SOLVENT DEGREASING
AP-42 Section 4.6
Reference Number
4

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Contract No. 68-02-1329
Task Order No. 9

Appendix Reports

June 30, 1976

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APPENDIX - A

Study to Support New Source Performance Standards for Solvent Metal Cleaning Operations

EMISSION SURVEY

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Office of Air Quality Planning
U.S. Environmental Protection Agency

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I

INTRODUCTION

THIS STUDY HAS IDENTIFIED THE USE OF ORGANIC SOLVENTS IN THE METAL WORKING INDUSTRY. THE STUDY EMPHASIZES HOW THE SOLVENTS ARE USED, THE VOLUMES, THE USE OF VAPOR RECOVERY AND CONTROL SYSTEMS, AND THE METHODS OF SOLVENT DISPOSAL.

EACH ASPECT OF THE STUDY HIGHLIGHTS THE RELEVANCY OF THE TYPE OF INDUSTRY, THE PLANT SIZE, AND THE GEOGRAPHIC LOCATION.

IT IS IMPORTANT TO NOTE THIS IS A SURVEY OF THE METAL WORKING INDUSTRY (SEE SECTION II FOR DEFINITION) AND THE DATA CONTAINED HEREIN PERTAINS ONLY TO THIS SEGMENT OF ALL INDUSTRY.

THE AREAS INCLUDED IN THIS STUDY ARE REPORTED AS FOLLOWS:

1. A DEFINITION OF THE METAL WORKING INDUSTRY WITH REGARD TO ITS SIZE, SCOPE, AND GEOGRAPHIC CONCENTRATION. (SECTION II)

2. THE EXTENT OF METAL CLEANING OUTLINING THE SEGMENT OF THE METAL WORKING INDUSTRY USING A METAL CLEANING SYSTEM WITH EMPHASIS ON THE PORTION OF THAT SEGMENT WHICH USES ORGANIC SOLVENTS. (SECTION III)

3. THE EXTENT OF SOLVENT CLEANING INCLUDING THE PROPORTION OF VAPOR DEGREASING AND ROOM TEMPERATURE CLEANING. (SECTION IV)

4. THE EXTENT OF VAPOR DEGREASING REPORTING THE NUMBER OF UNITS IN USE AND TYPES OF UNITS. (SECTION V)

5. THE QUANTITY OF SOLVENTS USED FOR CLEANING IN THE METAL WORKING INDUSTRY. (SECTION VI)

6. THE USE OF VAPOR RECOVERY AND CONTROL SYSTEMS INCLUDING THE TYPES OF SYSTEMS AND THE EXTENT OF THEIR USE. (SECTION VII)

7. THE METHODS USED FOR SOLVENT DISPOSAL AND THE QUANTITIES OF SOLVENTS DISPOSED OF BY THESE VARIOUS ROUTES. (SECTION VIII)

8. THE PROCEDURES USED IN OBTAINING THE DATA PRESENTED IN THIS STUDY ARE REPORTED IN SECTION IX.

II

THE METAL WORKING INDUSTRY

ORGANIC SOLVENTS ARE USED IN A WIDE VARIETY OF WAYS. SINCE OUR CONCERN IS WITH THE USE OF SOLVENTS IN INDUSTRIAL DEGREASING AND CLEANING, A DEFINABLE POPULATION TO SURVEY HAD TO BE SELECTED. THIS POPULATION NOT ONLY HAD TO DEMONSTRATE THE END USE, BUT REPRESENT THE LARGEST SEGMENT OF THE END USE. WITH THE CRITERIA MENTIONED, THE METAL WORKING INDUSTRY WAS SELECTED.

SCOPE OF METAL WORKING

THE UNITED STATES GOVERNMENT CLASSIFIES ALL INDUSTRY BY THE STANDARD INDUSTRIAL CLASSIFICATION (S.I.C.) CODE SYSTEM. IN 1972 THE METAL WORKING INDUSTRY WAS CLASSIFIED INTO THESE EIGHT S.I.C.S:

25	FURNITURE AND FIXTURES
33	PRIMARY METAL INDUSTRIES
34	FABRICATED METAL PRODUCTS
35	MACHINERY EXCEPT ELECTRICAL
36	ELECTRICAL AND ELECTRONIC EQUIPMENT
37	TRANSPORTATION EQUIPMENT
38	INSTRUMENTS AND RELATED PRODUCTS
39	MISCELLANEOUS MANUFACTURING INDUSTRIES

THESE 2-DIGIT S.I.C. CODES ARE FURTHER SUBDIVIDED TO 4-DIGIT AND 6-DIGIT CODES GIVING EACH INDUSTRY A FURTHER SUBDIVISION THAN LISTED IN THE ABOVE 2-DIGIT CODES. FOR THE PURPOSE OF THIS STUDY, DATA IS PROVIDED TO THE 2-DIGIT CLASSIFICATION.

SIZE OF METAL WORKING *

NEARLY HALF OF THE TOTAL INDUSTRIAL ACTIVITY IN THE U.S. IS CLASSIFIED AS THE METAL WORKING MARKET. IT IS THE LARGEST SINGLE MARKET ACROSS ALL U.S. INDUSTRY.

THE METAL WORKING MARKET:

> SPENDS 45% OF TOTAL DOLLARS EXPENDED BY INDUSTRIAL PLANTS FOR MATERIALS.
>
> MAKES 40% OF ALL CAPITAL EXPENDITURES.
>
> EMPLOYS 47% OF ALL INDUSTRIAL WORKERS.
>
> PAYROLL IS 53% OF THE TOTAL FOR ALL INDUSTRIES.

AS SHOWN IN EXHIBIT II-A, THERE ARE 43,562 PLANTS EMPLOYING 20 OR MORE WITH A TOTAL EMPLOYMENT OF 10,147,834 IN THE METAL WORKING INDUSTRY. YOU WILL NOTE IN THE STUDY THE NUMBER USED FOR TOTAL PLANTS IS 41,670. THIS IS DUE TO THE ELIMINATION OF SITES NOT MANUFACTURING LIKE R&D SITES OR HEADQUARTER LOCATIONS.

IT IS ALSO IMPORTANT TO NOTE THERE ARE 83,074 PLANTS EMPLOYING LESS THAN 20. THE DATA IN THIS SURVEY EXCLUDES THIS SEGMENT OF THE METAL WORKING INDUSTRY. BECAUSE OF THE LIMITED AVAILABILITY OF INFORMATION ON THIS SEGMENT OF THE METAL WORKING INDUSTRY, SAMPLING WAS NOT POSSIBLE. WHEN CONSIDERING TOTALS IN THIS REPORT, KEEP IN MIND IT DOES NOT INCLUDE THE 83,074 PLANTS EMPLOYING LESS THAN 20.

EXHIBIT II-B IS A COLOR CODED MAP BY COUNTY REFLECTING THE CONCENTRATION OF METAL WORKING PLANTS.

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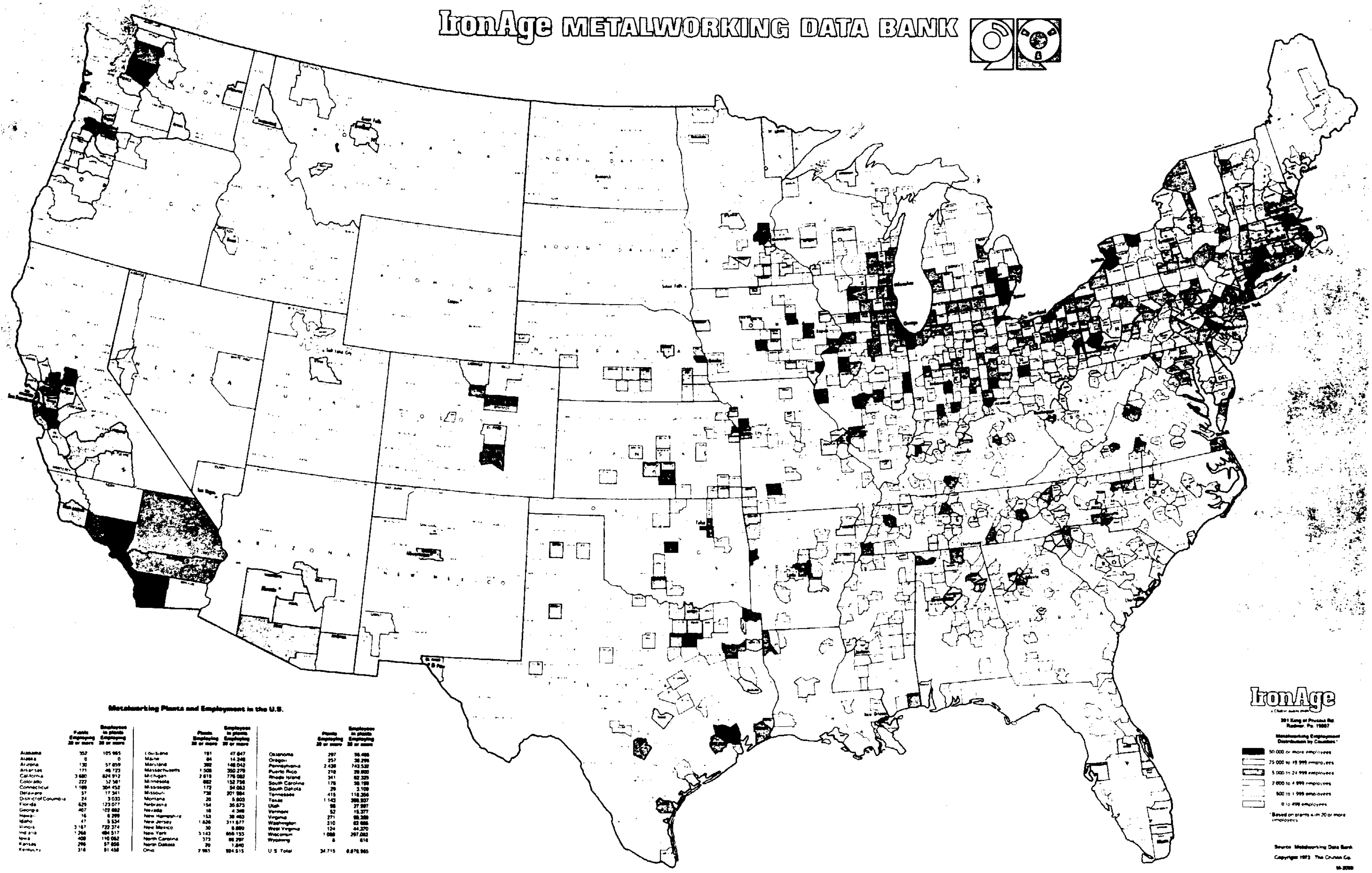
*SOURCE: 1972 CENSUS OF MANUFACTURING

EXHIBIT: II-A

SIZE OF METAL WORKING

STANDARD INDUSTRIAL CODE	TITLES OF INDUSTRY GROUPS	LESS THAN 20 EMPLOYEES	BY PRIMARY PRODUCT				
			TOTAL PLANTS EMPLOYING 20 OR MORE	TOTAL EMPLOYMENT IN PLANTS EMPLOYING 20 OR MORE	NUMBER OF PLANTS BY EMPLOYMENT SIZE		
					20-99	100-499	500 OR MORE
25	FURNITURE & FIXTURES	8,218	893	140,033	472	352	64
33	PRIMARY METAL INDUSTRIES	3,019	3,672	1,325,592	1,828	1,304	494
34	FABRICATED METAL PRODUCTS	17,355	11,686	1,646,258	7,722	3,324	554
35	MACHINERY EXCEPT ELECTRICAL	28,046	12,263	2,135,282	8,180	3,049	870
36	ELECTRIC & ELECTRONIC EQUIP.	4,922	7,042	2,137,686	3,563	2,374	925
37	TRANSPORTATION EQUIPMENT	4,836	3,806	2,042,694	1,930	1,129	642
38	INSTRUMENTS & RELATED PRODUCTS	3,249	2,617	486,755	1,532	781	227
39	MISC. MANUFACTURING INDUSTRIES	13,429	1,583	233,534	946	527	98
	U. S. TOTAL	83,074	43,562	10,147,834	26,173	12,840	3,874

IronAge METALWORKING DATA BANK



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a Chilton publication

201 King of Prussia Rd.
Radnor, Pa. 19087

Metalworking Employment Distribution by Counties*

■	50 000 or more employees
▨	25 000 to 49 999 employees
▨	5 000 to 24 999 employees
▨	2 000 to 4 999 employees
▨	500 to 1 999 employees
□	0 to 499 employees

*Based on plants with 20 or more employees

Source: Metalworking Data Bank

M-2080

Metalworking Plants and Employment in the U.S.

	Plants Employing 20 or more	Employees in plants Employing 20 or more		Plants Employing 20 or more	Employees in plants Employing 20 or more		Plants Employing 20 or more	Employees in plants Employing 20 or more
Alabama	352	105 985	Louisiana	191	47 647	Oklahoma	297	56 466
Alaska	0	0	Maine	84	14 248	Oregon	257	38 296
Arizona	130	57 659	Maryland	392	148 042	Pennsylvania	2 438	743 532
Arkansas	171	46 723	Massachusetts	1 508	350 278	Puerto Rico	218	29 800
California	3 680	824 912	Michigan	2 619	776 082	Rhode Island	341	82 325
Colorado	227	52 581	Minnesota	682	152 756	South Carolina	178	50 198
Connecticut	1 169	304 452	Mississippi	172	54 063	South Dakota	29	3 108
Delaware	57	17 341	Missouri	738	201 984	Tennessee	415	118 356
District of Columbia	24	3 033	Montana	20	5 803	Texas	1 143	286 937
Florida	629	123 077	Nebraska	154	36 673	Utah	98	27 987
Georgia	407	107 682	Nevada	18	4 388	Vermont	52	15 377
Hawaii	16	6 299	New Hampshire	153	38 463	Virginia	271	88 288
Idaho	47	5 534	New Jersey	1 826	311 677	Washington	310	82 686
Illinois	3 167	722 374	New Mexico	30	6 880	West Virginia	124	44 370
Indiana	1 264	484 517	New York	3 143	859 153	Wisconsin	1 088	297 082
Iowa	408	110 062	North Carolina	373	86 297	Wyoming	6	614
Kansas	296	57 856	North Dakota	20	1 840			
Kentucky	318	91 458	Ohio	2 985	924 515	U.S. Total	34 715	8 678 965

GEOGRAPHICAL BREAKDOWN

GEOGRAPHIC ZONES

In several sections of this study, the data is reported with reference to geographic locations. Exhibit II-c shows this breakdown.

The following lists the states in each zone:

NORTHEAST

- Connecticut
- Delaware
- Maine
- Maryland
- Massachusetts
- New Hampshire
- New Jersey
- New York
- Pennsylvania
- Rhode Island
- Vermont

SOUTHEAST

- Alabama
- Florida
- Georgia
- No. Carolina
- So. Carolina
- Tennessee
- Virginia
- Arkansas
- Washington, D.C.
- Puerto Rico

MID-WEST

- Illinois
- Indiana
- Kentucky
- Michigan
- Ohio
- West Virginia
- Wisconsin

FAR WEST

- Arizona
- California
- Hawaii
- Idaho
- Nevada
- Oregon
- Washington
- Utah

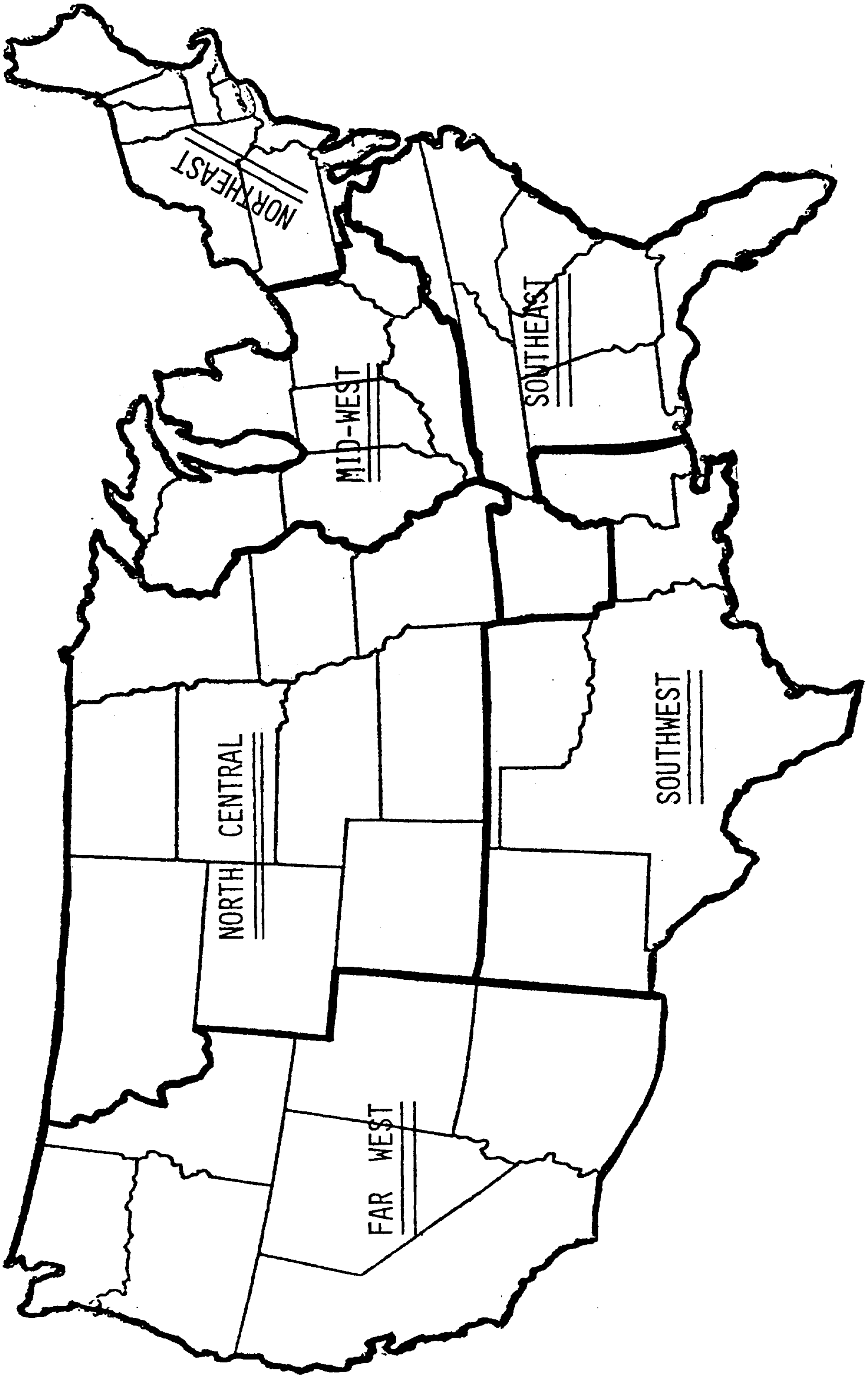
NORTH CENTRAL

- Colorado
- Iowa
- Kansas
- Minnesota
- Missouri
- Montana
- Nebraska
- North Dakota
- South Dakota
- Wyoming

SOUTHWEST

- Louisiana
- Mississippi
- Oklahoma
- Texas
- New Mexico

EXHIBIT II-C


NORTHEAST
MID-WEST
SOUTHEAST
NORTH CENTRAL
SOUTHWEST
FAR WEST

III

PLANTS METAL CLEANING

TO BEST DETERMINE THE NUMBER OF PLANTS USING ORGANIC SOLVENTS, A COMPARISON IS MADE WITH THE OTHER OPTION A PLANT HAS FOR METAL CLEANING, THE USE OF AN ALKALINE WASH SYSTEM.

EXHIBIT III-A: IT CAN BE SEEN IN THIS GRAPH THAT SOME OF THE PLANTS USE BOTH SOLVENT AND ALKALINE SYSTEMS, WHILE OTHERS USE JUST ONE SYSTEM. 34% OF THE PLANTS REPORTED THEY DO NOT USE ANY SYSTEM.

THE FOLLOWING IS SEEN IN THIS GRAPH:

- 66% OR 27,315 PLANTS IN THE METAL WORKING INDUSTRY DO METAL CLEANING.
- 29% OR 12,015 PLANTS USE A SOLVENT SYSTEM ONLY FOR METAL CLEANING.
- 20% OR 8,305 PLANTS USE A SOLVENT SYSTEM AND ALSO DO ALKALINE WASHING.
- THEREFORE, 49% OF THE METAL WORKING INDUSTRY OR 20,320 PLANTS USE SOLVENTS.

EXHIBIT III-A

PLANTS METAL CLEANING

TOTAL PLANTS: 41,670

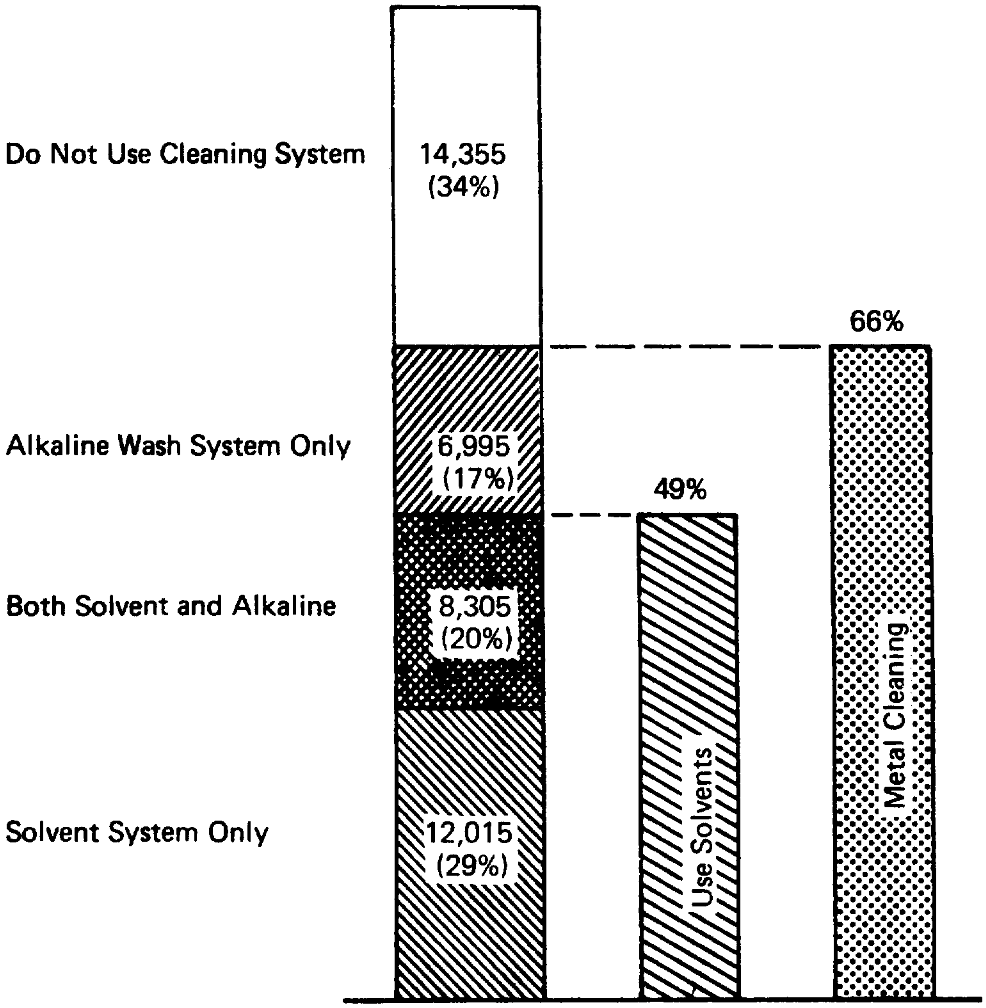


EXHIBIT III-B: THIS TABLE IS A BREAKDOWN OF THE PLANTS METAL CLEANING BY S.I.C. GROUP. IMMEDIATELY BELOW EACH GROUP TITLE IS THE NUMBER OF PLANTS REPRESENTED IN THAT GROUP. AS EXPECTED, THE GROUPS WITH THE GREATEST NUMBER OF PLANTS (34, 35, AND 36) HAVE THE MAJORITY OF PLANTS METAL CLEANING.

NUMBER OF PLANTS METAL CLEANING

THIS MANY PLANTS IN THESE S.I.C. GROUPS ...

	TOTAL	25 METAL FURNITUR	33 PRIMARY METALS	34 FABRICTD PRODUCTS	35 NON-ELEC MACHINRY	36 ELECTRIC EQUIPMNT	37 TRANSPTN EQUIPMNT	38 INSTMTS & CLOCKS	39 MISC INDUSTRY
	41,670	878	3,531	11,474	11,812	6,532	3,462	2,430	1,551
USE THIS TYPE OF METAL CLEANING PROCESS:									
ALKALINE WASH SYSTEMS ONLY	6,995	221	635	2,428	1,620	909	699	141	342
SOLVENT SYSTEMS ONLY	12,015	252	733	2,653	3,923	2,208	745	1,028	473
BOTH TYPES OF SYSTEMS	8,305	147	684	2,151	2,265	1,408	957	562	131
DO NOT USE A CLEANING SYSTEM:	14,355	258	1,479	4,242	4,004	2,007	1,061	699	605

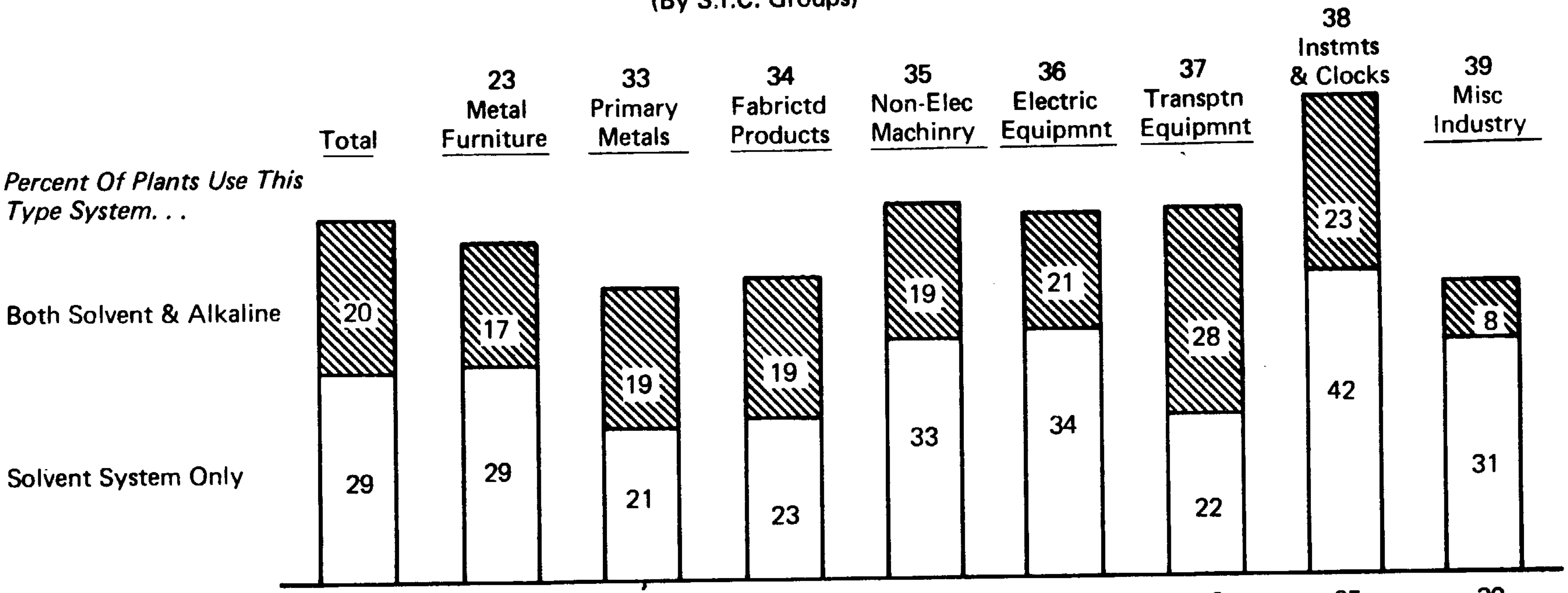
EXHIBIT III-B

EXHIBIT III-c: This graph gives a better picture of the use of solvent cleaning in each S.I.C. group. It shows the percent of plants in each group using solvents. Looking at "Total Solvents", it can be seen that groups 35, 36, 37, and 38 are above the industry average of 49%. Group 38, Instruments and Clocks, have a large percent of plants using solvents at 65%.

If you note group 25 you can see that although this group shows 71% of the plants metal cleaning, only 46% use solvents. This is due to the extensive use of alkaline cleaning in this group.

EXHIBIT III-c

PERCENT OF PLANTS METAL CLEANING
(By S.I.C. Groups)



	Total	23 Metal Furniture	33 Primary Metals	34 Fabrictd Products	35 Non-Elec Machinry	36 Electric Equipmnt	37 Transptn Equipmnt	38 Instmts & Clocks	39 Misc Industry
Total Solvents:	49	46	40	42	52	55	50	65	39
Alkaline Only	17	25	18	21	14	14	20	6	22
Total Metal Cleaning:	66	71	58	63	66	69	70	71	61
Do Not Use Metal Cleaning System:	34	29	42	37	34	31	30	29	39

EXHIBIT III-D: IT IS EASILY SEEN IN THIS GRAPH THAT THE LARGER THE PLANT, THE GREATER THE CHANCE SOLVENTS ARE USED. BUT, THE LARGER PLANTS (EMPLOYING 500+) REPRESENT A SMALL PORTION OF TOTAL PLANTS. ESPECIALLY WHEN WE CONSIDER 83,074 PLANTS EMPLOYING LESS THAN 20.

EXHIBIT III-D

METAL CLEANING — PLANT SIZE

		This Many Plants Employing. . .		
	Total	20-100 People	100-500 People	500+ People
	41,670	25,641	12,408	3,621
Use This Type Of Metal Cleaning System:				
Alkaline Wash Systems Only	6,995	3,843	2,430	722
Solvent Systems Only	12,015	7,557	3,581	877
Both Types Of Systems	8,305	3,762	3,022	1,521
Do Not Use A Cleaning System:	14,355	10,479	3,375	501

This Percent Of Plants. . .

Use A Solvent Cleaning System

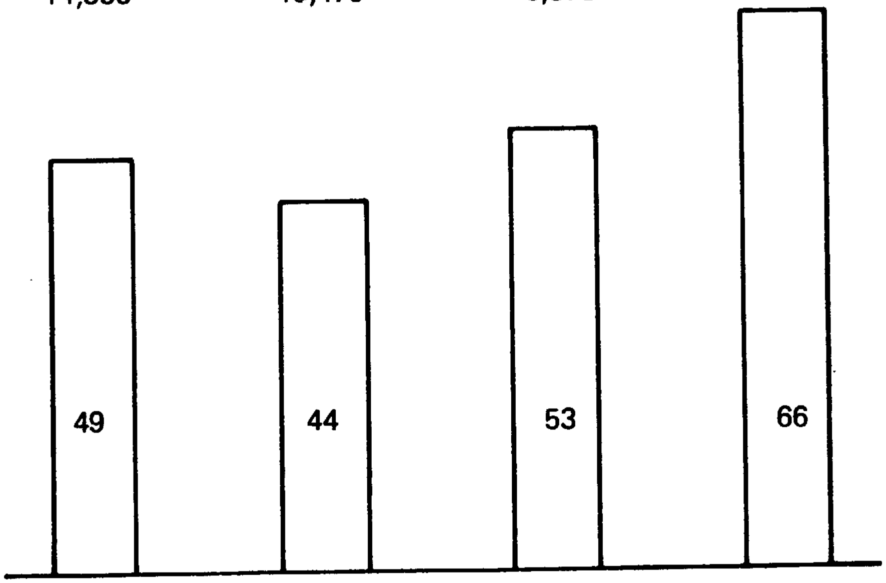


EXHIBIT III-E: THIS SHOWS THE NUMBER AND PERCENT OF PLANTS IN EACH ZONE USING SOLVENTS. BY TAKING THE PLANTS IN THREE OF THE SIX ZONES, THE NORTHEAST, THE MID-WEST, AND THE FAR WEST, IT REPRESENTS 78% OF THE TOTAL PLANTS. THESE THREE ZONES ALSO SHOW THE HIGHEST PERCENT OF PLANTS USING SOLVENTS.

EXHIBIT III-E

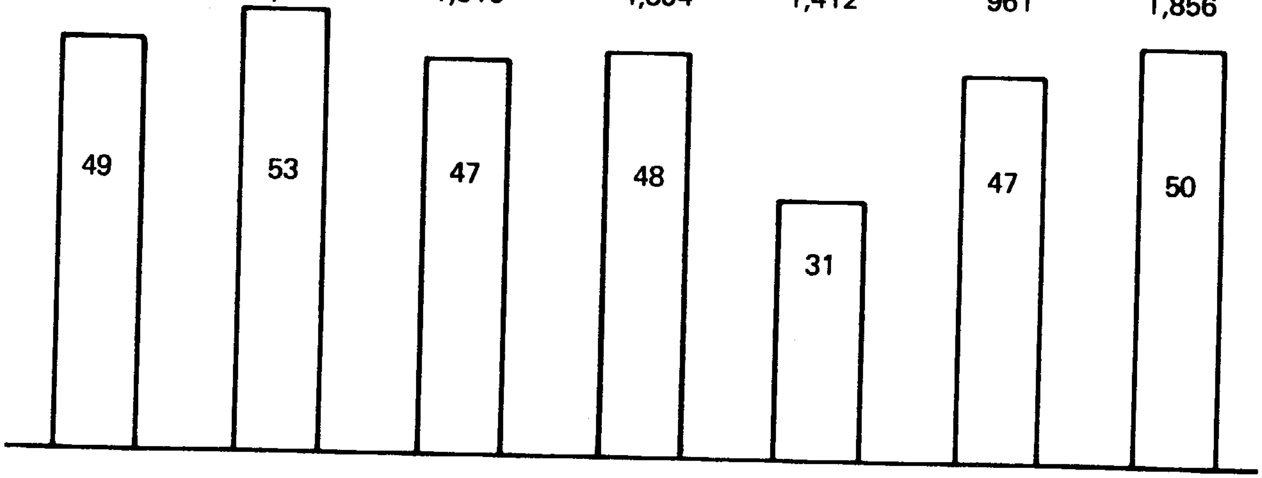
METAL CLEANING – LOCATION

Of Plants Located In The. . . . .

	Total	Northeast	Southeast	Mid-West	Southwest	North Central	Far West
	41,670	13,050	3,957	13,492	2,403	2,960	5,808
This Many Use:							
Alkaline Wash Systems Only	6,995	1,864	563	2,686	246	613	1,023
Solvent Systems Only	12,015	4,173	948	3,920	377	775	1,822
Both Types Of Systems	8,305	2,797	930	2,492	368	611	1,107
Do Not Use A Cleaning System	14,355	4,216	1,516	4,394	1,412	961	1,856

This Percent Of Plants....

Use A Solvent Cleaning System



IV

TYPES OF SOLVENT CLEANING

Solvents are used to clean or degrease in basically two ways, at room temperature or in a vapor state. Room temperature or "cold cleaning" (most used term) is done by several methods. These would include wiping the area to be cleaned, spraying or dipping in a tank of solvent. Vapor degreasing involves the heating of a solvent to its boiling point and creating a vapor zone. The parts to be cleaned are placed into this vapor zone. The solvent vapor condenses on the part and the soils drip off with the solvent.

EXHIBIT IV-A: Of the total 20,320 plants using solvents, 54% or 11,028 use room temperature cleaning only, 25% or 5,365 vapor degrease only, and 20% or 3,927 use both systems.

From this, it can be determined that of the 20,320 plants 74% or 14,955 use room temperature cleaning while 46% or 9,292 vapor degrease.

EXHIBIT IV-A

SOLVENT CLEANING
ROOM TEMPERATURE & VAPOR DEGREASING

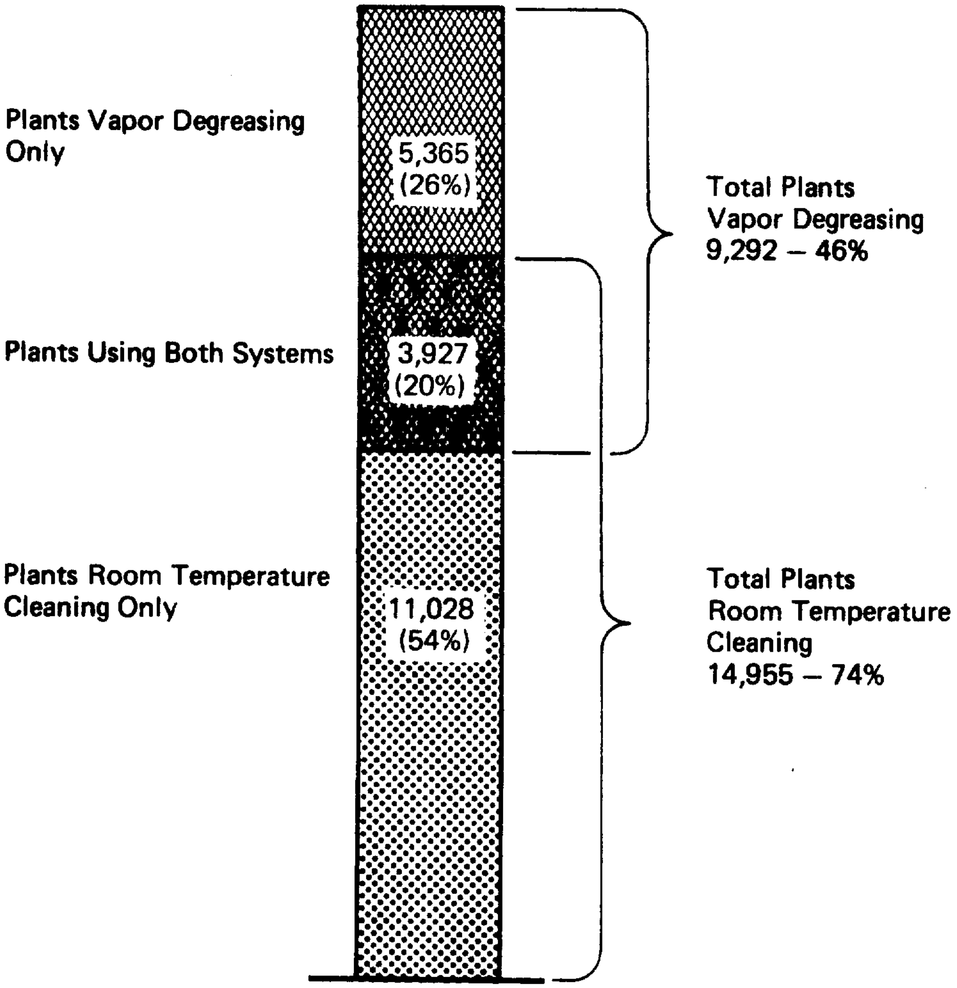


EXHIBIT IV-B: THIS TABLE GIVES THE TOTAL NUMBER OF PLANTS IN EACH S.I.C. GROUP USING SOLVENTS. IT ALSO GIVES THE TYPE OF SOLVENT CLEANING DONE IN EACH GROUP. GROUP 35, WITH THE LARGEST NUMBER OF PLANTS USING SOLVENTS, HAS 4,147 PLANTS ROOM TEMPERATURE CLEANING. THIS BY FAR EXCEEDS ANY OF THE OTHER GROUPS. THE MAJORITY OF THE PLANTS VAPOR DEGREASING FALL IN GROUPS 34, 35, AND 36.

EXHIBIT IV-B

SOLVENT CLEANING
ROOM TEMPERATURE & VAPOR DEGREASING

THIS MANY PLANTS IN THESE S.I.C. GROUPS ...

	TOTAL	25 METAL FURNITUR	33 PRIMARY METALS	34 FABRICTD PRODUCTS	35 NON-ELEC MACHINRY	36 ELECTRIC EQUIPMNT	37 TRANSPTN EQUIPMNT	38 INSTRMTS & CLOCKS	39 MISC INDUSTR
	20,320	399	1,417	4,804	6,188	3,616	1,702	1,590	604
USE THIS TYPE OF SOLVENT SYSTEM FOR METAL CLEANING:									
VAPOR DEGREASING SYSTEMS ONLY	5,365	95	348	1,282	1,086	1,367	376	575	236
ROOM TEMPERATURE SYSTEMS ONLY	11,028	209	825	2,826	4,147	1,191	962	605	263
BOTH TYPES OF SYSTEMS	3,927	95	246	696	955	1,058	362	410	105

EXHIBIT IV-c: THIS GRAPH SHOWS THE RELATIONSHIP BY S.I.C. GROUPS OF THE TYPE OF SOLVENT CLEANING SYSTEM USED. THE PERCENT OF ONE OF THE SYSTEMS ONLY AND THE PERCENT USING BOTH SYSTEMS TOGETHER GIVE THE TOTALS AT THE BOTTOM FOR EACH GROUP.

GROUPS 36 (ELECTRIC EQUIPMENT) AND 38 (INSTRUMENTS & CLOCKS) SHOW LARGEST USE OF VAPOR DEGREASING. WHILE GROUP 35 (NON-ELECTRIC MACHINERY) IS LOW WITH ONLY 33% OF THE PLANTS USING SOLVENTS VAPOR DEGREASING AND THEREFORE HAS THE HIGHEST PERCENT OF PLANTS USING ROOM TEMPERATURE CLEANING AT 82%.

