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NFPA 36

Solvent Extraction Plants

1993 Edition

National Fire Protection Association 1 Batterymarch Park, PO Box 9101, Quincy, MA 02269-9101

NFPA 36

Standard for

Solvent Extraction Plants

1993 Edition

This edition of NFPA 36, *Standard for Solvent Extraction Plants*, was prepared by the Technical Committee on Solvent Extraction Plants, released by the Correlating Committee on Flammable Liquids, and acted on by the National Fire Protection Association, Inc. at its Fall Meeting held November 16-18, 1992, in Dallas, Texas. It was issued by the Standards Council on January 15, 1993, with an effective date of February 12, 1993, and supersedes all previous editions.

The 1993 edition of this document has been approved by the American National Standards Institute.

Changes other than editorial are indicated by a vertical rule in the margin of the pages on which they appear. These lines are included as an aid to the user in identifying changes from the previous edition.

Origin and Development of NFPA 36

This standard was developed at the request of the solvent extraction industry in an effort to achieve greater uniformity in fire protection practices for extraction plants. The purpose of this standard is to provide reasonable standards for the design and operation of solvent extraction processes and extraction plants.

NFPA 36 was tentatively adopted at the 1957 Annual Meeting of the Association. A revision of this tentative edition was adopted at the 1958 Annual Meeting. NFPA 36 was officially adopted by the Association at its 1959 Annual Meeting. Amendments were adopted in 1962, 1964, 1967, 1972, 1973, 1974, 1978, 1983, 1985, and 1988.

This 1993 edition of NFPA 36 incorporates the following amendments to the previous edition:

- A complete rewrite of the Scope of the document to eliminate inconsistencies and potential misapplication of the document to alcohol-based extraction processes.
- Addition of appropriate statements to Chapter 1, General, to address equivalency and retroactivity.
- A complete editorial rewrite of Chapter 1 to comply with NFPA's Manual of Style.
- A complete revision of Subsection 3-1.2, Bulk Solvent Storage Tanks, to address the need for more explicit requirements for aboveground storage tanks.
- An amendment to Paragraph 5-2.7.1 to achieve greater flexibility in siting of cooling towers having fill of limited-combustible materials.
- Amendments to Paragraphs 5-8.2.3 and 5-8.2.4 to clarify the purpose of the vapor seal on the extractor and to recognize that a single seal is adequate in most cases.
- A complete rewrite of Appendix B, General Description of Solvent Extraction Process, to reflect current practices and technology in the industry.
- A complete rewrite of Appendix C, Operational Practices, to reflect current industry practices and certain rules of the U.S. Occupational Safety and Health Administration.

Appendix B General Description of Solvent Extraction Process

This Appendix is not a part of the requirements of this NFPA document, but is included for information purposes only.

General. The removal of vegetable oils from oil-bearing materials by solvent extraction involves almost exclusively the use of solvents. The preparation processes that are employed depend on the oil content of the seed, the physical characteristics of the seed, the type of extraction system used, and the desired end products.

Extraction Solvents and Their Properties. The primary solvent used for the extraction of vegetable oils is the petroleum hydrocarbon fraction sold commercially as "hexane." This is because of hexane's low cost, stability, excellent thermal characteristics, and selectivity for oils and fats. Other solvents, such as pentane, heptane, and trichloroethylene, have been tried, but have not been widely used. Table B-1 shows hexane's physical properties. Table B-2 shows a typical distillation analysis for hexane.

Table B-1 Physical Properties of Hexane

Flammable limits (percent by vol.)	1.2-6.9
Ignition temperature °F	437
Flash point °F closed cup	- 15
Molecular weight	86.2
Melting point	-137°F
Coefficient of expansion	0.00135
Boiling point at 14.7 psia	156.1°F
Specific gravity at 60°F	0.664
A.P.I. gravity at 60°F	81.6
Pounds per gallon at 60°F	5.536
Vapor density (air equals 1)	2.975
Cubic feet vapor per gallon liquid, 60°F, 14.7 psia	25.5
Vapor weight per cu ft (lb at 60°F)	0.217
Vapor weight, cu ft per lb at 60°F	4.61
Latent heat of vaporization at 760 mm Btu/lb	143.3
Heat of combustion, Btu/lb (gross)	20,970
Btu per cu ft vapor (gross)	4,762
Btu per pound (net)	19,420
Vapor pressure at 100°F, psia	5.0
Specific heat liquid at 60°F	0.531
Specific heat vapor at 60°F	0.339
Solubility in water, moles per liter at 60°F	0.0016

Table B-2 Distillation Analysis of Hexane (°F)

Percent Distilled	
Initial Boiling Point	146
5	148
10	148
20	149
30	149
40	149
50	149
60	150
70	150
80	151
90	152
95	153
Dry end point	156

Two of hexane's more important properties are also safety concerns: its flash point and its vapor density. As shown in Table B-1, the flash point is -15°F (-26°C) and the boiling point is 156°F (69°C), making hexane a Class IB Flammable Liquid. The vapors of hexane are about three times more dense than air, which accounts for their tendency to flow across a surface and to collect in low spots and confined areas.

