K = T_{°R}/1.8$

kelvin per watt

kelvin square meter per watt

degree Celsius; °C.

kelvin	degree Celsius	$t_{°C} = T_K - 273.15$
degree Fahrenheit	degree Celsius	$t_{°C} = (t_{°F} - 32)/1.8$

TIME

second; s; S.I. basic unit.

day (mean solar)	second (s)	8.640 000*E+04
day (sidereal)	second (s)	8.616 409 E+04
hour	second (s)	3.600 000*E+03
hour (sidereal)	second (s)	3.590 170 E+03
minute	second (s)	6.000 000*E+01
minute (sidereal)	second (s)	5.983 617 E+01
second (sidereal)	second (s)	9.972 696 E−01
shake	second (s)	1.000 000*E−08
year (365 days)	second (s)	3.163 600*E+07
year (sidereal)	second (s)	3.155 815 E+07
year (tropical)	second (s)	3.155 693 E+07

To convert from	*To*	*Multiply by*

TORQUE (See BENDING MOMENT)

VELOCITY (Includes SPEED)

meter per second; m/s; velocity; S.I. derived unit.

ft/h	meter per second (m/s)	8.466 667 E−05
ft/min	meter per second (m/s)	5.080 000*E−03
ft/s	meter per second (m/s)	3.048 000*E−01
in/s	meter per second (m/s)	2.540 000*E−02
km/h	meter per second (m/s)	2.777 778 E−01
knot (international)	meter per second (m/s)	5.144 444 E−01
mi/h (international)	meter per second (m/s)	4.470 400*E−01
mi/min (international)	meter per second (m/s)	2.682 240*E+01
mi/s (international)	meter per second (m/s)	1.609 344*E+03

radian per second; rad/s; angular velocity; S.I. derived unit.

kilometer per hour; km/h.

mi/h (international)	kilometer per hour (km/h)	1.609 344*E+00

VISCOSITY

pascal second; Pa•s; dynamic viscosity; S.I. derived unit.

centipoise	pascal second (Pa•s)	1.000 000*E−03
poise	pascal second (Pa•s)	1.000 000*E−01
poundal•s/ft²	pascal second (Pa•s)	1.488 164 E+00
lb/ft.h	pascal second (Pa•s)	4.133 789 E−04
lb/ft•s	pascal second (Pa•s)	1.488 164 E+00
lbf•s/ft²	pascal second (Pa•s)	4.788 026 E+01
lbf•s/in²	pascal second (Pa•s)	6.894 757 E+03

meter² per second; m²/s.

centistokes	meter² per second (m²/s)	1.000 000*E−06
ft²/s	meter² per second (m²/s)	9.290 304*E−02
stokes	meter² per second (m²/s)	1.000 000*E−04

1 per pascal second; 1/Pa.s.

rhe	1 per pascal second	1.000 000*E+02

VOLUME (Includes CAPACITY)

meter³; m³; cubic meter; S.I.•derived unit.

acre-foot	meter³ (m³)	1.233 489 E+03
barrel (oil, 42 gal)	meter³ (m³)	1.589 873 E−01
board foot	meter³ (m³)	2.359 737 E−03
bushel (US)	meter³ (m³)	3.523 907 E−02
cup	meter³ (m³)	2.365 882 E−04
fluid ounce (US)	meter³ (m³)	2.957 353 E−05
ft³	meter³ (m³)	2.831 685 E−02
gallon (Canadian liquid)	meter³ (m³)	4.546 090 E−03
gallon (UK liquid)	meter³ (m³)	4.546 092 E−03
gallon (US dry)	meter³ (m³)	4.404 884 E−03
gallon (US liquid)	meter³ (m³)	3.785 412 E−03
gill (UK)	meter³ (m³)	1.420 654 E−04
gill (US)	meter³ (m³)	1.182 941 E−04
in³	meter³ (m³)	1.638 706 E−05
liter	meter³ (m³)	1.000 000*E−03
ounce (UK fluid)	meter³ (m³)	2.841 307 E−05
ounce (US fluid)	meter³ (m³)	2.957 353 E−05
peck (US)	meter³ (m³)	8.809 768 E−03
pint (US dry)	meter³ (m³)	5.506 105 E−04
pint (US liquid)	meter³ (m³)	4.731 765 E−04
quart (US dry)	meter³ (m³)	1.101 221 E−03

To convert from	*To*	*Multiply by*
quart (US liquid)	meter³ (m^3)	9.463 529 E−04
stere	meter³ (m^3)	1.000 000 E+00
tablespoon	meter³ (m^3)	1.478 676 E−05
teaspoon	meter³ (m^3)	4.928 922 E−06
ton (register)	meter³ (m^3)	2.831 685 E−10
yd^3	meter³ (m^3)	7.645 549 E−01

cubic meter per kilogram; m^3/kg; specific volume; S.I. derived unit.