IT CAN BE ASSURED THAT THE GROUPS WITH THE HIGHEST PERCENT USING BOTH SYSTEMS HAVE MORE SOPHISTICATED CLEANING REQUIREMENTS. EXAMPLES OF THIS WOULD BE GROUPS 36, ELECTRIC EQUIPMENT, AND 38, INSTRUMENTS & CLOCKS.

EXHIBIT IV-c

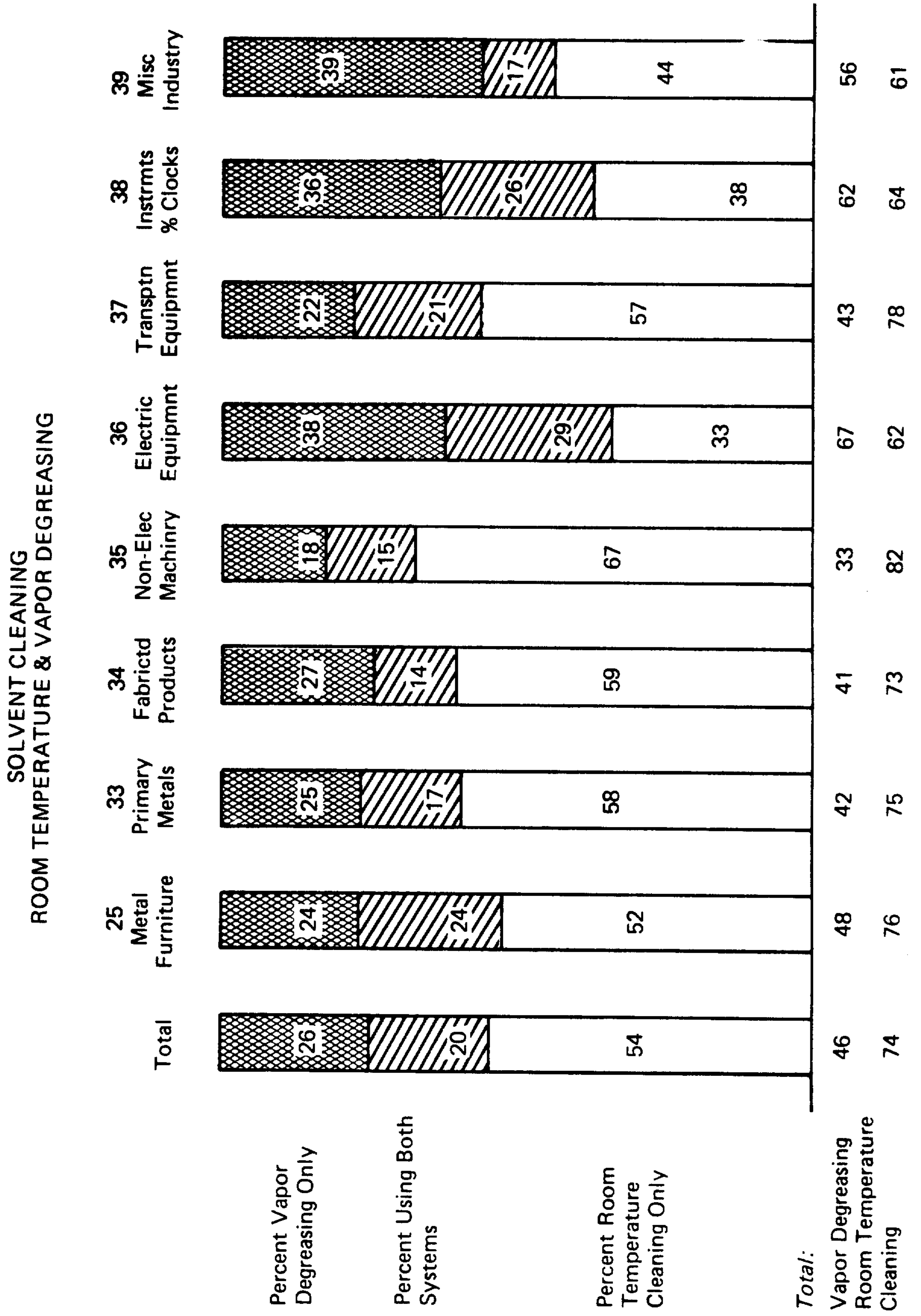

SOLVENT CLEANING
ROOM TEMPERATURE & VAPOR DEGREASING
Total
25 Metal Furniture
33 Primary Metals
34 Fabrictd Products
35 Non-Elec Machinry
36 Electric Equipmnt
37 Transptn Equipmnt
38 Instrmts % Clocks
39 Misc Industry
Percent Vapor Degreasing Only
Percent Using Both Systems
Percent Room Temperature Cleaning Only
Total: Vapor Degreasing
Room Temperature Cleaning
26 20 54 46 74
24 24 52 48 76
25 17 58 42 75
27 14 59 41 73
18 15 67 33 82
38 29 33 67 62
22 21 57 43 78
36 26 38 62 64
39 17 44 56 61

EXHIBIT IV-D: PLANT SIZE HAS A DIRECT RELATIONSHIP TO THE TYPE OF CLEANING DONE. THE GRAPH CLEARLY INDICATES THE FOLLOWING:

- THE LARGER THE PLANT, THE GREATER THE PERCENT OF VAPOR DEGREASING ONLY
- THE LARGER THE PLANT, THE SMALLER THE PERCENT OF ROOM TEMPERATURE CLEANING.
- THE LARGER THE PLANT, THE GREATER PERCENT OF USING BOTH TYPES OF SYSTEMS

EXHIBIT IV-D

SOLVENT CLEANING
ROOM TEMPERATURE & VAPOR DEGREASING

This Many Plants Employing. . .

	Total	20-100 People	100-500 People	500+ People
	20,320	11,319	6,603	2,398
Use This Type Of Solvent System For Metal Degreasing:				
Vapor Degreasing Systems Only	5,365	2,562	1,883	920
Room Temperature Systems Only	11,028	7,494	2,868	666
Both Types Of Systems	3,927	1,263	1,852	812

This Percent Of Plants. . .

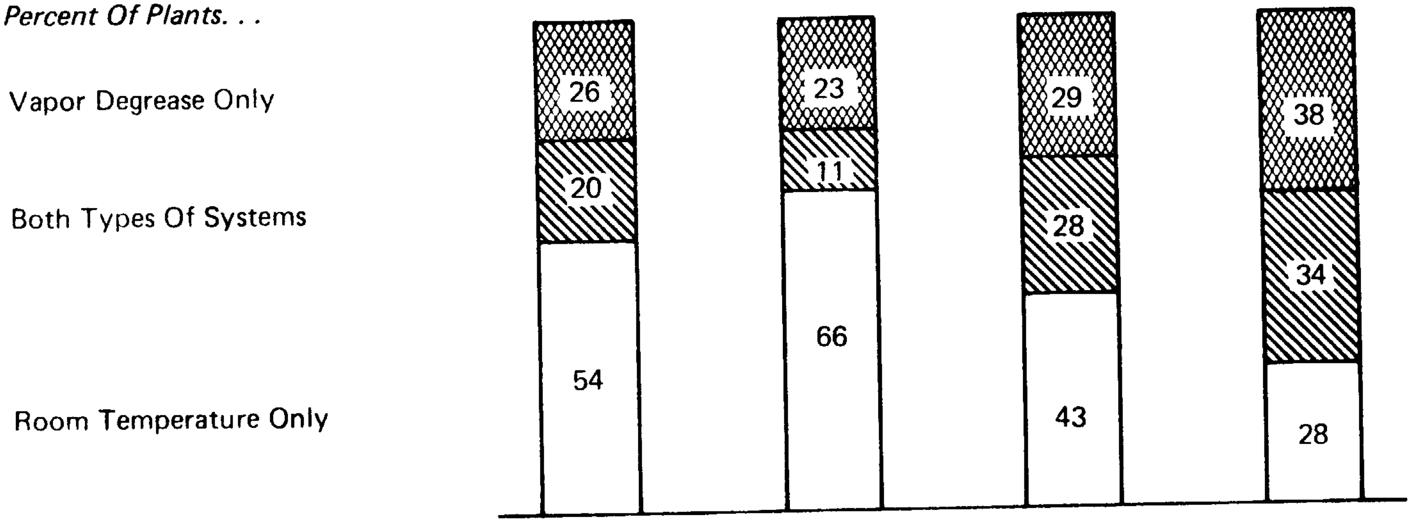


EXHIBIT IV-E: THE DIFFERENT REGIONS OF THE COUNTRY VARY TO A GREAT DEGREE ON THE TYPES OF SOLVENT CLEANING SYSTEM USED. THE NORTHEAST AND FAR WEST HAVE THE LARGEST PERCENT OF PLANTS IN THEIR ZONES VAPOR DEGREASING, 50% AND 55%. THE SOUTHWEST HAS THE LEAST PERCENT VAPOR DEGREASING AT 29%. OTHER THAN THE SOUTHWEST, THE BALANCE OF THE COUNTRY HAS AN ALMOST IDENTICAL PERCENT OF ROOM CLEANING SOLVENT USE.

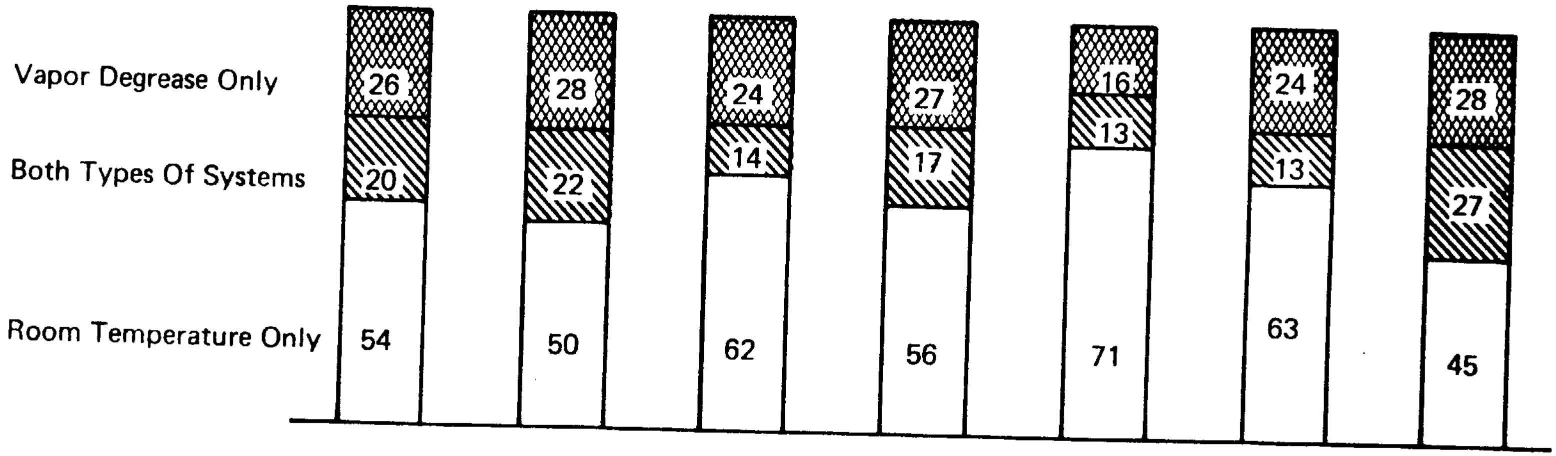
EXHIBIT IV-E

SOLVENT CLEANING
ROOM TEMPERATURE & VAPOR DEGREASING

Of Plants Located In The. . .

	Total	Northeast	Southeast	Mid-West	Southwest	North Central	Far West
	20,320	6,970	1,878	6,412	745	1,386	2,929
This Many Use:							
Vapor Degreasing Systems Only	5,365	1,910	442	1,738	119	328	828
Room Temperature Systems Only	11,028	3,511	1,178	3,614	530	872	1,323
Both Types Of Systems	3,927	1,549	258	1,062	96	186	778

This Percent Of Plants. . .



V

VAPOR DEGREASERS

IT HAS BEEN SHOWN THAT 20,320 OF THE 41,670 PLANTS IN THE METAL WORKING INDUSTRY USE SOLVENTS. ALSO, THAT 9,292 OF THE PLANTS USING SOLVENTS, VAPOR DEGREASE. THIS SECTION IDENTIFIES THE NUMBER OF VAPOR DEGREASERS AND THE TYPE USED IN THE DIFFERENT S.I.C. GROUPS, BY PLANT SIZE AND BY GEOGRAPHIC LOCATION.

EXHIBIT V-A: THERE ARE 18,090 VAPOR DEGREASERS IN THE 41,670 PLANTS INCLUDED IN THIS SURVEY. THE 18,090 VAPOR DEGREASERS ARE FOUND IN 9,292 PLANTS GIVING AN AVERAGE NUMBER OF 1.95 UNITS PER PLANT.

EXHIBIT V-A

NUMBER OF VAPOR DEGREASERS

TOTAL NUMBER OF PLANTS
VAPOR DEGREASING: 9,292

TOTAL NUMBER OF VAPOR
DEGREASING UNITS: 18,090

AVERAGE NUMBER OF UNITS
PER PLANT: 1.95

EXHIBIT V-B: GROUPS 34, 35, AND 36 HAVE 69% OF THE PLANTS VAPOR DEGREASING AND 63% OF THE TOTAL VAPOR DEGREASERS. OTHER GROUPS LIKE 33, 37, AND 38 HAVE FEWER PLANTS VAPOR DEGREASING BUT THE AVERAGE NUMBER OF UNITS PER PLANT IS MUCH HIGHER.

EXHIBIT V-B

NUMBER OF VAPOR DEGREASERS
(BY S.I.C. GROUP)

	TOTAL	25 METAL FURNITUR	33 PRIMARY METALS	34 FABRICTD PRODUCTS	35 NON-ELEC MACHINRY	36 ELECTRIC EQUIPMNT	37 TRANSPTN EQUIPMNT	38 INSTRMTS & CLOCKS	39 MISC INDUSTRY
NUMBER OF PLANTS VAPOR DEGREASING:	9,292	190	594	1,978	2,041	2,425	738	985	341
NUMBER OF VAPOR DEGREASING UNITS:	18,090	376	1,315	3,214	3,285	4,919	2,400	2,233	348
AVERAGE NUMBER OF UNITS PER PLANT:	1.95	1.98	2.21	1.62	1.61	2.03	3,25	2.27	1.02

EXHIBIT V-c: As THE PLANT SIZE INCREASES, THE NUMBER OF PLANTS VAPOR DEGREASING FALLS OFF CONSIDERABLY. THE NUMBER OF VAPOR DEGREASERS REMAINS FAIRLY CONSTANT IN EACH PLANT SIZE GROUPING. THEREFORE, AS EXPECTED, THE LARGER PLANTS HAVE MORE UNITS PER PLANT.

EXHIBIT V-C

NUMBER OF VAPOR DEGREASERS
(BY PLANT SIZE)

		EMPLOYMENT		
	TOTAL	20-100 PEOPLE	100-500 PEOPLE	500+ PEOPLE
NUMBER OF PLANTS VAPOR DEGREASING:	9,292	4,287	3,414	1,591
NUMBER OF VAPOR DEGREASING UNITS:	18,090	6,149	5,966	5,975
AVERAGE NUMBER OF UNITS PER PLANT:	1.95	1.43	1.75	3.76

EXHIBIT V-D: By looking at the number of plants vapor degreasing by geographic location, the Northwest, Mid-West, and Far West regions respectively have the largest number. This is also true with regard to number of units. But, the Far West has a much higher average number of units per plant at 2.58. This brings the balance of the regions below the national average of 1.95.

NUMBER OF VAPOR DEGREASERS
(BY PLANT LOCATION)

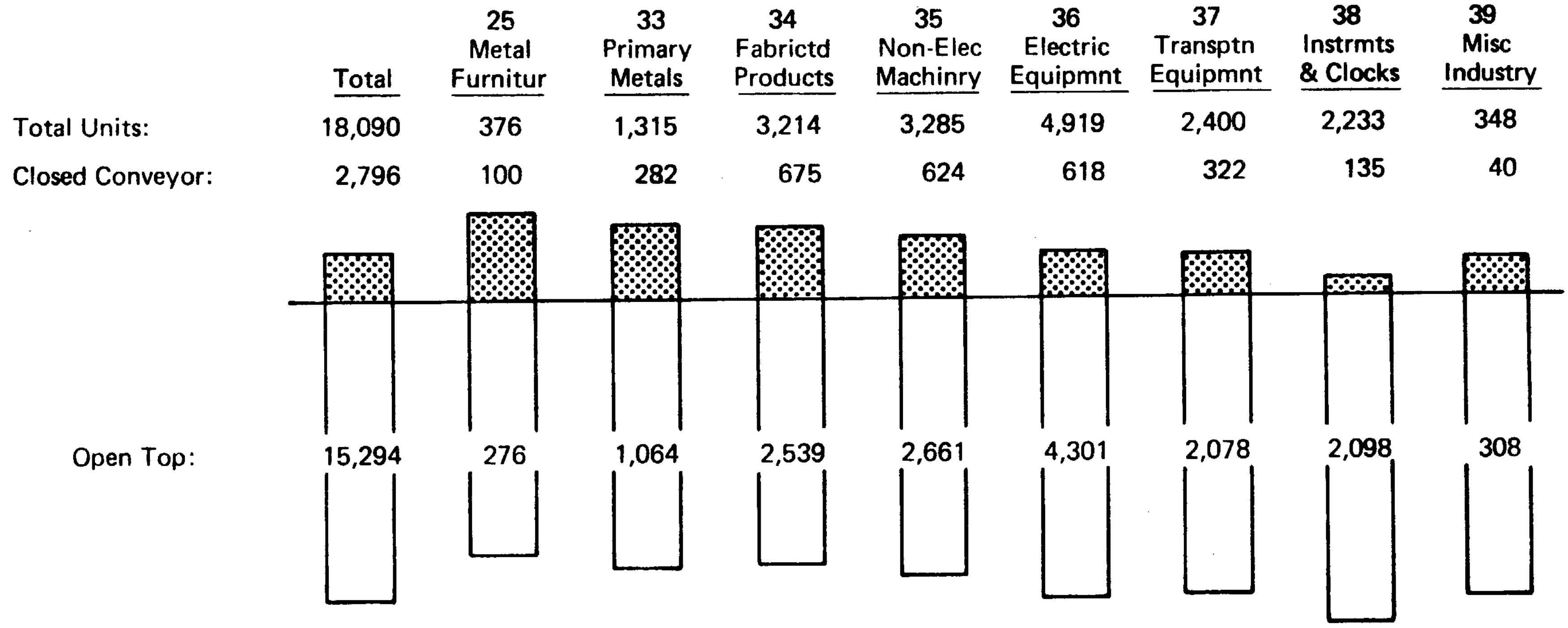
	TOTAL	NORTHEAST	SOUTHEAST	MID-WEST	SOUTHWEST	NORTH CENTRAL	FAR WEST
UMBER OF PLANTS APOR DEGREASING:	9,292	3,452	698	2,798	212	506	1,626
UMBER OF VAPOR EGREASING UNITS:	18,090	6,357	1,206	5,090	377	870	4,190
VERAGE NUMBER OF IITS PER PLANT:	1.95	1.84	1.73	1.82	1.78	1.72	2.58

EXHIBIT V-D

EXHIBIT V-E: IN CONSIDERING THE TYPES OF VAPOR DEGREASERS, THEY WERE CLASSIFIED IN TWO GENERAL CATEGORIES: CLOSED CONVEYOR AND OPEN TOP. THERE ARE A TOTAL OF 18,090 VAPOR DEGREASERS. 2,796 OR 15% ARE CLOSED CONVEYOR AND 15,294 OR 85% ARE OPEN TOP. IN THE GRAPH, THE BAR REPRESENTS 100% OF THE DEGREASERS. THE AMOUNT OF THE BAR ABOVE OR BELOW THIS LINE REPRESENTS THE PERCENT OF DEGREASERS OF THE CLOSED CONVEYOR OR OPEN TOP TYPE. THIS DEMONSTATES THAT GROUPS 25, 33, AND 34 HAVE THE LARGEST PERCENT OF CLOSED CONVEYOR TYPE DEGREASERS WITH GROUP 38 THE LEAST.

EXHIBIT V-E

TYPES OF VAPOR DEGREASERS
(BY S.I.C. GROUP)



	Total	25 Metal Furnitur	33 Primary Metals	34 Fabrictd Products	35 Non-Elec Machinry	36 Electric Equipmnt	37 Transptn Equipmnt	38 Instrmts & Clocks	39 Misc Industry
Total Units:	18,090	376	1,315	3,214	3,285	4,919	2,400	2,233	348
Closed Conveyor:	2,796	100	282	675	624	618	322	135	40
Open Top:	15,294	276	1,064	2,539	2,661	4,301	2,078	2,098	308

EXHIBIT V-F: IT WOULD BE ASSUMED THAT THE LARGER PLANTS WOULD HAVE THE HIGHER PERCENTAGE OF CLOSED CONVEYOR DEGREASERS, BUT BY LOOKING AT THIS GRAPH, IT CAN BE SEEN THAT PLANT SIZE HAS NO EFFECT.

EXHIBIT V-F

TYPES OF VAPOR DEGREASERS
(BY PLANT SIZE)

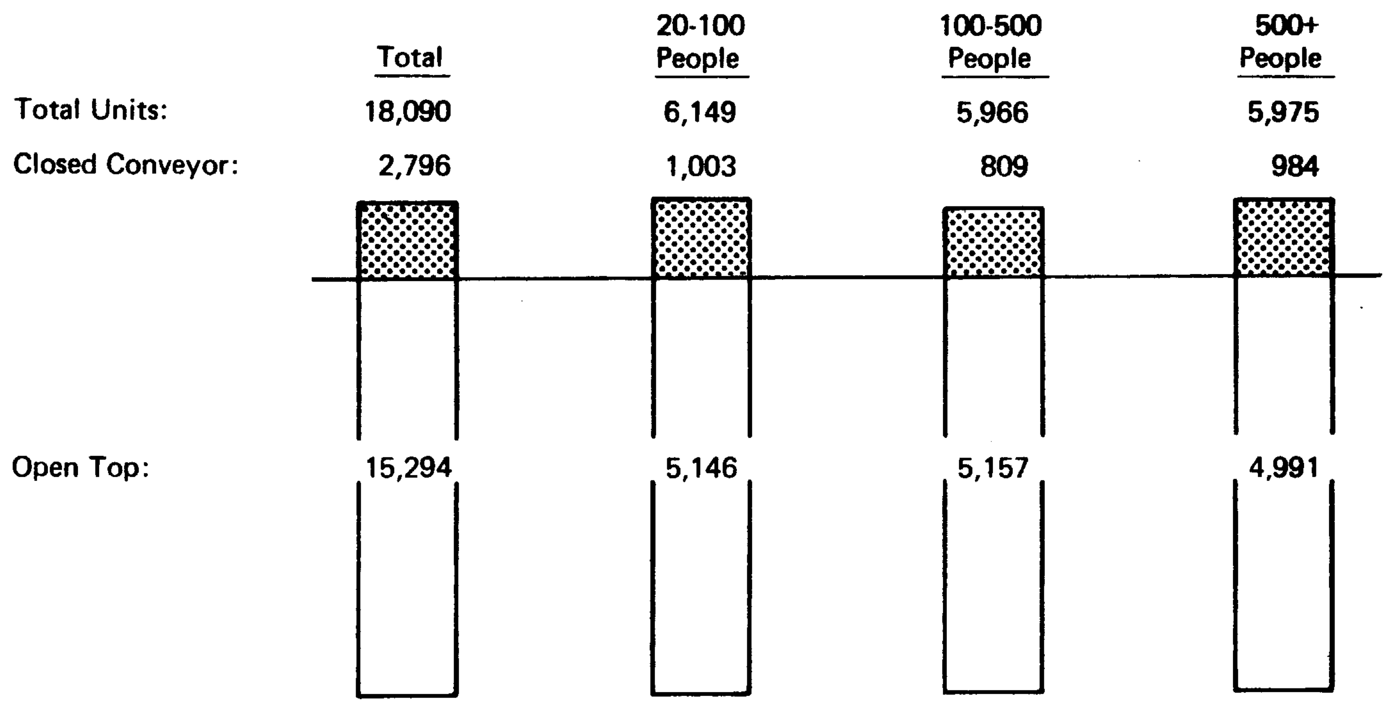
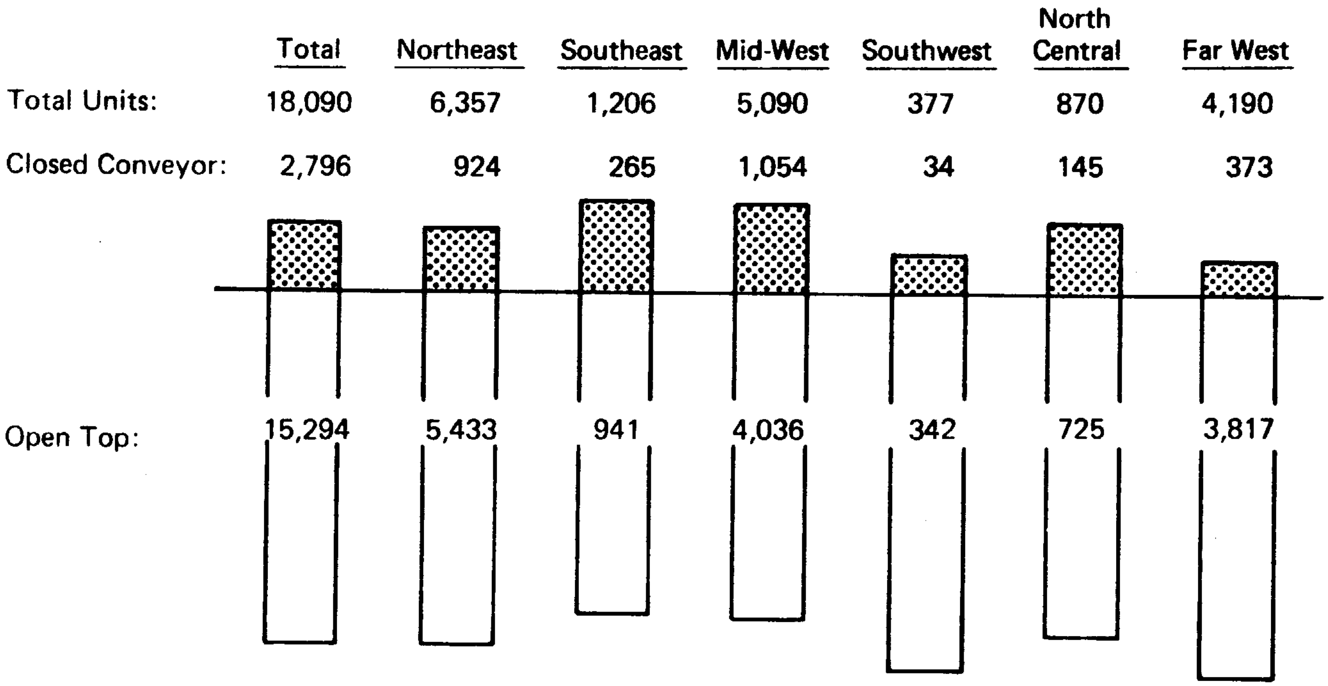


EXHIBIT V-G: THE TYPE OF VAPOR DEGREASER USED DOES VARY WITH REGARD TO GEOGRAPHIC LOCATION. BY LOOKING AT THAT BAR GRAPH, YOU CAN SEE THE REGIONS WITH A LOW PERCENT OF OPEN TOP (SOUTHWEST AND FAR WEST). WHILE THE SOUTHEAST AND MID-WEST HAVE A HIGH PERCENT OF CLOSED CONVEYOR.

EXHIBIT V-G

TYPES OF VAPOR DEGREASERS
(BY PLANT LOCATION)



VI

QUANTITY OF SOLVENT USED

THIS SECTION OUTLINES WHICH SOLVENTS ARE USED IN VAPOR DEGREASING AND ROOM TEMPERATURE CLEANING. IT IS DETERMINED HOW MANY PLANTS USE A PARTICULAR SOLVENT AND THE QUANTITY USED.

EXHIBIT VI-A: TRICHLOROETHYLENE IS THE MOST WIDELY USED SOLVENT FOR VAPOR DEGREASING WITH 5,447 PLANTS OR 59% OF THE TOTAL 9,292 PLANTS VAPOR DEGREASING USING IT. 1,1,1-TRICHLOROETHANE IS USED IN 21% OF THE PLANTS FOR VAPOR DEGREASING.

EXHIBIT VI-A

SOLVENTS USED FOR VAPOR DEGREASING

SOLVENT:	NUMBER OF PLANTS USING	PERCENT OF PLANTS USING
TRICHLOROETHYLENE	5,447	59
1,1,1-TRICHLOROETHANE	1,910	21
PERCHLOROETHYLENE	1,486	16
METHYLENE CHLORIDE	142	1.5
FLUOROCARBONS	1,014	11

EXHIBIT VI-B: THIS TABLE GIVES THE QUANTITY OF EACH VAPOR DEGREASING SOLVENT USED AND THE AVERAGE USE PER PLANT IN A YEAR. THIS TABLE SHOWS THAT ALTHOUGH TRICHLOROETHYLENE AND 1,1,1-TRICHLOROETHANE USAGE IS SIMILAR, THE AVERAGE QUANTITY USED PER PLANT OF 1,1,1 IS THREE TIMES GREATER THAN TRI. THIS INDICATES THAT PLANTS WITH A LARGE SOLVENT REQUIREMENT HAVE SWITCHED FROM TRICHLOROETHYLENE TO 1,1,1-TRICHLOROETHANE.

Exhibit VI-B

SOLVENT USAGE FOR VAPOR DEGREASING

SOLVENT:	PLANTS USING	GAL/MO $(x10^3)$	LBS/YR $(x10^3)$	AVERAGE LBS/YR/PLANT
TRICHLOROETHYLENE	5,447	774	111,456	20,461
1,1,1-TRICHLOROETHANE	1,910	899	118,668	62,130
PERCHLOROETHYLENE	1,486	283	44,148	29,709
METHYLENE CHLORIDE	142	62	8,184	57,634
FLUOROCARBONS	1,014	235	36,660	36,154

EXHIBIT VI-c: THERE ARE 14,955 PLANTS, OR 74% OF THE PLANTS SOLVENT CLEANING, WHICH ARE ROOM TEMPERATURE CLEANING. MANY OF THESE PLANTS USE MORE THAN ONE OF THE SOLVENTS, SO THE NUMBER OF PLANTS USING WILL NOT TOTAL 14,955. PETROLEUM SOLVENTS ARE THE MOST WIDELY USED AT 42%. TRICHLOROETHYLENE, 1,1,1-TRICHLOROETHANE AND SAFETY BLENDS ARE A FAR SECOND CHOICE.

EXHIBIT VI-c

SOLVENTS USED FOR ROOM TEMPERATURE CLEANING

SOLVENT:	NUMBER OF PLANTS USING	PERCENT OF PLANTS USING
TRICHLOROETHYLENE	2,295	15
1,1,1-TRICHLOROETHANE	2,106	14
PERCHLOROETHYLENE	702	5
METHYLENE CHLORIDE	324	2
FLUOROCARBONS	1,026	7
PETROLEUM SOLVENTS	6,344	42
ACETONE	1,215	8
METHYL-ETHYL-KETONE	648	4
TOLUENE	837	6
ALCOHOLS	945	6
ETHERS	27	-
SAFETY BLENDS	2,079	14
CARBON TETRACHLORIDE	162	1

EXHIBIT VI-D: BY TAKING THE POUNDS PER YEAR USED BY ANY SOLVENT AND DIVIDING IT BY THE PLANTS USING THAT PARTICULAR SOLVENT, THE AVERAGE POUNDS USED PER YEAR PER PLANT IS GIVEN. THE LARGEST SINGLE USE IS PETROLEUM SOLVENTS AT 73 MILLION POUNDS PER YEAR, BUT THE AVERAGE PLANT USES ONLY 11,560 POUNDS PER YEAR WHILE THE AVERAGE PLANT USING 1,1,1-TRICHLOROETHANE USES 32,028 POUNDS PER YEAR.

EXHIBIT VI-D

SOLVENT USAGE FOR ROOM TEMPERATURE CLEANING

SOLVENT:	PLANTS USING	GAL/MO $(x10^3)$	LBS/YR $(x10^3)$	AVERAGE LBS/YR/PLANT
TRICHLOROETHYLENE	2,295	299	43,056	18,760
1,1,1-TRICHLOROETHYLENE	2,106	511	67,452	32,028
PERCHLOROETHYLENE	702	61	9,516	13,556
METHYLENE CHLORIDE	324	52	6,864	21,185
FLUOROCARBONS	1,026	125	19,500	19,006
PETROLEUM SOLVENTS	6,344	926	73,339	11,560
ACETONE	1,215	110	8,712	7,170
METHYL ETHYL KETONE	648	86	6,811	10,511
TOLUENE	837	138	11,923	14,245
ALCOHOLS	945	77	6,098	6,453
ETHERS	27	2	-	-
SAFETY BLENDS	2,079	180	16,200	7,792
CARBON TETRACHLORIDE	162	12	1,584	9,778

VII

VAPOR RECOVERY AND CONTROL SYSTEMS

THIS SECTION GIVES THE NUMBER AND PERCENT OF PLANTS USING A VAPOR RECOVERY OR CONTROL SYSTEM. SOLVENT VAPORS CAN BE RECOVERED FROM AIR BY THE USE OF A CARBON ABSORPTION UNIT. VAPORS ARE CONTROLLED BY REFRIGERATION, A WATER BARRIER, AND VAPOR BURNING.

EXHIBIT VII-A: 76% OF THE PLANTS USING SOLVENTS USE NO VAPOR RECOVERY OR CONTROL SYSTEM. THE ONLY SYSTEMS USED TO ANY EXTENT IS REFRIGERATION AT 12%. 8% OF THE PLANTS ARE LISTED IN THE "OTHER" CATEGORY, BUT CANNOT BE CONSIDERED AS USING GOOD RECOVERY AND CONTROL SYSTEMS. (A PLANT MIGHT CONSIDER THE LEAVING OF THE BACK DOOR OPEN, A VAPOR CONTROL SYSTEM.)

EXHIBIT VII-A

USE OF VAPOR RECOVERY AND CONTROL SYSTEMS

	NUMBER OF PLANTS	PERCENT OF PLANTS
TYPE OF SYSTEM:		
CARBON ABSORPTION	305	1.5
REFRIGERATION	2,477	12
VAPOR BURNING	351	2
WATER BARRIER	37	.2
OTHER	1,632	8
NONE	15,519	76

PLANT USING SOLVENTS: 20,320

EXHIBIT VII-B: THIS TABLE PROJECTS THE NUMBER OF PLANTS USING VAPOR RECOVERY AND CONTROL SYSTEMS BY S.I.C. GROUP. AREAS LEFT BLANK INDICATE TOO SMALL A SAMPLING TO PROJECT A TOTAL. THE LARGEST USER OF VAPOR RECOVERY AND CONTROL SYSTEMS IS GROUP 36, ELECTRIC EQUIPMENT.

USE OF VAPOR RECOVERY & CONTROL SYSTEMS

THIS MANY PLANTS IN THESE S.I.C. GROUPS...

	TOTAL	25 METAL FURNITUR	33 PRIMARY METALS	34 FABRICTD PRODUCTS	35 NON-ELEC MACHINRY	36 ELECTRIC EQUIPMNT	37 TRANSPTN EQUIPMNT	38 INSTRMTS & CLOCKS	39 MISC INDUSTRY
	20,320	399	1,417	4,804	6,188	3,616	1,702	1,590	604
USE THIS TYPE OF SYSTEM:									
CARBON ABSORPTION	305	-	57	40	100	83	-	25	-
REFRIGERATION	2,477	18	95	436	477	712	310	350	79
VAPOR BURNING	351	36	-	99	59	42	-	75	40
WATER BARRIER	37	18	-	-	-	-	19	-	-
SOME OTHER TYPE	1,631	18	210	415	379	356	39	175	39
DO NOT USE RECOVERY OR CONTROL SYSTEMS:	15,519	309	1,055	3,814	5,173	2,423	1,334	965	446

EXHIBIT VII-B

EXHIBIT VII-c: THIS GRAPH GIVES A CLEAR PICTURE OF THE USE OF VAPOR RECOVERY AND CONTROL SYSTEMS BY S.I.C. GROUP. GROUPS 36 AND 38 HAVE THE LARGEST PERCENT OF PLANTS USING RECOVERY AND CONTROL SYSTEMS.

EXHIBIT VII-C

PERCENT OF PLANTS USING VAPOR RECOVERY & CONTROL SYSTEMS
(By S.I.C. GROUP)

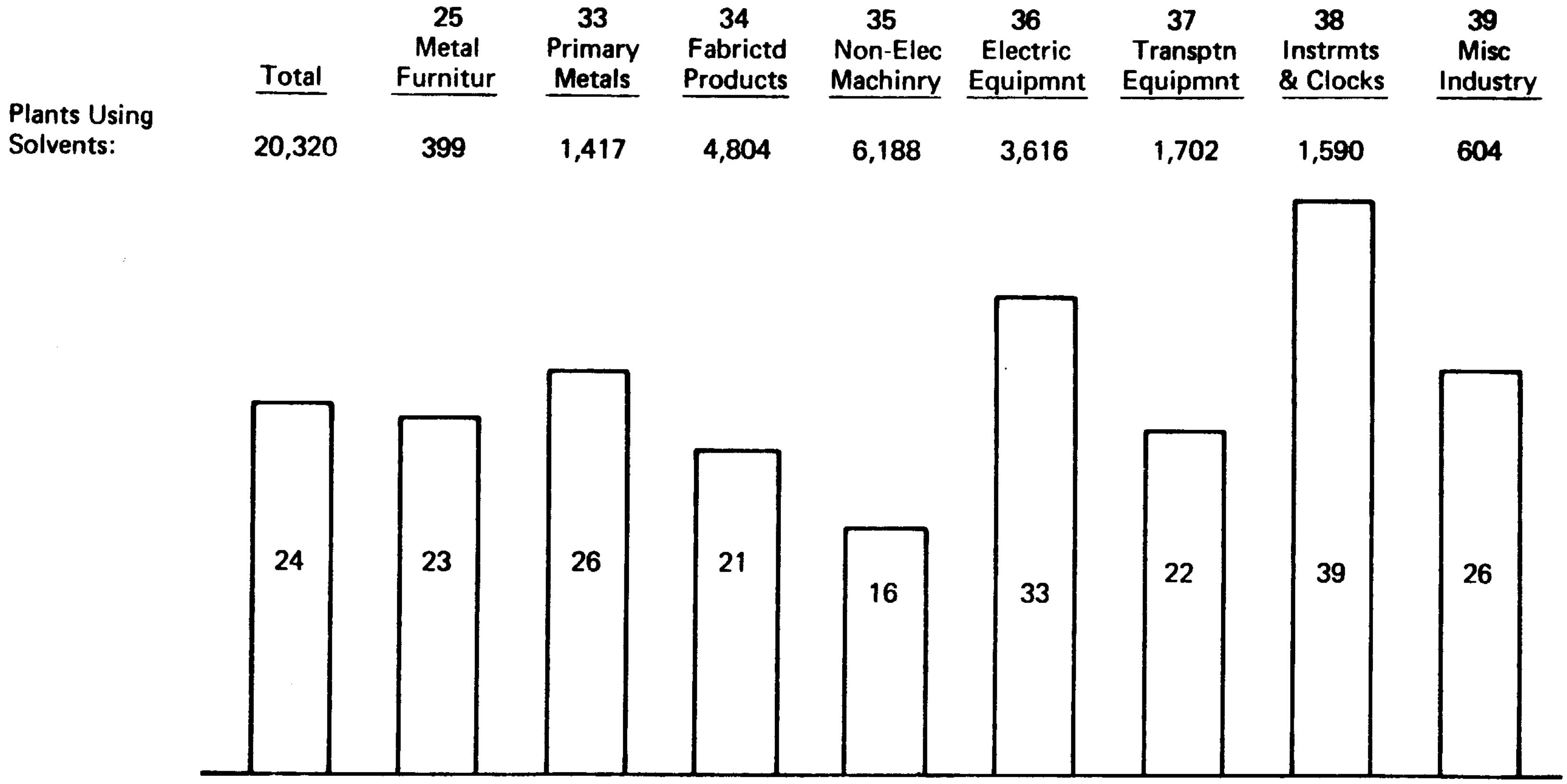


EXHIBIT VII-D: THE LARGER THE PLANT, THE GREATER THE USE OF VAPOR RECOVERY AND CONTROL SYSTEM. IN PLANTS EMPLOYING FROM 20 TO 100 PEOPLE ONLY .8% USE CARBON ABSORPTION, WHILE PLANTS EMPLOYING OVER 500 PEOPLE HAVE 4.6% OF THE PLANTS USING SOLVENTS USING CARBON ABSORPTION.

EXHIBIT VII-D

USE OF VAPOR RECOVERY AND CONTROL SYSTEMS

(BY PLANT SIZE)

THIS MANY PLANTS EMPLOYING ...