Preparation and Pre-Treatment. Oilseeds that have a high oil content, such as sunflower seed, rapeseed (canola), peanut, and corngerm, are difficult to prepare and do not allow economical direct extraction. Experience has shown that it is less costly to remove some of the oil by first pressing the seeds prior to solvent extraction. This is called "prepressing" and is done in screw presses. The oil from this prepressing is screened to remove fine material called "foots" and is then filtered or centrifuged to produce a clear oil. The foots are returned to the inlet of the screw press. Before pressing, the oilseeds are sometimes cracked, then flaked. Generally, the seeds must be cooked before pressing. This cooking step varies widely, depending on the variety of seed and the type of press. Some screw presses are able to handle whole, uncooked seeds. In any case, the cake produced by the prepressing is the raw material for the solvent extraction process. This cake may be granulated prior to the extraction step.

There are many ways to prepare soybeans for solvent extraction, although certain operations are common to all preparation processes. Soybeans generally bypass the prepressing step. The four major steps in preparing soybeans for solvent extraction invariably follow a sequence of cleaning, cracking, heating, and flaking. The demand for high protein soybean meal for poultry feed has led some extraction plants to add a process to remove the hulls from the cracked beans, using air separation. Sometimes the soybeans are dried to a relatively low moisture level and then dehulled before any heating takes place. Another method processes the beans at their normal moisture content, preheats them quickly in a fluidized bed drier, then cracks and dehulls the beans while they are still hot. The resulting meats are then conditioned in a second fluidized bed drier, prior to flaking.

Some processors, whether they dehull the beans or not, add an additional step that uses an extrusion device called an expander to produce pellets from the flakes. This process is said to improve the extractability of the flakes and to reduce the amount of hexane holdup in the flakes that leave the extractor.

Cottonseed and other soft seeds are prepared both with and without prepressing. Some processors consider the prepressing step to be essential, while others consider it costly and unnecessary. The preparation of cottonseed by the direct extraction method, i.e., without prepressing, consists of cleaning, delinting, cooking, and flaking. The last two steps may be adjusted to optimize the efficiency of the extraction step and to overcome the toxic effects of a pigment gland, called gossypol, that is usually present in the seed. Prepressing of cottonseed produces higher-than-normal temperatures because of the cooking step prior to the prepressing and the frictional heat developed in the press. As with other soft seeds, moisture adjustment and granulation, flaking, or both, of the press cake usually follows.

Extraction. Modern systems for the extraction of vegetable oils and fats by solvents appear rather complex. This complexity, however, is largely due to the systems for control, safety, automation, and energy recovery. The basic extraction process is rather simple, as shown in Figure B-1.

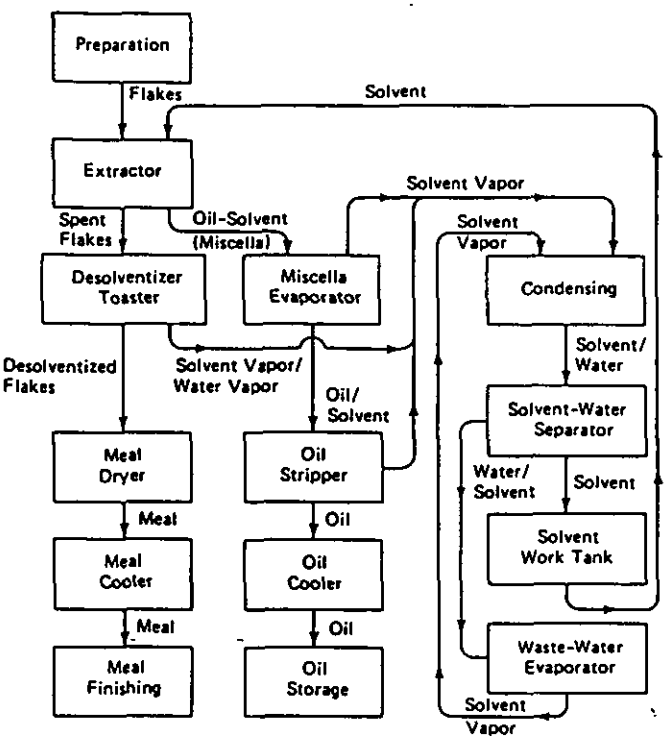

Figure B-1 Flow Diagram of Extraction Process.

Despite the seemingly complicated array of equipment, the process consists of just four major functions: extraction, desolventizing, distillation, and solvent recovery.

In the extraction step, the oil is removed from the oil-bearing material. The extraction of the oil or fat leaves the oil-bearing material saturated with solvent. This solvent is removed in the desolventizer, which drives off the solvent by direct and indirect steam heating. The miscella, or oil-bearing solvent, goes to the evaporation or distillation system, where the solvent is removed from the oil by means of heat, direct steam, and vacuum. The evaporation of the solvent from the miscella is not difficult because the solvent has the relatively low boiling point of about 156°F (69°C), compared to the boiling point of the oils, which can usually withstand temperatures as high as 250°F (121°C) without discoloration or polymerization. Thus, a wide temperature differential between the solvent and the oil, plus the use of stripping steam and vacuum in the final stage, facilitates desolventizing of the oil. The solvent recovered in this process is reused.