VOLUME PER UNIT TIME (Includes FLOW)

meter³ per joule; m^3/J.

gal (U.S. liquid)/(hp•h) (SFC, specific fuel consumption)	meter³ per *joule*	1.410 089 E−09

meter³ per second; m^3/s.

ft^3/min	meter³ per second (m^3/s)	4.719 474 E−04
ft^3/s	meter³ per second (m^3/s)	2.831 685 E−02
in^3/min	meter³ per second (m^3/s)	2.731 177 E−07
yd^3/min	meter³ per second (m^3/s)	1.274 258 E−02
gal (US liquid)/day	meter³ per second (m^3/s)	4.381 264 E−08
gal (US liquid)/*min*	meter³ per second (m^3/s)	6.309 020 E−05

WORK. (See ENERGY)

S.I. Prefixes

Multiplication Factor	Prefix	Symbol
1 000 000 000 000 000 000 = 10^{18}	exa	E
1 000 000 000 000 000 = 10^{15}	peta	P
1 000 000 000 000 = 10^{12}	tera	T
1 000 000 000 = 10^{9}	giga[a]	G
1 000 000 = 10^{6}	mega	M
1 000 = 10^{3}	kilo	k
100 = 10^{2}	hecto[b]	h
10 = 10^{1}	deca[b]	da
0.1 = 10^{-1}	deci[b]	d
0.01 = 10^{-2}	centi[b]	c
0.001 = 10^{-3}	milli	m
0.000 001 = 10^{-6}	micro	μ
0.000 000 001 = 10^{-9}	*nano*	n
0.000 000 000 001 = 10^{-12}	*pico*	p
0.000 000 000 000 001 = 10^{-15}	femto	f
0.000 000 000 000 000 001 = 10^{-18}	atto	a

[a] Pronounced "jiga."

[b] To be avoided when practical.

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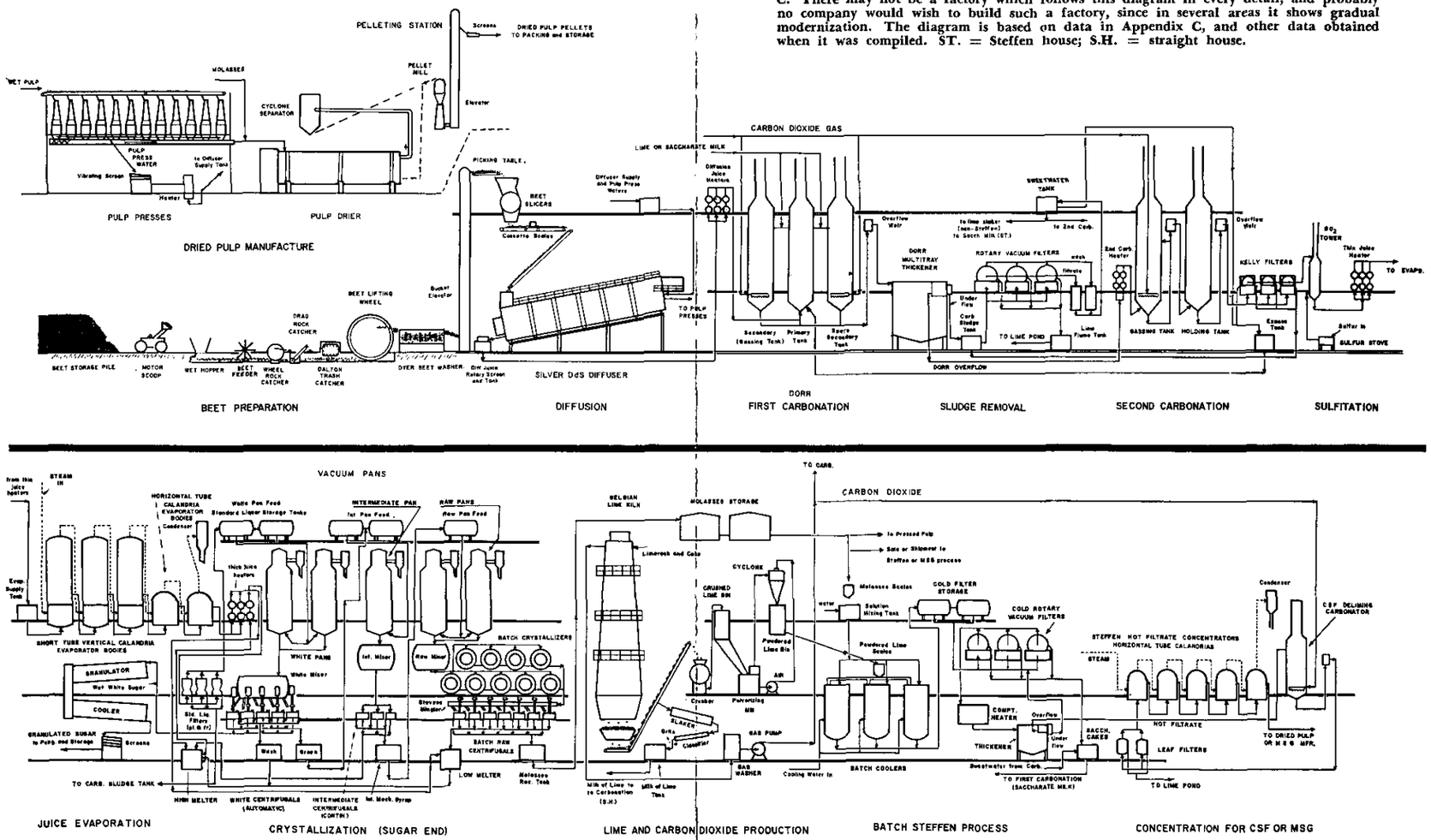

Figure 5-21. Flow diagram of the composite North American beet-sugar factory. It is intended that each unit operation or material flow path be that used by more factories than any other. The only purposeful violation of this principle is the presence of the Steffen process, which most factories do not incorporate. Within the Steffen process, however, the composite concept is followed. The diagram does not necessarily represent majority usage. For example, if 20 factories use machine A, another 20 factories use machine B, and the remaining 23 factories use machine C, the drawing will show machine C. There may not be a factory which follows this diagram in every detail, and probably no company would wish to build such a factory, since in several areas it shows gradual modernization. The diagram is based on data in Appendix C, and other data obtained when it was compiled. ST. = Steffen house; S.H. = straight house.