	TOTAL	20-100 PEOPLE	100-500 PEOPLE	500+ PEOPLE
	20,320	11,319	6,603	2,398
... USE THIS TYPE OF SYSTEM:				
CARBON ABSORPTION	305	90	104	111
REFRIGERATION	2,477	976	1,030	471
VAPOR BURNING	351	183	93	75
WATER BARRIER	37	37	-	-
OTHER	1,632	722	675	234
... DO NOT USE RECOVERY ON CONTROL SYSTEM	15,519	9,310	4,701	1,508

EXHIBIT VII-E: THE PERCENT OF PLANTS USING ANY TYPE OF RECOVERY OR CONTROL SYSTEM INCREASES DRAMATICALLY AS PLANT SIZE INCREASES.

EXHIBIT VII-E

PERCENT OF PLANTS USING VAPOR RECOVERY & CONTROL SYSTEMS (BY PLANT SIZE)

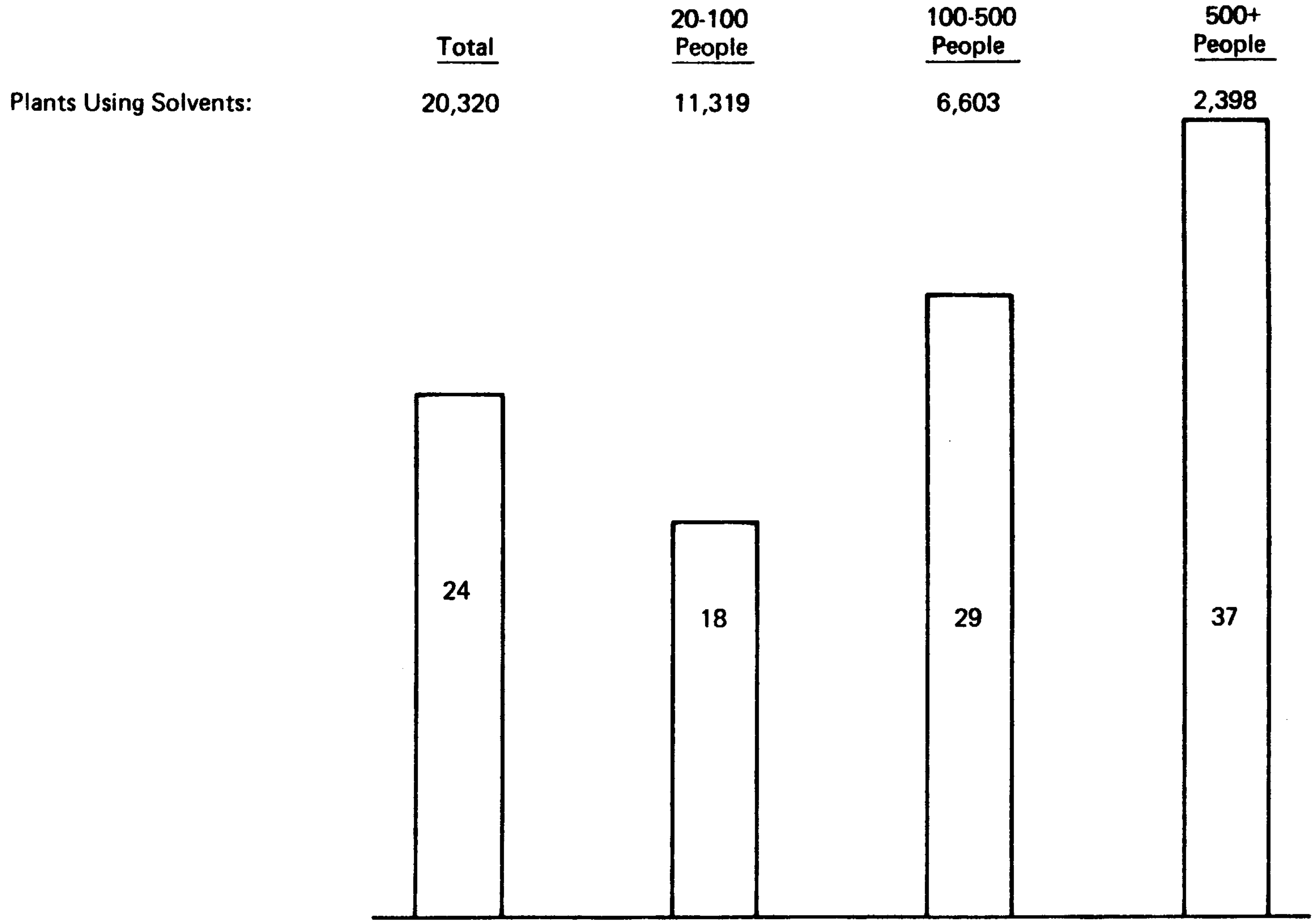


EXHIBIT VII-F: THIS TABLE PROJECTS THE NUMBER OF PLANTS USING THE RECOVERY AND CONTROL SYSTEMS IN THE VARIOUS GEOGRAPHIC ZONES. SOME PROJECTIONS ARE NOT MADE DUE TO AN INADEQUATE SAMPLE SIZE.

EXHIBIT VII-F

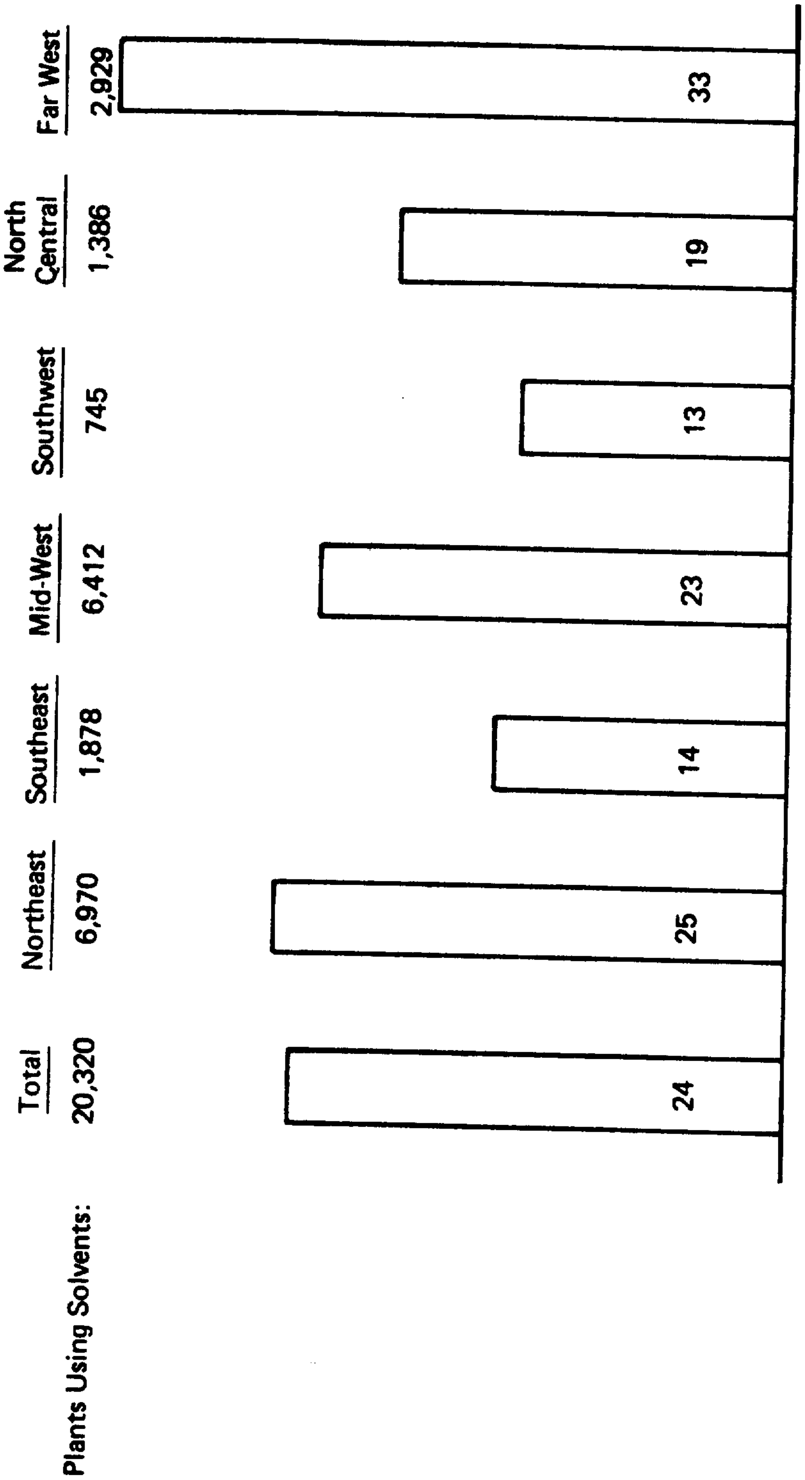
USE OF VAPOR RECOVERY & CONTROL SYSTEMS

	OF PLANTS LOCATED IN THE ...						
	TOTAL	NORTHEAST	SOUTHEAST	MID-WEST	SOUTHWEST	NORTH CENTRAL	FAR WEST
	20,320	6,970	1,878	6,412	745	1,386	2,929
.. USE THIS TYPE OF SYSTEM:							
CARBON ABSORPTION	305	127	-	141	-	-	37
REFRIGERATION	2,477	861	173	717	36	124	566
VAPOR BURNING	351	142	26	136	26	-	21
WATER BARRIER	37	19	-	18	-	-	-
SOME OTHER TYPE	1,631	612	73	439	36	143	328
. DO NOT USE RECOVERY OR CONTROL SYSTEMS	15,519	5,209	1,606	4,961	647	1,119	1,977

EXHIBIT VII-G: THIS GRAPH INDICATES THE PERCENT OF PLANTS USING VAPOR RECOVERY AND CONTROL SYSTEMS BY GEOGRAPHIC ZONE. THE FAR WEST HAS THE LARGEST PERCENT OF PLANTS USING RECOVERY AND CONTROL SYSTEMS WHILE THE SOUTHEAST AND SOUTHWEST HAS THE LEAST PERCENT.

EXHIBIT VII-G

PERCENT OF PLANTS USING
VAPOR RECOVERY & CONTROL SYSTEMS
(BY PLANT LOCATION)



VIII

SOLVENT DISPOSAL

SOLVENTS CAN BE DISPOSED OF BY SEVERAL ROUTES: BURNING, FLUSHING, LAND FILL, DISPOSAL SERVICE AND RECLAIMER. THIS SECTION LOOKS AT THESE ROUTES GIVING THE NUMBER AND PERCENT OF PLANTS USING THE VARIOUS DISPOSAL ROUTES. THE QUANTITY OF SOLVENTS DISPOSED OF BY THESE ROUTES IS ALSO REPORTED.

EXHIBIT III-A: THIS TABLE INDICATES THE NUMBER OF PLANTS USING THE VARIOUS DISPOSAL ROUTES AND THE PERCENT EACH IS OF THE TOTAL. (IT DOES NOT TOTAL 100% SINCE SOME PLANTS MAY USE MORE THAN ONE ROUTE). THE LARGEST METHOD OF DISPOSAL IS DONE WITH A DISPOSAL SERVICE AT 39% OR 7,989 PLANTS. ONLY 2% OF THE PLANTS BURN THEIR SOLVENTS AS THEIR METHOD OF DISPOSAL.

Exhibit VIII-A

DISPOSAL ROUTES FOR SOLVENTS

DISPOSAL ROUTE:	NUMBER OF PLANTS	PERCENT OF PLANTS
BURNING	382	2
FLUSHING	2,667	13
LAND FILL	3,674	18
DISPOSAL SERVICE	7,989	39
RECLAIMER	4,323	21

EXHIBIT VIII-B: Although more plants use a disposal service, the largest quantity of solvents goes to reclaimers. The average gallons of solvents per plant going to a reclaimer is 2,509 gallons per year.

By totaling the gallons per month disposed and the quantity of solvents used, the following relationship can be seen:

	Gal/Month
Quantity of Solvents Used:	4,832,000
Quantity of Solvents Disposed:	1,706,000
Quantity Lost	3,126,000

65% of the solvent used does not go to one of the disposal routes.

EXHIBIT VIII-B

QUANTITY OF SOLVENT BY DISPOSAL ROUTES

	THIS MANY PLANTS ...	... DISPOSE OF THIS MUCH SOLVENT		
		GALLON/MONTH $(x\ 10^3)$	GALLON/YEAR $(x\ 10^3)$	AVERAGE GALLON/YEAR PER PLANT
... BY THIS ROUTE:				
BURNING	382	8	96	251
FLUSHING	2,667	112	1,344	504
LAND FILL	3,674	135	1,620	441
DISPOSAL SERVICE	7,989	547	6,564	822
RECLAIMER	4,323	904	10,848	2,509

EXHIBIT VIII-c: THIS TABLE LISTS THE PROJECTED NUMBER OF PLANTS IN EACH GROUP USING THE VARIOUS DISPOSAL ROUTES. SOME AREAS ARE LEFT BLANK DUE TO AN INSUFFICIENT SAMPLING TO MAKE A PROJECTION.

DISPOSAL ROUTES FOR SOLVENTS

THIS MANY PLANTS IN THESE S.I.C. GROUPS ...

	TOTAL	25 METAL FURNITUR	33 PRIMARY METALS	34 FABRICTD PRODUCTS	35 NON-ELEC MACHINRY	36 ELECTRIC EQUIPMNT	37 TRANSPTN EQUIPMNT	38 INSTRMTS & CLOCKS	39 MISC INDUSTRY
	20,320	399	1,417	4,804	6,188	3,616	1,702	1,590	604
USE THESE DISPOSAL ROUTES:									
BURNING	382	18	54	56	132	56	66	-	-
FLUSHING	2,667	19	156	617	840	533	278	224	-
LAND FILL	3,674	114	276	965	1,319	481	281	101	137
DISPOSAL SERVICE	7,989	114	630	1,959	2,382	1,366	534	867	137
RECLAIMER	4,323	171	319	927	870	1,147	287	410	192

EXHIBIT VIII-c

EXHIBIT VIII-D: THE PERCENT OF PLANTS USING THE VARIOUS DISPOSAL ROUTES IN THIS TABLE SHOWS THE USE OF BURNING TO BE FAIRLY CONSTANT AMONG THE INDIVIDUAL S.I.C. GROUPS. A SMALLER PERCENTAGE OF PLANTS IN METAL FURNITURE USE FLUSHING AS A MEANS OF DISPOSAL. WHILE THE USE OF LAND FILL IS WIDELY USED IN GROUP 25 AND LITTLE USED IN GROUP 38, 55% OF THE PLANTS IN GROUP 38 USE A DISPOSAL SERVICE AND ONLY 14% USE THIS SERVICE IN GROUP 33. THE LARGEST PERCENT OF RECLAIMING IS DONE IN GROUP 25.

DISPOSAL ROUTES FOR SOLVENTS

THIS PERCENT OF PLANTS IN THESE S.I.C. GROUPS...

	TOTAL	25 METAL FURNITUR	33 PRIMARY METALS	34 FABRICTD PRODUCTS	35 NON-ELEC MACHINRY	36 ELECTRIC EQUIPMNT	37 TRANSPTN EQUIPMNT	38 INSTRMTS & CLOCKS	39 MISC INDUSTRY
	20,320	399	1,417	4,804	6,188	3,616	1,702	1,590	604
. USE THESE DISPOSAL ROUTES:									
BURNING	2	5	4	1	2	2	4	-	-
FLUSHING	13	5	11	13	14	15	16	14	-
LAND FILL	18	29	19	20	21	13	17	6	23
DISPOSAL SERVICE	39	29	14	41	38	38	31	55	23
RECLAIMER	21	43	23	19	14	32	17	26	32

EXHIBIT: VIII-D

EXHIBIT VIII-E: THE NUMBER OF PLANTS USING THE VARIOUS DISPOSAL ROUTES ACCORDING TO PLANT SIZE REFLECTS THE CATEGORIES CONTAINING THE MOST PLANTS, IN TURN, HAVE THE GREATEST NUMBER OF PLANTS USING THE VARIOUS DISPOSAL ROUTES.

EXHIBIT VIII-E

DISPOSAL ROUTES FOR SOLVENTS

THIS MANY PLANTS EMPLOYING ...

	TOTAL	20-100 PEOPLE	100-500 PEOPLE	50+ PEOPLE
	20,320	11,319	6,603	2,398
... USE THESE DISPOSAL ROUTES:				
BURNING	382	182	128	72
FLUSHING	2,667	1,627	889	151
LAND FILL	3,674	2,487	976	211
DISPOSAL SERVICE	7,989	4,207	2,798	984
RECLAIMER	4,323	2,062	1,666	595

EXHIBIT VIII-F: IT CAN BE SEEN THAT THE LARGER THE PLANT, THE BETTER THE PRACTICE OF SOLVENT DISPOSAL. THE USE OF BURNING, A DISPOSAL SERVICE AND A RECLAIMER INCREASES WITH PLANT SIZE. WHILE THE PRACTICE OF USING LAND FILL AND FLUSHING DECREASE AS PLANT SIZE INCREASES.

EXHIBIT VIII-F

DISPOSAL ROUTES FOR SOLVENTS

THIS PERCENT OF PLANTS EMPLOYING...

	TOTAL	20-100 PEOPLE	100-500 PEOPLE	50+ PEOPLE
	20,320	11,319	6,603	2,398
... USE THESE DISPOSAL ROUTES:				
BURNING	2	2	2	3
FLUSHING	13	14	13	6
LAND FILL	18	22	15	9
DISPOSAL SERVICE	39	37	42	41
RECLAIMER	21	18	25	25

EXHIBIT VIII-G: THIS TABLE LISTS THE NUMBER OF PLANTS IN EACH GEOGRAPHIC REGION USING THE VARIOUS DISPOSAL ROUTES. THE GREATEST NUMBER OF PLANTS BURNING APPEAR IN THE NORTHEAST REGION. THE NORTHEAST AND MID-WEST WITH THE LARGEST NUMBER OF TOTAL PLANTS HAVE THE LARGEST NUMBER OF PLANTS USING A DISPOSAL SERVICE OR A RECLAIMER.

DISPOSAL ROUTES FOR SOLVENTS

	OF PLANTS LOCATED IN THE ...						
	TOTAL	NORTHEAST	SOUTHEAST	MID-WEST	SOUTHWEST	NORTH CENTRAL	FAR WEST
	20,322	6,970	1,878	6,412	745	1,386	2,929
USE THESE DISPOSAL ROUTES:							
BURNING	382	200	36	73	37	18	18
FLUSHING	2,667	927	359	700	170	208	303
LAND FILL	3,674	880	457	1,263	249	364	459
DISPOSAL SERVICE	7,989	2,837	444	2,663	405	403	1,177
RECLAIMER	4,323	1,507	139	1,388	178	218	892

EXHIBIT VIII-G

EXHIBIT VIII-H: THE PERCENT OF PLANTS GIVE A BETTER PICTURE OF THE DISPOSAL ROUTES USED IN THE DIFFERENT GEOGRAPHIC LOCATIONS. THE SOUTHWEST HAS THE GREATEST PERCENT OF PLANTS USING DISPOSAL ROUTES. RECLAIMING IS DONE IN THE LARGEST PERCENTAGE OF PLANTS IN THE FAR WEST AND THE LEAST IN THE SOUTHEAST. THE SOUTHEAST ALSO IS LOW ON THE USE OF A DISPOSAL SERVICE, AND ABOVE AVERAGE ON THE USE OF LAND FILL AND FLUSHING. THE NORTHEAST AND FAR WEST APPEAR TO USE THE BEST DISPOSAL PRACTICES.

DISPOSAL ROUTES FOR SOLVENTS

THIS PERCENT OF PLANTS LOCATED IN THE ...

	TOTAL	NORTHEAST	SOUTHEAST	MID-WEST	SOUTHWEST	NORTH CENTRAL	FAR WEST
	20,322	6,970	1,878	6,412	745	1,386	2,929
USE THESE DISPOSAL ROUTES:							
BURNING	2	3	2	1	5	1	1
FLUSHING	13	13	19	11	23	15	10
LAND FILL	18	13	24	20	33	26	16
DISPOSAL SERVICE	39	41	24	42	54	29	40
RECLAIMER	21	22	7	22	24	16	30

EXHIBIT VIII-H

IX

SURVEY PROCEDURES

THE RESULTS REPORTED IN THE ACCOMPANYING TABLES ARE DERIVED FROM PERSONAL INTERVIEWS CONDUCTED WITH A RESPONSIBLE PERSON AT EACH OF 2,578 PLANT SITES ENGAGED IN A MANUFACTURING ACTIVITY WITHIN ONE OF THE METALWORKING INDUSTRIES. ALL INTERVIEWS WERE CONDUCTED BY TELEPHONE. THE FINDINGS MUST BE INTERPRETED WITHIN THE LIMITS SUGGESTED BY THE FOLLOWING DETAILS:

THE STUDY POPULATION: MANUFACTURING LOCATIONS WITHIN THE EIGHT INDUSTRIAL CLASSES COMPRISING THE METALWORKING INDUSTRY, AT WHICH 20 OR MORE PEOPLE ARE EMPLOYED, WERE DESIGNATED AS THE POPULATION FOR STUDY. (THE EIGHT INDUSTRIAL CLASSES ARE THOSE NAMED IN THE VARIOUS TABLES.) A PRESUMED COMPLETE LISTING OF THIS POPULATION WAS OBTAINED FROM THE CHILTON MARKET/PLANT DATA BANK, CHILTON COMPANY, RADNOR, PENNSYLVANIA. THE LIST NAMED 41,670 MANUFACTURING PLANT SITES. (ALSO INCLUDED IN THE LIST WERE A NUMBER OF NON-MANUFACTURING LOCATIONS AND DISTRIBUTORS. THESE WERE IGNORED.)

SAMPLING THE POPULATION: THE OBJECTIVE OF SAMPLING WAS TO OBTAIN AN ADEQUATE REPRESENTATION OF THAT SEGMENT OF THE METALWORKING INDUSTRY WHICH MAKES USE OF EITHER CHLORINATED OR PETROLEUM SOLVENTS IN SOME TYPE OF METAL CLEANING OPERATION. ON THE BASIS OF PRIOR KNOWLEDGE WE GUESSED THAT SOMEWHAT LESS THAN 50% OF THE STUDY POPULATION MIGHT BE INCLUDED IN THIS SEGMENT. FOR THE PURPOSES OF THE ANALYSIS WE SOUGHT TO COMPLETE APPROXIMATELY 1,000 INTERVIEWS WITHIN THE SEGMENT. WE THEREFORE DETERMINED TO DRAW A SAMPLE OF 2,300 SITES FOR SCREENING.

THE POPULATION LIST WAS FIRST ORDERED IN TERMS OF THE STANDARD INDUSTRIAL CLASSIFICATION OF EACH PLANT, AND FOR NUMBER OF EMPLOYEES WITHIN INDUSTRIAL CLASS. A NEW LIST WAS MADE OF EVERY 18TH PLANT NAMED IN THE REORDERED POPULATION LIST. THIS WAS DESIGNATED AS THE "PRIMARY SAMPLE". AT THE SAME TIME, PARALLEL LISTINGS OF EVERY 19TH AND EVERY 20TH PLANT WERE CREATED AND WERE DESIGNATED AS "ALTERNATIVE SAMPLE #1 OR #2". BECAUSE OF THE WAY THE ORIGINAL POPULATION LIST HAD BEEN ORDERED, A PLANT IN EITHER OF THE ALTERNATIVE SAMPLES WAS SIMILAR TO THE CORRESPONDING PLANT IN THE PRIMARY SAMPLE IN BOTH SIZE AND TYPE OF MANUFACTURING ACTIVITY. IF, FOR ANY REASON, AN INTERVIEW COULD NOT BE COMPLETED WITH A SPECIFIC PLANT IN THE PRIMARY SAMPLE, AN INTERVIEW WAS ATTEMPTED WITH THE CORRESPONDING PLANT IN ALTERNATIVE SAMPLE #1. IF THIS FAILED, ANOTHER ATTEMPT WAS MADE WITH THE CORRESPONDING PLANT IN ALTERNATIVE SAMPLE #2.

THE CHOICE OF SPOKESMEN FOR THE PLANT SITES: IN PLANTS EMPLOYING BETWEEN 20 AND 100 PEOPLE, THE SPOKESMAN SOUGHT WAS THE OWNER OR GENERAL MANAGER; IN PLANTS EMPLOYING BETWEEN 100 AND 500 PEOPLE, THE SPOKESMAN SOUGHT WAS THE PLANT MANAGER; AND IN PLANTS EMPLOYING MORE THAN 500 PEOPLE, THE SPOKESMAN SOUGHT WAS THE PRODUCTION SUPERVISOR OR PRODUCTION SUPERINTENDENT. IN ANY CASE, IF THE DESIGNATED PERSON SEEMED NOT TO HAVE THE INFORMATION WE WERE SEEKING (I.E., ANSWERED "DON'T KNOW" TO CERTAIN KEY QUESTIONS), HE WAS ASKED TO NAME SOMEONE ELSE WHO MIGHT BE KNOWLEDGEABLE IN THE AREA OF THE INQUIRY. THIS REQUEST USUALLY PRODUCED A REFERRAL TO A SUBORDINATE MORE FAMILIAR WITH THE DETAILS OF THE MANUFACTURING OPERATIONS (AS DISTINGUISHED FROM THE BUSINESS OPERATIONS) OF THE PLANT.

THE INTERVIEW: A DETAILED INTERVIEWING GUIDE WAS DRAWN UP BY MEMBERS OF THE NATIONAL MARKETING SURVEYS STAFF IN CO-OPERATION WITH THOSE MEMBERS OF THE INORGANIC CHEMICALS DEPARTMENT WHO WERE KNOWLEDGEABLE ABOUT THE OBJECTIVES OF THIS STUDY. THE INTERVIEWS WERE CONDUCTED BY SKILLED INTERVIEWERS OF NATIONAL MARKETING SURVEYS, WHO HAD BEEN THOROUGHLY BRIEFED ABOUT THE INTERVIEWING REQUIREMENTS AND PROCEDURE FOR THIS STUDY. ALL INTERVIEWS WERE COMPLETED BETWEEN JANUARY 9 AND FEBRUARY 3, 1975.

EDITING OF COMPLETED INTERVIEWS: IN THE COURSE OF THE INTERVIEW THE SPOKESMEN WERE ASKED TO NAME THE SPECIFIC SOLVENTS USED IN HIS PLANT, AND TO STATE THE QUANTITIES USED AND DISPOSED OF BY VARIOUS ROUTES. RESPONSES CONCERNING SOLVENTS USED WERE MOST FREQUENTLY GIVEN AS BRAND NAMES. THESE WERE EDITED TO ONE OF THE FOURTEEN GENERIC CLASSES (NAMED IN THE TABLES) BY A MEMBER OF THE INORGANIC CHEMICALS DEPARTMENT. RESPONSES CONCERNING QUANTITIES COULD BE GIVEN EITHER IN TERMS OF "DRUMS", "POUNDS", OR "GALLONS". ALL RESPONSES WERE EDITED TO THEIR EQUIVALENT IN GALLONS. ALL QUANTITATIVE RESPONSES WERE EDITED TO REASONABLE INTERVALS, FOR EASE OF KEYPUNCHING AND LATER TABULATING AND ANALYSIS.

TABULATING AND PROJECTING TO POPULATION: THE RAW DATA WERE KEYPUNCHED AT NATIONAL MARKETING SURVEYS, AND CAST INTO SUMMARY TABLES AT THE COMPUTATIONS RESEARCH LABORATORY ACCORDING TO SPECIFICATIONS DRAWN UP BY THE NATIONAL MARKETING SURVEYS STAFF.

IN PROJECTING THE SAMPLE TO THE POPULATION, EACH INDUSTRIAL CLASS WAS TREATED SEPARATELY. A "MULTIPLIER" FOR EACH INTERVIEW AS DETERMINED BY DIVIDING THE POPULATION SIZE BY THE NUMBER OF INTERVIEWS COMPLETED WITHIN THAT POPULATION. NOT SURPRISINGLY, THE MULTIPLIER IN MOST CASES WAS APPROXIMATELY 18. HOWEVER, FOR MOST QUESTIONS WITHIN THE QUESTIONNAIRE THE PLANT'S SPOKESMAN WAS ALLOWED THE OPTION OF REPLYING "I DON'T KNOW". IN SUCH CASES HE WAS ASKED TO PROVIDE HIS "BEST ESTIMATE", BUT IN SOME CASES THE SPOKESMAN WAS UNABLE TO ESTIMATE OR HAD NO BASIS FOR AN ESTIMATE. THESE "DON'T KNOWS" WERE DROPPED FROM THE NUMBER OF COMPLETED INTERVIEWS IN THE TREATMENT OF THE QUESTIONS INVOLVED. IN EFFECT, THIS PROCEDURE IS IDENTICAL TO MAKING THE ASSUMPTION THAT WHAT <u>IS</u> <u>NOT</u> KNOWN BY THOSE WHO DON'T KNOW IS THE SAME AS WHAT <u>IS</u> KNOWN BY THOSE WHO DO KNOW.

THIS STUDY WAS DESIGNATED AS PROJECT #221 IN NATIONAL MARKETING SURVEYS, AND ALL MATERIALS RELEVANT TO ITS EXECUTION ARE MAINTAINED UNDER THAT FILE NUMBER.

APPENDIX B

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Review of Analytical Methods and Techniques

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Prepared For:

Emission Standards and Engineering Division
Office of Air Quality Planning
U.S. Environmental Protection Agency

TITLE

REVIEW OF ANALYTICAL METHODS AND TECHNIQUES - SUB TASK 2 OF PROJECT PLAN FOR EPA CONTRACT NO 68-02-1329

INFORMATIVE SUMMARY WITH CONCLUSIONS

An EPA contract to conduct a "Study to Support New Source Performance Standards for Industrial Degreasing Operations" has been awarded The Dow Chemical Company. As part of this contract a review of analytical methods and techniques for sampling and analyzing industrial degreasing solvents emissions from stationary sources has been conducted. Literature sources include EPA methodology, ASTM, ISC, instrument manufacturers literature, books and journals.

This report describes how a sampling and measurement task should be undertaken to obtain the best information possible from current public technology. These steps for an analytical chemist are:

1. Obtain preliminary information
2. Choose analytical technique and sampling methodology
3. Prepare and implement choices
4. Conduct flowrate measurements
5. Complete the analysis and suitable report.

Review of Analytical Methods and Techniques - Sub Task 2 of EPA Contract "Study to Support New Source Performance Standards for Industrial Degreasing Operations."

I. Preliminary Information

In a pre-sampling visit to a customer the analytical chemist should gather much information. A general knowledge of the process or operation, the variety of solvents used, the physical arrangement of the venting systems and control systems, cost factors, local legislative requirements, and the nature of the data required by the customer will guide the choice of sampling and analytical techniques to be employed. The ultimate choice of techniques may be a compromise resulting from the above factors plus the expertise of the analytical chemist. Three approaches are available:

1. Analytical technique for specific compounds
2. Analytical technique for general organics
3. Techniques when the analytical facilities are remote from the sampling site.

However, flowrate measurement techniques and compatable sampling methodology must accompany any analytical approach.

II. Flow Rate Measurements

The emission rate of a compound from a stationary source is the product of the volumetric flow rate and the concentration (by volume) If stationary sources are to include exhaust fans and hood vents with blowers, flowrate measurements and data should also be included.

Flow Rate Measurement References

"Determination of Stack Gas Velocity and Volumetric Flow Rate" Method 2 Federal Register 36 (247) 24884 Dec. 23, 1971.

Average Velocity in a Duct (Pitot Tube Method) D3154 ASTM Annual Standards Part 26 pp 642-652 1974.

Average Velocity in a Duct (Thermal Anemometer Method) Method being drafted in ASTM Committee D-22.

Rotometer Calibration D3195
ASTM Annual Standards Part 26 pp 690-693, 1974

Air Sampling Instruments
ACGIH 4th Ed. 1972 Section B

III Sampling Methodology

For on-site analysis the analyzer can be piped up on a semi-permanent basis, samples can be brought to the analyzer, or the analyzer can be taken to the sampling point (SAFETY). If a minimum number of sampling points are needed and can be monitored individually for a period of time, a sample transport line can be installed between the sampling point and the analyzer. If a large number of sampling points are to be examined simultaneously or in a timed sequence, instantaneous or "grab" samples can be taken to the analyzer. If the sampling point is in a convenient and non-hazardous location, the analyzer could be used at the sampling point without long sample lines and many sample "grabbers". For remote sampling, see Section VI.

Sampling Methodology References

Stern, A.C., "Air Pollution" Vol. II 2nd Ed. Academic Press, New York, N.Y., 1968, Chapter 16, p. 3-54.

Sampling Atmospheres for Analysis of Gases and Vapors D1605 ASTM Annual Standards Part 26 pp 285-306, 1974.

Sampling and Storage of Gases and Vapors, Methods of Air Sampling and Analysis - Intersociety Committee APHA Part 1, pp 43-55 1972.

Air Sampling Instruments, ACGIH 4th Ed. Section A, 1972.

Ledbetter, J., "Air Pollution Analysis" Part A, Marcel Dekker Inc. New York, N.Y. 1972 Chapter 6, page 195.

IV. Analytical Technique for Specific Compounds

An instrumental analytical technique for specific compounds would be an easily movable gas chromatograph with a flame ionization detector (GC-FID), temperature programming capability and/or switching valves with a variety of analytical columns for the various groups of compounds. This analysis could be done on-site in an approved (safe) atmosphere.

This approach would probably be of "high" initial investment incorporating a preventive maintainance program and some initial selection of appropriate analytical columns along with a compilation of gas chromatographic operating conditions and retention times for compounds on various columns.

Gas Chromatography References
Gas Chromatograph Terms and Relationships E 355
ASTM Annual Standards Part 42 1974
General Gas Chromatography Procedure E 260 Ibid
Stern, A.C., "Air Pollution" Vol. II 2nd Ed.
Academic Press, New York, N.Y., 1968 Chap. 18 pp 120-145
Leithe, W., "The Analysis of Air Pollutants"
Ann Arbor-Humphrey, London, 1970 Chap. 4 pp 78-95

V. Analytical Technique for General Organic

Various detectors can be employed without sophisticated sample preparation to present a "total" measure of organic vapors or gases. These physical methods of detection include electric conductivity, coulometry, ionization, thermal conductivity, combustion, UV and IR spectroscopy, chemiluminescence, and flame photometry. As a detector alone they are not specific but can be calibrated for a single vapor or a known mixture. Many of these detectors are incorporated as "direct reading" instruments.

General Organic References
Total Hydrocarbons by GC-FID ISC 43101-02-71T
Methods of Air Sampling and Analysis - Intersociety Committee
APHA, Washington, D.C., 1972

"Flame Ionization Hydrocarbon Analyzer", R.A. Morris and R.L. Chapman; JAPCA 11 (10) 467-9 Oct. 1961

"Continuous Trace Hydrocarbon Analysis by Flame Ionization", A. J. Andreatch and R. Feinland; Anal. Chem. 32 (8) 1021-4 July, 1960

Stern, A. C., "Air Pollution," Vol II 2nd Ed.
Academic Press, New York, N.Y., 1968 Chap. 18 p 118.

Air Sampling Instruments
ACGIH, 4th Ed. 1972 Section U.

VI Remote Sampling

Because of some of the disadvantages of the portable gas chromatograph, such as high initial investment and the increased maintainance and training necessary, a second approach for the determination of specific compounds may be necessary. The remote sampling approach (i.e., taking a sample and transporting to a laboratory for specific quantitative analysis of the target compounds) may be a more practical approach for a wide range of solvent mixtures.

A disadvantage of the remote sampling approach would be the time lag between sampling and obtaining the finished results. There are, however, a number of advantages which would make this the preferred approach.

A number of different techniques have been used for taking remote samples. These are 1) whole air sample, 2) use of absorber solvents, and 3) solid adsorbents.

Whole air Samples. Whole air samples are taken by pulling the test air into an evacuated glass bulb, can, tube, syringe, etc., or by pumping the air into a container or plastic bag. Although these techniques may work well for many solvents when the samples can be analyzed the same day, such samples are more difficult to transport, may show loss of the compound due to adsorption, condensation, or diffusion over long periods and do not offer component concentration and therefore limit sensitivity.

Solvent Absorber. A widely used technique for the collection of organic vapor from air is the solvent absorber (K1, K2). The difficulty with this system is to find an absorber solvent which is compatable with all the compounds to be collected, low enough volatility so that it will not be lost during sampling, and it will not interfere with gas chromatographic analysis. One of the main disadvantages of this system is that many of the solvents used for collection cannot be mailed or transported by common carriers.

Solid Adsorbents. A number of solid adsorbents have been used for collection and concentration of organics. These adsorbents may be coated or non-coated and desorbed in the laboratory by heat or a suitable solvent. The advantages of solid adsorbents are:

1. Samples can be taken with existing equipment such as a small laboratory pump or portable battery-operated pump.
2. A minimum amount of time would be spent on-site by using the rapid sampling technique. Simultaneous samples can be taken with two or more pumps.
3. The samples could be transported easily or mailed back to the laboratory while the investigator continues with his sampling program.
4. A complete analysis can be made on the returned samples using the more versatile, sophisticated laboratory instrumentation.
5. The tubes containing the adsorbent can be prepared in advance and a relatively large supply can be carried with the investigator when traveling.

This technique generally involves pulling the test air through a small tube containing the adsorbent thereby collecting and concentrating. The tubes are then returned to the laboratory and the collected components are desorbed using either thermal or solvent techniques.

THERMAL DESORPTION OF SOLID ADSORBENTS

Thermal desorption necessitates the collection of organic vapors on a solid adsorbent contained in a rigid tube i.e. stainless steel. The tube is heated and back-flushed with carrier gas directly into a gas chromatograph. This technique offers very high sensitivity since the entire collected sample is injected and is advantagous for repetitive analysis of low concentrations. Difficulty may arise when a mixture of components especially with a wide volatility range is sampled. The adsorbent must be able to collect efficiently all vapors and efficiently desorb with heat.

Special equipment may be necessary to interface the collection tube to the gas chromatograph. The Bendix HS-10 Flasher is a commercially available unit that utilizes this technique.

SOLVENT DESORPTION OF SOLID ADSORBENTS

When samples are analyzed by solvent desorption, the adsorbent is removed from the tube and shaken with the solvent. The extract can then be analyzed. The advantages of solvent desorption are:

1. Repeated analysis can be made on the sample using different chromatographic conditions and columns.
2. Other techniques such as infrared can be used on the same sample.
3. No special additional equipment is necessary to analyze the samples by gas chromatography.
4. The desorbed samples can be set up with available automatic injection systems which can run a large number of samples with a minimum of attention.
5. The desorbed sample can be saved for a period of time in case additional analysis is required.

TYPES OF SOLID ADSORBENTS

The most widely used adsorbents are silica gel and charcoal, however, many other adsorbents have been reported for specific sampling problems.

SILICA GEL (References G1 - G14)

Good collection and recovery for a large number of organic vapors has been reported. Many variations as to the size of the collection tube, mesh size and type of silica gel, collection volumes and desorption solvents have been used. Table II in a publication by Feldstein et al. (G6) (Copy attached) indicates the collection and recoveries of a number of organic vapors.

The adsorption of water by silica gel is a serious problem in that high concentration of water deactivates silica gel and reduces the collection efficiency. However, with the proper balance of sampling rate, time and sample tube size, good recoveries may be obtained.

In addition to the desorption solvents described by Feldstein, ethanol, methanol, acetone, diethylether, and combinations of these solvents have been used with success.

The main difficulty arises in selecting a solvent which will give high desorption for all the organics collected and still not interfere with the analysis. The most troublesome class of compounds is the volatile, polar compounds. Methanol can be used to desorb most organic solvents efficiently, but then methanol itself cannot be determined. Feldstein has suggested dimethylsulfoxide as a solvent for polar compounds but no data is given for methanol.

CHARCOAL (References H1 - H10)

A review written by Mueller and Miller (H 10) effectively summarizes the use of charcoal for sampling organic vapors in air. A copy of this report is attached. The technique employed is based on work of Kupel and coworkers at the National Institute for Occupational Safety and Health (NIOSH) (H 6).

Good collection and recovery has been obtained for many organic vapors, however, several important facts must be considered.

1. Most of the testing has been done at concentrations relative to the recommended OSHA limits. The concentrations found in source emissions may be many factors higher and the use of larger tubes and smaller sample volumes may be necessary. For example, methylene chloride was found to break through the small NIOSH tube at 500 ppm when collected at the recommended rate of one liter per minute for ten minutes.
2. Carbon disulfide does not readily displace all organic compounds from carbon. The total recovery for volatile polar compounds is generally low, for example; methanol 10%, ethanol 67%, butanol 75%, and diacetone alcohol 60%.

ACTIVATED ALUMINA

Although there may be some difficulties, either silica gel or charcoal could be used to collect the solvents of interest except perhaps the volatile, polar compounds. Activated alumina has been suggested as a general adsorbent in some references, but no literature has been found which describes its practical use for ambient air testing. Recently it has been shown (M 26) that activated alumina will efficiently collect volatile polar organics and that the organics can be desorbed with water with high recovery. The water extract is then injected directly into a gas chromatograph for analysis.

VII DISCUSSION FOR REMOTE SAMPLING

From the preceding information, it appears that by using an adsorption tube packed with silica gel, charcoal, activated alumina, or other suitable adsorbent, or a combination of the tubes, all the organic solvents of interest can be collected and recovered with good efficiency. Gas chromatography can be used for analyzing mixtures of the solvents. Stoddard-type solvents which are composed of a mixture of hydrocarbons within a specified boiling range, present a special problem. This type of solvent can be determined by gas chromatography by selecting the strongest peaks of the mixture for calculation or by integrating the total area for the boiling point range. This type of solvent could also be determined by infrared by measuring absorption at 3.4 microns. In either case a standard of the actual solvent being used would be necessary to obtain satisfactory results. Interference corrections for other solvents present would be necessary.