The functions of various components of the extraction process are described in the sections that follow.

Basket-Type Extractors. Basket extractors come in three varieties: vertical, rectangular, and horizontal. The material to be extracted is carried through the extractor in individually-suspended, perforated or screen-bottomed baskets. The baskets are hung on longitudinal shafts located just above the center of gravity of the basket. The shaft ends are affixed to bearing brackets that are part of endless chains. The chains are supported and guided by large sprockets at the top and bottom of vertical extractors and at each end of horizontal extractors. This configuration is shown for a vertical extractor in Figure B-2.

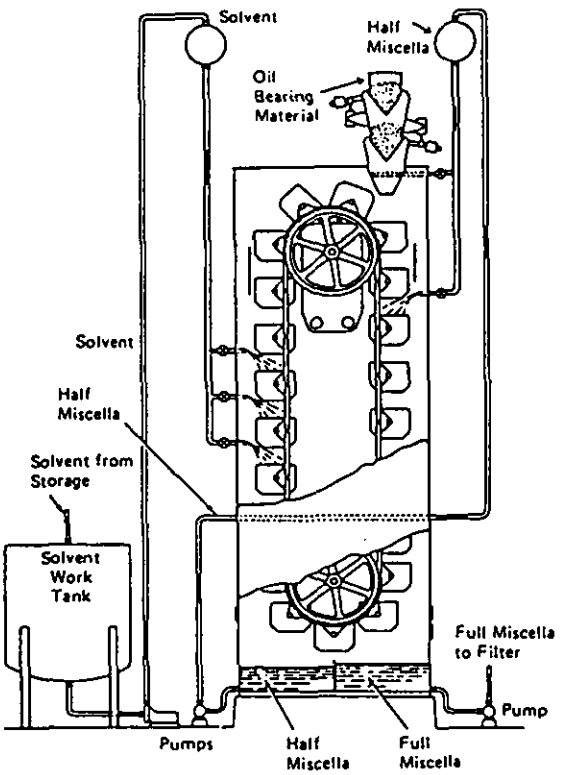

Figure B-2 Vertical Basket Extractor.

The baskets are guided by pins at the end of each basket that slide in a track fastened to the inside of the extractor. The track and pin arrangement prevents the basket from tipping until it reaches a point over a discharge hopper, where a mechanism inverts the basket and discharges the extracted material. As the basket passes the discharge position, it is righted and immediately recharged with oil-bearing material.

The number of baskets in the extractor depends on the through-put, the extraction time, and the design balance: 24 to 36 baskets would be normal. The most common type of charging system is a continuous screw conveyor that feeds a mixture of oil-bearing material and half-miscella to the baskets. The extracted material is removed from the discharge hopper by a paddle conveyor or a mass-flow type of conveyor that is set in the bottom of the hopper.

Vertical and rectangular basket extractors have both ascending and descending baskets traveling through a shower of solvent and miscella. A concurrent phase takes place from the time the baskets are filled until they reach the bottom of the extractor on the downward leg. Half-miscella from the bottom of the ascending side of the extractor is pumped to a basket near the top of the descending side and percolates down through the baskets

to the full-miscella chamber at the bottom. Raw solvent is sprayed onto one or more of the baskets near the top of the ascending side and flows concurrently through the baskets to the half-miscella chamber at the bottom of the ascending side.

Rectangular and horizontal basket-type extractors are quite similar to the vertical type, with the exception that a series of pumps continuously pump miscella to spray pipes above the baskets.

Rotary Extractors. (*See Figure B-3.*) A rotary extractor consists of a series of concentrically arranged cells supported from a vertical shaft. Extraction is effected in a manner similar to horizontal basket extractors, where the baskets pass beneath fixed nozzles fed from stage pumps recirculating various concentrations of miscella, as shown in Figure B-4. A primary difference is that the bed of material is much deeper in the rotary extractor; hence, the term "deep bed extractor" is sometimes applied to this type. Each cell has an open top and a hinged, perforated bottom door. As the cells travel around the track and pass beneath the feeding device, a slurry of oil-bearing material and half-miscella fills each cell. The rotation speed of the cells is variable to ensure that a continuous flow of slurry fills each cell to the desired level. While the cells are completing a revolution of the extractor, increasingly stronger concentrations of miscella, which is collected from the drain compartments that form the bottom of the extractor, are sprayed back onto the top of the cells. At approximately two-thirds of the distance around from the slurry inlet, raw solvent is sprayed onto the top of the cells, and the cells are allowed to drain free of excess solvent. After the drainage section, the cells pass over a discharge hopper. When each cell is directly over the hopper, a cut-out section of the track permits the cell bottom to be released, and the spent material drops into the hopper. Immediately after passing this position, the cell bottom is mechanically raised back to the closed position and is ready to be recharged. The spent material is continuously conveyed from the discharge hopper to the desolventizer at a uniform rate. This rate is regulated so that the discharge hopper is nearly empty when the next cell discharges.