It is difficult at this point to recommend one specific collection and analysis method for all compounds of interest. In practice, a diverse but smaller number of compounds would be present at any specific degreasing operation. Before actual sampling is undertaken the possible compounds at that location should be determined from customers application, and from this information the proper type of sampling tubes could be selected. This information should also be supplied to the laboratory so that the proper analytical conditions can be selected and calibrated.

VIII SUMMARY FOR REMOTE SAMPLING

Recommended steps for designing a system for remote collection and subsequent analysis.

1. The types of solvents and mixtures used in various degreasing operations should be determined.
2. The concentration range of interest should be suggested for each solvent.
3. A recommended sampling tube system could be determined from the above list of solvent mixtures and concentrations, and from the recovery data in the literature.

4. The retention times for the solvents of interest should be compiled for a number of suggested gas chromatographic columns so that the proper analytical condition can be determined for all combinations and mixtures. See References J 1 - J 4.
5. Collection and recovery data should be determined in the laboratory for the expected mixtures and concentration ranges.

   This study should also include time storage data.

SILICA GEL REFERENCES

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ATMOSPHERIC DETERMINATIONS OF CHLORINATED HYDROCARBONS, ESPECIALLY THE DETERMINATION OF 1,1,1-TRICHLOROETHANE. Text in Japanese. Japan J. Ind. Health (Tokyo), 7 (5):25-28, May 1965. 5 refs.

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SAMPLING AND ANALYSIS OF AROMATIC HYDROCARBON VAPORS IN AIR: A GAS-LIQUID CHROMATOGRAPHIC METHOD. Am. Ind. Hyg. Assoc. J. 25 (5) 464-9 Oct. 1964.

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GAS CHROMATOGRAPHIC ANALYSIS

J-1

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Application Bulletin 801

MODEL 800-VTP AIRFLOW MULTIMETER

A basic tool to help you meet the "Occupational Safety and Health Standards" (OSHA)

MEASURES
GAS FLOW
PARAMETERS:

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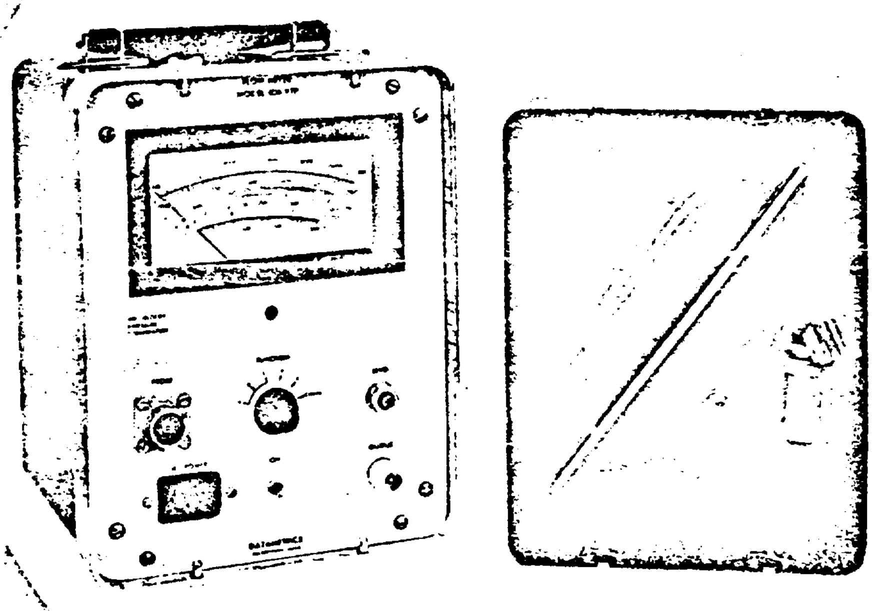

This Application Bulletin illustrates simple methods to measure a number of Ventilation Requirements in accordance with Subpart G, Section 1910 of the Rules and Regulations of the Occupational Safety and Health Standards.

(FIRST PAGE ONLY)

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SAMPLING SYSTEM GUIDELINES

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ABSTRACT

This paper covers basic design factors for typical sampling problems. Guidelines are given to provide the instrument engineer with a checklist of items to consider when designing sample handling systems. The guidelines are also useful to aid communication between instrument engineer and process engineer. Data is presented on system time lags and flow rates required for given lag times with various size sample lines. Several examples are given showing proper and improper installations.

INTRODUCTION

Much has been written on the subject of sampling. It's very interesting to read some of the earlier papers and see the progression of sampling ideas and methods. Recently, there have been several excellent papers given that cover specific sampling problems. This paper is intended to give general guidelines for approaching sampling problems.

STATEMENTS AND DEFINITIONS

First, let us cover some basic terms and state the problem a little more clearly. Analyzers and sampling systems are not in themselves important. Analyzers and sampling systems are only tools that make the analysis possible. The analysis cannot be accomplished without both a good analyzer and a good sampling system. The purpose of the sampling system is not to deliver to the analyzer a sample of the exact composition as that in the process line. The purpose is to deliver a representative sample suitable for the analyzer in which the variables to be measured vary according to the stream composition.

The sampling system and the analyzer should be complimentary of each other to accomplish the required analysis and not make impractical demands on either because of poor design in the other. All too often impractical and unnecessary demands are made of on-stream analyzer systems. Don't lower your necessary requirements but, "If you don't need it, don't do it". This is the basic philosophy of design to use throughout the analysis system.

Engineering neglect contributes to the problems of sampling. The specifications for analyzers are generally very detailed but the sampling system frequently receives only a casual mention.

A sampling system doesn't have to be high priced to be good, but it should be adequate to do the required job. This point will be shown throughout the discussion.

The requirements of an on-stream analysis (which involves both the analyzer and the sampling system) is to analyze the process stream:

1. as completely as required,
2. as fast as required,
3. as accurately as required,
4. and present the analysis in a desired and usable form.

Since the analyzer is so closely tied in with the sampling system, the analyzer location is very important. The proper location of the analyzer can sometimes lessen or even eliminate some of the difficult sampling problems and requirements. Some guidelines for locating the analyzer:

1. Locate as near as practical to the sampling point.
2. Locate where it will be convenient for servicing.
3. Locate where the required utilities are available.
4. Do not block accessibility to other equipment.
5. Provide adequate shelter from the elements.
6. Avoid locating in a hazardous working area or near hazardous equipment.

BASIC FUNCTIONS OF A SAMPLING SYSTEM

In view of the previous comments, now let us consider the sampling system portion of the analysis system. There are four basic functions required of the sampling system.

1. Obtain a representative sample from the process.
2. Condition the sample to make it suitable for the analyzer without modifying its basic characteristics.

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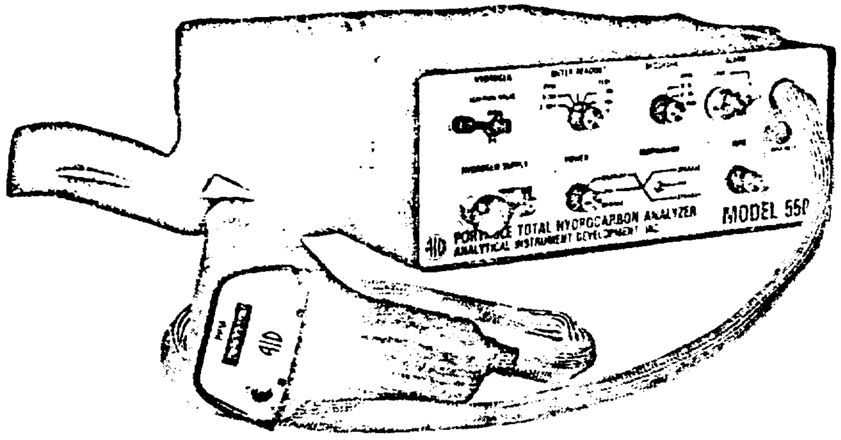
AID

MODEL 550 PORTABLE HEATED TOTAL HYDROCARBON ANALYZER



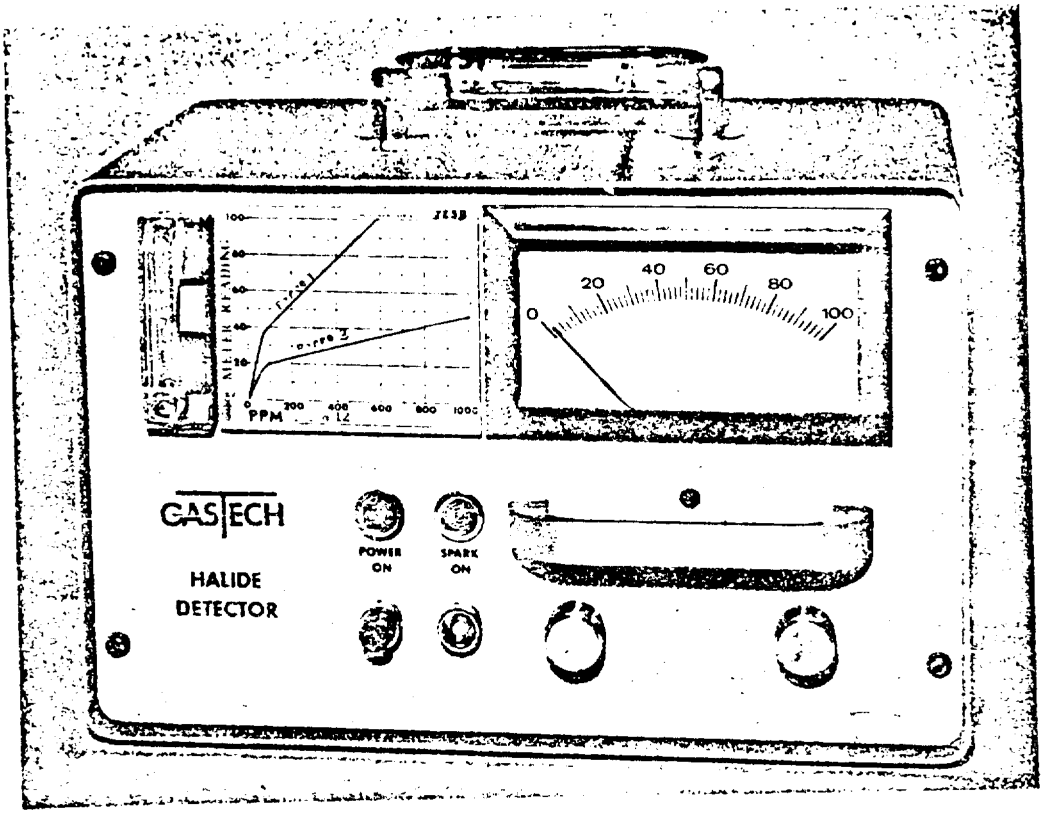
- **Gun-Type Analyzer Section**
- **Flame-Out Alarm.**
- **Minimum Detectable of 0.1 ppm.**
- **Recorder Output.**
- **Digital Readout in ppm.**
- **Completely Portable (self-contained).**
- **Heated Sample Probe and Detector.**
- **Adjustable Concentration Alarm System.**

The AID Model 550 is a completely portable HEATED Total Hydrocarbon Analyzer which determines the concentration of organic vapors in an air sample. An integral digital readout allows the operator to directly determine the concentration of the organic compounds. A range switch is used to select the readout to either 0-200 ppm or 0-2000 ppm full scale, with sensitivity to 0.1 ppm in the 0-200 ppm scale. Condensation of vapors is avoided on the Model 550 by having both the sampling probe and detector heated to above 70°C. An integral adjustable alarm system allows the operator to preselect a concentration which will activate an audible alarm system when the level is reached. In addition, a FLAME-OUT audible alarm is also built into the instrument.

For operator ease and comfort the Model 550 is designed in two sections. The first or analyzer section which is hand held consists of the heated probe and detector, supply batteries, ignitor and a digital readout. This is connected to the second or supply section by means of an umbilican cord which is used to supply power for heating purposes, vacuum for sampling and hydrogen for detector operation. The supply section contains the refillable hydrogen cylinder, pressure gauges and regulators, sample pump and rechargeable Nickel-Cadmium batteries. In addition, a jack is provided so that the signal may record continuously on a 2 millivolt recorder. There is sufficient power and hydrogen in the supply section to operate the instrument in the portable mode for at least 8 hours. The instrument may be operated indefinitely using A.C. line and an external hydrogen supply.

FIRST PAGE ONLY

THE Gas Tech HALIDE DETECTOR



A new portable instrument for detection and measurement of all airborne halogen compounds. Light weight and small size are attained by careful design of detection and flow systems, and by use of solid-state circuitry throughout.

DETECTION PRINCIPLE

The instrument depends upon the long-recognized fact that the ultraviolet spectral content of an electrical spark is increased by the presence of halogenated compounds, and that the degree of spectral enhancement is related to the concentration of halogens in the atmosphere surrounding the spark.

DESCRIPTION

A portable metal case with carrying handle has all the normal operating controls and indicators displayed on the front panel, and the 115AC power line and 10′ sample tube extend from the rear. Halide concentration is read out on a meter, graduated 0 - 100, and interpreted in terms of ppm of a specific compound by use of calibration curves.

A built-in diaphragm pump draws sample through the sample line and delivers it to the spark chamber, where the spark intensity is monitored by a photocell/UV filter combination. Cell output is amplified by a FET-input solid-state amplifier, and spark continuity is assured by a spark sensing and restartin circuit.

Recorder terminals are provided on rear of case, s that a continuous record can be made of variation in halide concentration. Zero stability is adequat for long term as well as short term monitoring.

APPLICATION

The instrument will give a useful reading over mos practical ranges of halogenated hydrocarbons, a used in research and industry. Threshold limit concentrations of carbon tetrachloride, trichloroethylene, chloroform and most other halogen-containin compounds are readily determinable on the meter yet expanded scales also permit readings up t 10,000 ppm. Response is a function of the type an number of halogen atoms, with greatest sensitivit obtainable on chlorine and fluorine compounds.

Primary field of application is by industrial hygienists in industrial, laboratory and government facilities. It will also be useful as a process monitor an as a leak detector in many industries where halogenated compounds are used; such as solvent degreasing, dry cleaning, painting, refrigeration an aerosol container plants.

Argus Supply Company
Industrial Safety & Fire Equipment
5940 CASMERE AVENUE
DETROIT, MICHIGAN 48212
(313) 892-8350

APPLICATION INFORMATION DATA

INSTRUMENT SENSITIVITY
MINIMUM DETECTABLE

GCAN 131

AID's Portable Gas Chromatographs have proven to be an effective tool in the Environmental Health and Air Pollution areas. Because of the varied types of analysis needed to be performed in these areas, the following table was prepared. Compounds are listed according to class. Each class is intended to give representative examples of what analysis detectability can be achieved with other compounds in the class. If the compound of interest is not present, it is reasonable to assume it will have sensitivity similar to a compound which is similar in structure. For further clarification AID should be contacted.

Each class is listed under the detector type which is suggested for that analysis. This does not mean to imply that the compound would not be detected with a differenct detector type, but that this detector type is the most widely used for that species.

In addition to minimum detectable (MD) in parts per million (ppm), the threshold level value (TLV) is stated in ppm. This gives the operator an indication as to analysis confidence. For example, in the case of Acetone with a TLV of 1000 ppm, minimum detectable of 0.2 ppm is unimportant. The table also lists suggested columns and column temperatures for the analysis of interest.

Several points should be made. The table is by no means meant to be all inclusive. It is representative of the type of compounds and the level of sensitivity that can be expected from a gas chromatographic analysis. The column and column temperature may be changed depending on the compound present in the sample to maximize system performance and eliminate interference. This analysis adjustment may result in a change in system response for an individual compound. It should be remembered that minimum detectable (MD) is affected by sample size and retention time. The following table illustrates compounds and conditions which give retention times of under 6 minutes and a sample size of 1 ml. in air.

FLAME IONIZATION DETECTOR MODEL 511

Material	TLV (ppm)	MD (ppm)	Column	Oven Tem
Ketones				
Acetone	1000	0.2	(1)	80°
Methyl Ethyl Ketone (2-Butanone)	200	0.2	(1)	80°
Methyl Isobutyl Ketone	100	0.10	(1)	80°
Methyl n-Butyl Ketone (2-Hexanone)	100	0.10	(1)	80°
2-Pentanone	200	0.10	(1)	80°
3-Pentanone	-	0.10	(1)	80°
Aromatics				
Benzene	10	0.05	(1)	80°
Toluene	200	0.04	(1)	80°
Xylenes (mixed isomers)	100	0.12 (p-xylene)	(1)	80°
Ethyl Benzene	100	0.1	(1)	80°
Chlorinated				
Methyl Chloride	100	0.11	(2)	140°
Ethyl Chloride	1000	0.08	(2)	140°
Methylene Chloride (Dichloromethane)	500	0.2	(2)	140°
Chloroform	25	0.4	(1)	55°
Vinylidene Chloride	10	0.2	(1)	50°
Carbon Tetrachloride	10	0.5	(1)	55°
1,2 - Dichloroethane	-	0.05	(1)	55°
Methyl Chloroform (1,1,1,-Trichloroethane)	350	0.1	(1)	55°
Trichloroethylene	100	0.15	(1)	80°

FLAME IONIZATION DETECTOR MODEL 511

Material	TLV (ppm)	MD (ppm)	Column	Oven Tem
Chlorinated (Cont'd)				
Tetrachloroethylene	100	0.3	(1)	80°
Vinyl Chloride	1.0	0.05	(2)	125°
Freon 12 (Difluorodichloromethane)	1000	0.62	(1)	80°
Freon 114 (1,2 Dichlorotetrafluoroethane)	1000	0.26	(1)	80°
Freon 11 (Fluorotrichloromethane)	1000	0.85	(1)	80°
Acetates				
Ethyl Acetate	400	0.04	(10)	100°
i-Propyl Acetate	250	0.03	(10)	100°
n-Propyl Acetate	200	0.05	(10)	100°
n-Butyl Acetate	150	0.09	(10)	125°
n-Amyl Acetate	100	0.09	(10)	125°
Alcohols				
Methanol	200	0.5	(2)	135°
Ethanol	1000	0.06	(2)	135°
1-Propanol	200	0.05	(10)	125°
2-Propanol	400	0.04	(10)	125°
1-Butanol	100	0.06	(10)	125°
2-Butanol	100	0.06	(10)	125°
1-Pentanol	-	0.09	(10)	150°
2-Pentanol	-	0.05	(10)	150°
3-Pentanol	-	0.05	(10)	150°
2-Hexanol	-	0.05	(10)	150°
3-Hexanol	-	0.07	(10)	150°

FLAME IONIZATION DETECTOR MODEL 511

Material	TLV (ppm)	MD (ppm)	Column	Oven Temp
Paraffins				
Methane	-	0.04	(2)	80°
Ethane	-	0.04	(2)	80°
Propane	1000	0.02	(2)	100°
i-Butane	-	0.02	(2)	130°
n-Butane	500	0.02	(2)	130°
n-Pentane	1000	0.01	(1)	60°
n-Hexane	500	0.02	(1)	60°
n-Heptane	500	0.02	(1)	80°
n-Octane	500	0.05	(1)	80°

FLAME IONIZATION DETECTOR W/CATALYSIS CHAMBER
MODEL 514 OR 511 OPTION 14

Material	TLV (ppm)	MD (ppm)	Column	Oven Temp
CO	50	0.3	(8)	140°
CH_4		0.2	(8)	140°
Total Hydrocarbon		.05	(9)	

FLAME PHOTOMETRIC DETECTOR MODEL 513

Material	TLV (ppm)	MD (ppm)	Column	Oven Temp.
SO_2	5	0.03	(3)	75°
H_2S	10	0.03	(3)	75°
COS		0.03	(3)	75°
CS_2	20	0.03	(3)	75°
Methyl Mercaptan	0.5	0.03	(3)	75°
Ethyl Mercaptan	0.5	0.06	(3)	100°
Dimethyl Sulfide		0.1	(3)	100°
Dimethyl Disulfide		0.1	(3)	100°

THERMAL CONDUCTIVITY DETECTOR MODEL 512 OR 511 OPTION 17

Material	TLV (ppm)	MD (ppm)	Column	Oven Temp.
Orsat type analysis				
CO	50	50	(4)	40°
CO_2	5000	200	(4)	40°
O_2	-	50	(4)	40°
N_2	-	200	(4)	40°
CH_4	-	40	(4)	40°
NO	25	70	(4)	40°
H_2	-	40	(5)	40°
NGAAA procedure				
Propane	1000	50	(6)	65°
Butane	500	50	(6)	65°
Propane	1000	200	(2)	100°
Butane	500	200	(2)	100°
Benzene	10	75	(1)	80°
Toluene	200	75	(1)	80°

ELECTRON CAPTURE DETECTOR MODEL 510 OR 511 OPTION 06

Material	TLV (ppm)	MD (ppm)	Column	Oven Temp
CCl_4	10	0.001	(1)	50°
Chloroform	25	0.02	(1)	50°
1,1,1, trichloroethane	350	0.005	(1)	50°
Chlorine	1	0.5	(3)	40°
Sulfur hexaflouride	-	0.0001	(8)	40°
Freon 112	500	0.001	(2)	
NITRATE ESTERS		0.005	(7)	125°
TNT		0.01	(7)	125°
Pesticides		0.005	(7)	190°

COLUMN REFERENCES

(1) 6' 10% DC200 on 80-100 mesh Chromosorb WHP

(2) 6' 80-100 mesh Chromosorb 102

(3) 6' 15% UCON 50 HB280X on 40-60 mesh Chromosorb T

(4) 2' 80-100 mesh Chromosorb 102 + 4' 45-60 mesh Molecular Sieve 13X

(5) Same as (4) except 2' 1/8" delay line on entrance of MS column

(6) 30' 27% DC 200 on 45-60 mesh Chromosorb P

(7) 6' 3% GC SE30 on 80-100 mesh Chromosorb W-HP in Glass

(8) 10' 45-60 mesh Molecular Sieve 5A

(9) No column. Sample direct to detector and corrected for "Zero" air Bl

(10) 6' 5% Carbowax 1540 on 80-100 mesh Chromosorb WHP

The Use of Silica Gel in Source Testing

M. FELDSTEIN, S. BALESTRIERI, and D. A. LEVAGGI

Bay Area Air Pollution Control District, 1480 Mission Street, San Francisco, California

This is a study of the adsorption of a large group of solvent vapors upon silica gel and their subsequent quantitative desorption. Esters, ketones, aromatic and aliphatic hydrocarbons, and halogenated hydrocarbons were among those studied. Except for certain low-molecular-weight hydrocarbons, the silica gel was extremely efficient for adsorbing organic solvents. In general, dimethylsulfoxide proved ideal for elution of adsorbed materials and for subsequent analysis by gas chromatography. It could not be used for hydrocarbon solvent mixtures or for higher boiling solvents. In these cases, carbon disulfide, alone or with water, was used for desorption.

Introduction

THE LITERATURE contains many references on the use of adsorbing materials for the collection of organic vapors. Charcoal,[1-3] silica gel,[4-6] and molecular sieves[7] are only a few of the adsorbing materials which have been suggested. Desorption has been accomplished by heating at elevated temperatures,[8] by using polar and nonpolar organic solvents,[1,4] and by using water and acid and alkaline solutions.[6] Analysis ranged from simple weighing[9] to complex instrumental methods including infrared,[8] gas chromatographic,[1] and chemical procedures.[4] Recovery data have shown values ranging from 0 to 100% for various types of organic materials.

This study was prompted by the need to collect and analyze organic vapors emitted by various industrial processes, including paint spraying and drying, lithographic operations, degreasing, drying cleaning, and petroleum refining operations. The range of organic vapors to be collected and analyzed included alcohols, esters, ketones, aromatic and aliphatic hydrocarbons, halogenated hydrocarbons, and a variety of other types of organic compounds. The study centered around two problems: the efficient collection of organic vapors on an adsorbing medium, and the quantitative removal of the adsorbed material for subsequent analysis.

Areation System

Prior to field testing of the procedure finally developed, a laboratory system was devised for the collection of organic vapors. The apparatus used is shown in Figure 1. Silica gel was chosen as the adsorbing material. Three probes (¾ inch × 6 inches) containing 20 gm of Davidson silica gel PA 400 (8 to 16 mesh, dried at 110° to 120°C for at least 2 hours prior to use) were arranged in series. The organic material was injected into the flask in quantities ranging from 25 to 50 μl, with the use of a constant-drive syringe pump. Larger volumes of sample (250 μl) were injected into the flask manually over a 5- to 10-minute period. The flask was heated by means of a heating mantle to 45° to 50°C. In the case of higher boiling materials the kettle temperature was raised to 120°C. Air was drawn through the kettle and the probes at a rate of 0.25 cfm for a period of 1 hour for a total of 15 cubic feet. The contents of the probes were then poured into separate 125-ml T/S stoppered Erlenmeyer flasks for desorption and analysis.

Desorption

A variety of desorbing materials was studied to determine the efficiency of desorption. Since infrared (IR) and gas-liquid-chromatographic (GLC) analytical procedures were

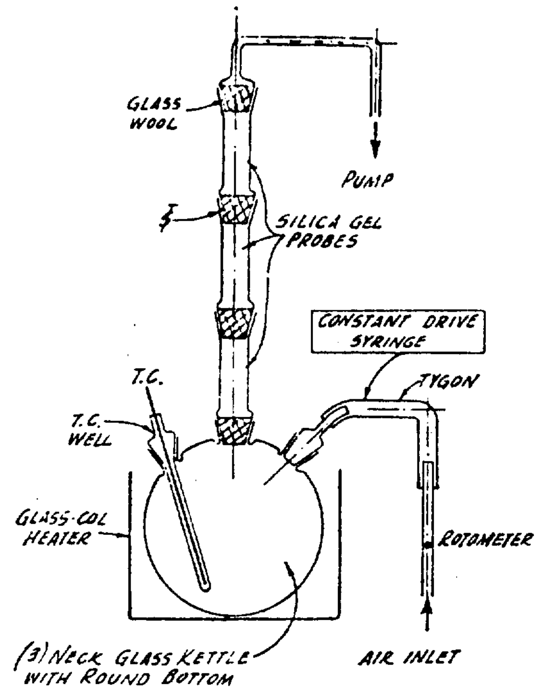


FIGURE 1. Apparatus used for adsorption of known concentrations of solvent vapors on silica gel.

to be used, it was necessary to select solvents compatible with the final analytical procedure. For gas chromatographic analyses, it was necessary to employ desorbing materials which did not interfere with the material being eluted. For infrared analysis, the solvent had to be nonabsorbing in the infrared region of interest (2 to 15 microns).

TABLE I
Gas Chromatographic Analytical Data
(4-foot Carbowax 20 M Column)

Compound	Temperature (C)	Approximate Flow Rate (ml/min)	Time for Peak (minutes)
Methyl acetate	100	78	1.05
Butyl acetate	100	78	2.8
Ethyl acetate	100	87	1.05
Vinyl acetate	100	75	1.2
Butyl Cellosolve acetate	100	90	20.0
Ethyl acrylate	100	75	1.65
Isopropyl alcohol	100	75	1.3
n-Butyl alcohol	100	75	3.6
Butyl Cellosolve	100	75	22.2
Benzene	100	61	0.9
Toluene	100	61	3.1
Xylene	100	61	5.9
Trichloroethane	70	81	2.65
Trichloroethylene	70	81	5.3
Tetrachloroethylene	70	81	6.5
Tetrachloroethane	70	81	8.1
Methylethyl ketone	100	87	1.2
Methyl isobutyl ketone	100	75	2.25
Dimethylsulfoxide	100	75	35.0

The choice of analytical procedure was determined by the nature of the collected material. Simple compounds like aromatic hydrocarbons, esters, and alcohols could be analyzed by GLC procedures, whereas complex materials like Stoddard solvent and V.M. P naphtha, which are mixtures of innumerable hydrocarbons of varying chain length, had to be analyzed by infrared procedures.

The desorbing procedure was as follows: To the flask containing the silica gel, 25 ml of desorbing solution was added. The flask was stoppered and allowed to stand for 2 hours with occasional shaking. The solvent was decanted from the gel and stored in a T/S stoppered test tube. Aliquots of the solvent were then taken for GLC or IR analysis.

Where CS_2 and H_2O were used as the desorbing material, the procedure was as follows: 25 ml of CS_2 was added to the flask contining the gel, as described above. After the two-hour period, 15 ml of water was added to the mixture in the flask. The flask was permitted to stand further for one hour with occasional shaking. The solvent was then filtered into a T/S stoppered test tube for subsequent analysis. Under these conditions, no water layer was present with the CS_2, since most of the water is retained on the silica gel.

Analytical Procedure

The GLC procedure consisted in injecting 1 to 5 μl of eluate into the gas chromatograph. A Beckman Model GC2A gas chromatograph with flame ionization detector was used. The column was a 4-foot × ¼-inch stainless-steel tube filled with 20% Carbowax × 20 M on firebrick 60/80 mesh. Carrier gas was N_2 at flow rates that varied depending on the compound being analyzed (Table I). Temperature was usually 100°C.

For IR analysis, a Beckman IR 4 spectrophotometer was used. The eluate was transferred to a 0.1-mm cell, and the absorption was determined at the wavelength of interest. For Stoddard-type solvents absorption at 3.4 microns was found to be proportional to concentration of solvent. For other solvents measured by IR spectrophotometry, the wavelength used depended on the absorption characteristics of the material. Thus the 5.7-micron band was used to measure concentration of ketones and esters. All IR analyses were done in CS_2 solution.

It was found that dimethylsulfoxide (DMSO) was an ideal solvent for elution of adsorbed materials from silica gel and for subsequent GLC analysis. Having a high boiling point, the DMSO peak appears after the appearance of the peaks of the solvent being studied. In two general cases, DMSO could not be used for GLC analyses. These included hydrocarbon solvent mixtures such as Stoddard and VMP naphtha, which contain many kinds of hydrocarbons, and higher boiling solvents which may appear with the DMSO peak. In these cases CS_2 and CS_2/H_2O mixtures were used for desorption, and analysis was accomplished by infrared.

Results of Analyses

The results of the experiments are shown in Table II. Analysis for each of the materials was performed separately on each silica gel probe. In all cases shown in the table, there were no solvents found on the second or third probes. Recovery figures shown for DMSO as an eluant indicate that all the material is adsorbed on the first probe. In field tests, where the concentration of solvent is unknown, and generally high, some of the materials were found on the second and third probes. Certain generalizations can be drawn from the table:

1. Carbon disulfide is a poor eluant for most of the materials studied. The notable exceptions are solvent mixtures such as Stoddard and naphtha and hydrocarbons of more than eight carbons.
2. Addition of water to the carbon disulfide in the eluting flask improves the elution of such compounds as toluene and xylene and certain halogenated hydrocarbons and ethyl acrylate. Poor recovery is still obtained with alcohols, ketones, most acetates, and some halogenated hydrocarbons.
3. DMSO is an efficient eluant for all the materials studied.

With the exception of certain low-molecular-weight hydrocarbons, the silica gel was ex-

TABLE II
Adsorption and Recovery of Solvents from Silica Gel

Solvent	Microliters injected	Recovery	
		Microliters	Percent
	Eluent — CS_2		
Ethyl acetate	125	—	—
	250	5	2
Vinyl acetate	250	13	5
Butyl acetate	125	48	38
	250	105	42
Methyl acetate	125	25	20
	250	53	21
Ethyl acrylate	250	12.5	5
Isopropyl alcohol	125	6	5
	250	—	0
n-Butanol	125	6	5
Butyl cellosolve	125	6	5
Benzene	100	5	5
	430	12	3
Toluene	100	39	39
	300	124	41
Xylene	100	38	38
	250	105	42
Stoddard[a]	100	98	98
	210	204	97
Naphtha[a]	100	95	95
	300	288	96
Trichloroethane	35	1.7	5
	100	5	5
Tetrachloroethylene	35	10.5	35
	100	35	35
Tetrachloroethane	35	27.5	79
	100	77	77
Methylethyl ketone	250	10	4
Methylisobutyl ketone	125	6.5	5
n-Octane	250	253	100.9
	Eluent — CS_2/H_2O		
Ethyl acetate	125	74	59
	250	152	61
Vinyl acetate	250	170	68
Butyl acetate	125	121	97
	250	244	98
Methyl acetate	125	41.5	33
	250	80	32
Ethyl acrylate	250	240	96
Isopropyl alcohol	125	6	5
n-Butanol	125	6	5
Butyl cellosolve	125	6	5
Benzene	100	95	95
	430	415	96
Toluene	100	98	98
	303	303	101
Xylene	100	97	97
	250	238	95
Stoddard[a]	100	97	97
	200	204	97
Naphtha[a]	100	93	93
	300	288	96
Trichloroethane	35	34	97
	100	95	95
Tetrachloroethylene	35	34	97
	100	98	98
Tetrachloroethane	35	34	97
	100	95	95
Methylethyl ketone	250	88	35
Methylisobutyl ketone	125	94	75
	Eluent — DMSO		
Ethyl acetate	75	70	93
	250	256	102
Vinyl acetate	250	243	97
Butyl acetate	250	244	97.6
Methyl acetate	250	251	100.2
Ethyl acrylate	250	240	96
Isopropyl alcohol	125	121	97
	250	240	96
n-Butanol	125	121	97
Butyl Cellosolve	125	121	96
Benzene	125	122.5	98
	430	425	99
Toluene	100	97	97
	300	303	101
Xylene	100	104	104
	250	258	103
Trichloroethane	100	96	96
Trichloroethylene	100	97	97
Tetrachloroethane	35	36	102
	100	96	96
Methylethyl ketone	250	242	97
Methylisobutyl ketone	125	123	93
n-Octane	250	250	100

[a] Infrared analysis.

TABLE III
Efficiency of Adsorption of Hydrocarbons on Silica Gel

Hydrocarbon	Efficiency of Adsorption (% adsorbed) First Probe	Second Probe	Carbon Probe
n-Pentane	<5	<5	90-100
n-hexane	<5	<5	90-100
n-heptane	15	60	20
n-Octane	90-100	<5	<5
n-Nonane	90-100	<5	<5
Cyclopentane	<5	<5	90-100
Cyclohexane	40-50	<5	90-100
Cyclohexene	90-100	<5	<5

TABLE IV
Effect of Moisture on Silica Gel Adsorption

Compound	First Probe	Second Probe	Third Probe
Isopropyl alcohol			
Dry air	95-100	<5	<5
Wet air	95-100	<5	<5
Butyl Cellosolve			
Dry air	95-100	<5	<5
Wet air	95-100	<5	<5
Ethyl acetate			
Dry air	95-100	<5	<5
Wet air	95-100	<5	<5
Perchloroethylene			
Dry air	95-100	<5	<5
Wet air	15	45	40
	14	20	40
	5	25	35

tremely efficient for adsorbing organic solvents. To test the efficiency of silica gel for C_5 to C_8 aliphatic hydrocarbons, a series of experiments was run in which the first two silica probes were followed by a charcoal probe. Elution of each of the probes was effected with CS_2 and CS_2 and water. The results are shown in Table III. From the table it can be seen that pentane, hexane, and heptane are poorly adsorbed on the first silica probe. Octane and higher hydrocarbons are efficiently adsorbed on the first silica probe. Among the cyclo compounds, cyclopentane and cyclohexane are poorly adsorbed, whereas cyclohexene is efficiently adsorbed on the first silica probe. Even though silica gel poorly adsorbs the lower molecular weight hydrocarbons such as pentane and hexane, the adsorption and recovery of such solvent mixtures as Stoddard and naphtha were excellent, as shown in Table II. This is primarily due to the fact that these solvent mixtures contain relatively small amounts of C_5 and C_6 compounds, and much larger quantities of C_8 and higher branched chain parafins, which are adsorbed by silica gel.

While charcoal appears to be quite efficient for adsorbing C_5 and C_6 hydrocarbons, the same affinity for the other solvents studied was noted. However, elution problems were much greater when carbon was used as the adsorbent. DMSO could not be used because of poor elution of organic compounds in some cases. Other eluants ($CHCl_3$, CCl_4, H_2O) varied widely in their efficiency to remove adsorbed materials. It was for these reasons that charcoal was not selected as the adsorbing material.

Effect of Excess Moisture

A series of aerations was run to determine the effect of moisture on the efficiency of adsorption of silica gel for various organic compounds. The aerating stream was saturated with water vapor at room temperature by passing the air through an impinger of water. Flow rates and times of aeration were the same as with previous aeration runs. The results are shown in Table IV. The only compound tested that was affected by excessive moisture in the air stream was perchloroethylene. In all the previous aerations, the relative humidity of the airstream was 40 to 55%.

It should be pointed out that in field studies, where sampling times may be much longer, and greater quantities of water may be present, the efficiency of adsorption of organic compounds is decreased. It is for this reason that three probes in series are used, rather than relying on a single probe. In actual field practice, sampling time is controlled so that excessive quantities of solvent are not collected. A simple rule of thumb is to sample until the third probe begins to become warm to the touch, indicating that adsorption is beginning to occur on the third probe.

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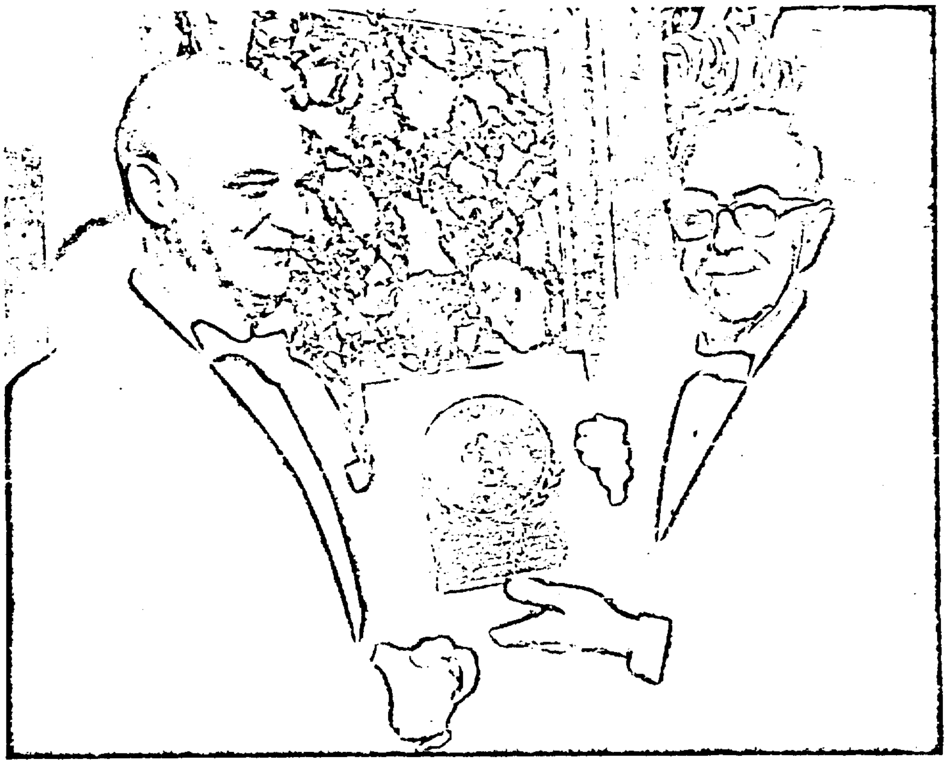


AIHA Presidents

At the annual banquet of the American Industrial Hygiene Association during the annual meeting in Chicago, Dr. Clyde M. Berry (right), President of AIHA for this year, presented to William T. McCormick, retiring President, a plaque symbolizing the appreciation of the American Industrial Hygiene Association for the leadership and devotion "Bill" has given to the Association over the years and particularly during his term in this office.

Determination of organic vapors in industrial atmospheres

SOLVENT VAPORS ARE ubiquitous components of the atmospheres in many manufacturing plants. Solvents used in degreasing, paint application, and other industrial processes contribute to this condition. Prior to 1970, solvent vapor concentrations in factory environments were voluntarily controlled to accepted limits, such as the threshold limit values of the American Conference of Governmental Industrial Hygienists. This control was exercised for worker safety as well as for the concomitant material economics. The Williams-Steiger Occupational Safety and Health Act of 1970 eliminated all options. Solvent vapor concentrations in the workplace came under statutory limits, and compliance with these OSHA limits became mandatory.

Semiquantitative solvent vapor concentration measurements have been made for many years utilizing manual sampling pumps and proprietary color indicator tubes. While such devices are useful as range-finding means, they usually lack the specificity, precision, and accuracy necessary to document worker exposure. In consequence of these limitations, a literature search was undertaken to uncover a suitably selective, quantitative method applicable to the needs of a large, multiplant manufacturing facility utilizing a variety of solvents.

Kupel et al.[1] had prepared a directly pertinent literature survey early in 1970. They found that difficulties in collection and concentration of air samples had long been an impediment to the successful evaluation of workplace solvent vapor concentrations. The gas chromatograph (GC) was found to be most often mentioned as the analytical tool, whereas sampling techniques ranged from solvent absorption to charcoal adsorption. Kupel and coworkers at the National Institute for Occupational Safety and Health (NIOSH) chose the charcoal adsorption technique of Otterson and Guy,[2] and then systematically evaluated collection efficiency, carbon disulfide desorption efficiency, and quantitative determination of 14 solvents by GC.[1] The sampling and analysis techniques developed by these workers were accepted by the Occupational Safety and Health Administration (OSHA) as standard methods for compliance investigations.[3] Kupel et al.[4] devised a technique for collection and identification of individual GC fractions applicable to the complex atmospheres likely to be encountered in factory situations. They utilized a mass spectrometer (MS) to identify the GC fractions which were individually collected in capillary charcoal traps.