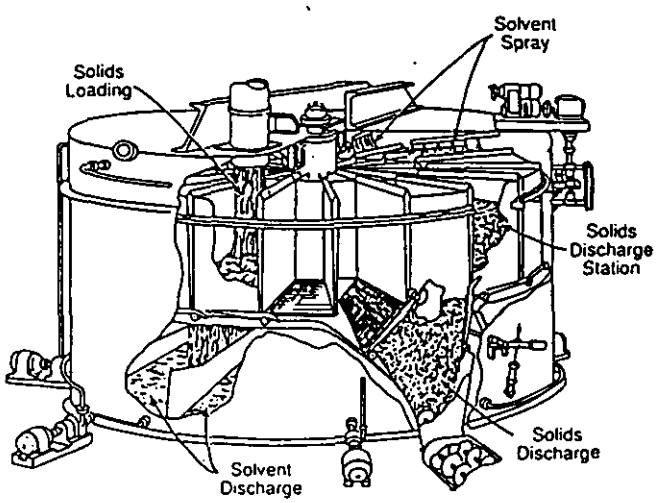

Figure B-3 Rotary or Deep Bed Extractor. (Courtesy *Chemical Engineering*, Vol. 57, No. 8, August, 1950, p. 109.)

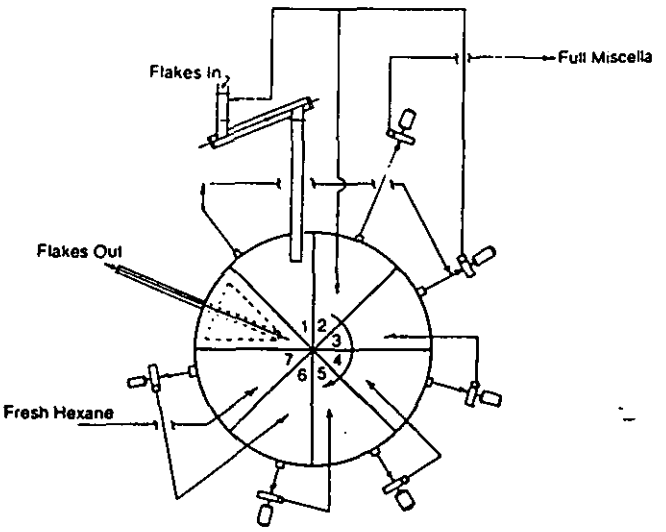

Figure B-4 Schematic of Operation of Rotary Extractor.

Stationary-bottom and sliding-plate extractors (Figure B-5) are variations of the deep bed rotary extractor. These extractors have concentric cells affixed to a central axis that is powered by a variable speed drive. The cells slide over a self-cleaning slotted bottom plate. Because of this feature, no doors are required to support the flakes. The cells are uniformly filled to a desired level. Several stage pumps are provided to recycle the solvent and gradient miscellas countercurrent to the mass as it is extracted. A drainage section is provided after the fresh solvent is added, and the extracted material drops into a discharge hopper, where it is conveyed to the desolventizer.

The stationary cell extractor is another type of rotary extractor. This type provides countercurrent extraction without moving the cells. Instead, the fill spout, spray nozzles, bottom screens, and miscella collection compartments rotate on a central shaft. The thrust load is carried on a circular track mounted on the bottom of the extractor shell. The oil-bearing material remains in the stationary basket until drained. As the bottom screen rotates under the basket, the material discharges into the internal discharge hopper from which it is conveyed from the extractor.

Perforated Belt Extractor. (*See Figure B-6.*) Another type of extractor is the horizontal perforated belt extractor in which the flakes are fed in a uniform depth onto one end of a slowly moving perforated belt. As the oil-bearing material, which is not as deep as in the rotary-type extractor, travels the length of the extractor, gradient miscellas are sprayed over the moving bed from stage pumps, in much the same fashion as the horizontal basket and rotary extractors. The belt is comprised of a pair of endless chains running on a set of large sprockets at each side of the extractor. Attached across the chains and forming a flat surface are a series of perforated plates or screens. Chambers or pans are arranged beneath the belt for the entire length of the extractor for collection of various concentrations of miscellas. The spent material is continuously discharged from the end of the belt into a hopper, from which it is conveyed to the desolventizer.