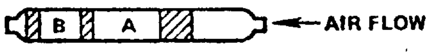
Two other methods have appeared in the literature which merit consideration; both portable infrared spectrophotometers[5] and portable gas chromatographs[6] have been used to good advantage by workers in field evaluations. Each of these methods requires a significant capital equipment outlay, which, in the case of our laboratory, would have duplicated more versatile, sophisticated laboratory instrumentation already available. Therefore, the decision was made to meld the techniques of the NIOSH researchers with existing laboratory instrumentation.

Experimental

Solvent vapors are collected on activated carbon, desorbed with a suitable agent, and separated by gas chromatographic procedures. Individual peaks appearing in the chromatograms are identified by retention times and/or by mass spectrometer analysis; concentrations are calculated from calibration curves.

Dr. Mueller is Chemist, Air Quality, and Mr. Miller is Environmental Scientist, Major Appliance Laboratories, General Electric Company.

SAMPLING TUBE, COMMERCIAL VERSION



A. MAIN SECTION FOR ANALYSIS, 100 MG. CARBON.
B. BACK-UP SECTION FOR TEST VALIDATION, 50 MG. CARBON.

Figure 1 *Charcoal tube employed to trap solvent vapors.*

Table 1

Sampling and elution

Sampling procedure using charcoal tubes

1. Remove plastic caps from charcoal sampling tube and break off both glass tips from the tube.
2. Install sampling tube in holder. The arrow on the tube indicates the correct direction of sample flow. *Note:* Keep tube vertical while sampling. Air should not pass through any tubing before entering carbon tube.
3. Adjust personal sampling pump so that the flow rate through the tube is 1 liter/min for quantitative samples, and at maximum flow for bulk qualitative samples.
4. When the desired sample volume (10 liters for quantitative analysis, 100–200 liters for qualitative analysis) has passed through the charcoal tube, shut off the sample flow. Remove the tube and reinstall the plastic caps to protect the sample until it is analyzed.
5. One sampling tube is opened and then resealed at the sampling site. No air is drawn through this tube, which serves as a blank.

Elution of solvents from charcoal sampling tubes

1. Remove carbon granules from sampling tube and place in glass-stoppered weighing bottles (15 X 50 mm). The two carbon sections, A and B (see Figure 1), are treated separately. (If organic material is detected in the B section, the test must be repeated using a smaller sample volume.)
2. Place weighing bottles in an ice bath.
3. Rapidly add 1 ml carbon disulfide* to each weighing bottle; then stopper tightly.
4. Agitate weighing bottles repeatedly for 30 min; alternatively, bottles may be agitated momentarily and then refrigerated overnight.

Sample collection and manipulation

The procedure for sampling in the field is given in *Table 1*. Collection tubes (*Figure 1*) may be prepared as described by Kupel et al.[1] or can be purchased from several sources (Mine Safety Appliance Co. and SKC, Inc.). Sampling tubes are returned to the laboratory for processing. Tightly sealed sampling tubes can be stored at, or below, room temperature for several days. Sample desorption is described in Table 1. The carbon disulfide solutions containing the desorbed contaminants should be analyzed without delay, and in no case should storage time exceed two days.

Component identification

Equipment and conditions employed for GC separation of eluted samples are given in *Table 2*.

Table 2

GC analysis

Apparatus

Varian Aerograph gas chromatograph model 1860-10 equipped with thermal conductivity and flame ionization detectors
Infotronics Corp. CRS 108 digital electronic integrator with Victor Digit Matic printer
Leeds and Northrup Speedomax XL dual-pen strip-chart recorder
Column (20 ft × ⅛ in.) stainless steel packed with 10% FFAP stationary phase on 80/100 mesh acid-washed DMCS Chromosorb W solid support for aromatic solvents. Column (5 ft × ⅛ in.) 3% SE-30 on 100/120 Varaport #30 for Polycat 8
Zero gases — hydrogen, helium, and air from Matheson Gas Products
Hamilton 700 Series syringes — 10 and 100 μl

Conditions

24 cm^3/min helium carrier flow
20 cm^3/min hydrogen gas and 310 cm^3/min air flow to FID detector
215°C injector temperature
250°C detector temperature
Oven temperatures:
  Isothermal, 120°C
  Temperature programs:
  1) 100°C for 6 min, 100°–140°C at 20°C/min
     5 min at 140°C
  2) 120°C for 18 min, 120°–200°C at 20°C/min

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*Carbon disulfide is a volatile, flammable, toxic material and must be handled with care in an efficient laboratory fume hood.

Table 3

Retention data and FID relative sensitivities for organic compounds

Carrier flow rate, 24 ml/min

FFAP column Organic solvent analyzed	FID relative sensitivities Literature values[a]	 Experimental[b]	Relative retention times[c] Literature values	 Experimental
1, 1, 1-trichloroethane		0.18		0.83
i-propyl alcohol	0.53			0.86
Methyl ethyl ketone	0.61	0.61	0.85	0.86
Benzene	1.12	1.21	1.00	1.00
Trichloroethylene		0.21	1.16	1.10
Perchloroethylene		0.16	1.38	1.28
Toluene	1.07	1.07	1.51	1.45
n-butyl alcohol	0.66			1.90
Methylene chloride				1.93
m-xylene	1.04	1.09	2.10	2.14
Styrene				3.41

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[a] Response factors reported by Dietz[7] with factors relative to heptane.

[b] Response factors calculated from a seven-component mixture with the toluene response set equal to 1.07, facilitating comparison with literature values.

[c] Benzene employed as internal standard. Literature data calculated from retention times given by Kupel.[1]

Chromatographic peaks of interest can in some cases be characterized by retention times, and retention data for several chemicals are listed in *Table 3*. Aliquots of eluted samples are injected into the instrument using a backflush of desorbing reagent to clean the syringe barrel. The solvent flush injection technique increases the accuracy and reproducibility of the injected sample volume.[1]

Response factors for the Varian flame ionization detector (FID), obtained from a mixture of solvents, were in excellent agreement with literature data (Table 3). The FID is less sensitive to chlorocarbons, and this is readily evident from the rather low response factors exhibited by these compounds. The FFAP column used by NIOSH effectively separates aliphatic and aromatic compounds but is not generally recommended for polar compounds where tailing or poor resolution can be a problem. The resolution of peaks experienced by Kupel et al.[1] was reproduced in our laboratory (*Figure 2*).

Unknown peaks in chromatograms can be trapped in capillary collection tubes and positively identified by mass spectrometry. Capillary collection tubes were constructed from glass tubing (2 mm × 75 mm) by partially closing one end. One piece of carbon from an unused sampling tube was placed in each capillary tube. A collector tube

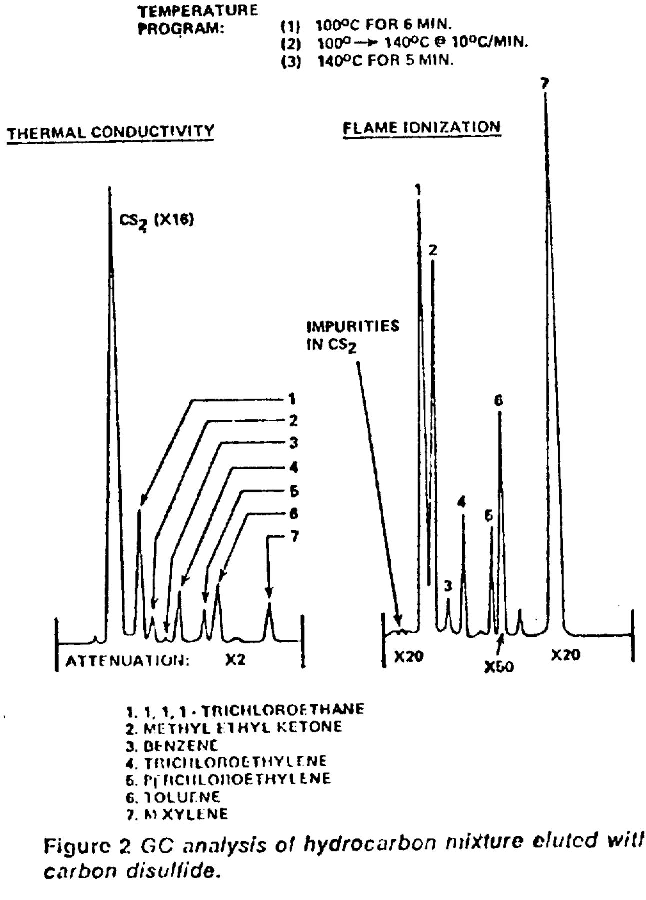

Figure 2 *GC analysis of hydrocarbon mixture eluted with carbon disulfide.*

was inserted in the exit port of the GC as the peak of interest appeared on the chromatogram and was withdrawn on the downside of the peak prior to reaching the baseline. The carbon particle was transferred to the mass spectrometer direct inlet probe. A Bendix inlet probe (model MA016) was connected through a vacuum lock into the ion chamber of a Bendix time-of-flight mass spectrometer model 12-107). The probe temperature was raised gradually to release the material from the carbon for recording of the mass spectrum. The limits of detection for several organic compounds using this procedure have been determined.[4]

Quantitative analysis

The NIOSH method[1] employs an absolute calibration approach to achieve quantitative analysis of eluted samples. Standard solutions are prepared volumetrically in the elution solvent. Concentrations equivalent to 0.5, 1.0, 2.0, and 5.0 times the OSHA limit were prepared using Eq. (1):

$$\mu\text{l material in 1.0 ml standard solution} = \frac{F \times L \times V \times M}{\rho \times 24.45 \times D} \qquad (1)$$

where:
F = fraction or multiple of OSHA limit
L = OSHA limit, ppm
V = air sample volume, m^3
M = molecular weight, g/mole
ρ = density, g/ml
24.45 = liters/mole @ 25°C, 760 mm Hg
D = volume desorbing agent, ml

5-μl portions of the standards and samples are analyzed by GC. Peak areas of the standards are related to concentrations to obtain calibration curves; linear relationships were obtained in every

Table 4

Desorption percentages of organic solvents

Compound	OSHA Limits (Ref. 9), ppm	Desorption percentages at OSHA limit[b] Average	Range	Reference[c]
i-amyl acetate	100	97	95–99	GE
n-amyl acetate	100	96	94–99	GE
Benzene	10	96	94–100	NIOSH, GE
2-butanone	200	96	90–99	NIOSH, GE
n-butyl acetate	150	94	88–97	NIOSH
n-butyl alcohol	100	91	87–97	GE
Butyl carbitol Dowanol DB	None established[a]	98	95–100	GE
Carbon tetrachloride	10	101	96–113	NIOSH
Chloroform	50	100	98–101	NIOSH
Diacetone alcohol	50	97	95–100	GE
p-dioxane	100	88	84–98	NIOSH
Ethanol	1000	67	64–72	NIOSH
Ethyl ether	400	90	86–96	NIOSH
Iso-octane	None established[a]	95	92–97	NIOSH
Perchloroethylene	100	96	95–97	NIOSH, GE
i-propyl alcohol	400	80	75–87	GE
Pyridine	5	48	46–50	NIOSH
Toluene	200	98	93–99	NIOSH, GE
Trichloroethylene	100	97	93–99	NIOSH, GE
SC-100 (aromatic distillate)	None established[a]	97	94–100	GE
Xylene	100	98	97–100	NIOSH, GE

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[a] Concentrations employed for compounds with no established limits were as follows: butyl carbitol = 50 ppm, iso-octane = 400 ppm, SC-100 = 100 ppm.
[b] Percentages represent the average value of three to twelve determinations.
[c] NIOSH data taken from Ref. 1; GE values determined experimentally in our laboratory.

Table 5

Efficiency of charcoal technique

	Aluminum odor chamber concentration, ppm	OSHA Limit (Ref. 9), ppm	Peak areas (counts × 10^{-5})[a] Carbon tubes	Standard solution[b]	Analytical efficiency, %[c]
Benzene	18.7	10	0.91±0.01	1.01±0.08	90
Toluene	103	200	5.65±0.12	5.65±0.33	100
m-xylene	87	100	5.97±0.13	5.15±0.30	115
I-propyl alcohol	153	400	1.99±0.20	2.29	87
n-butyl alcohol	61.8	100	0.90±0.05	1.20	75
Diacetone alcohol	26.3	50	0.29±0.02	0.48	60
Butyl carbitol	11.1	[d]	0.13±0.01	0.15	87
SC-100 and xylene (14 peaks)	100	100[e]	10.8±0.1	11.3	96
Styrene	90	100	9.5±0.4	11.5±0.1	83
Methylene chloride (3 carbon tubes in series)	600	500	7.8±0.2	8.4+01	93

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[a] Average values of peak areas and standard deviation for three separate determinations: $X \pm S$, with $N=3$.
[b] Standard solutions prepared volumetrically in carbon disulfide equivalent to aluminum odor chamber concentrations.
[c] Analytical efficiency calculated from Eq. (2).
[d] No OSHA limit.
[e] 100 ppm OSHA limit for xylene; no OSHA limit for SC-100, a complex mixture of aromatics.

case. Sample solution peak areas are converted to concentrations using the calibration curves.

Some disadvantages of this absolute calibration approach are that standards and samples must be reproducibly injected and detector sensitivity must remain constant.[8] After initial calibration, one standard should be run at the time of analysis to verify instrument sensitivity.

Method evaluation

Desorption efficiency

Adsorbed organic compounds may or may not be readily displaced from carbon by carbon disulfide, and it is necessary to know the amount desorbed to avoid inaccurate interpretation of analytical results. Kupel et al.[1] list desorption percentages for fourteen compounds, and another seven materials were evaluated in this laboratory (*Table 4*). To obtain desorption efficiency, a known volume of the substance was injected directly into the carbon sampling tube. The tube was capped and left sitting overnight to provide ample time for sorption equilibrium to be attained. The carbon was eluted, and the desorbed material was analyzed by GC. The desorption efficiency for each material was calculated as the ratio of the amount desorbed to the amount added to the carbon tube.

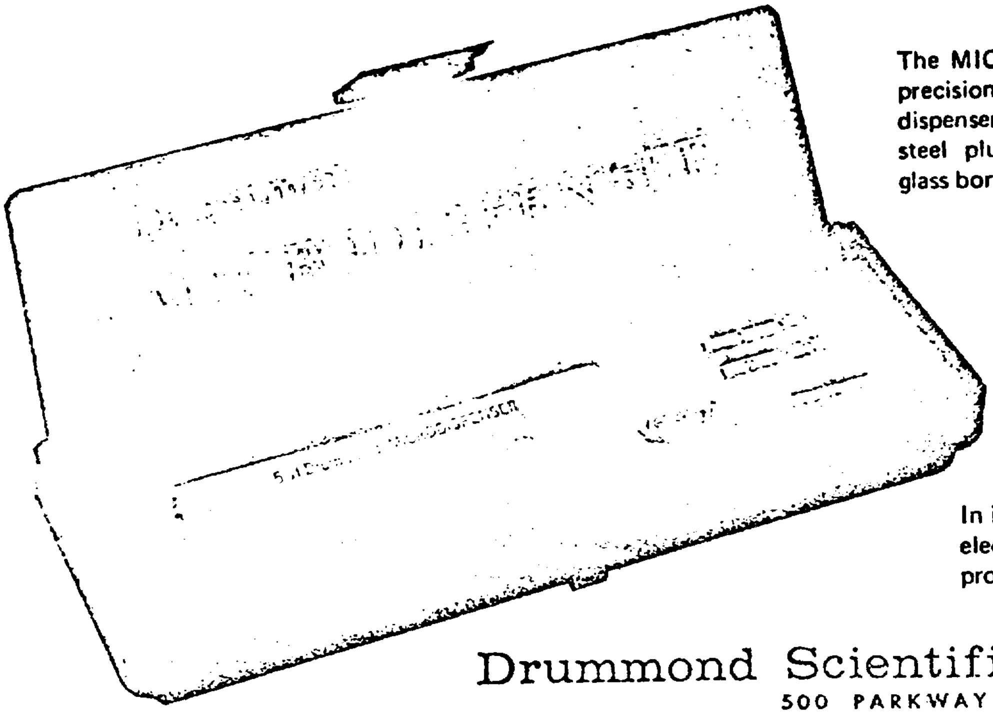
Analytical efficiency

A test was devised to measure the overall precision and accuracy of this method, including sampling, adsorption, desorption, and gas chromatographic analysis. Known concentrations of organic vapors were generated in a 76.6-ft^3 test chamber.[9] Thorough mixing was provided by fans located within the chamber. Hydrocarbon levels in the chamber were monitored with a Beckman total hydrocarbon analyzer (model 109A) and a Varian gas chromatograph to ensure that the proper quantities were present for sampling. Three 10-liter air samples were taken from the chamber through carbon tubes, desorbed, and analyzed by GC. Analytical efficiencies were calculated using the following relationship:

$$\text{Analytical efficiency (\%)} = \frac{\text{Organic vapor conc. from carbon tube analysis} \times 100}{\text{Known organic vapor conc. in chamber}} \qquad (2)$$

Analytical efficiencies for several materials are given in *Table 5*. Results obtained for benzene, toluene, and xylene were in agreement with chamber desorption percentages reported by Kupel.[1]

Polar compounds, such as alcohols and amines, exhibit lower desorption percentages with carbon disulfide (CS$_2$), and the overall efficiency of the



ORGANIC VAPORS *continued*

method is reduced.[1] Polycat 8 (N,N'-dimethylcyclohexylamine) was examined as a polar air contaminant, using the aluminum test chamber. Only a fraction of its original concentration was evident from desorbed carbon samples. The efficiency of measurement varied with the choice of desorption solvent, with chloroform being more effective than CS_2 in removing this amine from carbon. The limit of detection for Polycat 8 with chloroform as the eluting solvent is between 4.2 and 9.6 ppm, since the amine was not detected in chromatograms obtained from samples at the lower level.

There was some question about the applicability of this method to polymerizable monomers or substances with relatively low adsorptivity on carbon. Styrene vapor sorbed on carbon could polymerize due to surface catalysis, or polymer formation could occur on the column packing of the gas chromatograph. Neither reaction prohibited valid analysis by this method, and styrene was effectively determined using the carbon tube technique (Table 5). A single carbon tube (Figure 1) did not adequately trap methylene chloride vapors at the OSHA limit of 500 ppm, and the backup carbon section contained a considerable amount of this contaminant. Increasing the quantity of carbon available for sorption by utilizing three carbon tubes in series resulted in a high analytical efficiency for methylene chloride (Table 5). For substances with relatively high OSHA limits and appreciable vapor pressures at room temperature, it may be necessary to increase the amount of activated carbon or lower the volume of air sampled in order to trap the material efficiently.

Practical applications

Four separate experiences will serve to illustrate our use of the carbon tube method in industrial situations.

Case 1 — Simple mixture of readily identified components

An employee performing a spraying operation complained of exposure to odorous vapors. Carbon tube samples were obtained from the breathing zone of this operator. Normal and isoamyl acetates were identified by chromatographic retention times and were the only substances found in the desorbed air samples. The spray applied by this worker contained a mixture of the acetate isomers, and MS analysis was not required. The desorption efficiency of the carbon disulfide elution was greater than 95% for both normal and isoamyl acetates, and

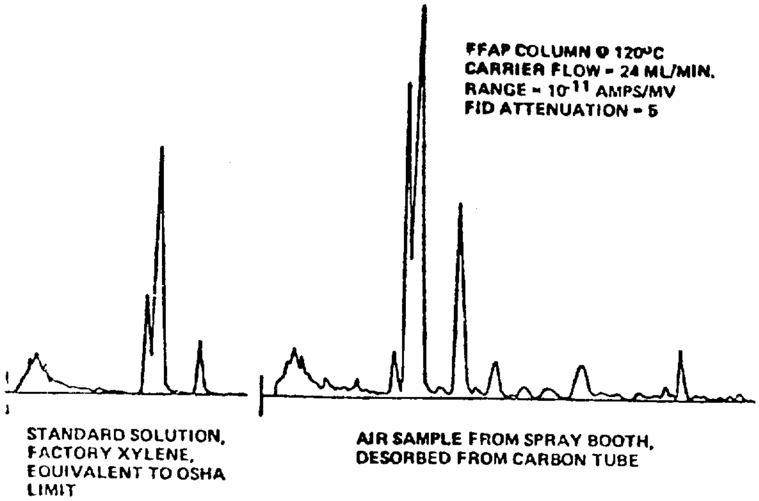


Figure 3 *Comparison of desorbed air sample with standard solution.*

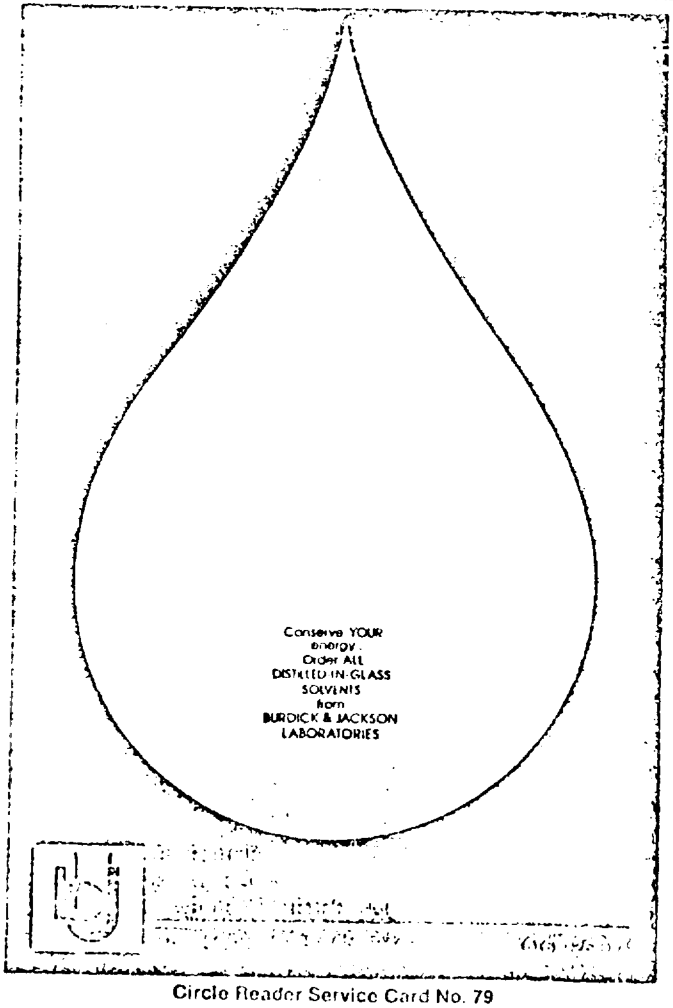
GC analysis revealed acceptable workroom vapor concentrations.

Case 2 — Complex mixture with single component above OSHA limit

A spray booth was surveyed for airborne organic vapors because employees were concerned about persistent disagreeable odors present in the area. A chromatogram of the desorbed sample contained over twenty separate peaks. Comparison with chromatograms of bulk liquid samples revealed that three of the major peaks were due to xylene. The concentration of xylene alone was above the OSHA limit (*Figure 3*), and it was not necessary to identify the smaller unknown peaks. This information was reported to the responsible manager, and corrective ventilation action was immediately taken.

Case 3 — Mixture of solvents identified by mass spectrometry

A paint mix area was surveyed for vapor concentrations of six organic materials: methyl ethyl ketone (MEK), isopropyl alcohol (IPA), butyl alcohol, xylene, diacetone alcohol, and butyl carbitol. CS_2 desorption percentages of the four alcohols were determined (Table 4) since they were not available from the literature. Analytical efficiency for these alcohols was in the range of 60–90% (Table 5). These values can be compared with Kupel's desorption percentages of 10% for methyl alcohol[10] and 67% for ethyl alcohol.[1] GC analysis of a qualitative air sample indicated that the factory atmosphere contained a number of compounds (more than 20 peaks in chromatogram). Peaks assigned to diacetone alcohol, butyl alcohol, and xylene were present, but there was no evidence of butyl carbitol in the atmosphere. IPA and MEK were not separated satisfactorily, the two giving a single chromatographic peak under the analytical conditions used. The single peak was evaluated as



MEK, which has a lower OSHA limit than does IPA. MS analysis was employed to identify unknown peaks and to confirm assignment of peaks according to retention times. Identification of the minor peaks was not attempted, and only the larger peaks were considered. MS analysis identified the presence of IPA, MEK, toluene, *n*-butyl alcohol, and xylene. It is interesting to note that toluene contributed to the total worker exposure although its presence was not considered prior to qualitative analysis.

Case 4 — Paint diluents containing a variety of aromatic compounds

A finishing operation was monitored for OSHA compliance, and as suspected, the solvents were multicomponent mixtures. Air samples contained a multitude of peaks, and retention time comparisons with bulk liquid samples indicated the atmospheres in the various spray booths were mixtures of benzene, toluene, xylene, and SC-100. Concentrations varied with the sampling location, and different chromatograms were realized for different parts of the finishing area (*Figure 4*). These results demonstrate the complexity of industrial atmospheres where the process materials are mixtures and where the composition of the air can vary with locality or time of sampling. To calculate the concentration of SC-100, a single clean peak was chosen for comparison with standard solutions prepared volumetrically. This approach was suggested by Kupel[10] as an alternative to attempting identification of a number of peaks in a solvent which is a distillation cut. The variety of aromatic compounds potentially present is aptly demonstrated by the work of Deans and Scott[11]; their chromatogram of a heart-cut naphtha showed the presence of benzene, toluene, ethyl benzene, *m*- and *p*-xylene, isopropylbenzene, *o*-xylene, 3- and 4-ethyl toluene, mesitylene, 2-ethyl. toluene, and pseudo cumene. An OSHA limit has not been established for SC-100, and so a limit of 100 ppm was assumed for this material. Analytical efficiency for SC-100 was 95%.

Discussion

The carbon tube method cannot be properly used or interpreted without understanding the following:

1. Some air contaminants are poorly adsorbed by carbon and would not be efficiently collected by this method. The relative capacity of carbon for

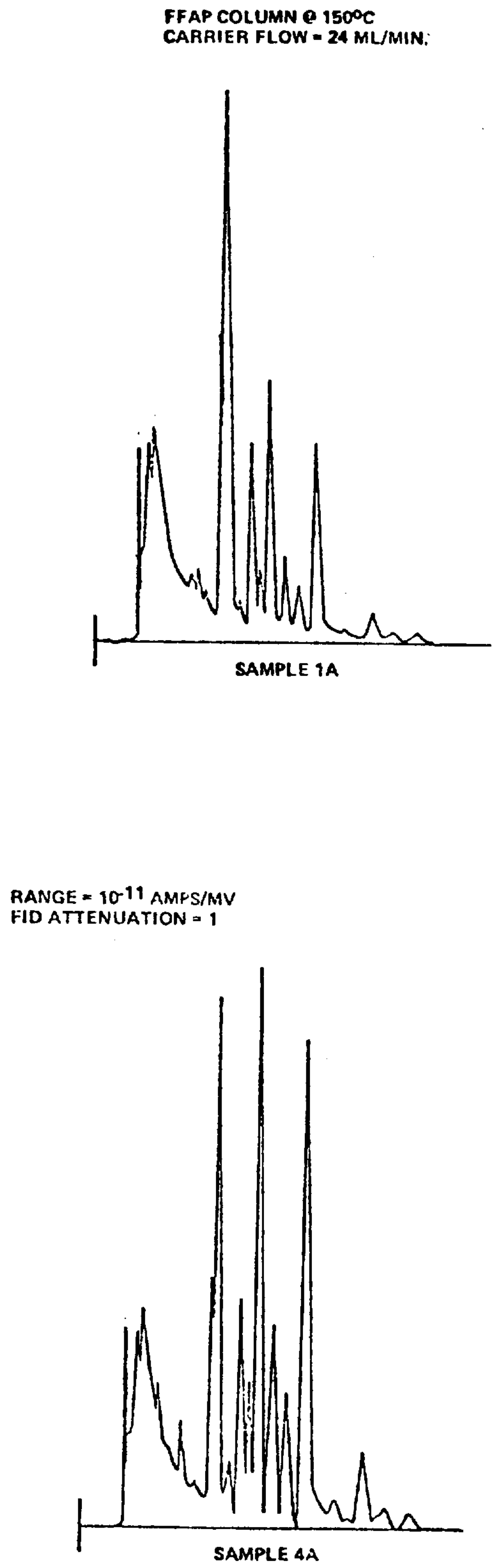


Figure 4 Chromatograms of desorbed air samples from adjacent factory areas.

various vapors is available from the literature[12] and can be used to estimate adsorption efficiency.

2. Chemically reactive gases can be converted to other species on the carbon surface and would not be properly identified. This would be an important consideration if the area of interest contained airborne species formed by thermal, radiant, or chemical decomposition.

3. Carbon disulfide does not readily displace all organic compounds from carbon, and a different desorption solvent would be necessary for some compounds to obtain acceptable overall efficiency.

4. Organic compounds can be displaced from charcoal tubes by the presence of chemical species that are more strongly adsorbed. If organic materials are present on the backup carbon section of the sample tube, the data should be carefully scrutinized.

5. The 180-mg "A" section of a carbon sampling tube has a saturation limit of 28–45 mg (approximately 1000–1500 ppm) of total solvents.[1] Each section of the sampling tube collects solvent vapors until the saturation limit is reached.

Summary

The adsorption of organic vapors by activated carbon followed by liquid desorption and GC analysis is a reliable method for assessing occupational hazards, provided certain limitations inherent in the procedure are realized. The method is not universally applicable to all organic vapors, and each determination must be considered a separate case.

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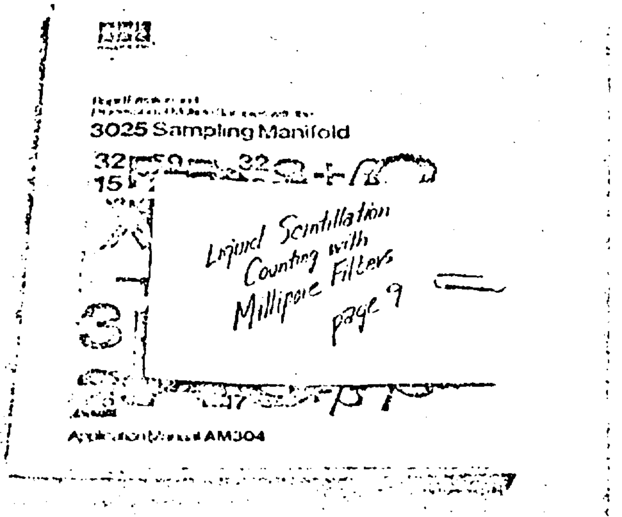
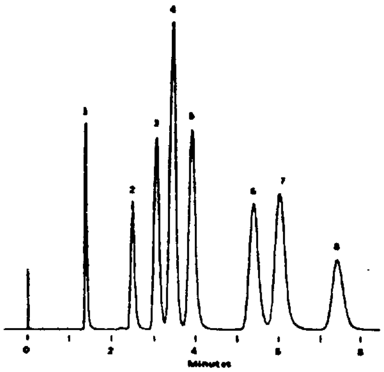


Liquid Scintillation Counting with Millipore Filters page 9



GC Separation of Solvent Mixtures

& Determination of Water in Solvents

Bulletin 747

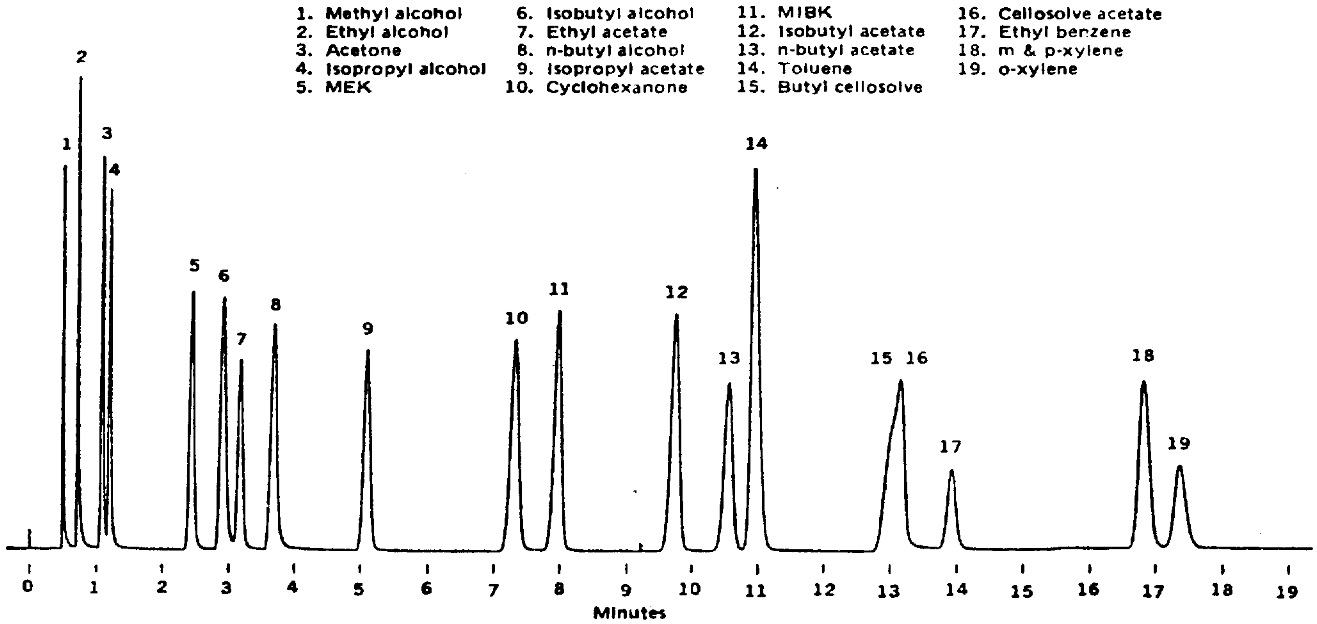
Introduction

This bulletin deals with columns useful for the complex mixtures of solvents. There are several columns which may serve as a general purpose column and these may be supplemented by one or more special purpose columns. No single packed column will be able to separate every possible solvent combination; consequently, more than one column may be needed to handle a variety of solvent problems.

General Purpose Column A

Carbopack C/0.1% SP-1000 is a good general purpose column; a major feature is the ability to separate alcohols without tailing of the peaks. It can be used to separate complex mixtures of alcohols, ketones, esters, aromatics and chlorinated hydrocarbons as illustrated in Figure 1 with the separation of a 19 component mixture. The column can be used isothermally as shown in Figure 2; however, if a wide boiling range mixture is to be separated, this is done better with temperature programming rather than isothermally. Table 1 lists the retention data for a variety of compounds.

Figure 1 — Solvent Mixture



Carbopack C/0.1% SP-1000, 6 ft. x 2mm ID, Glass, Col. Temp.: 100-225°C @ 8°C/min., Flow Rate: 20 ml/min., Nitrogen @ 50 psi, Sample Size: 0.15 μl, Detector: FID, Sensitivity: 32×10^{-10} AFS.

Table 1 – Retention Data (minutes)

	Col. Temp.			
	175°C	100°C	100°C	100°C
Alcohols	Carbopack C ▲ 0.1% SP-1000	20% SP-2100 ▲	20% SP-2401 ▲	18% THEED ▲
Methyl	0.65	1.12	1.1	3.0
Ethyl	0.75	1.24	1.25	3.36
i-propyl	0.88	1.42	1.4	3.0
n-propyl	0.95	1.82	1.68	4.89
i-butyl	1.35	2.6	2.15	6.48
n-butyl	1.55	2.98	2.6	7.66
Diacetone	Decomposes	8.0	14.38	Not Eluted
Cellosolves				
Methyl	1.3	2.67	2.85	12.12
Ethyl	2.1	3.98	3.78	12.29
Butyl	Late	12.35	9.7	26.95
Ketones				
Acetone	0.9	1.38	2.18	1.43
MEK	1.25	2.1	3.12	1.75
MIBK	1.71	4.56	6.12	2.28
Mesityl Oxide	6.25	6.35	7.56	4.1
Cyclohexanone	3.4	11.1	18.26	Not Eluted
Isophorone	Not Eluted	43.9	–	Not Eluted
Acetates				
Ethyl	1.45	2.33	2.51	1.3
i-propyl	2.10	2.95	3.05	1.3
n-propyl	2.70	3.88	3.95	1.69
i-butyl	5.05	5.43	5.46	1.81
n-butyl	5.90	6.8	6.48	2.25
Cellosolve	9.8	11.14	11.95	6.48
Formates				
Methyl	0.75	1.18	1.32	–
Ethyl	1.00	1.55	1.78	–
Chloroalkanes				
Methylene Chloride	0.90	1.68	1.05	1.15
1,1,1,-trichloroethane	1.65	3.0	1.68	1.33
Aromatics				
Benzene	2.23	3.25	2.22	1.41
Toluene	6.35	5.79	3.47	1.91
m & p-xylene	Late	10.54	5.5	2.65
o-xylene	Late	12.1	6.66	3.3
Aliphatics				
n-hexane	2.85	2.4	1.33	0.79
n-heptane	6.5	4.0	1.78	0.81
n-octane	16.6	7.12	2.55	0.91
n-nonane	Late	13.0	3.89	1.09
n-decane	Late	24.3	6.25	1.31
Miscellaneous				
THF	1.19	2.72	–	1.65
DMF	1.95	5.45	–	–
2-nitropropane	1.55	3.6	7.05	3.5

▲Carbopack Column - 6 ft. x 2mm, all other columns - 10 ft. x 1/8" SS

The Carbopack C column is best used with small sample sizes and consequently a flame ionization detector rather than a thermal conductivity detector should be used. Sample sizes of 0.1 to 0.5 µl are recommended, but with the smaller volume preferred. If large sample sizes are used, the column is overloaded and poor peak shapes and poor separation results. Although with properly packed stainless steel columns, 700 plates/foot can be obtained, the best column efficiencies are obtained using a glass column; however, with stainless steel columns, there is some tailing of peaks.

The Carbopack C/0.1% SP-1000 has the ability to separate isomers very readily. As a result, if complex mixtures of hydrocarbons are to be separated, this is done very well. However, if alcohols, ketones, etc. are present as well as complex mixtures of aliphatic hydrocarbons, there will be considerable overlap of the peaks. Upper column temperature limit is 225°C. Carbopack C/0.1% SP-1000 can be used interchangeably with Carbopack A/0.2% SP-1000. For more details on the use of Carbopacks, see Bulletin 738. BULLETIN

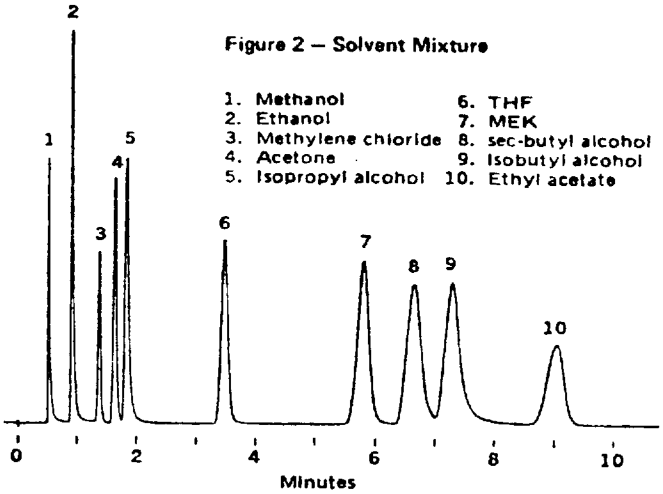


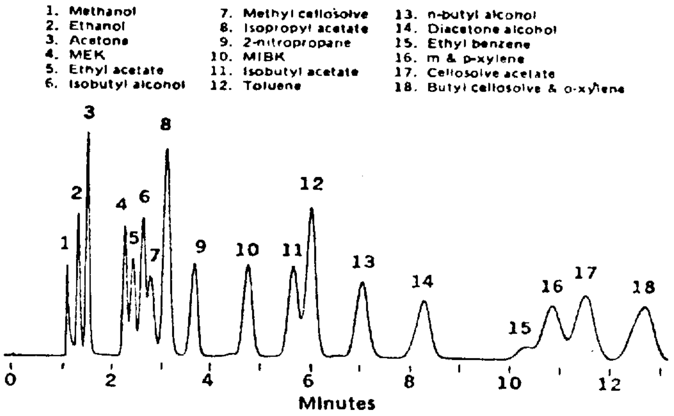
Figure 2 – Solvent Mixture

Carbopack C/0.1% SP-1000, 6 ft. x 2mm Glass, Col. Temp.: 65°C, Inlet & Det. 100°C, Flow Rate: 20 ml/min., N_2 @ 34 psi, Sample Size: 0.1 µl, Det. FID, 16 x 10^{-10} AFS.

General Purpose Column B

Our SP-2100, methyl silicone, makes a very good general purpose column for a variety of solvents, with the components eluted generally in order of their boiling points. Figure 3 shows the separation of an 18 component mixture of alcohols, esters, ketones, aromatics, etc. In Table 1, we list the retention time of these and a number of other common solvents. We have been able to solve a large number of different solvent problems with this column.