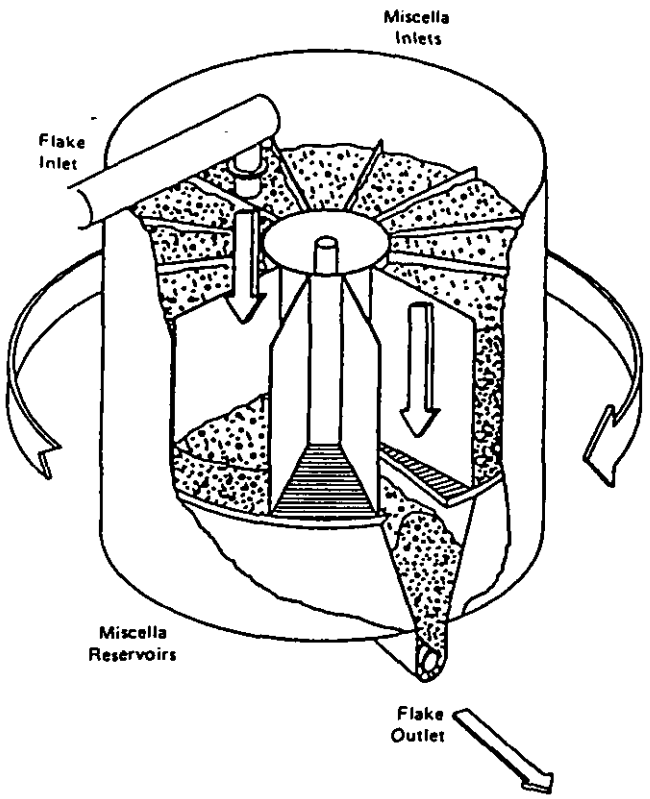

Figure B-5 Stationary-Bottom or Sliding-Plate Extractor.

Another type of perforated belt extractor, the sliding cell extractor (Figure B-7), provides two extraction surfaces where the flakes are separated by rectangular compartments, while traveling along the moving slotted screens or stationary vee-bar screen plates. This unit achieves countercurrent extraction by employing stage pumps and a series of collecting pans to direct the flow of the various miscella concentrations. Because there is an upper and a lower screen belt or plate, the filling device can be located over, but separated from, the discharge hopper. When the partially extracted material reaches the end of the top belt, it falls from the upper cell into a similar compartment on the lower belt. The mass continues to be sprayed with miscella from the stage pumps until it reaches the fresh solvent wash prior to draining and feeding into the discharge hopper.

Rectangular Loop Extractor. (*See Figure B-8.*) The same basic principles are used in the rectangular loop extractor as in the sliding cell extractor. However, the overall shape of this extractor utilizes an "en masse" type conveyor instead of individual baskets. The bottom part of the conveyor uses stationary, linear vee-bar screens. Flakes enter near the top of the extractor at the inlet hopper. A conveyor chain carries the flakes away from this hopper and down the first leg of the loop, where the flakes receive their first wash. In the bottom horizontal run of the loop, the flakes are washed with progressively weaker concentrations of miscellas. As the flakes travel up the vertical part of the loop, they are further extracted by a countercurrent wash of miscella flowing down the loop. On the sloping top run of the extractor, they receive a fresh solvent wash, are allowed to drain, and are discharged continuously from the unit.

The extractor conveying chain is open at the top, bottom, and sides. This allows easy loading and emptying of the material and for passage of the solvent through the flake bed as it is turned during its extraction wash cycle. The speed of the chain is automatically controlled by the level of flakes in the inlet hopper. A sensor in the feed hopper measures the level of flakes in the hopper and sends a signal to the extractor's variable speed drive.

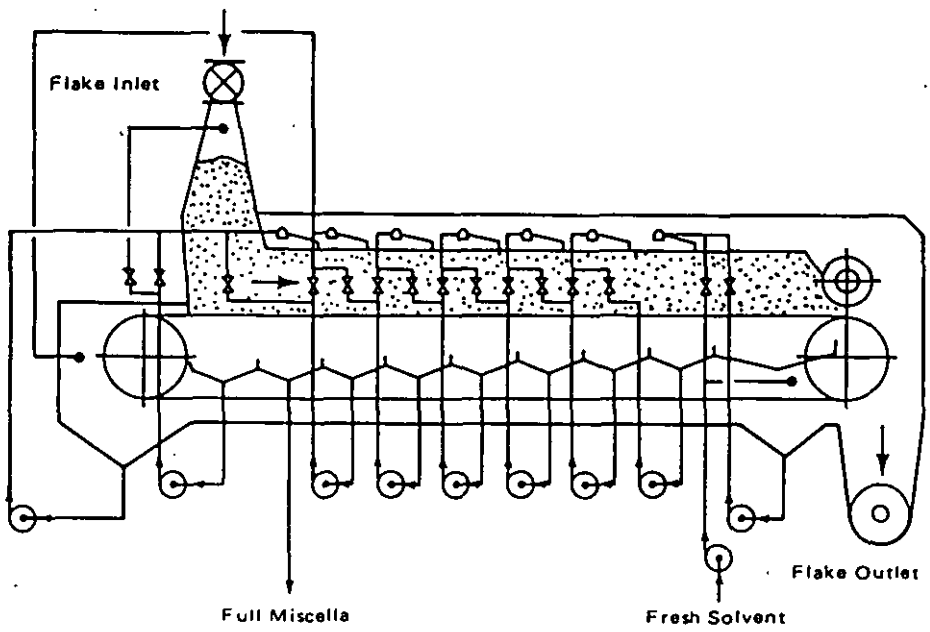

Figure B-6 Perforated Belt Extractor.

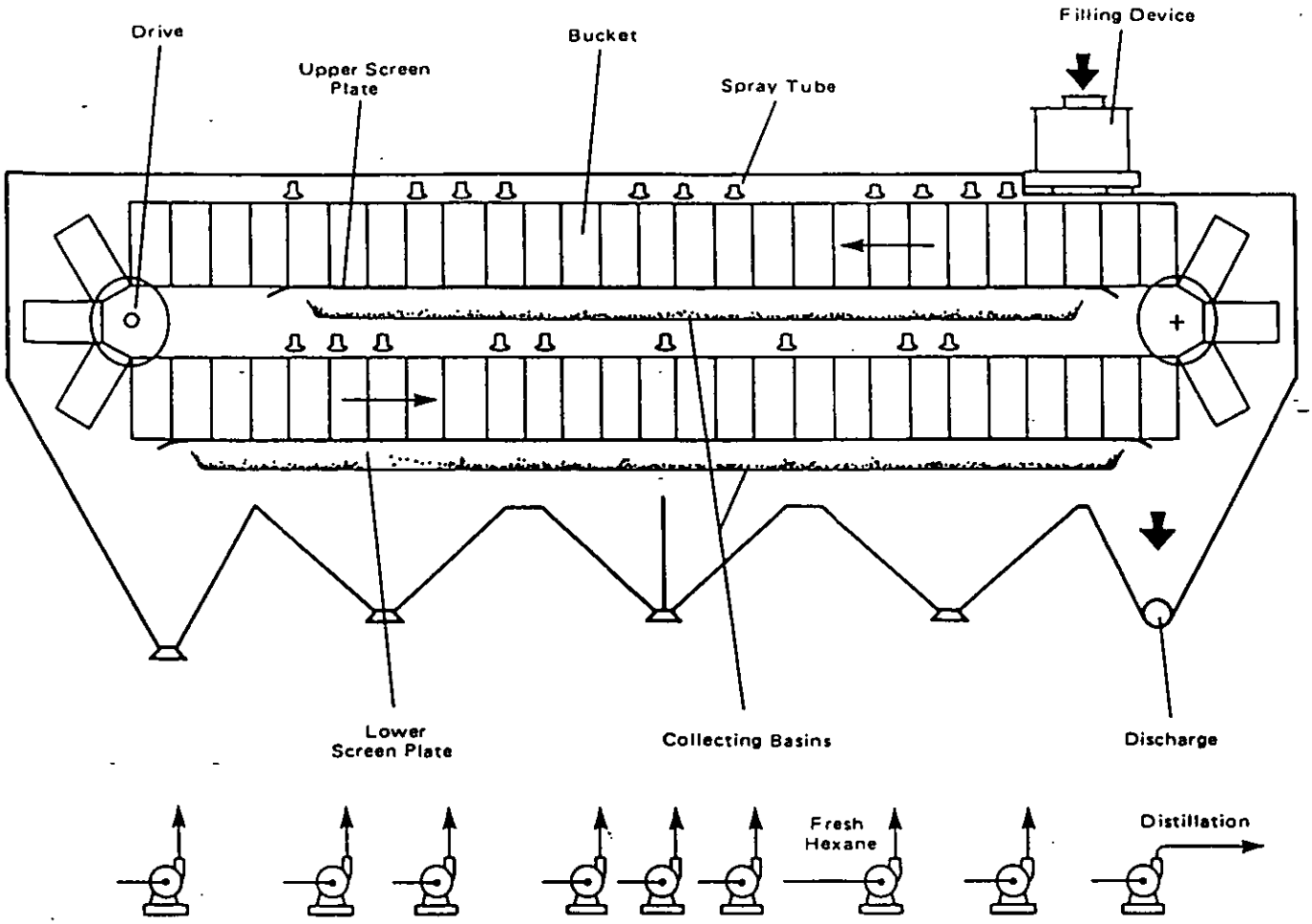

Figure B-7 Sliding Cell Extractor.

Other Types of Extractors. There are other types of extractors for extracting oil from oil-bearing materials, but they are not in general use. These other types include vertical immersion, filtration, and batch extractors. Some are used only for special applications.

Distillation. The distillation system of an extraction plant provides the means for evaporating and stripping the solvent from the oil. There are numerous methods for accomplishing this, from the early pot-type batch stills and pan-type evaporators to the currently popular long-tube rising film evaporators, followed by high vacuum stripping. Few of the pot stills or pan-type evaporators are still in use.

At this time, nearly all extraction plants use two long-tube rising film evaporators, with or without recirculation. The first evaporator usually operates under vacuum, while the second operates either under vacuum or at atmospheric pressure. Heat from the desolventizer vapor is recovered and used to supply heat to the first evaporator, while indirect steam supplies heat for the second evaporator. The choice of the type of oil stripper used depends on a number of factors, including the design and size of the other components in the system and the material being processed.