Figure 3 – Solvent Mixture



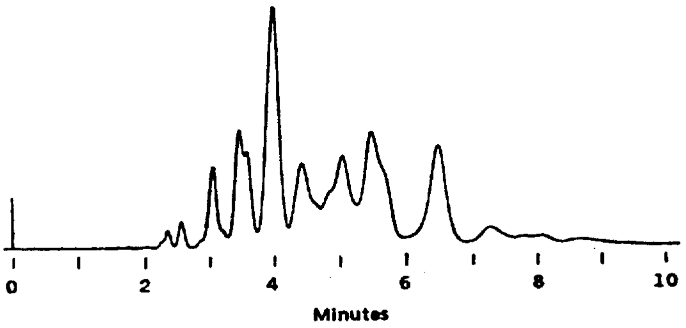
20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport, 10 ft. x 1/8" SS, Col. Temp.: 100°C, Flow Rate: 20 ml/min., N_2, Sample Size: 0.5 µl, Det. FID.

The 20% SP-2100 packing contains 0.1% Carbowax 1500 as a tail reducer to reduce the tailing of alcohols. The column generally does a good job with most alcohols, but the methyl and ethyl do tail when present in low concentrations. There are better columns for trace alcohols and these are described elsewhere. The SP-2100 column is not well suited for separation of complex mixtures of aliphatic solvents along with alcohols, ketones, etc. The hydrocarbons tend to be eluted along with the other components, obscuring the results, unless the aliphatics are either lower or higher boiling than the other components. Because of the presence of Carbowax 1500, the upper temperature limit of this packing is limited to 175°C.

Hydrocarbon "Fingerprint"

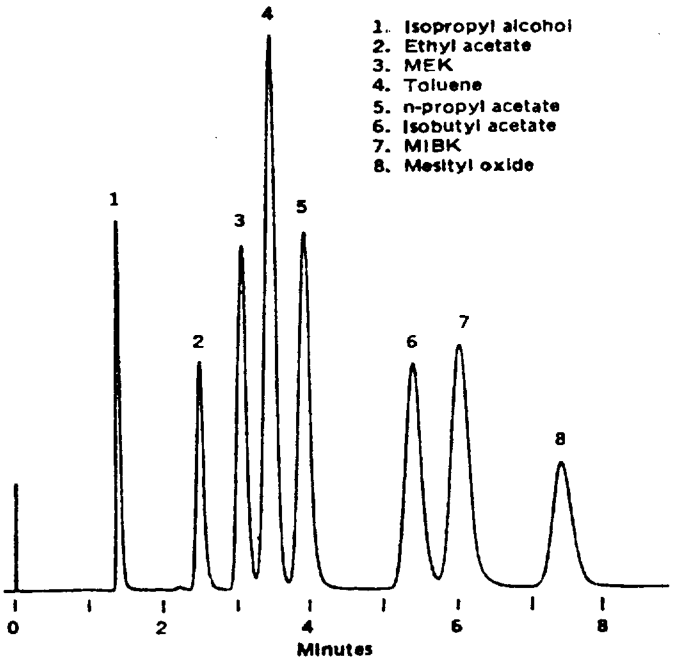
The SP-2100 can be used as a special purpose column to examine the composition of hydrocarbon mixtures such as naphthas. One can obtain a "fingerprint" of the hydrocarbon mixture and compare it to other batches of the material, as shown in Figure 4 which is a naphtha sample analyzed with the 20% SP-2100 column. If better resolution of the mixture is needed, a 10% SP-2100 is a good choice. For details see Bulletin 743, Section 7. BULLETIN

Figure 4 — Naptha Sample



20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport, 10 ft. x 1/8" SS, Col. Temp.: 125°C, Flow Rate: 20 ml/min., N_2, Sample Size: 0.5 µl, Det. FID.

Figure 5 — Ketone Retarder Column



20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport, 10 ft. x 1/8" SS, Col. Temp.: 125°C, Flow Rate: 20 ml/min., N_2, Sample Size: 0.5 µl, Det. FID.

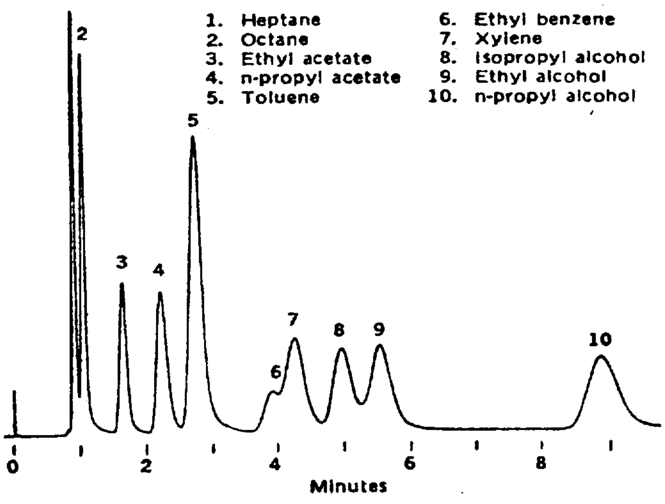
Ketone Retarder

The SP-2401 is a stationary phase which has the ability to elute ketones relatively later when compared to other stationary phases. As an example, SP-2401 elutes MEK after ethyl acetate with good separation of the two, as shown in Figure 5 along with several other compounds. In contrast with SP-2100, this pair is barely separated with MEK eluted first (Figure 3). Isophorone, a ketone, is eluted from the column, but one must increase the column temperature to elute it in a reasonable length of time. We recommend SP-2401 as a special purpose phase rather than a general purpose one. Our 20% SP-2401 packing also contains 0.1% Carbowax-1500 as a tail reducer to improve alcohol peak shapes. Upper temperature limit is 175°C. Retention data for many common solvents is shown in Table 1.

Alcohol Retarder

Tetrahydroxyethylenediamine (THEED) is a stationary phase which retards the elution of alcohols relative to other classes of compounds. It also has the ability to elute hydrocarbons very rapidly compared to other compounds boiling in the same temperature range. As an example of this, Figure 6 shows the rapid elution of heptane and octane, then esters, aromatics and finally alcohols. The 15% THEED is coated on 100/120 Chromosorb W AW. We do not recommend the use of a silanized support with THEED because of the wetting problem. Upper temperature limit for this column is 135°C. Retention data for common solvents is listed in Table 1.

Figure 6 — Alcohol Retarder

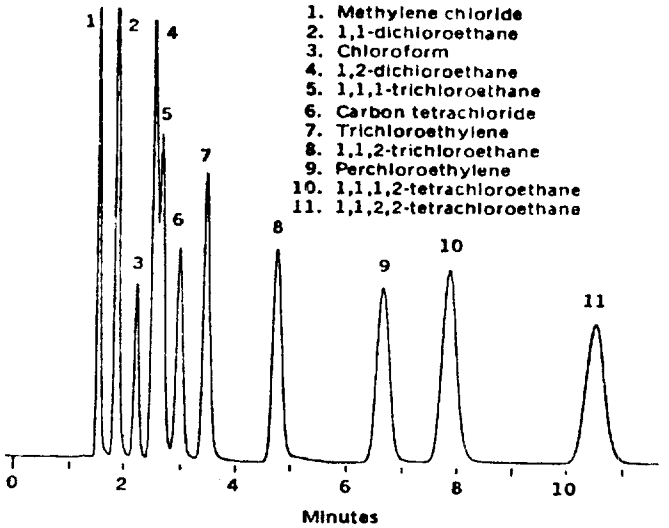


15% THEED on 100/120 Chromosorb W AW, 10 ft. x 1/8" SS, Col. Temp.: 80°C, Flow Rate: 20 ml/min., N_2, Sample Size: 0.5 µl, Det. FID.

Chlorinated Solvents

The 20% SP-2100/0.1% Carbowax 1500 on Supelcoport makes a good column for separation of complex mixtures of chlorinated solvents or for determination of impurities in a single chlorinated solvent. Figure 7 shows the separation of a mixture of eleven components made with a 10 ft. x 1/8" SS column at 100°C.

Figure 7 — Chlorinated Solvents



20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport 10 ft. x 1/8" SS, Col. Temp.: 100°C, Flow Rate: 20 ml/min., Nitrogen, Det. FID.

Packings

1-1820	GP Carbopack C/0.1% SP-1000, 25g	$75
1-1821	20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport, 25g	50
1-1822	20% SP-2401/0.1% Carbowax 1500 on 100/120 Supelcoport, 25g	50
1-1823	15% THEED on 100/120 Chromosorb W AW, 25g	30

Packed Columns (except Carbopack & Carbosieve)

1/4", 3/16" or 1/8" Stainless Steel	$25 + $3.50/ft.

Packed Columns (Carbopack & Carbosieve)

1/4" or 3/16" Stainless Steel	$30 + $15/ft.
1/8" Stainless Steel	$30 + $ 8/ft.

Carbopack/Glass Column

See our special 8-page glass column catalog and our latest GC catalog.

DETERMINATION OF WATER IN SOLVENTS

Water can be rapidly determined in solvents by using a Chromosorb 101 porous polymer, packed into a short metal tube; Figure 8 shows 0.08% water in toluene. The work shown here was carried out with a Fisher Model 2400 GC equipped with a thermal conductivity detector. The Chromosorb 101 column can be used specifically for the water determination or used to separate some solvent mixtures.

The determination of water by GC is difficult because the water peak will tail severely with many columns making quantitative results very poor. The tailing of the water peak can be caused by a variety of factors including the column packing, the column tubing and the inlet of the GC instrument. The various diatomite supports, even when AW-DMCS treated, still cause tailing, whereas the porous polymers, such as Chromosorb 101, are much more inert and water does not tail on them.

Metal column tubing has also been blamed for the tailing but this depends on both the quality of the tubing and the concentration of water to be determined. Thus far, we have tested our stainless steel tubing successfully down to 0.01% water. Some workers prefer either glass or Teflon column tubing to avoid tailing of the water peak. The overall column efficiency obtained with a glass column is higher than obtained with stainless steel, but that obtained with Teflon is rather poor. The metal inlet of an instrument can also be highly adsorptive causing tailing. This can be avoided by using a glass insert in the inlet or by injecting the sample directly into the column, avoiding the inlet.

A calibration curve for water can be obtained by injecting the same size sample of each of several standards containing known amounts of water into the chromatograph. Plot peak heights versus concentration of water as in Figure 9. The calibration mixtures should be prepared with a solvent which is free of water. This can be done by drying the solvent with a good drying agent such as Molecular Sieve 5A. The syringe used to inject the sample should also be free of water so as not to contaminate the standards or sample.

Figure 8 — 0.08% Water in Toluene

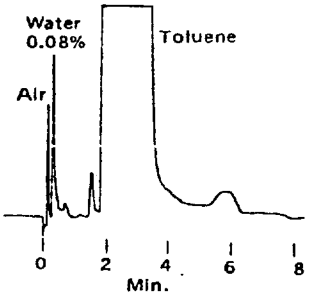
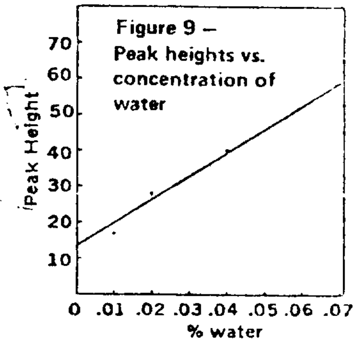


Figure 9 — Peak heights vs. concentration of water



Chromosorb 101, 60/80 mesh, 3 ft. x 1/8" SS, Col. Temp.: 165°C, Flow Rate: 20 ml/min., helium, Sample Size: 1 μl, Det. TC @ 250MA, Sensitivity 2X, Recorder 1MV.

For details on the use of an internal standard procedure for water, see Hogan et al. (3). Other useful references dealing with the determination of water are 1, 2 and 4.

Packed Columns (except Carbopack or Carbosieve)

1/4", 3/16" or 1/8" Stainless Steel	$25 + $3.50/ft.
1/4", 3/16" or 1/8" Teflon	$40 + $4.00/ft.

Tubing

2-0526	1/8" Stainless Steel, 50 ft. coil	$80
2-0532	1/8" Teflon, 50 ft. coil	40

Chromosorb 101

2-0213	60/80, 50g	$33
2-0214	80/100, 50g	33
2-0215	100/120, 50g	31

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APPENDIX - C1

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

COMPARISON OF ALKALINE WASHING AND VAPOR DEGREASING

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Grand Rapids, Michigan

PREPARED BY:

K. S. Surprenant
The Dow Chemical Company

PREPARED FOR:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

A cost and energy comparative study between alkaline washing and vapor degreasing was made. The equipment involved a spiral alkaline washer and a Vibra degreaser. The cost of cleaning a ton of work was found to be the same as to 17% less with vapor degreasing than alkaline washing. The energy demand for the washer was just over twice that of the degreaser.

Both alkaline washing and vapor degreasing satisfy unique cleaning needs and are not interchangeable metal cleaning processes. This fact was repeatedly brought out in seeking a comparative evaluation testing site and during the evaluation itself. The substitution of alkaline washing for solvent metal cleaning to control hydrocarbon emissions would be possible in relatively few operations.

Objective

Alkaline washing is another means of metal cleaning. No hydrocarbon emissions result from this cleaning method because it depends on water detergent solutions rather than organic solvents. The purpose of this evaluation is to define the cost and energy relationships between alkaline washing and solvent metal cleaning, especilly vapor degreasing. In those instances where either cleaning process might be used, this background information may be valuable in evaluating the choice.

Introduction

The qualitative parameters involved in choosing between alkaline washing and solvent metal cleaning have been outlined in the body of the main report. In most cases, the cleaning requirement defines the most practical choice between alkaline washing and vapor degreasing. Because these cleaning processes satisfy different cleaning needs, it is difficult to locate industrial applications where both processes are employed to clean essentially similar production.

This evaluation site was located through the assistance of Detrex Chemical Industries Incorporated who manufacture both alkaline washing and vapor degreasing equipment. Potential sites were sought with several other firms manufacturing equipment for both processes. However, this location was the only recommendation received.

In addition to the performance characteristics of the processes, there are other causes for selecting one or both cleaning methods. Some of these reasons would include: the availability of a surplus washer or degreaser within the corporation, changes in the cost of energy, equipment, or chemicals or a reassessment of the hazard of alkaline washing compound or chlorinated hydrocarbons. The reasons for the existence of both cleaning processes at Diesel Equipment were not explored but could have been any of the above or other valid causes. Even though the work processed by the washer and degreaser at this location is largely similar, there are a large number of specific cleaning operations which are done exclusively by one or the other process. It is also valuable to note that this manufacturing location utilizes a number of vapor degreasers of several different design types as well as a number of washers. In spite of this variety of equipment, it is quite unusual for parts being cleaned in one operation to be cleaned by the other cleaning process either routinely or even alternatively.

Equipment

Vapor Degreasing Operation - The vapor degreasing operation studied was a Detrex Vibra degreaser Model No. RV918-8-75 Special. This degreaser is 64" x 64" x 110" high and uses trichloroethylene. The attached specification sheet shows its diagramatic construction. The parts are introduced through the loading chute to the flooded bottom pan and are conveyed up the spiral by the vibratory motion of the spiral. With the exception of the vibratory conveying means, the degreaser operates similarly to other vapor degreasing operations in producing solvent vapors and condensing and controlling them. This degreaser is equipped with two Detrex Model S185 stills. The still design is shown on the specification sheet also attached. Both stills were located on a storage tank 65" x 78" x 33". The degreaser is identified as Serial No. 54715 and the stills are identified by Serial No.'s 54515A and 54515B.

Alkaline Washer - The spiral washer evaluated was supplied by Ransohoff Company, Serial No. 10443 (Figure 1). The Equipment No. is 36948. This washer has three stages: wash, rinse and dry. The overall equipment dimensions are 21' x 6' with a machine height of about 7'10".

Further design information is listed on the attached Ransohoff specification sheet. Parts are fed into this machine through a loading chute to the inside of a rotating drum with a spiral screw. The spiral screw causes the parts to move through a spray washing station to a spray rinse station and a drying zone before being discharged. The drum is perforated to allow the drainage of wash and rinse water as well as the flow of hot air for drying. The operating conditions are spelled out on the washer maintenance specification sheet supplied by Diesel Equipment and attached to this report.

Experimental Design - This evaluation differs from other emission testing in that no emission control devices were a portion of this study. Rather, an objective cost and energy comparison between alkaline washing and vapor degreasing was the purpose of this effort. For this reason, no attempt was made to improve the operation of either the alkaline washer or the vapor degreasing system. However, in a number of ways both processes studied demonstrated excellent operation.

Briefly, the concept involved in this evaluation was simply to observe and measure each operation for a sufficient time to permit reliable estimates of their consumption of chemicals and utilities and their production workloads. Equipment cost data was provided by the original equipment suppliers.

Data Discussion

The method of comparison between these two cleaning processes is on a cost per ton basis. The following data is organized to develop the cost of each input expressed in dollars per ton. Costs per unit of energy, chemical, etc. are selected to represent national averages, not Diesel Equipment costs.

Work Processed

The work process record for the Vibra degreaser is summarized below.

Vibra Degreaser Work Record

Date	Gondolas Cleaned
9-16-75	9 (1 Shift Only)
9-17-75	9 (1 Shift Only)
9-18-75	34
9-19-75	11 (1 Shift Only)
9-23-75	17 (1 Shift Only)
9-24-75	16 (1 Shift Only)
9-25-75	29
9-26-75	32
9-27-75	36
9-29-75	27
9-30-75	28
10-2-75	34
10-3-75	22
10-4-75	34

A similar record for the spiral washer follows:

Washer Work Record

Date	Gondolas Cleaned
8-5-75	21
8-6-75	21
8-7-75	11 (1 Shift Only)
8-8-75	13
8-9-75	16
8-10-75	20 1/2
8-11-75	24 1/2
8-12-75	25
8-13-75	25 1/4
8-14-75	10 3/4

In determining the average work processed, only those days having production on two shifts were used. The plant schedule during both operations was two shifts per day, six days per week. The average number of gondolas per day was 19.8 for the washer or 5940 gondolas per year. The degreaser averaged 30.7 gondolas/day or 9210/years. The weight of a gondola load was found to be essentially one ton (1980 lbs.). In the calculation of costs per ton, the gondolas were assumed to weigh a ton each.

Capital Investment - The current prices for each of the process equipment were obtained from the original suppliers. An installation cost of 15% of the equipment price was assumed. The building capital cost was charged on 1 1/2 x the square foot area occupied by each process x $25.9 per square foot. This information is summarized in Tables 1 and 2.

The capital linear Annual Payment for capital is calculated by:

$$LAP = \frac{I(1 + I)^n P}{(1 + I)^n - 1}$$

Where I = Interest Rate
n = Equipment or Building Life
P = Borrowed Principle.

The function $\frac{I(1 + I)^n}{(1 + I)^n - 1}$ equals:

0.131476 for 15 yrs.
0.11746 for 20 yrs.
0.110168 for 25 yrs.

when the interest rate is 10%.

Using the equipment and installation cost of $61,502 for the Vibra degreaser and a 20 year life factor, the annual cost for equipment is $7224. The cost per ton is obtained by dividing that value by 9210 tons per year or $0.784 per ton. The total equipment capital for the alkaline washer ($42,320) x the factor for a 20 year equipment life, yields a value of $4971 per year. At 5940 tons per year the cost per ton is $0.837 per ton. The building capital is calculated at a 25 year life and amounts to $0.030 for the degreaser and $0.091 for the washer on a tonnage basis.

Insurance - The insurance costs for both processes was figured at 2% of the total equipment and building capital, without the installation costs. The capital costs ($55,951 for the degreaser and $41,695 for the washer) was multiplied by 0.02 and divided by the tons processed per year. Thus, the interest cost per ton was found to be $0.122 per ton for the degreaser and $0.140 per ton for the washer.

Maintenance - The Maintenance Department estimated the montly cost for the degreaser at $715. When this number is multiplied by 12 to obtain the cost per year and divided by 9210 tons be year cleaned, a value of $0.932 per ton is obtained for the degreaser. The maintenance cost for the washer was based on 100 man-hours per year. Assuming a $7.00 per man-hour, an annual cost of $700 per year is obtained. This divided by the 5940 tons of work cleaned yields $0.118 per ton.

The maintenance costs as stated above represent 16% and 2% of the equipment prices per year respectively for vapor degreasing and alkaline washing. Although the vibratory conveyor means can be expected to cause more wear than is experienced in other metal cleaning equipment, 16% of the original equipment price per year is regarded as a high estimate. Based on conversations with Detrex and the operational schedule for degreaser and still clean-outs, the following maintenance estimate was prepared.

MAINTENANCE SCHEDULE

Still Operation 4 hrs./50 x 50 wks./yr x $7.00/hr.		--- $1400
Degreaser Clean-out (2 Per Yr.)		
2 men x 3-8 hr. shifts x 2/yr. x $7.00/hr.		--- $ 672
Bearing Replacements (2 Per Yr.)		
2 men x 2-8 hr. shifts x 2/yr. x $7.00/hr.		--- $ 448
Bearing Cost ($200 ea.)		--- $ 400
Replacement of Spiral and 2 Motors Every 6 Yrs.		
Spiral	$7500	
2 Motors	$4000	
	$11,500	
Installation (15%)	$ 1,725	
	$13,225 - 6 yrs.	---$2204
		$5124

This maintenance estimate would be excessive for most Vibra degreaser operations. However, the work process load handled by the Diesel Equipment Vibra degreasers is extraordinarily high and requires extra maintenance activity. The $5124 estimated is reasonably close to 10% ($5348) of the original purchase price. If 10% of the original purchase price is taken as the maintenance cost per year, the extra dollars would provide for the replacement of small components of the degreaser system. This value divided by the 9210 tons per year equals $0.581 as a cost of maintenance per ton of product.

Chemicals - The Vibra degreaser was studied for three weeks beginning September 15, 1975. No solvent was added to the system during this interval. The storage tank (78" x 65") had an initial level of 25 5/8 inches and a final level of 17 7/8 inches. Each inch within the storage tank was equivalent to 21.95 gallons. Thus, the 7 3/4 inches difference in level is equal to 170 gallons. This level could be determined within 1/4 of an inch or ± 5.5 gallons. The degreaser level at the start of the trial was 11 1/8 inches and at the end was 9 7/8 inches. This tank measures 64 inches square and 1 inch of elevation is equal to 17.73 gallons. The 1 1/4 inch level change in the degreaser equals 22 gallons of solvent consumed. An error of ± 1/4 inch is equal to ± 4.4 gallons. The total consumption for the three weeks was 192 gallons ± 10 gallons. The degreaser was operated two shifts per day and scheduled six days per week. Only five days of operation occurred in the third week, however, this difference is corrected by expressing all costs on a tonnage of product cleaned basis. When the 192 gallons used is divided by 17 workdays, 11.3 gallons consumption per workday is found. When this is multiplied by $2.15 per gallon and divided by 30.7 tons per day, a cost per ton of $0.791 is obtained.

The Ransohoff washer used Detrex 75LN detergent solution. This product weighs 10.6 pounds per gallon and has a value of $.26 per pound according to Detrex. When the alkaline washer was filled initially on August 5, 20 gallons of 75LN were added to the wash tank and 6 gallons to the rinse tank. A total of 39 gallons were added during the course of the next week to maintain the cleaning solution concentrations. The 75LN drum was removed from the operation between additions to prevent use for other purposes. Although the clean-out schedule was weekly, the washer was being cleaned on a two week basis. The second week consumption should be the same as the first week with the exception of the initial fill chemical requirements. Therefore, 104 gallons would be consumed in two weeks for an average of 52 gallons per week on a six day work week. The average consumption per day is 8.67 gallons or 91.9 pounds. A cost per ton of $1.206 is obtained by multiplying by $0.26 per pound and dividing by 19.8 tons per day.

Steam - The steam condensate from both stills and the degreaser were plumbed to a condensate collection tank and pumped to the boiler. To determine the steam condensate use a water meter was installed in the boiler return line. This meter read 686590 gallons on September 15, and 700229 on October 6, for a total of 13,639 gallons of steam condensate used. The degreaser is heated only during operating shifts. Thus, 802 gallons were used per day for the 17 operating days. A value of $0.493 per ton as a cost for steam is obtained by using $2.30 per thousand pounds of steam condensate, 8.2 pounds per gallon and 30.7 tons per day.

The washer steam condensate was collected four times. The average of these readings was 554 pounds of steam per hour. The cost of steam per ton was calculated as follows:

554 lbs./hr. x 16 hrs./day x $2.3/1000 lbs. x 1/19.8 tons/day.

By this means, a value of $1.030 per ton is identified as the cost of steam. This value appears to be quite low when compared to the equipment engineering steam consumption specification of 1620 pounds per hour or a verbal report from Ransohoff that this washer would be estimated to consume 1000 pounds of steam per hour. Consequently, the actual steam consumption could be 2-3 times higher than that determined. A measurement of the steam condensate required for the dryer section alone was found to be 260 pounds per hour. Another significant difference between the washer and the degreaser is the pressure of the steam used. The degreaser uses 15 psig steam whereas the washer uses 100 psig steam.

<u>Electric Power</u> - The electric power demand for the Detrex Vibra degreaser was found to be 10 amperes without work being processed and 11 amperes with work. The empty power demand for the Ransohoff washer was 17 amperes and 18 amperes were found with work being processed. In both cases, the current voltage was 460 volts. The average ampere demand was used along with the formula below.

$$\frac{\text{Amperes} \times \text{Volts} \times 1.73 \times \text{Power Factor}}{1000} = \text{Kilowatt Hours}$$

$$\frac{\text{Amperes} \times 460 \times 1.73 \times 0.85}{1000} = \text{Kilowatt Hours}$$

Using this formula, the kilowatt hours consumed by the degreaser were 7.10 KWH while that consumed by the washer was 11.84 KWH. The cost per ton calculated using 16 hours per day, $0.025 per kilowatt hour and the tons per day by each process. The electric power cost per ton was $0.093 for the degreaser and $0.239 for the washer.

Water - The water consumption for the Vibra degreaser and two stills was measured by water meters. The total measured flow for the 21 days of testing was 1,355,540 gallons. This consumption rate is approximately three times higher than that needed based on the equipment specifications. A water consumption rate of 2597 gallons per ton is obtained by dividing by 17 workdays and 30.7 tons per day. Using $0.04 per 1000 gallons, a water cost of $0.104 per ton is obtained.

A water meter on the Ransohoff washer showed a consumption of 4769 gallons per week excluding 1190 gallons to fill the equipment. The water requirement for two weeks of operation including the initial fill was 10,728 gallons. Dividing by 12 workdays and 19.8 tons per day, 45.2 gallons per ton was used. This equates to $0.002 per ton.

Waste Water Treatment - No waste water treatment charge is assigned to the degreaser system because this water can be immediately recycled through a cooling tower for reuse or can be used in other plant water requirements.

The gallonage of waste water from the Ransohoff washer was determined by a water meter to be 1391 gallons per week. At the end of two weeks, the washer capacity (1190 gallons) would be discharged to the Waste Water Treatment Plant. Thus, the total quantity of waste water requiring treatment per two weeks is estimated at 3972 gallons. Using 12 workdays per two weeks and 19.8 tons per day, 16.7 gallons per ton of water must be processed by the waste water treatment plant. A cost of $0.100 per ton is obtained as the water treatment cost when a value of $6.00 per thousand gallons is taken.

Solvent Disposal - The quantity of residue obtained from the stills was measured and sampled. This information is summarized in the table following.

STILL RESIDUES

Date	Weight % Non-Volatile	Gals. Res.	Wt. of Residue
9-18	60.0 51.1	100	965 Lbs.
9-22	81.5 87.6	(42.5)	414 Lbs.
9-25	88.2 84.0	40	496 Lbs.
9-29	89.8 86.3	55	532 Lbs.
10-1	88.3	100	726 Lbs.
10-6	80.9		
		338 Gals.	3133 Lbs.

The gallons (inserted for September 22, 1975) were calculated based on the average density of the other residues. All other results are as reported. The gallons of waste for disposal per ton of work processed was 0.648. The cost per ton was $0.130 using a disposal cost of $0.20 per gallon.

Assuming that the volatile portion of each sample is trichloroethylene, the quantity of trichloroethylene in the residues from the still amounted to about 60 gallons or about 31% of the total solvent consumed during the evaluation.

Make-up Air Heat Requirement - The Detrex Vibra degreaser required no ventilation. Thus, no charge is assessed to heating plant make-up air for this process.

The high water content of the exhaust ventilation from the washer prevented the actual measurement of the ventilation rate. The Ransohoff specification indicates a ventilation rate of 2240 cfm. The exhaust temperature was found to be 182°F. For estimating purposes, the mean temperature during the four cold months of the year in Michigan is estimated at 30°F. The expansion of the air due to heating within the washer decreases the air make-up to approximately 1700 cfm.

The heat requirement for this air is estimated by

$$1700 \text{ cfm} \times 0.0808 \frac{\text{lb.}}{\text{Ft.}^3} \times 0.25 \frac{\text{Btu}}{\text{lb.-}^\circ\text{F}} \times 40^\circ\text{F} \times 60 \frac{\text{mins.}}{\text{hr.}}$$

$$= 82416 \text{ Btu/hr.}$$

On a 16 hour workday, 1,318,656 Btu's per day would be needed. A daily cost of $1.648 is found using $1.25 per million Btu's for natural gas heating. However, this heating requirement would be needed only about four months per year. Annualized, the cost would amount to $0.550 per day or $0.028 per ton of work.

Two cases for an operating cost comparison are offered in this report. The first (Figure) is a direct summation of the cost developed in the preceding sub-paragraphs. The second (Figure) develops slightly different costs which present the case least favorable to vapor degreasing. In the least favorable case, the equipment life is recalculated on a 15 year basis. In addition, the maintenance cost is based on $715 per month or 16% of the equipment purchase price as discussed in the maintenance sub-paragraph earlier. The waste water treatment plant and the cost of heating make-up air is treated as a general overhead in the second case rather than assigned to the operation requiring these services and no charge is assessed.

No cost of direct labor was identified for either process. However, the material handling operations before and after both the spiral washing operation and the Vibra degreasing operation are identical and the direct labor should be exactly the same on a cost per ton basis. The differences in maintenance labor are charged appropriately to each process.

Conclusions

1. In the realistic case (Figure), the cost comparison between alkaline washing and vapor degreasing shows a savings of 17% by vapor degreasing.

2. The energy requirement of alkaline washing was found to be approximately twice as large as vapor degreasing. A number of literature sources indicate the energy demand of alkaline washing to be six to eight times that of vapor degreasing.

3. Alkaline washing equipment costs have been reported to be higher than comparable vapor degreasing equipment in earlier studies. Although the results may be specific to the case studied, the vapor degreasing equipment costs were 45% higher than the alkaline washing equipment.

4. In the cost comparison least favorable to vapor degreasing (Figure), alkaline washing and vapor degreasing are about equal in cost per ton of work processed.

5. Solvent consumption per ton of work was 0.37 gallons. This level of consumption is exceptionally low.

High production loads, good operating practices, free draining parts and the confinement provided by the degreasing equipment make this possible in this specific operation. However, this level could not be achieved in most vapor degreasing operations.

6. Of the total solvent consumed, 31% could be accounted for in the still residues in spite of good distillation practices. The oil content of still residues exceeded 80% by weight. The quantity of solvent remaining in the residues could not be recovered by any of the standard emission control equipment such as carbon adsorption.

SPECIFICATIONS - - VIBRA DEGREASERS

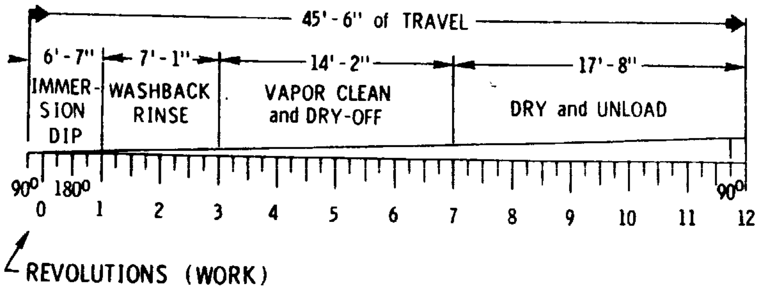
STD. SIZE	MODEL NO.	OVERALL DIMENSIONS	LOAD HGT.	UNLOAD HEIGHT	PROD. LBS STEEL/HR	SOLVENT CAP. GAL	*STEAM LBS/HR @ 15 psi	*WATER GALS/HR @ 50°F RISE	*DILUTION GALS/HR	APPROX. WEIGHT	STILL SIZE	**COMBINED BOILER HP
1	RV118-40-15-0	5'-0" x 4'-9" x 5'-8"	42"	34"	1200	40	100	200	50	1500 lb	S 60	7
2	RV218-40-11-7	5'-0" x 4'-9" x 5'-8"	42"	34"	1200	40	100	200	50	1600 lb	S 60	7
3	RV218-50-21-0	5'-0" x 4'-9" x 6'-0"	46"	39"	2400	40	150	305	60	3300 lb	S 185	15
4	RV312-50-15-7	5'-0" x 4'-9" x 6'-0"	46"	39"	2400	40	150	305	60	3450 lb	S 185	15
5	RV312-60-25-0	5'-10" x 4'-9" x 6'-7"	46"	41"	4500	60	210	430	75	3800 lb	S 185	16
6	RV312-60-21-7	5'-10" x 4'-9" x 6'-7"	46"	41"	4500	60	210	430	75	3950 lb	S 185	16
7	RV918-80-35-0	6'-10" x 5'-7" x 9'-0"	63"	54"	7500	95	325	665	100	5500 lb	S 185	20
8	RV918-80-30-7	6'-10" x 5'-7" x 9'-0"	63"	54"	7500	95	325	665	100	5050 lb	S 185	20

NOTE: *Degreaser Only. **Degreaser and Still.

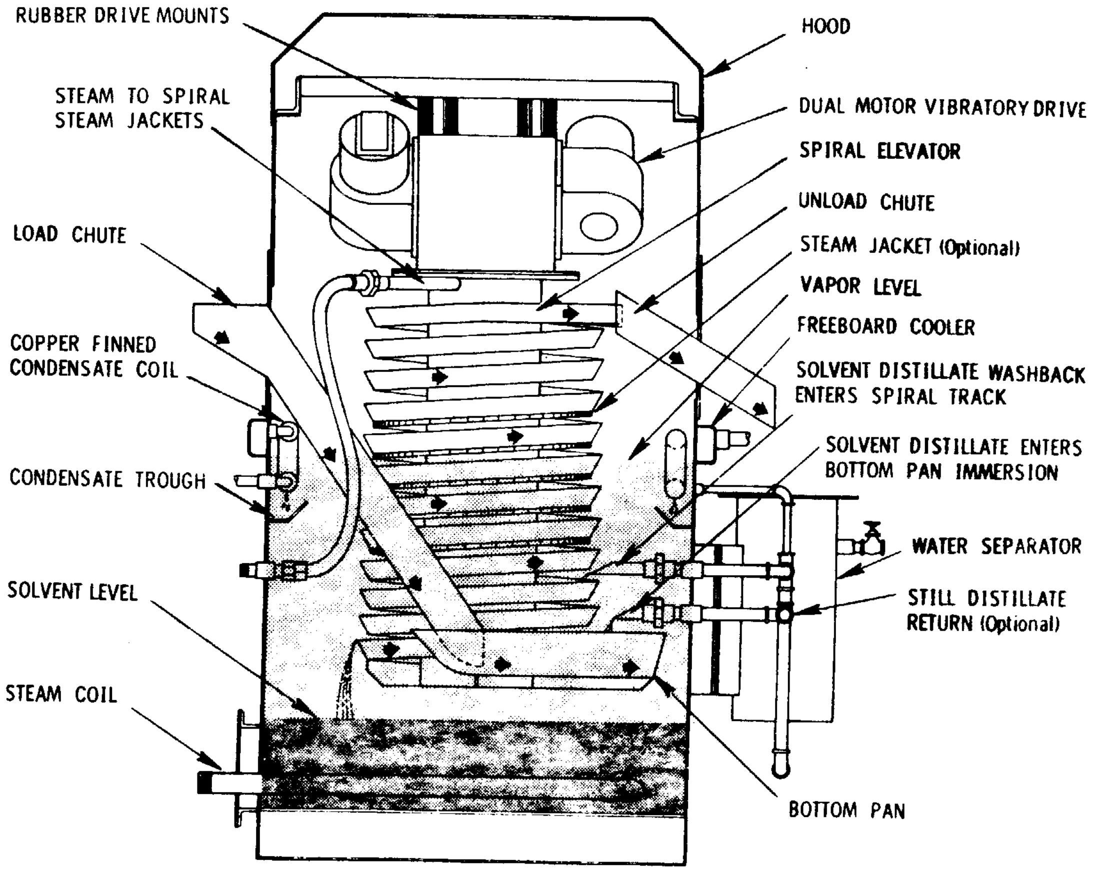
Standard Vibra Units can be supplied with a tote pan cleaning or basketed work section.

Submit sample or drawing of tote pan or basket and advise number per hour to be cleaned. This section can also be used for occasional work load of work not feasable to process thru Vibra Unit.

Optional Model S 225 recommended where soils that tend to cause "foaming" upon distillation are present.



Work Cleaning Cycle



Schematic Arrangement of Vibra Degreaser

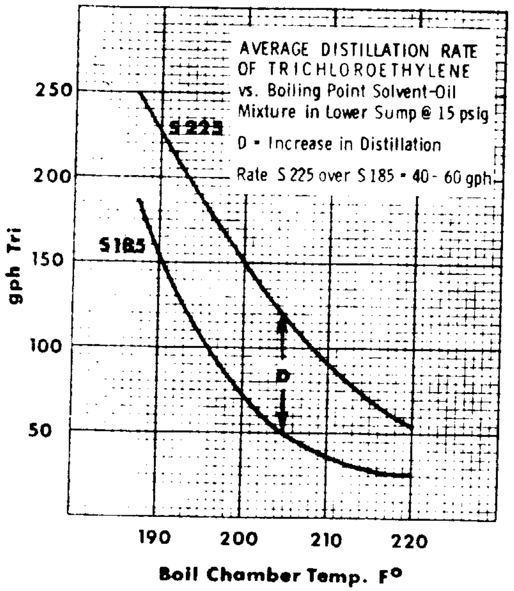
SPECIFICATIONS -- DETREX MODEL S185 and S225 STILLS for HALOGENATED SOLVENTS

SOLVENT	*DISTILLATION RATE gph		STEAM REQ'D. LB/HR.		*WATER gph MAX.	
	Model S185	Model S225	Model S185	Model S225	Model S185	Model S225
Trichloroethylene (Perm-A-Clor NA)	185	225	275 @ 15 psig	330 @ 15 psig	620	750
Perchloroethylene (Perk)	175	200	265 @ 50 psig	300 @ 50 psig	565	640
1,1,1-Trichloroethane (Perm-Ethane)	175	185	225 @ 7 psig	235 @ 7 psig	525	550
Trichlorotrifluoroethane (Detron "D")	70**	70**	70 @ 3 psig	70 @ 3 psig	400	400
Methylene Chloride	35**	35**	65 @ 3 psig	65 @ 3 psig	710	710

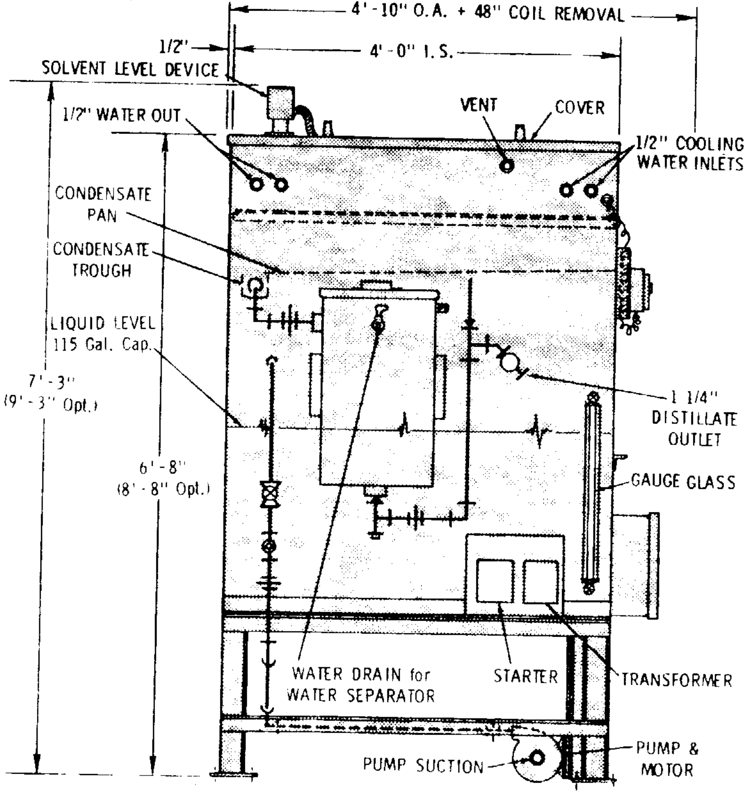
*Based on 60°F water inlet 50°F rise - Trichloroethylene, Perchloroethylene and 1,1,1-Trichloroethane; 10°F rise for Methylene Chloride, 20°F rise for Trichlorotrifluoroethane.