Stripping columns commonly used are either the packed column type or the disk-and-doughnut type. The stripping column distributes the oil into a very thin film on a large surface area with a relatively high velocity of dry steam passing over and through the film. A countercurrent flow is established by introducing the oil at the top of the column and allowing it to pass downward through the tower against the flow of the steam, which is introduced at the bottom of the tower. The tower must be operated under a vacuum of 22 to 28 in Hg (559 to 711 mm Hg) to achieve highest efficiency. The mixture of steam and solvent vapor passes from the top of the tower to a condenser from which the condensate is pumped to a solvent/water separator. The solvent flows from the separator to a work tank. Water flows from the separator to the waste water evaporator. Finished oil is usually pumped from the stripping column by a rotary positive displacement pump.

Desolventizing and Toasting. Desolventizing of the spent material is accomplished in several ways. In one of these, spent material is desolventized by passing through a series of steam-jacketed rotary conveyors commonly called "schneckens." These conveyors are usually stacked one above the other, and the material to be desolventized

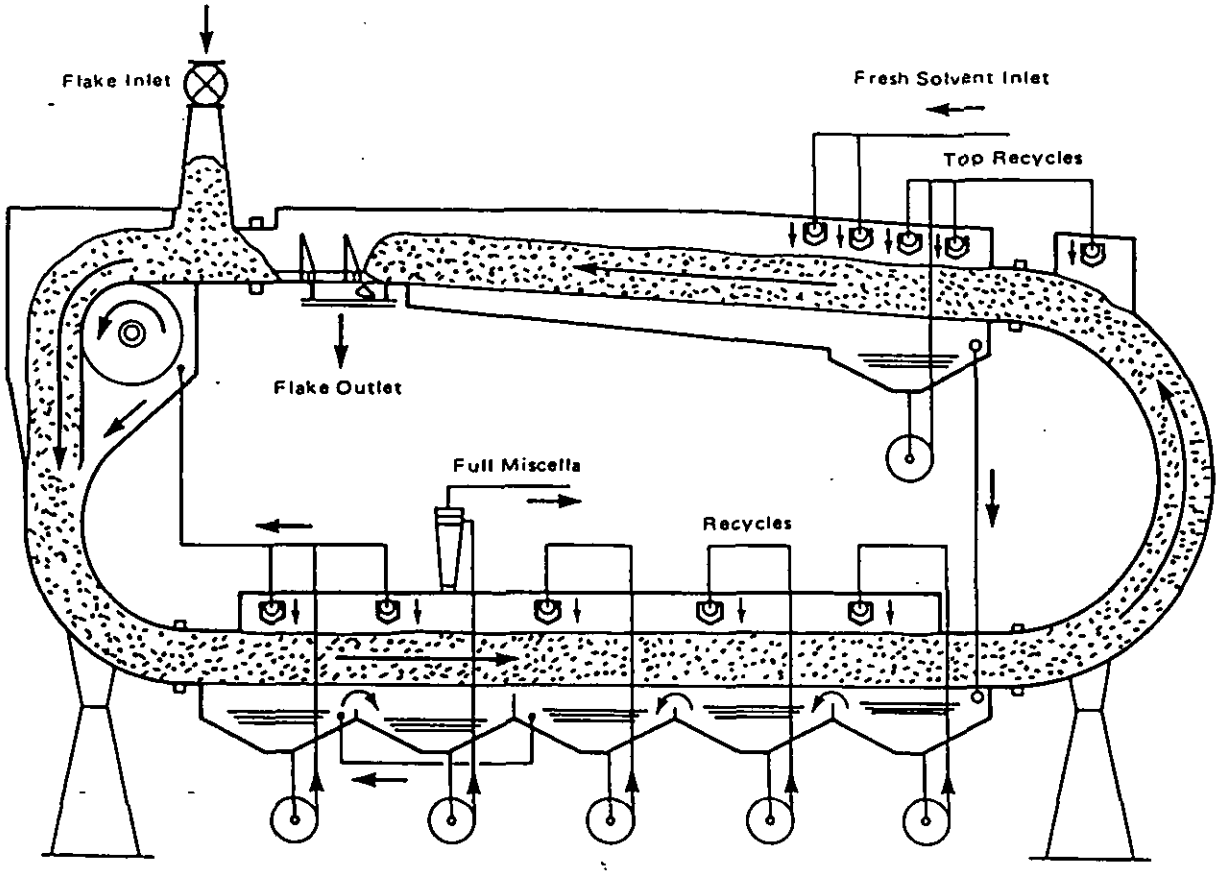

Figure B-8 Rectangular Loop Extractor.

drops by gravity from one to the next. The last conveyor often has a direct flow of steam to remove the final traces of solvent. Vapors are piped to a scrubbing and condensing system. The desolventized material is discharged from the bottom conveyor through a vapor seal.