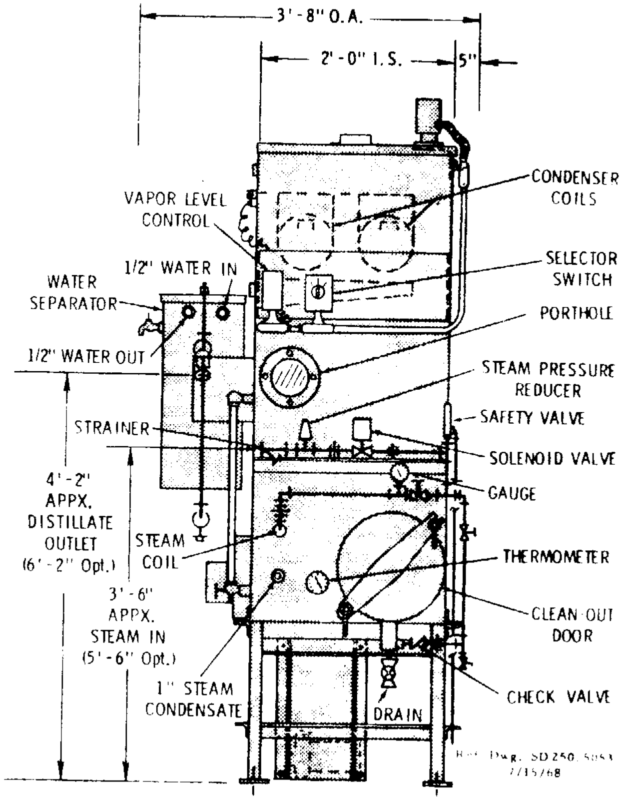
	MODEL S185	MODEL S225
Overall Dimensions	Length 4'-10" Width 3'-8" Std. Height: 7'-3" Opt. Height: 9'-3"	Length 4'-10" Width 3'-8" Std. Height: 7'-3" Opt. Height: 9'-3"
Distillate Discharge Height	Std. Height: 4'-2" Opt. Height: 6'-2"	Std. Height: 4'-2" Opt. Height: 6'-2"
Weight - Approx.	1,500 lbs.	1,600 lbs.
Liquid Capacity - Gal.	115	90
Pump Capacity	10 gpm @ 15 ft.	10 gpm @ 15 ft. Hd.
Current Characteristics S185 & S225	110/220/1/60 - 1/3 hp, 1750 rpm TEBB 230/460/3/60 - 1/4 hp, TEBB	

**Distillation rate can be increased by supplying chilled (40°F) water.



DISTILLATION RATE CURVES





ENGINEERING SPECIFICATIONS
RANSOHOFF COMPANY
HAMILTON, OHIO

DESIGN

Washer Size (Lgth. & Width) 21'-0" x 6'-0"
Washer Type Drum
Description Continuous
No. of Stages 3 Parts/hour 45 cu. ft.

RECIRCULATING SYSTEM

Pump Manufacturer Deming
Pump Type Vertical
Pump Capacity (G.P.M. & Head)
Wash Section 150 @ 60
Rinse Section 100 @ 60

BLOW-OFF SYSTEM

Blower Manufacturer New York
Blower Capacity (CFM & SP) 4800 @6"
Blow-off System Type Heated

SPRAY NOZZLES

Nozzle Manufacturer Ransohoff
Nozzle Type deflector
No. of Spray Nozzles 30

CONSTRUCTION

Washer Housing (Gauge) 10 ga. M.S.
Washer Tanks (Gauge) 1/4" M. S.
Tank Capacity (Gallons)
Wash 670
Rinse 520

EXHAUST SYSTEM

Fan Manufacturer American Fan
Fan Capacity (CFM & SP) 2240 @ 3/4

INSULATION

Insulation Manufacturer
Insulation Type

TANK HEATING

Steam X Gas Elec.
Manufacturer Tranter
Type 12 ga.M. S.

TEMPERATURE CONTROLS

Manufacturer Trerice
Type 9100
Capacity 900

PRESSURE REGULATOR

Manufacturer
Type
Capacity

TRAPS

Manufacturer Yarway
Type #131
Capacity 4860

SERVICE REQUIREMENTS

WATER

Initial Filling 1190
Consumption 12 G.P.H.

AIR

Quantity (CFM) @ PSI

STEAM

Consumption 1620 lbs./hour

CONDENSATE

Consumption 1620 lbs./hour

Consumption CFM @

HORSEPOWER (EACH MOTOR)

Wash Pump 5
Rinse Pump 3
Conveyor Drive
Drum Drive 1
Exhaust Fan 1
Blow-off System 7.5
Other

Total HP 17.5

Investment Capital

	Vibra Degreaser	Washer
Equipment	$53,480	$36,800
Installation (15%)	8,022	5,520
	$61,502	$42,320
Building	$ 2,471	$ 4,895
Total	$63,973	$47,215

TABLE I

Industrial Building*

Shell (M&L) Cost	\$ 4.09/Ft.^{2}
Lighting and Electrical	1.75
Heating and Ventilating	1.50
Plumbing	1.70
Fire Prevention	1.10
	\$10.14 (1968 Base)
	\$17.3/Ft.^{2} (8%/Annum 1.71 Multiple in 1975)
Sub-Contract Cost (1.3)	\$22.5/Ft.^{2}
Contingency (15%)	\$25.9/Ft.^{2}

*Derived from "Modern Cost-Engineering Techniques by H. Popper. The 8% inflation rate was estimated by the author.

REVISED 10/

WASHER MAINTENANCE

TYPE WASHER RANSOHOFF 2 STAGE ROTARY DRUM WASHER DED NO.

LOCATION PRESS & MISC. DEPT. NO. 951 BAY

TYPE OPERATION CLEANING COLD FORMED PARTS

TYPE CLEANER DETREX 75 LB

WASHER INFORMATION:

	WASH TANK	RINSE TANK
1. OPER. TAMP.	160/170°F	130/140°F
2. TANK CAPACITY	670 GAL	520 GAL.
3. CLEANER CONCENTRATION	3% by Vol.	1% by Vol.
(A) NEW CHARGE	20 GAL.	5.5 GAL.
(B) ADDITIONS	2-3 GAL/8 HRS	0.5 - 1. GAL/8 HRS

4. CHANGE PERIOD WEEKLY.

NOTES:

Figure

Case 1

Operating Cost Comparison
(Cost Per Ton)

	Vibra Degreaser	Washer
Equipment	$0.784	$0.833
Building	0.030	0.091
Insurance	0.122	0.140
Maintenance	0.581	0.118
Chemicals	0.791	1.206
Steam	0.493	1.030
Electric Power	0.093	0.239
Water	0.104	0.002
Waste Water	---	0.100
Solvent Waste Disposal	0.130	---
Make-Up Air Heat	---	0.028
	$3.128	$3.787

Figure

Case 2

Operating Cost Comparison
(Cost Per Ton)

	Vibra Degreaser	Washer
Equipment	$0.878	$0.837
Building	0.030	0.091
Insurance	0.122	0.140
Maintenance	0.932	0.118
Chemicals	0.791	1.206
Steam	0.493	1.030
Electric Power	0.093	0.239
Water	0.104	0.002
Waste Water	---	---
Solvent Waste Disposal	0.130	---
Make-Up Air Heat	---	---
	$3.573	$3.663

APPENDIX - C2

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Evaluation of Vapor Degreaser Covers

Eaton Corporation
Saginaw, Michigan

Prepared By:

K. S. Surprenant
The Dow Chemical Company

Prepared For:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

INTRODUCTION

Chlorothene NU (inhibited 1,1,1-trichloroethane) was developed as an improved grade of Chlorothene, more easily recovered by distillation and exhibiting much improved corrosion characteristics. Due to the similarity in the conditions of distillation and vapor degreasing, it was thought that this solvent might find further markets in this application. In fact, several companies have successfully used this solvent in vapor degreasing for some time.

The laboratory vapor degreasing studies made on Chlorothene NU showed sufficient success to encourage field evaluations. For the purpose of these field tests, Chlorothene NU is identified as MC154 to prevent damage to present Chlorothene markets in the event of failure and to conceal from competition the entry of this solvent into the degreasing market.

The foremost purpose of this test was to determine the relative operating costs of trichloroethylene and MC154. In an effort to insure our obtaining this information, the test site conditions were chosen to reduce the probability of solvent decomposition. This test was designed to provide data on the stability of the solvent and stabilizer system, the relative toxicity and cleaning performance, and to define the equipment changes necessary to use this solvent in a degreaser designed to use trichloroethylene.

PROCEDURES AND EQUIPMENT

The equipment used in this test consisted of a Detrex open-top degreaser with only one compartment- a boiling chamber. The accessory equipment consisted of a water separator, storage tank, and spray equipment. This unit was heated electrically and held about two drums of solvent. The degreaser had developed considerable rust on the walls.

All the parts processed by this equipment were steel. These parts were given a heat treatment and oil quench just prior to cleaning. Infra-red analysis of the quench oil revealed it to be mostly aliphatic hydrocarbon with 5-10% aromatic hydrocarbon.

In general, the procedures normally practiced by this concern were not changed so that the cost information developed would not be prejudiced whether these techniques were good or bad. Their cleaning cycle was vapor-spray-vapor and the machine was operated three shifts each day, five days per week. On Monday mornings the equipment was boiled down, the residues removed, and clean solvent added. The only change in their operating practice was the operation of the degreaser with one half of their electrical coils. Four coils were used for trichloroethylene while only two coils were used with MC154. This was necessary to contain the vapors. Trichloroethylene and MC154 were operated in the degreaser for periods of three to four weeks with the top left open all the time in each case and with the top closed during the down time to determine the savings resultant from simply closing the top. It was their normal practice to leave it open.

All of the solvent added to or taken from the machine was weighed - including the samples and residues. A record of the parts processed was also kept. This data was used to prepare the cost comparison developed in this report. Samples were taken daily during the MC154 phases of the operation for analysis of the solvent and stabilizer system. The analytical procedures used are enclosed in Report AA-1370, "Chlorothene NU Laboratory Vapor Degreasing Evaluations."

DISCUSSION:

Economics

There were two aspects of this cost study - one, the relative costs of heating, the other a comparison of the consumption of the solvent All other costs either were the same or were assumed to be the same.

As mentioned, four electrical heating elements were required for trichloroethylene while only two were required for MC154. Each of these elements operated on a 460 volt circuit and drew about 10 amperes. They are wired in parallel. Since the difference between operating with the two solvents was two elements, the power these would have consumed is a measure of the heating savings experienced by operating with MC154.

$$\frac{\text{2 elements (460 volts)(10 amperes)(24 hrs)}}{1000} = \frac{\text{220.8 KWH}}{\text{day}}$$

$$\text{220 KWH (\$.0132/KWH)} = \frac{\text{\$2.90 saved}}{\text{day}}$$

or about $710.00 per year

Tables I through IV are the records of the solvent consumed, hours of operation, and the pounds of parts processed. Again, it should be noted that the tables identified as "degreaser top closed" refers to closing the top only during down periods. In order to cancel out the differences in the amounts of solvent remaining in the residues after boildown, nonvolatile content determinations were made on each sample. The volatile matter was assumed to be solvent and was subtracted from that added to the equipment to obtain the net solvent consumption. This technique assumes that both solvents can be recovered equally well from the oil; this should be approximately correct. In the case of trichloroethylene, only the solvent in the residues and the amount remaining in the machine at the completion of the test were subtracted from the total added because no samples were taken. In calculating the consumption of MC154, the estimated weight of the samples taken was also subtracted to get the net consumption.

On Table V the four phases of this test are summarized. It was interesting to note that closing the degreaser top in down periods saved between 30-31 pounds of solvnet per ton of work and that under both operating conditions MC154 used from 18-19 pounds of solvent per ton of work less than trichloroethylene.

Toxicity

Table VI shows the results of the Halide Meter Surveys run on trichloroethylene and MC154 under similar conditions. The rectangles represent the surface of the open top areas of the degreasers and the numbers are the parts per million (ppm) concentration of the vapors found at each location. Note: The American Conference of Governmental Industrial Hygenist suggest MAC values of 100 ppm for trichloroethylene and 500 ppm for MC154. While some of the readings were above the recommended values, these were all taken just above the lip of the degreaser and not in the air which the operators would be inhaling. The values obtained for the air the operator would be breathing were safe for both solvents: 45 ppm for trichloroethylene and 50 ppm for MC154.

Cleaning Performance

The general opinion of the operation personnel on the cleanliness of the parts was that it was equivalent to that of trichloroethylen No more detailed study of this attribute was made.

Stability

Boiling Sump Samples:

On Figure I the graphs of Acidity, Specific Gravity, and the Stabilizers are plotted. The stabilizer concentrations are expressed as percent by volume of the total sample and not corrected for the oil content. The encircled points correspond to the boildown residue samples. It should be noted that all three stabilizers maintained a satisfactory concentration throughout the test period except for these residue samples. However, the condition represented by the residue samples is prevalent in the degreaser only briefly once each week. In addition, high concentrations of oils inhibit some of the solvent decomposition reactions e.g. the aluminum reaction.

The graph plotting the acid concentrations shows the residue samples to be somewhat high in acidity. The oil in these samples interfered with the titration and the silver nitrate qualitative test showed little or no chloride. Therefore, the acidity fourd is not due to HCl and not a result of solvent decomposition.

Warm Dip Samples:

Acidity, Chloride, and the stabilizer concentrations are plotted on Figure II. These four graphs demonstrate the excellent stability of the solvent under the conditions of this test.

It should be noted that much of the variation in the stabilizer concentrations of both warm dip and boiling sump samples is due to the gradual increase of soils in the boiling sump. This increase in oil concentration reduces the specific gravity and increases the boiling temperature of the boiling sump. The distribution of the concentrations of the stabilizers between the warm dip and the boiling sump is a function of the temperature of the boiling sump

In this instance, the boildown cycle was once each week and this cycle is observable in the stabilizer concentrations of both sets of samples.

CONCLUSIONS:.

1. \$2.90 per day (\$710 per year based on 245 days/year) was saved in the cost of heating the degreaser by using MC154.
2. When the equipment was operated with the top open all the time, MC154 used 18 pounds of solvent less than trichloroethylene per ton of work, or saved \$1.4 per ton of work processed (using Dow list prices).

3. When the equipment was operated with the top closed in down periods, MC154 used 19 pounds of solvent less than trichloroethylene per ton of work, or saved $1.8 per ton.
4. Comparing trichloroethylene operations with open and closed top, 30 pounds solvent per ton less was consumed when the top was closed, or a savings of $3.7 dollars per ton.
5. Comparing MC154 operations with open and closed top, 31 pounds solvent , or $4.1, was saved by closing the top.
6. Neither solvent presented a toxicity hazard under normal operating conditions. However, in an accident (e.g. as spill), MC154 would provide a greater degree of safety.
7. The cleaning performance of MC154 was considered equal to that of trichloroethylene.
8. The solvent stability was excellent under the operating conditions found in this test site.

RECOMMENDATIONS:

1. Field testing of MC154 should be continued and the conditions of operation expanded with regard to metals processed and soils cleaned.
2. Further field tests should be found in other types of equipment to further document the cost of operating with MC154 as compared to that of trichloroethylene.

TABLE I

Consumption of Trichloroethylene
(Degreaser Top Closed in Down Time)

Date	Tri Added (lbs)	Boildown Residues (lbs)	% tri in Residues	Wt. of tri in Residues (lbs)	Operating Time (hrs)	Work Load (pounds)
2-6-61	1152				20	2720
2-7-61					24	2681
2-8					24	1459
2-9					24	1537
2-10					20	2033
2-13	410	127	27	34	20	1022
2-14					24	2044
2-15					24	1287
2-16					21	1905
2-20	470	125	61	76	19	1644
2-21					24	2922
2-22					24	2830
2-23					20	3089
2-27		112	7	8		
	2032 lbs used gross			118 lbs	288 hrs	27,173 lbs
	-118 lbs in residues					13.59 Tons
	1914 lbs					
	-576 lbs good tri left					
	1338 lbs used net					

99 lbs tri used per ton of work

TABLE II

Consumption of Trichloroethylene
(Degreaser Top Open)

Date	Tri Added (lbs)	Boildown Residues (lbs)	% tri in Residues	Wt. of tri in Residues (lbs)	Operating Time (hrs)	Work Load (pounds)
10-31-60	1062				17	911
11-1-60					24	1538
11-2	258				24	1673
11-3					16	1875
11-4					20	1667
11-7	448	166	28.3	47	21	1248
11-8					24	2180
11-9					24	1183
11-10					24	1954
11-11					20	1683
11-14	370	117	9.4	11	21	980
11-15					24	2059
11-16					24	2433
11-17					24	1225
11-18		106	11.4	12	20	1323
Totals	2138 lbs used gross -70 lbs in residues 2068 lbs -524 lbs good tri left 1544 lbs used net			70 lbs	327 hrs	23,932 lbs 11.97 tons

129 lbs tri used per ton of work

TABLE III

Consumption of MC-154
(Degreaser Top Closed)

Date	MC-154 Added (Lbs)	Boildown Residue (Lbs)	% MC-154 in Residue	Wt. of MC-154 in Residue (Lbs)	Operating Time (Hours)	Work Load (Pounds)
11-21-60	922				20	1229
11-22					24	2385
11-23					22	915
12-1					24	448
12-2					20	336
12-5	353	72	20	14	20	1089
12-6					24	1593
12-7					24	1784
12-8					24	1132
12-9					24	1993
12-12	373	54	9	5	19	1112
12-13					24	1005
12-14					24	1383
12-15					24	1848
12-16					24	2646
12-19	345	161	27	43	20	803
12-20					24	1763
12-21					24	2152
12-22					23	1870
12-27	329	207	19	39	10	655
12-28					24	1900
12-29					24	2949
12-30		95	72	68	20	2000
				169 Lbs	510 Hours	34,990 Lbs 17.50 Tons

2322 lbs used gross in residues
-169 lbs
2153 lbs
-750 lbs good MC-154 left
1403 lbs
-12 lbs (30 samples 0.4 lbs/sample)
1391 lbs MC-154 used net

80 Lbs MC-154
used per
ton

TABLE IV
Consumption of MC154
(Degreaser Top Open)

Date	MC154 Added (lbs)	Boildown Residues (lbs)	% MC154 in Residue	Wt. MC154 in Residues (lbs)	Operating Time (Hours)	Work Load (Pounds)
1-3-61	1020				19	1154
1-4-61					24	1834
1-5-61					24	2170
1-6-61					20	2928
1-9-61	406	92	39	36	19	574
1-10-61					24	1773
1-11-61					24	1060
1-12-61					24	556
1-13-61					20	601
1-16-61	254	91	13	12	20	465
1-17-61					24	755
1-18-61					24	1980
1-19-61					21	1204
1-23-61	362	85	42.5	36	21	1250
1-24-61					24	2054
1-25-61					24	1608
1-26-61					20	1908
1-30-61		67	17	11	--	----
	2042 lbs used gross			95 lbs	376 hrs	23,883 lbs 11.94 Tons

2042 lbs used gross
- 95 lbs in residues
1947
-610 lbs good MC154
1337 lbs
- 12 lbs (30 samples at 0.4 lbs/sample)
1325 lbs

111 lbs MC154 used per Ton of work

TABLE V

SUMMARY OF CONSUMPTION DATA

Solvent	Consumption (Open Top) Lbs solvent/ton work	Consumption (Closed Top) Lbs solvent/ton work	Difference
Trichloroethylene	129	99	30
MC154	111	80	31
Difference	18	19	

COST PER TON OF WORK

Cost of MC154 (truckload)	$0.1325 per lb.
Cost of Trichloroethylene (truckload)	0.1250 per lb.

TRICHLOROETHYLENE - OPEN TOP

129 lbs per ton $\left(\frac{\$0.1250}{\text{lb}}\right)$ = $16.1 per ton of work

TRICHLOROETHYLENE - CLOSED TOP

99 lbs per ton $\left(\frac{\$0.1250}{\text{lb}}\right)$ = $12.4 per ton of work

MC154 - OPEN TOP

111 lbs per ton $\left(\frac{\$.1325}{\text{lb}}\right)$ = $14.7 per ton of work

MC154 - CLOSED TOP

80 lbs per ton $\left(\frac{\$.1325}{\text{lb}}\right)$ = $10.5 per ton of work

APPENDIX - C3

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Evaluation of Two Refrigerated Freeboard Chillers

Hamilton Standard
Windsor Locks, Connecticut

Prepared By:

K. S. Surprenant
The Dow Chemical Company

Prepared For:

Emission Standards and Engineering Div.
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

Two open top degreasing operations equipped with refrigerated freeboard chillers were evaluated to determine the emission control efficiency achieved by this device. One system was operated consistently with a cover while the other was left open.

The freeboard chillers effected 43 percent and 40 percent lower emission rates of methylene chloride than the same equipment without the emission control operating. A 50 percent ± 5 percent reduction in solvent losses to the atmosphere was derived from the use of a cover.

The added energy required by either emission control method is negligible compared to the basic degreasing operation.

Introduction

Autosonics Inc. recommended Hamilton Standard as a suitable site for the evaluation of their refrigerated freeboard chiller equipment. This device is referred to as a Cold Trap when manufactured by Autosonics. Two Cold Traps were in operation at this location. Both were reported to be recent installations with the most up-to-date design factors.

The Cold Trap installations at Hamilton Standard are a portion of a program to comply with Connecticut Air Pollution Control Regulations. Earlier, the degreasers at this location were operated with trichloroethylene. Currently, the degreasing operations are being converted over to exempt solvents, including methylene chloride. Due to the lower boiling point of methylene chloride (104°F), vapor degreaser equipment design must be modified. The equipment modifications include: 1) a reduced heat input, 2) an assured supply of coolant for vapor condensation and an extended freeboard to width ratio (0.75). These equipment modifications were sufficiently extensive to cause the purchase of new equipment rather than reconstruction of existing degreasers. Cold Traps were purchased with the new vapor degreasing equipment to assure minimum solvent emission rates. Methylene chloride was the solvent used in this equipment.

Objective

The purpose of this test program is to evaluate two refrigerated freeboard chillers (Cold Traps) as a means of controlling solvent emissions from two open top degreasers. The information needed for this evaluation includes determining:

1. the efficiency of this device in reducing solvent emissions to the atmosphere,
2. the cost/benefit relationship of this emission control system,
3. the energy requirement of the emission control system,
4. any alternate emissions created by the emission control system.

This data base is being developed to forecast the magnitude of emission reductions which can be achieved nationally and the effect on businesses involved. This information combined with the results of other testing will be used to design emission control regulations which effectively limit air pollution and are practical for industrial application.

Equipment

Department 203

Crest Ultrasonics Corp. supplied the open top degreaser used in this plant location. This stainless steel degreaser has an open top area of 41" x 65". The inside working area is 36" x 60". The degreaser is supplied coolant from a five horsepower Tecumseh refrigeration system, Serial CL522HT and is electrically heated. It is Hamilton Standard No. E30407. The distance from the top of the vapor zone to the lip of the degreaser (freeboard) is 30 inches. The refrigerated freeboard chiller or Cold Trap is an Autosonics Model No. 75, Serial No. 2558. This 3/4 horsepower refrigeration system supplies coolant to dual heat exchange coils constructed of copper tubing with fins. These two coils are located immediately above the primary condenser coils around the perimeter of the degreaser. This construction is shown along with the rough degreaser design in Figure 1.

This degreaser works three shifts per day and six days per week. When shut down, both the heat and the primary condenser refrigeration system are turned off. However, the Cold Trap is left on. This degreaser is equipped with a counterbalanced metal cover. The degreaser is covered whenever it is not in immediate use.

Department O&R

The stainless steel degreaser is this department is another Crest Ultrasonics open top design. The open top work area of this degreaser is 45" x 45" while the internal work area is 40" x 40". Again, the freeboard was found to be 30 inches. This degreaser is supported with a small 20-30 gallon per hour stainless steel still and both pieces of equipment are steam heated. Both the degreaser and the still are equipped with separate refrigeration systems to provide coolant for the primary solvent condensation. E29957 is the Hamilton Standard identification number for this equipment. The cover on this O&R Department degreaser is not hinged to the equipment as in the case of the Department 203 degreaser. Neither degreaser (Department 203 or O&R) is equipped with a lip exhaust system and both have a spray pump and spray lance. The degreaser and still operate eight hours per day and five days per week but the Cold Trap supporting it operates continuously.

The Cold Trap is installed exclusively on the vapor degreaser and has three finned heat exchange tubes around the periphery of the degreaser just above the primary condenser coils. The refrigeration system for the Cold Trap is one horsepower and was supplied by Autosonics Inc. It is identified by Model 100 and Serial No. 2487. The essentials of this equipment design are shown in Figure 2.

Experiment Design

It was planned to operate both degreasers with the Cold Trap "on" from June 25 through July 16 and with the Cold Trap "off" from July 16 to August 1. A longer operating time with the Cold Trap on was deliberately planned due to the expected lower consumption during this operating interval. The solvent emission rate was determined by difference in solvent inventory in the equipment. Since all chambers within both degreasers overflow to the boiling chamber, the difference in solvent inventory could be obtained by measuring the solvent level in the boiling chamber when the other compartments were full to the overflow level. The only additional precaution needed for the degreasing system in Department O&R was to assure that the still was full to the upper level of the float control. This was done manually before each measurement. With the heat turned off, the solvent level in each of the degreasers could be measured to plus or minus 1/8 of an inch.

Both degreasers were operated in their normal manner and on the schedules described earlier. However, the Department O&R degreaser was left open at all times throughout the test period. Department 203 degreaser was covered except when work was being processed. This use and nonuse of covers was deliberate to provide further information on the emission control which can be experienced through the consistent use of a cover. Due to the inconvenience of using most covers, most open top vapor degreasing equipment is left open most of the operating time. The better operations cover the degreasers during down shifts and weekends. The practice of covering open top degreasers when not in immediate use, as is done at Hamilton Standard, is a rare practice.

Data Discussion

Department 203

The measurement of solvent emission rates was made by difference from solvent inventory within the equipment as discussed earlier. Using the inside dimensions of the degreaser (36" x 60") a factor of 9.35 gallons per inch was calculated. This value was confirmed by measuring the solvent level before (20.5 inches) and after (26.5 inches) a 55 gallon drum of solvent was added to the degreaser. The six inch difference in level times 9.35 gallons per inch provides an estimated volume of solvent at 56 gallons. The volume of solvent contained in this drum was probably higher than normal (54 gallons) because it was loaded quite full from the Hamilton Standard bulk tank without precise measurement. The solvent level could be determined within plus or minus 1/8 on an inch. This means that errors in measurement from this source would be plus or minus 1.2 gallons.

Table 1 summarizes the solvent use record for this department with the Cold Trap "on" and "off". No records were kept between July 16, 1975 and July 24, 1975 due to the failure of an electric resistance heater. This resistance heater burn out caused the entire solvent to be withdrawn from the equipment so that the heater could be replaced. When resistance heaters short out, solvent decomposition often occurs. In many cases, this solvent decomposition requires the disposal of the solvent. This solvent loss is not recorded in the data of this report because it is not related to the performance of the emission control device. However, it does illustrate a type of solvent loss which is not recoverable by any emission control method.

The degreaser was recharged and the test re-initiated early on the morning of July 24. The test was aborted on the late evening shift of July 30, 1975 soon after the installation of ceiling fans which caused high solvent vapor odors in the area.

The average solvent consumption for total days of testing and per operating day are derived on Table 2. Percent emission control achieved is the difference between the consumption rate with the Cold Trap "on" versus that with the Cold Trap "off" on a daily basis.

The number of workloads processed with the Cold Trap "on" totaled 723 whereas 170 workloads were processed with the Cold Trap "off". Thus, the workloads processed on a daily basis with the Cold Trap "on" was somewhat higher than the operation with the Cold Trap "off". This tends to make the percent emission control determined slightly conservative. When this information is weighted with the fact that this equipment is in use three shifts per day and six days per week the emission control (43 percent) obtained on a total day basis is more realistic.

The capital and operating costs for the Cold Trap are outlined in Table 3. Again referring to Table 2, a volume of 2.76 gallons per day were conserved through the use of the Cold Trap. Thus, the reduced solvent consumption can be calculated by:

2.76 Gals./Day x 7 Days/Wk. x 50 Wks./Yr. = 966 Gals./Yr.

The July 7, 1975 issue of Chemical Marketing Reporter shows the price of methylene chloride at $0.165 per pound. Using this value and 11 pounds per gallon, the dollar value of 966 gallons per year equals $1753. A net profit of $977 is obtained when the operating cost of $776 is subtracted from the annual savings. This represents 2.26 times the operating cost per year.

Department O&R Degreaser

The method of emission measurements for this degreasing system was the same as that for Department 203 with the exception that the still was manually filled to a constant volume before each reading. The inside working dimensions of this degreaser are 40" x 40". One inch of level change in the degreaser therefore equals 6.93 gallons of methylene chloride. An error of plus or minus 1/8 of an inch in measuring the solvent level would result in the error of 0.87 gallons in this degreaser.

The gallons used, total days of testing and operating days are summarized on Table 3. No data was taken during a shut-down period between July 16 and July 22 because of the failure of the refrigeration system for the primary condenser coils on the degreaser during this interval. During the testing between July 10 and July 16, a drum of methylene chloride was added to the degreasing operation. None of the other test intervals on this degreasing operation or that of Department 203 included an estimate of solvent delivered from bulk storage. As mentioned, these drums are not filled to a specific volume. This combined with the fact that the primary refrigeration system failed toward the end of this test interval reduces the confidence level of this data. Due to these facts, this portion of the data was disregarded in developing the average consumption rate per day of testing and per operating day in Table 4. Again, on Table 5 the emission control efficiency is calculated as before.

This degreaser was left open 100 percent of the time even though it was used only one shift per day and five days per week. As the result of this method of operation, the emission control (40 percent) obtained on a total test day basis provides a more factual evaluation of the Cold Trap.

The number of workloads processed with the Cold Trap "on" was 45 and with it "off" was 23. Thus, the loads processed per operating day are essentially the same as shown below.

Cold Trap	Loads Per Day of Testing	Loads Per Operating Day
"on"	2.14	3.21
"off"	2.30	2.88

Table No. 6 outlines the capital investment and operating costs. A volume of 1.17 gallons per day was prevented from being lost with the Cold Trap "on". On an annual basis (350 days), 410 gallons of solvent would have been prevented from being lost from the system. The value of this solvent is obtained by:

410 Gals./Yr. x 11 Lbs./Gal. x $0.165/Lbs. = $744

Since the operating cost was estimated at $1016 per year, this installation achieves emission control at a loss of $272 per year or an annual savings to cost ratio of 0.73.

Comparison of Covered Vs. Open Operation

The solvent emission rate in pounds per square foot-hour is calculated from the following formula.

$$\text{Lbs./Ft.}^2\text{-Hr.} = \frac{\text{Gals./Oper. Day x 11 Lbs./Gals.}}{\text{Oper. Hrs./Day x Ft.}^2\text{ of Open Top Area}}$$

The emission rates determined by this formula are summarized in Table 7. The final column of this table indicates the emission control efficiency experienced with the covered degreaser versus the degreaser operating in the open condition. The available data permits the calculation of this emission control efficiency both with and without the operation of a Cold Trap. Of course, this calculation is hypothetical to the extent that it presumes that both operations are similar. In fact, the operations are similar with regard to their general open top design and their mutual use of methylene chloride. They are dissimilar in their dimensions, the parts processed through them and the rooms (air movement) in which they operated.

In spite of these variances between the two operations, this data can be interpreted to say that a 50 percent emission control ± five percent could be expected by covering a methylene chloride open top degreasing system. It should be noted that the higher evaporation rates of methylene chloride or Fluorocarbon 113 will cause greater emission rates from open equipment at room temperature than the other vapor degreasing solvents. In addition, methylene chloride has the lowest vapor density. This results in greater susceptibility to vapor disturbance as the result of air movement in the work area.

Conclusions:

1. Two Cold Traps operating on open top degreasers were found to be accomplishing a reduced emission rate of methylene chloride vapors of 43 percent and 40 percent.

2. Based on present replacement pricing obtained from the original supplier (Autosonics Inc.), the Department 203 Cold Trap was returning 266 percent per year on the total annual operating cost while the O&R Department unit per year year was controlling emissions at a loss of $272.

3. By contrasting the two degreasing operations, a forecast of a 50 percent ± 5 percent emission reduction was determined by the consistent use of a cover versus leaving the degreaser open.

4. The energy consumption by either the Cold Trap or covers is nominal when compared to the energy demand of basic operation.

5. No alternate air, water or solid pollution is created by use of these emission control techniques.

TABLE 1

DEPT. 203 SOLVENT RECORDS

Cold Trap	Dates	Gallons	Total Days	Operating Days
"on"	6/25/75-7/10/75	58	15	12
"on"	7/10/75-7/16/75	19	6	5
"off"	7/24/75-7/30/75	45	7	6

TABLE 2

DEPT. 203 - SUMMARY OF DATA

Cold Trap	Gallons	Total Days	Gals./Day	Operating Days	Gals./Oper. Day
"on"	77	21	3.67	17	4.53
"off"	45	7	6.43	6	7.50
Emission Control			43%		40%

TABLE 3

DEPT. 203

Capital Investment

Price	$3300
Installation (Included)	-
Floor Space (6.2 Ft.^{2} x $25.9/Ft.^{2})	161
TOTAL	$3461

Operating Costs

Capital	
Equipment (15 Yrs.)	$ 434
Building (25 Yrs.)	18
Insurance (2 of Capital)	
Equipment	66
Building	3
Maintenance (4% of Capital)	132
Utilities	
Electricity (3/4 Hp. Motor)*	123
Labor	0
Return on Investment	0
TOTAL COST/YEAR	$ 776

*3/4 Hp. X 0.746 KWH/Hp. X 24 Hrs./Day X 365 Days/Yr. x $0.025/KWH

TABLE 4

DEPT. O&R - SOLVENT RECORD

Cold Trap	Dates	Gallons	Total Days	Operating Days
"on"	6/25/75-7/10/75	26	15	10
"on"	7/10/75-7/16/75	7	6	4
"off"	7/22/75-8/01/75	29	10	8

TABLE 5

DEPT. O&R - SUMMARY OF DATA

Cold Trap	Gallons	Total Days	Gals./Day	Operating Days	Gals./Oper. Day
"on"	26	15	1.73	10	2.60
"off"	29	10	2.90	8	3.63
Emission Control			40%		28%

TABLE 6

DEPT. O&R

Capital Investment

Price	$4460
Installation (Included)	-
Floor Space	-

Operating Costs

Capital	
Equipment (15 Yrs.)	$ 586
Insurance (2% of Capital)	89
Maintenance (4% of Capital)	178
Utilities	
Electricity (1 Hp. Motor)*	163
Labor	0
Return on Investment	0
TOTAL COST/YEAR	$1016

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*1 Hp. x 0.746 KWH/Hp. x 24 Hrs./Day x 365 Days/Yr. x $0.025/KWH

TABLE 7

COVERED VERSUS OPEN OPERATION

LOSS RATE PER OPERATING HOUR

Cold Trap	Covered (Dept. 203)	Open (Dept. O&R)	Emission Control
"on"	0.112 lbs./ft^2.-Hr.	0.254 lbs./ft^2.-Hr.	56%
"off"	0.186 lbs./ft^2.-Hr.	0.354 lbs./ft^2.-Hr.	47%

Figure 1
DEPT. 203 NO E 30407

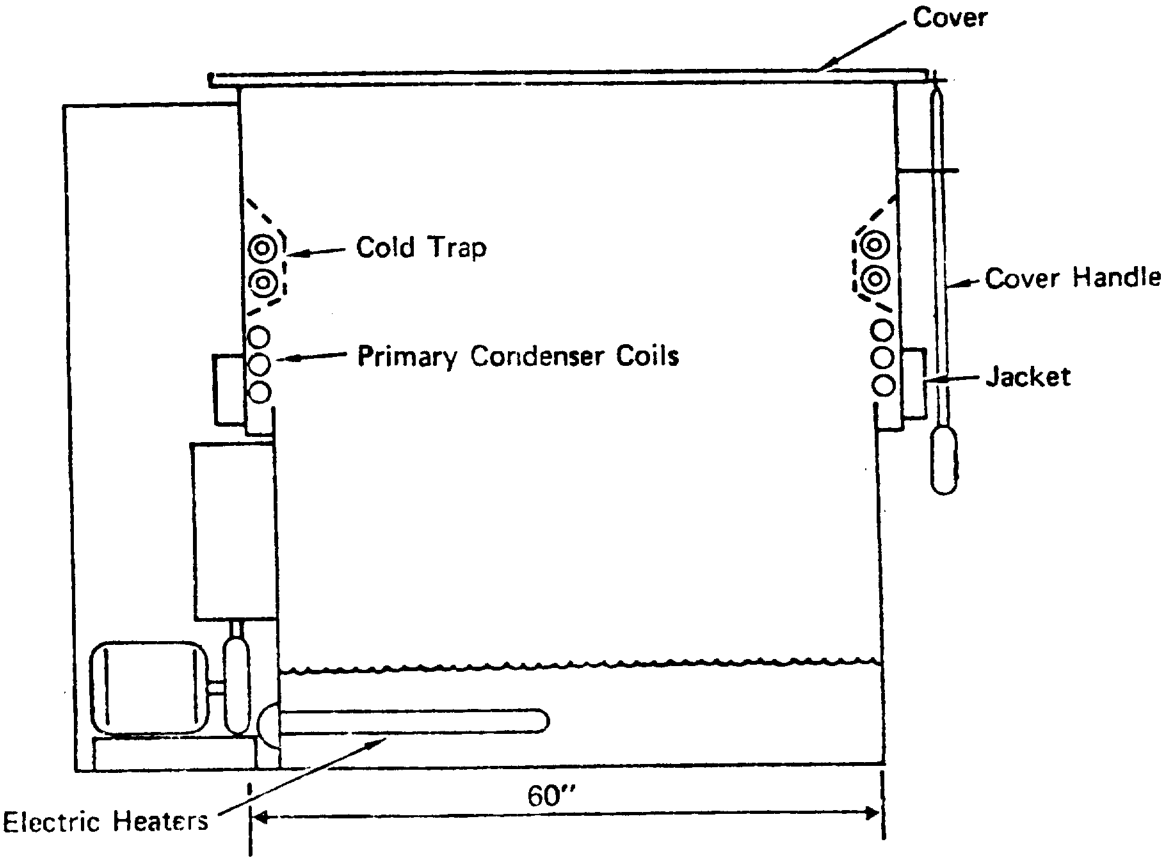
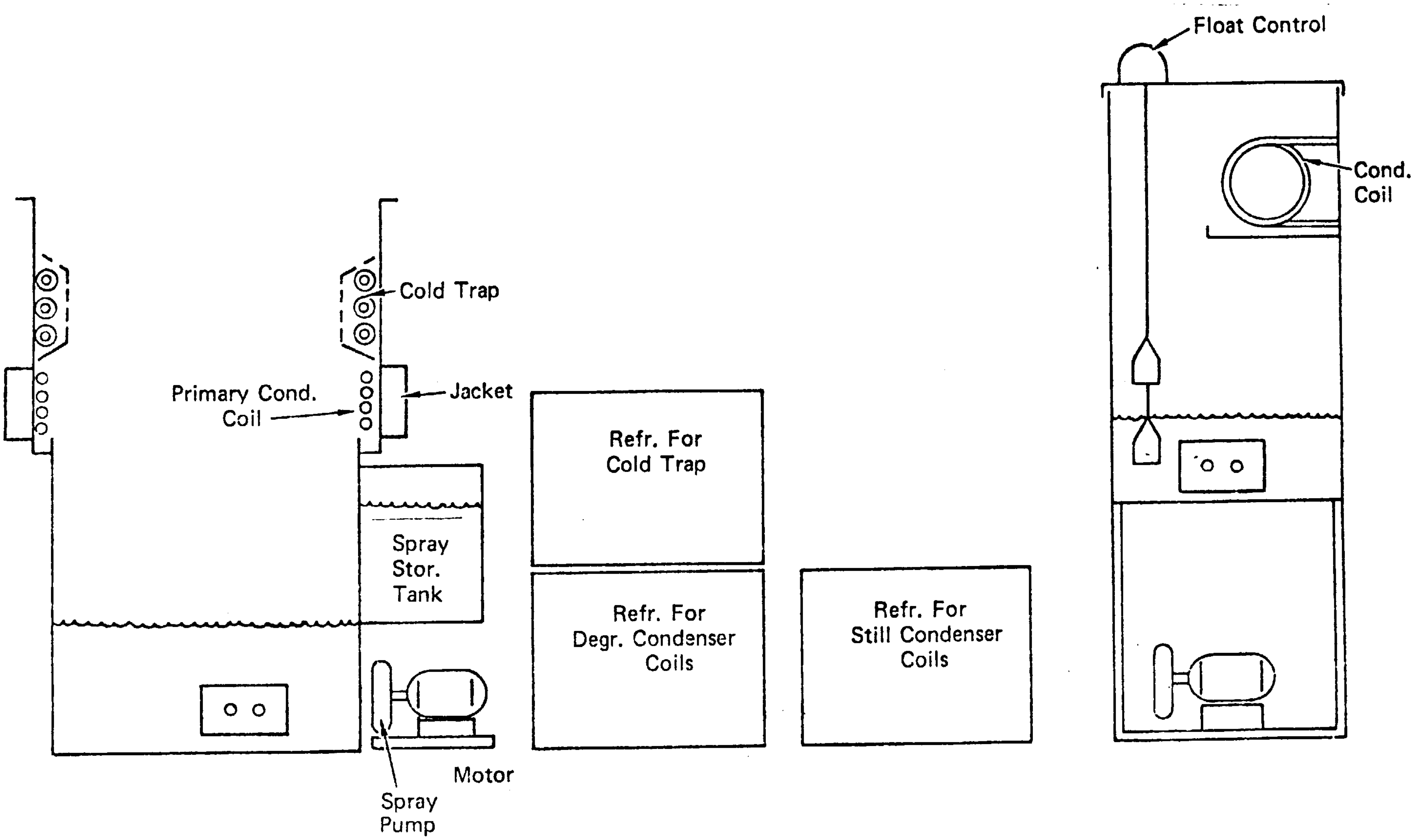


Figure 2
DEPT. O&R NO E 29957



Appendix C-4

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS OF SOLVENT METAL CLEANING OPERATIONS

EVALUATION OF CARBON ADSORPTION RECOVERY AT HEWLETT PACKARD CORPORATION IN LOVELAND, COLORADO

PREPARED BY:

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The Dow Chemical Company

PREPARED FOR:

Emission Standards and Engineering Division
Office of Air Quality Planning
U.S. Environmental Protection Agency

Summary

The evaluation of a carbon adsorption system as an emission control device for 1,1,1-trichloroethane used in a Riston® system was conducted with the assistance of Mr. Virgil Hebert at Hewlett Packard Corporation in Loveland, Colorado. It was found that the installation of the carbon adsorption unit reduced the overall emissions per unit of production by 21%. Eighty-five gallons of 1,1,1-trichloroethane were saved per week at a total recovery cost of $1.14 per gallon. When all costs are expressed on an annual basis, the savings (in solvent) equals 1.89 times the annual cost.