Another type of desolventizer is the recycled vapor type, which consists of a single cylindrical vessel with a rotating element that tumbles the spent material from the inlet to the discharge end. The vessel is steam-jacketed, and part of the solvent vapor that is driven off is superheated and passed directly back to the vessel. The superheated solvent vapor provides the energy to desolventize the material. This type of desolventizer is usually followed by some type of stripper, often using heat, vacuum, and direct steam to remove the final traces of solvent. The desolventized material is then discharged through a vapor seal.

Many extracted oilseed products require toasting or cooking in order to deactivate enzymes and other constituents and yield a nutritional product. The moisture and heat of the toasting process also agglomerates the fine particles of meal into a more granular form. This helps to reduce the dust problem in subsequent cooling and milling steps. Toasting can be carried out as a separate stage of the process. The meal is conveyed from the desolventizer to the toaster, which may be a vertical stacked cooker or a jacketed stationary drum-type unit with an internal agitator. The toasting section of the combination desolventizer-toaster, often called a "D-T," is essentially a stacked cooker. The function of all toasters is to retain the spent material for a sufficient time at a temperature above 212°F (100°C) and at a specific moisture content. The length of time that the meal is held at these conditions determines the degree of cooking or toasting.

From a fire protection standpoint, the development of the desolventizer-toaster, with the elimination of the intermediate vapor seals, conveyors, etc., represents a significant advance. The D-T is now used in most large capacity solvent extraction systems and accomplishes two important processing steps with a minimum of moving machinery and maximum safety against escape of solvent vapors. The D-T consists of individual kettles or trays placed one above the other, each kettle containing a layer of spent material to a depth of 1 to 4 ft (0.3 to 1.2 m). The feeding of material from one kettle to the next lower one is accomplished by an automatic gate mechanism or spout. The trays that form the floor of each kettle are steam-jacketed and direct steam is sparged into the material in the top kettle or kettles. Some of this steam condenses as it evaporates the solvent, raising the temperature and the moisture of the material to the levels required for toasting in the lower kettles. The top kettle is provided with a large pipe to conduct hot vapors through a scrubber to the condensing system. In normal operation, at least half of the kettles (trays) of the D-T are filled with desolventized material, providing an effective seal against fluctuating pressures and other changes in plant performance.

More recently, the fully counterflow D-T has been introduced. In this unit, the trays have a relatively uniform pattern of perforations that allow the passage of steam through each upper level tray and bed of material. Most or

all of the direct steam is introduced at the bottom of the D-T and flows up through the beds in counterflow to the downward flow of material. This method desolventizes and toasts effectively, reduces the danger of solvent escaping from the bottom of the D-T, and is more energy efficient.

Condensing System. Condensing of solvent vapors and steam is usually accomplished by the use of shell-and-tube condensers. The tubes are normally made of stainless steel. Water flows through the tubes, and the solvent vapor and steam condense on the outer surfaces of the tubes. The cold water used in the condensers may be supplied from deep wells, cooling towers, spray ponds, or some other source that can supply water cool enough to operate the condensers efficiently and clean enough to prevent fouling of the tubes. Solvent vapor from the desolventizer is usually passed through a scrubber to remove any solids entrained in the vapor stream. The vapors are normally washed with liquid solvent, and this scrubbing liquid, along with the removed fines, is returned to the desolventizer. Water has also been used as a scrubbing liquid.

Vent Vapor Recovery System. It is standard practice in extraction plants to vent each piece of processing equipment to a common vent header. This header contains hexane vapor, water vapor, and air, which flow to the final solvent recovery system. At a minimum, the solvent recovery system consists of a water-cooled shell-and-tube condenser and a mineral oil absorption recovery system. The noncondensible components are scrubbed in a mineral oil absorption column and are discharged through a flame arrester to atmosphere by a vent fan. The mineral oil is heated, stripped of its hexane, cooled, and returned to the absorption column. The recovered vapors are condensed and returned to the solvent separator. By today's standards of efficiency, the solvent losses in a large, well-designed, and well-run plant will not exceed one-half gallon (1.9 L) of solvent per ton of material processed. In a soybean processing plant, this is equivalent to a solvent loss of 0.14 percent by weight, based on the weight of the soybeans processed. Many plants have solvent losses that are considerably lower than this.

Waste Water Evaporator. Water is continuously produced in the extraction process from the condensation of direct sparge steam that is used in many sections of the plant. This water is continuously removed from the solvent-water separator and sent to a vessel called the waste water evaporator, where direct steam is introduced to raise the temperature to at least 185°F (85°C). Often, the direct steam is provided by the final stripper vacuum ejector (and possibly other ejectors in the process). The purpose of this waste water evaporator is to heat the waste water well above the boiling point of the solvent, thus evaporating any remaining solvent in the waste water stream. This waste water flows to a large outdoor separation sump that is also connected to the floor drains of the extraction building. This sump serves two purposes: it provides an additional level of safety by separating any remaining oils, solvents, and miscella from the waste water prior to its discharge; it also provides containment for any spills.