The recovered solvent contained good levels of "acid acceptor" stabilizer and acceptable water concentrations. However, the stabilizers designed to protect the solvent from reaction with aluminum were recovered in totally inadequate quantities. The water effluent from the water separator of the carbon adsorber contained from 2,000 ppm to 14,000 ppm of total hydrocarbons at the point of discharge to the drain.

®Riston is a registered trademark of the E. I. DuPont Company.

Objective

The objective of this test was to evaluate carbon adsorption as a means of controlling 1,1,1-trichloroethane vapor emissions in air. The information required for this evaluation required the development of specific data concerning:

1. Efficiency of this system.
2. Financial impact or cost/benefit of this system.
3. Energy requirement of the system.
4. Effect of the system on the present stabilizer levels.
5. The effect of the system as a potential water pollutant.

Introduction

Carbon adsorption has been offered to the solvent metal cleaning users since 1958. Other than specialty processes for example, film cleaning or textiles, metal cleaning carbon adsorption has been limited to perchloroethylene, trichloroethylene, methylene chloride, and Freons®. 1,1,1-trichloroethane was omitted from carbon adsorption due to the corrosion of the vapor adsorption equipment and the reactivity of the recovered solvent to aluminum.

Hewlett Packard was selected as an emission control test site for 1,1,1-trichloroethane for three main reasons.

1. The unit was new having started operation in January of 1975. This meant a system at optimum working order.

2. The unit was commercially available.

3. The unit was constructed of materials specifically designed for the recovery of 1,1,1-trichloroethane as inhibited for the metal cleaning industry.

®Registered trademark of the E.I. DuPont Company.

Equipment

The developer (see Figure 1) was built by Hewlett Packard. It is a long narrow spray chamber with a conveyorized monorail. Circuit boards are hung from the monorail and conveyed past various spray tanks of both CHLOROTHENE NU and water. After the developer, the boards are dried by blowing them with air and passing them by heat lamps prior to further treatment. Lip vent exhausts from both the entrance and exit lead to a Vic Model 536AD carbon adsorber. The cost of the develop line to Hewlett was $13,361.60 (parts and labor). The ovens were installed at an additional cost of $5,847.00.

Production was scheduled so as to maximize idling time by running only when a sufficient backlog of boards warrants the developer operation, usually two hours per day, but recently four hours per day. When idling, the spray tanks were turned off and approximately two inches of water settled out on top of the solvent in the develop tank.

The "Riston C100" still was made by DuPont and resembles an open top vapor degreaser with a cover. Its solvent level is maintained by float controls. The rate of distillation is dependent on contaminant level and varied from 5 gals./hr. to 45 gals./hr. The average was 9 gals./hr. based on 13 random sample rates.

The primary function of the carbon adsorption system was to recovery solvent used in the Riston system. Its secondary purpose was to capture the evaporation losses from the recovered solvent holding tank (see Figure 2). This adsorption system had a three horsepower fan, about 350 pounds of carbon per bed and operates with 85 psig air pressure and 10 psig steam pressure. Approximately 340-360 gallons per hour of condenser water was required and the effluent remained fairly steady at 145°F. The cost of the adsorption system was $16,042.00 and the installation was $1,940.00 additional. This adsorption system was higher in price than normal for a model 536AD due to the special materials of construction used. These materials included Hastelloy for the shell of the beds, Monel condenser tubes, Heresite coated milesteel for the water separator and stainless steel for the holding tank.

Experiment Design

This evaluation was designed to determine the following:

1. Efficiency--overall solvent recovery efficiency was determined by comparing the solvent use rate per unit of production for six months before and six months after the installation of the carbon adsorption system. The efficiency of the carbon bed proper was determined by measuring the vapor concentrations of the air stream before and after passage through the beds.

2. Effect of the adsorption system on stabilizer levels: The stabilizer concentrations in the recovered solvent and the Riston process equipment were analyzed and compared to the stablizer levels in fresh solvent.

3. Evaluation of the adsorption system as a source of water contamination: Steam condensate samples were taken from the water separator discharge to the drain and analyzed for total hydrocarbon content.

4. Energy: An energy balance was determined for both phases of operation by measuring solvent recovered, the steam condensate used and the volume of condenser water.

5. Recovery cost per gallon of solvent conserved.

DISCUSSION

Overall Efficiency

Production records were kept for six months prior to the installation date and six months after startup of the carbon adsorption equipment. These records included both the number of circuit boards processed and the total amount of 1,1,1-trichloroethane used in the development process. Fortunately, all circuit boards are of uniform size eliminating this variable. Consumption was calculated by dividing the number of boards processed into the number of gallons used. The overall emission control efficiency was based upon the comparison of these two six month periods. This data is summarized below.

Solvent Use Record

Without Carbon Adsorption	0.33 gals./board
With Carbon Adsorption	0.26 gals./board
Recovered Solvent	0.07 gals./board
Recovery Efficiency	21%

Bed Efficiency

Solvent vapor concentrations in air were analyzed by means of a "Siphon" pump connected to a carbon tube from

"Organic vapor SKC, Inc.". The "Siphon" pump has a counter and each count was calibrated for the cubic centimeters of air sampled. The total number of counts in each air sample was recorded and converted to an air volume. The solvent vapors were adsorbed in the carbon tube, extracted, and the total weight of 1,1,1-trichloroethane in the sample was determined by a gas chromatograph. Air flow measurements were taken using an "Alnor" thermo-anemometer. These data are summarized on Table 1.

TABLE 1

Solvent Air Vapor Concentrations

Develop Line "Off"

	PPM Inlet	PPM Exit	AV. % Bed Efficiency
	131		
	154	11	
	158	24	
Average	148	18	88%

Develop Line "On"

	Inlet	Exit	
		73 ppm	
	4395 ppm	133 ppm	
	4912 ppm	145 ppm	
		205 ppm	
	4927 ppm	496 ppm	
		789 ppm	
Average	4745	307	94%

Thus, the carbon bed efficiency ranged between 88-94%. The percent recovery efficiency overall reflects the actual savings to the customer and the emission control achieved. The percent bed efficiency is the maximum potential of the system if 100% of the vapors were captured by the ventilation system and directed through the carbon adsorption beds. The difference in the two percentages demonstrates that the efficiency of the carbon adsorption system as a whole is a function of its ability to capture the vapors emitted rather than the ability of the bed to recover solvent delivered to it.

Relying principally on the same data, a solvent material balance for the total system can be constructed (see Figure 3). Although the developed line operates only 2-4 hours per day, the carbon adsorption system functions eight hours per day. The material balanced describes these two modes of operation: (A) Development line "Off" and (B) Development line "On".

Effect of Adsorption on Solvent Stabilizers

Solvent stabilizers are needed for essentially all of the chlorinated solvents. Each of the stablizer systems are designed to protect the individual weaknesses of a specific chlorinated solvent. In the case of 1,1,1-trichloroethane three stabilizers will be discussed. The "acid acceptor" protects the solvent by removing any strong acids present in it. Two aluminum stabilizers prevent the reaction between 1,1,1-trichloroethane and aluminum. These stablizers will be referred to as the primary and secondary aluminum stablizers.

Stablizer concentrations were determined from daily samples of solvent taken from seven points shown on Figure 4. These daily samples were analyzed over an interval of approximately six months. Samples of solvent recovered by the carbon adsorption system were found to contain 80% of the original concentration of "acid acceptor" but a 87% loss in the primary aluminum stablizer concentration was experienced. A serious loss in the secondary aluminum stablizer was also experienced, but an exact percent loss could not be defined due to the low levels present. Since only about one gallon in five of solvent required for the developed system was recovered solvent, the stablizer lost effect on the process solvent inventory was much less. The following observations were made on the effect of the carbon adsorption system on the stablizer levels in the develop line after six months.

1. No decomposition products are seen in the development line either before or after the installation of the carbon adsorption system.

2. The acid acceptor level in the developed system was down 15% from original solvent levels. However, this amount of stabilizer loss is not detrimental to the system.

3. In the developed system, the primary aluminum inhibitor was reduced only 15% from the fresh solvent level although 87% of this inhibitor was not recovered with the solvent from the carbon adsorber.

4. The secondary aluminum inhibitor concentration was reduced 53%. Although the solvent could be used in this specific operation due to the large dilution with fresh solvent, the recovered solvent by itself was inadequately stabilized for use in any aluminum operations or systems containing aluminum as a material of construction.

Examination of Water Contamination From the Carbon Adsorption System

Samples of steam condensate from the carbon adsorber system were collected at the drain during two desorption cycles. One desorption cycle was representative of the operation with the developed line on, the other with the develop line off. These samples were analyzed for total hydrocarbon content and the data is summarized on Table 2. Although chlorinated solvents are essentially insoluble in water, their stablizer systems are frequently soluble in both water and solvent. As the steam in solvent condenses during the adsorption cycle, intimate mixing takes place. This mixing is continued until separation takes place in the water separator. This results in solublizing significant quantities of the stablizers in the water phase which is discharged to the drain. The volume of steam condensate generated per hour averaged 22.6 gallons and ranged in hydrocarbon content from 2,000-14,000 ppm. This volume of steam condensate was diluted approximately 15 times by the 350 gallon per hour discharge of condenser water. In most cases, this stream would then be diluted perhaps a 1,000 or more times from other plant water uses. It should be possible to remove much of this potential water contamination by sparging air through the steam condensate prior to discharging it at the drain.

Energy Balance

The gallons of recovered solvent and steam condensate were measured from several desorption cycles. These quantities are reviewed in Table 3. It should be noted that the volume of steam condensate used is related to the time of desorption rather than the volume of solvent retained by the bed. Other measurements determined that the condenser water flow rate ranged between 340-360 gallons per hour. The energy balances on Figures 4 and 5 for the adsorption and desorption cycles were developed from this information. The carbon adsorption energy balances are also tabulated on Table 4. The overall costs of operations are developed on Tables 5, 6, and 7. The calculations for utility costs are reviewed on Figure _. Prior to the installation of the carbon adsorption system, 412 gallons of 1,1,1-trichloroethane were consumed per week. With the carbon adsorption system in operation, the solvent consumption was reduced to 327 gallons per week. Thus, 85 gallons of solvent were conserved per week by the carbon adsorption system or 4,250 gallons per year (50 weeks per year). The value of that solvent at $2.12 per gallon is $9,010.00. This is compared to the $4,838.00 in cost per year developed on Table 7, a savings to cost ratio of 1.86 is found. This means that for every dollar invested in cost per year, $1.86 worth of solvent was recovered. Alternately, a cost per gallon of solvent recovered can be calculated by dividing the annual cost of $4,838.00 by the 4,250 gallons per year. This results in a cost per gallon of recovered solvent of $1.14.

Conclusions

1. The overall recovery efficiency of the carbon adsorption system was 21%. This represents the ability of the system to capture and recover solvent vapors from the develop operation.

2. The carbon bed efficiency was found to be 88% during develop line "off" operation and 94% with the develop line "on".

3. The cost of recovered solvent was \$1.14 per gallon when all costs were expressed on an annual basis. Similarly, \$1.86 worth of solvent was recovered per \$1.00 of annual cost.

4. Serious losses of solvent stabilization were experienced in the recovered solvent. However, the recovered solvent could be re-used in this operation without restabilization. If the operation was more demanding or the recovery efficiency higher, the solvent would require restabilization.

5. A small volume (20 gals./hr.) of water is contaminated with 2,000-14,000 ppm of total hydrocarbon.

TABLE 2

Drain Water Contamination Levels

Desorption Time (Min.)	Develop Line "Off"	Develop Line "On"
	Total Hydrocarbons Concentrations (PPM) In Steam Condensate (At Drain)	
4	1,951	
12	1,995	
20		4,121
23	1,867	
31		6,053
33	6,619	
40		5,199
42	9,693	
44	14,342	
48		4,049
55		2,805

TABLE 3

	Gal. Solvent	Gal. Steam Condensate
Develop Line "Off"	1.2	24.2
	2.6	16.2
Av.	1.8	
Develop Line "On"	9.9	24.5
	7.9	23.9
	8.7	14.3
Av.	8.8	

TABLE 4

Energy Balance on Carbon Adsorber (1 Bed)

	Input	Output
Adsorption (Solvent Recovery)		
Solvent Latent Heat (20.8 Lbs. A)	2,122 Btu's A	
(9617 Lbs. B)		
Air Flow @ 700 Cfm	(9,863 Btu's B)	2,122 Btu's A
		(9,863 Btu's B)
Desorption		
Steam 113 Lbs.	113,000 Btu's	
Heating Tank (900 Lbs.)		13,860 Btu's
Heating Carbon (300 Lbs.)		10,500 Btu's
Condenser Water (8 GpM)		88,640 Btu's
Vaporizing Solvent*	2,122 Btu's A	2,122 Btu's A
	9,863 Btu's B	9,863 Btu's B
Adsorption (Drying and Cooling Bed)		
Cooling Tank	13,860 Btu's	
Cooling Carbon	10,500 Btu's	
Air Flow		24,360 Btu's

*Vaporizing solvent from bed requires heat from steam but this same heat is given up to the condenser water when the vapor is condensed to liquid solvent.

A) Develop line off

B) Develop line on

TABLE 5

Industrial Building*

Shell (M&L) Cost	$ 4.09/Ft.^{2}
Lighting and Electrical	1.75
Heating and Ventilating	1.50
Plumbing	1.70
Fire Prevention	1.10
	$10.14 (1968 Base)
	$17.3/Ft.^{2} (8% Annum 1.71 Multiple in 1975)
Sub-Contract Cost (1.3)	$22.5/Ft.^{2}
Contingency (15%)	$25.9/Ft.^{2}

*Derived from "Modern Cost-Engineering Techniques" by H. Popper. The 8% inflation rate was estimated by K. S. Surprenant.

TABLE 6

Carbon Adsorber Capital Costs

Building Space

Direct	100 Ft.^{2}
Indirect (50%)	50 Ft.^{2}
Total	150 Ft.^{2}

Value @ \$25.9/Ft.^{2} = \$3,885 (Table 1)

Cost/Annum = \$3,885 x .11017*[1] = \$428

Direct Capital

Price	\$16,042.00
Shipping and Installation	\$ 1,940.00
Total Capital	\$17,982.00

Cost/Annum \$17,982.00 x .13147*[2] = \$2,364

*[1] Factor for linear return of capital with 10% interest.

*[2] Factor for linear return of capital with 10% interest.

TABLE 7

MODEL 536 AD CARBON ADSORBER

OPERATING COST PER ANNUM

Capital	
Building	$ 428
Equipment	$2364
Insurance (2%)	
Equipment	$ 321
Building	$ 78
Maintenance (4%)	$ 642
Utilities	
Steam	$ 865
Electricity	$ 112
Water	$ 28
Compressed Air	Nil
Labor	Nil
ROI	Nil
Total Cost/Annum	$4838
Solvent Recovered/Annum	4250/Gal.
Cost/Gal. Recovered	$1.14

Figure 1

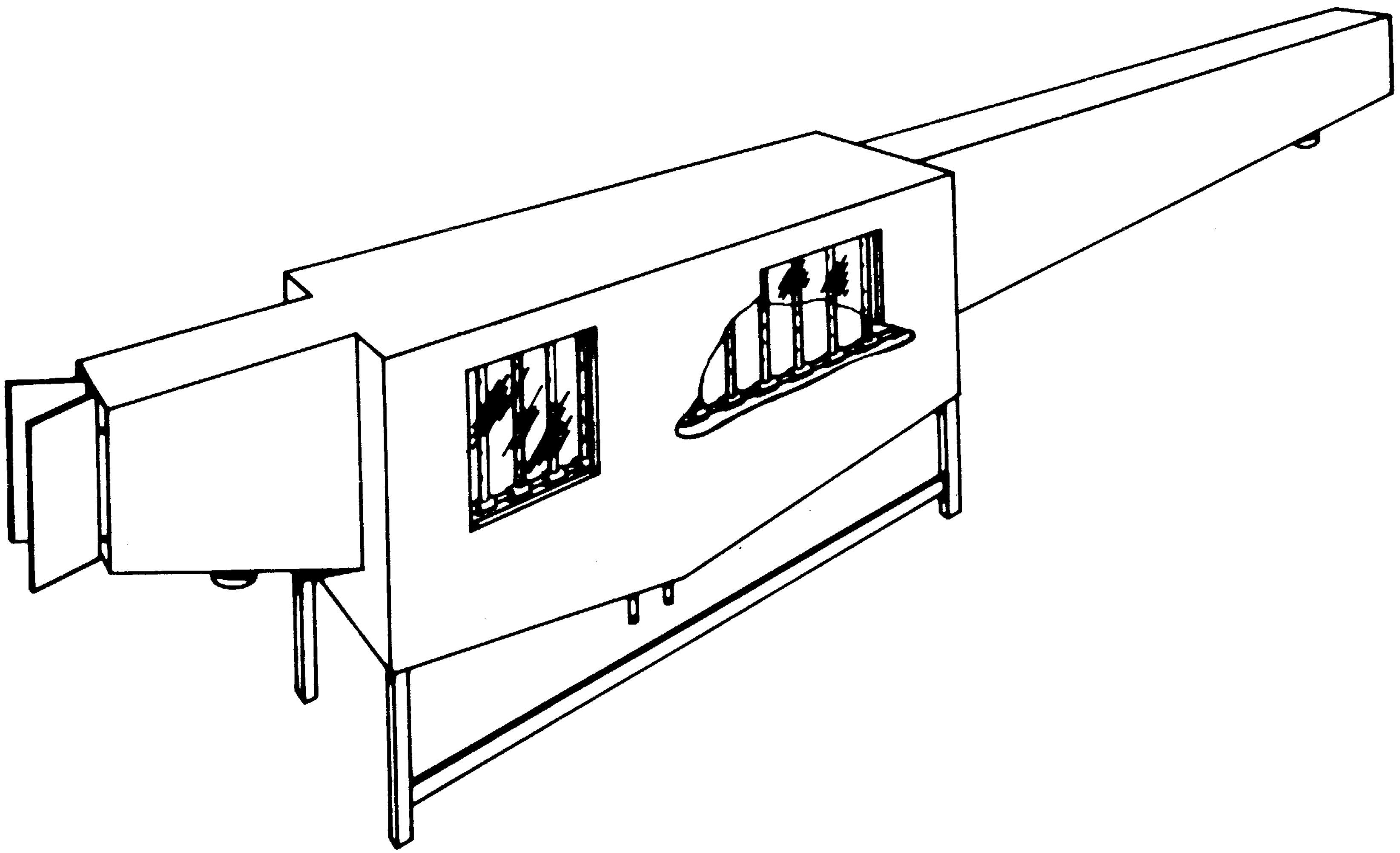


Figure 2

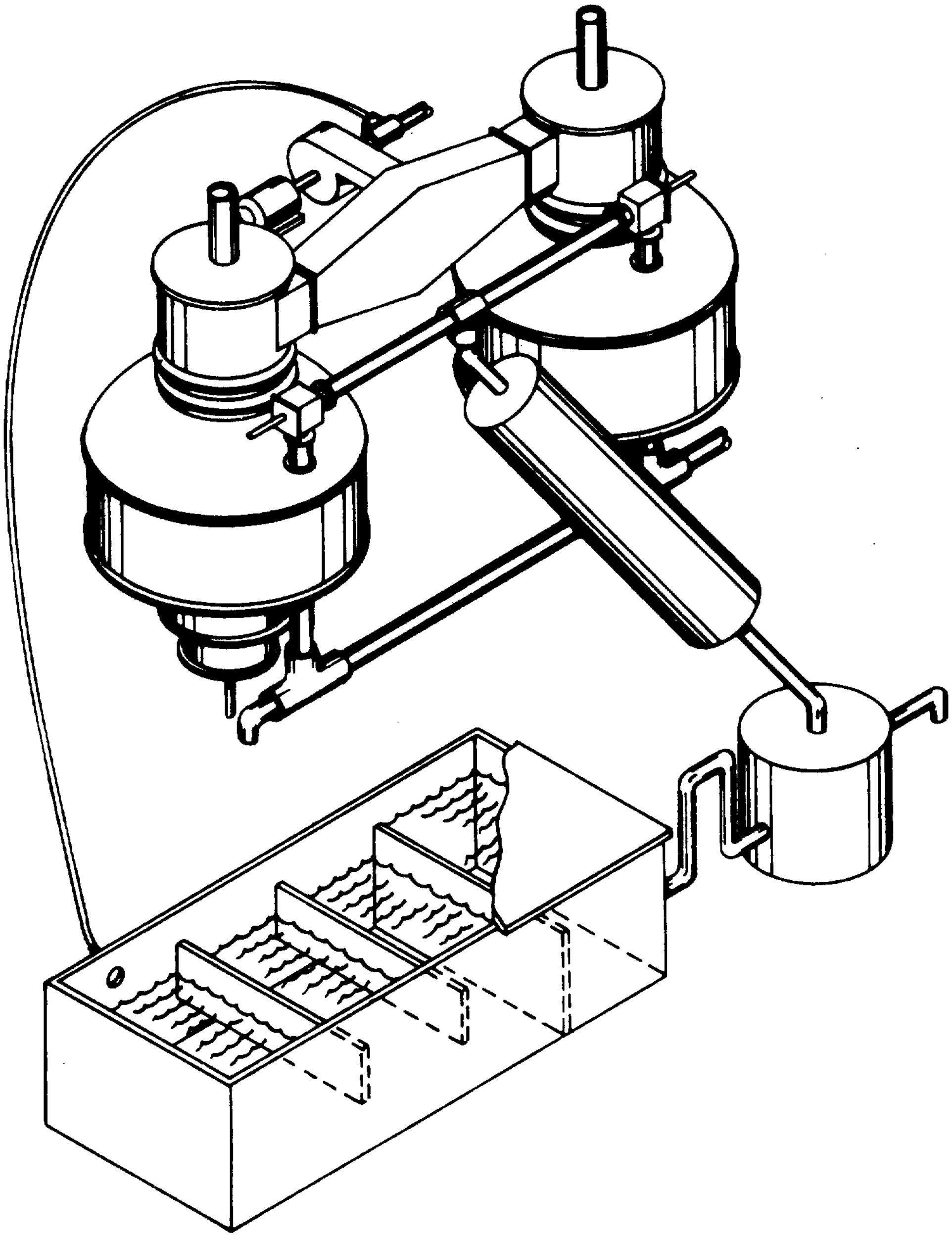


FIGURE 3

Utility Cost Calculations

Steam

22.6 gals/hr x 2000 hrs/yr x 8.33 lbs/gal x 1000 Btu/lb

= 377 x 10^6 Btu/yr

377 x 10^6 Btu/yr x \$2.3/10^6 Btu ÷ 10^6 = \$866

Electricity (3 Hp fan)

3 Hp x 0.746 KWH/Hp x 2000 hr/yr x \$0.025/KWH = \$112

Water

350 gal/hr x 2000 hrs/yr x \$0.04/10^3 gal ÷ 1000 gal = \$28

APPENDIX - C5

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Evaluation of (1) A Pneumatic Cover (2) Refrigeration to Control Solvent Emissions at

Pratt Whitney
Hartford, Connecticut

Prepared By:

K. S. Surprenant
The Dow Chemical Company

Prepared For:

Emission Standards and Engineering Div.
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

Two emission control methods for solvent metal cleaning operations were evaluated at Pratt Whitney. A pneumatic cover was found to effect a 40 percent reduction in emissions from an open top degreaser using 1,1,1-trichloroethane. This emission control requires little investment and energy and yields a positive return on investment (156 percent per year).

A refrigerated freeboard chiller was studied on another open top degreaser. This degreaser was equipped with a manually operated cover. A 16 percent lower emission rate was indicated with the refrigeration system in service then without it. The capital investment is about twice that of the pneumatic cover. The solvent conserved by this device would amortize about 78% of the investment and operating costs. The energy demand is less than carbon adsorption but greater than a pneumatic cover.

Objective

The purpose of this test program is to evaluate:

1. a pneumatic vapor degreaser cover, and
2. a refrigerated freeboard chiller (Cold Trap) as a means of controlling solvent emissions from metal cleaning operations.

The information needed for this evaluation includes determining:

1. the efficiency of this device in reducing solvent emissions to the atmosphere,
2. the cost/benefit relationship of this emission control system,
3. the energy requirement of the emission control system,
4. any alternate emissions created by the emission control system.

This data base is being developed to forecast the magnitude of emission reductions which can be achieved nationally and the effect on businesses involved. This information combined with the results of other testing will be used to design emission control regulations which effectively limit air pollution and are practical for industrial application.

Introduction

Pratt Whitney Division of The United Technologies, Inc. was recommended by Dow technical representatives because of their wide use of solvent metal cleaning and highly qualified technical staff. These assets made the prospect of locating one or more techniques of controlling solvent emissions likely at this location.

A preliminary meeting with Pratt Whitney people revealed two opportunities to evaluate emission control technology. The first of these was the use of a pneumatically actuated cover on an open top degreaser. Although essentially all open top degreasers are manufactured with covers, most are equipped with heavy metal covers which are seldom used. In contrast to industry wide normal procedures, Pratt Whitney reported a well disciplined use of canvas covers. Further, they found the installation of a pneumatic cover had increased the effectiveness and decreased the labor associated with manually closing the cover. The second technique employed to control solvent emissions was a refrigerated freeboard chiller (Cold Trap). This device reduces solvent diffusion loss from the vapor zone by establishing a cold air mass immediately above the vapors. This cold air mass acts as a barrier to the transport of solvent vapor out of a vapor degreaser. Both systems were regarded as worthy of examination.

Each of these evaluations will be treated separately through the balance of this report.

Pneumatic Cover Degreaser

Equipment

The degreaser proper is a moderately large open top Detrex Model 1 DVS-800-S, Serial No. 52301-A. This degreaser is constructed of stainless steel, steam heated and equipped with a spray lance. It is 65 inches wide x 110 inches long and is operated with a 40 inch freeboard (the distance from the top of the vapor zone to the top of the degreaser). The degreaser is divided to provide a solvent immersion chamber and a boiling chamber. This equipment is diagramed in Figure 1.

The cover is constructed of canvas and is supported by rods approximately every 18 inches.

Experiment Design

Degreaser operation was unchanged except for the use of the cover. Two weeks, as a minimum, were planned with the cover in normal use. The cover was to be deactivated and the degreaser left open for a minimum of two weeks. The efficiency of the emission control device could then be determined directly by comparing the solvent losses experienced with and without it.

This degreasing operation is used three shifts per day and five and one-half days per week. The standard Pratt Whitney procedure for operation of the degreaser involves closing the cover after every use. Obviously, the equipment would be closed on weekends and holidays. During the test period without the cover, the degreaser was left open 100 percent of the time including weekends. This method of operation provided the maximum control of test conditions and avoided possible human errors of various operators using the equipment.

Data Discussion

The standard practice at Pratt Whitney includes pumping essentially all the dirty solvent out of the boiling chamber of the degreaser once every two weeks. Clean solvent is added to the degreaser as needed during the two week intervals. For convenience in making test measurements, the degreaser was pumped out on a weekly basis and no solvent additions were made during the week. This was done in all cases except for the two week interval beginning July 17, 1975. During this test period no solvent was pumped from the degreaser or added to the degreaser for the full two weeks.

A cart containing two chambers is used to deliver fresh 1,1,1-trichloroethane and remove dirty solvent from the operation. Each chamber is 34 inches square by approximately 31 inches deep. Consequently, an inch of solvent in one of these chambers is equal to 5.00 gallons. The depth of solvent was measured before and after dirty solvent was pumped from the boiling chamber as well as before and after the clean solvent was pumped into the degreaser. The solvent depth in these chambers could be measured to within one quarter of an inch easily. Thus, the error in measurement of solvent volume from this source would be less than plus or minus 1.25 gallons. Variance due to the completeness of pumping solvent from the degreaser is estimated at plus or minus two gallons. The solvent consumed was taken to be the total solvent added minus the total volume of solvent removed. This information is reported in Table 1 along with the number of parts processed on a biweekly basis.

The degreaser was operated three shifts per day, five and one-half days per week. Two exceptions to this schedule occurred. July 4th was celebrated as a holiday and the degreaser was not operated, during a two week interval of testing with the cover off. Less than one day of operation was lost with the cover operating during the two week interval beginning July 17, 1975. This interruption was caused by the need to repair the solvent pump for the spray lance. No need to adjust solvent consumption is indicated because one shut down occurred with the cover in operation while the other without the cover.

The work parts processed through this equipment are large sections of turbine motors. A typical piece might be roughly 40 inches in diameter and 10 inches thick. These pieces contain numerous large cavities. Often these cavities present difficulty in obtaining good solvent drainage to avoid drag-out losses. The variance in the quantity of work processed with the cover on versus that with the cover off was approximately 4 percent. This variance yields a slightly higher emission control efficiency (42 percent) on a work piece basis versus the 40 percent efficiency obtained on a weekly solvent consumption basis shown on Table 2. The volumes of solvent consumed per unit piece are shown below:

0.159 gallons per piece with cover
0.276 gallons per piece without cover

An estimate of $2,300 was provided by Detrex Chemical Industries, Inc. as a replacement cost for the pneumatically operated canvas cover. Shipping and installation is estimated at $345 (15 percent of capital). Since no floor space is required for this equipment, no building capital is assessed. With a 15 year write-off and a 10% time value of money, the annual cost of capital is $348 ($2645 x 0.13147). Maintenance (4 percent of capital) $106 and insurance (2 percent of capital) $53 increase the annual operating cost to $507. The only power used on this equipment is plant compressed air. No measurement of air requirement was made. However, the power requirement is minimal since it is used only about 10 to 15 seconds per load and only about 360 loads are processed per week. Thus, a value of $25 per year is assigned for the utility expense. The total operating cost would be approximately $532.

The "Chemical Marketing Report," July 7, 1975 price of 1,1,1-trichloroethane is $2.12 per gallon. The total operating cost of $532 for the pneumatic cover would be recovered by conserving 251 gallons of solvent. The actual solvent saved by this device was 39 gallons per week. On a yearly basis, 1,950 gallons of solvent are being conserved or $4134. Thus, the value of the solvent saved is 7.77 times the annual operating cost, including capital.

Conclusions

1. A 40 percent reduction in solvent emissions was found to occur through the use of a pneumatic cover on an open top degreaser.

2. The pneumatic cover yielded a return on investment of 156 percent per year. The savings to annual cost ratio including capital was 7.77.

3. The energy requirement for this emission control is minimal.

4. No adverse effect on the solvent stabilization is observed as a result of this emission control means.

Figure 1
DETREX MODEL IDVS-800-S R 511853

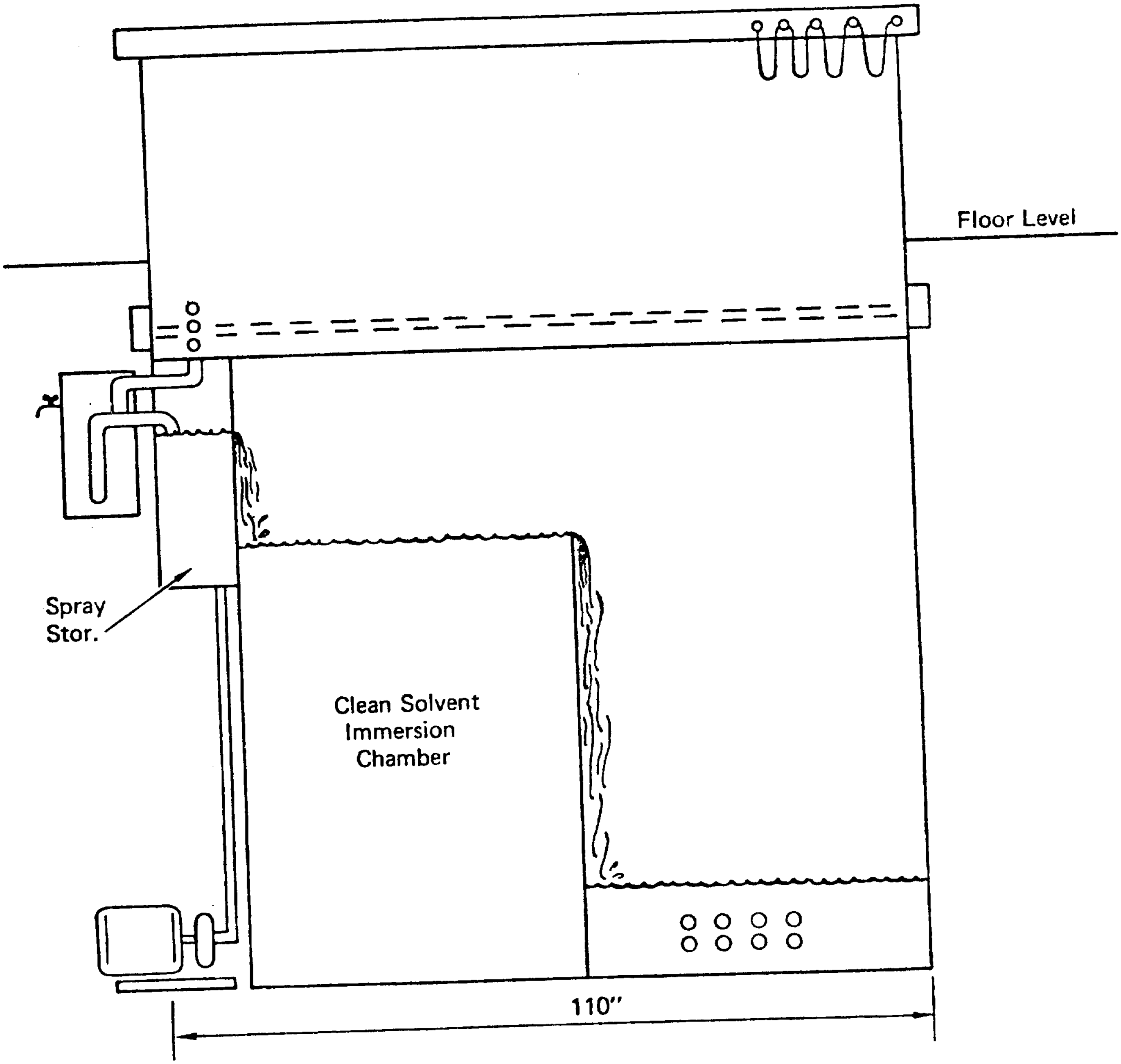


TABLE 1

Pneumatic Cover Degreaser Records

Operational Mode	Two Weeks Beginning	Solvent Used	Parts Processed
With Cover	May 29, 1975	116 Gals.	851
Without Cover	June 12, 1975	202 Gals.	729
Without Cover	June 26, 1975	188 Gals.	681
With Cover	July 10, 1975 (1 Week Only)	67 Gals.	357
With Cover	July 17, 1975	109 Gals.	630

TABLE 2

Summary of Pneumatic Cover Degreaser Data

Operational Mode	Total Test Time	Average Solvent Used	Average Parts Processed
With Cover	5 Weeks	58.4 Gals./Week	368/Week
Without Cover	4 Weeks	97.5 Gals./Week	353/Week

$$\% \text{ Emission Control} = \frac{(97.5 - 58.4)}{97.5} 100 = 40\%$$

Refrigerated Freeboard Chiller

Equipment

The vapor degreaser used in this evaluation is sketched in Figure 2. The degreaser is a Detrex 1 DVS-800-S model, Serial No. 52298. The Pratt Whitney equipment number is R511854. Again, this open top degreaser is equipped with a canvas cover and a spray lance and is steam heated. The view shown in Figure 2 is from the end of the degreaser. This equipment had an open top surface area of 56 inches wide by 90 inches long. The overall depth of the equipment was 139 inches and the freeboard was found to be approximately 33 inches. As was the case with the pneumatic cover degreaser, this equipment is divided to provide a clean immersion chamber so that parts can be rinsed in the liquid solvent (1,1,1-trichloroethane). Unlike the earlier degreaser, the cover on this equipment is manually operated.

The refrigerated freeboard chiller on this equipment is a Cold Trap Model 150, Serial No. 2171. This portion of the equipment was supplied by Autosonics Incorporated in August of 1968. The present value of this one and one-half horsepower equipment ($4,900) was provided by the original supplier. This price includes the equipment cost, shipping and installation. At the time of installation Autosonics advised that the expected reduction in solvent usage would not be achieved unless:

1. A steam condensate return pump was installed to avoid flooding the steam coils and disturbing the degreaser heat balance.

2. The steam line supplying the heating coil should enter the degreaser below the vapor zone. This steam line was originally piped through the freeboard, vapor interface and vapor zone to the heating coils. This method of supplying steam disturbed both the cold air barrier and the vapor zone.

Both of these modifications had been made prior to this evaluation.

It should be noted that current freeboard chiller equipment supplied by Autosonics employs different design parameters than exist on this equipment. These more advanced design parameters reportedly provide more efficient vapor emission control. Other evaluations are designed to determine the effectiveness of the improved parameters.

This evaluation provides valuable data on the effective life of this equipment and its performance several years after original installation. In addition, other suppliers of refrigerated freeboard chillers offer equipment with still different design parameters.

Experiment Design

Two weeks of testing were planned as a minimum with the refrigerated freeboard chiller on and another two weeks with it off. No other operational procedures were changed. As noted earlier, it is standard practice at Pratt Whitney to cover all degreasers when they are not in immediate use. Thus, this evaluation determined what additional emission control could be achieved with a refrigerated freeboard chiller when an effective emission control (the degreaser cover) was already in use.

All solvent handling and measurement techniques employed with the pneumatic cover degreaser were used in this evaluation as well.

Data Discussion

All of the test records are summarized in Table 3. The data accumulated during the week of June 20, 1975 was discarded because of the short six day week with only four operating days. Again, the week of June 26, 1975 was discarded from analysis due to the three working days in the week caused by shut downs and the low part process load. Some consideration was given to discarding the data for the week of July 3 due to the holiday. However, it was decided to incorporated this data since the part process load was more than comparable to the succeeding work weeks.

The averages reported in Table 4 represent the two weeks of July 3 and July 10, 1975 with the refrigeration on and the next two weeks with the refrigeration off. The percent emission control is caluclated on the weekly solvent consumption. The parts processed with the refrigeration system on were 10 percent higher than the workload processed with the refrigeration off. However, one less day of work was experienced with the refrigeration on. These factors should tend to cancel one another. If the percent emission control is calculated on a workday basis, the efficiency is reduced. In contrast, if the efficiency were expressed on a work piece basis the efficiency of control would be increased.

Table 5 derives a capital cost per square foot of plant space. Table 6 provides a breakdown of the total capital and operating cost estimated for the refrigerated freeboard chiller. A value of $2.12 per gallon is taken for 1,1,1-trichloroethane from the July 7, 1975 issue of "Chemical Marketing Reporter". Using this value of solvent a total of 574 gallons of solvent would be needed to offset the annual cost of $1216 per year. If nine gallons of solvent are saved per week on an average, 450 gallons would be conserved in the course of the year. The value of this solvent would be $954 and would result in a $262 loss using the operating cost outlined in Table 6. These values can be expressed as a ratio of savings to cost ($954/$1216 = 0.785). Thus, 78.5% of the operating cost (including capital) per year was returned via solvent savings.

The refrigerated freeboard chiller cannot be expected to conserve solvent which is already being controlled through the use of a manual cover. If this degreasing operation was operated in the open condition as is common practice, the control achieved by the refrigerated freeboard chiller could be expected to be notably higher. This and the improved design parameters of newer equipment will be evaluated at other locations.

The dirty solvent removed from all of the Pratt Whitney degreasers is distilled in a central still. Two samples of the still residues from this system averaged 69 percent by weight nonvolatile matter. The individual readings were 75.9 percent and 61.5 percent. Since the oil content of the solvent withdrawn from the degreasing operations during test contain only low percentiles of oils, no significant solvent losses were experienced through solvent distillation. Solvent stabilization of both systems was found to be adequate. Water contamination was observed visually and by solvent analysis throughout the evaluation of the freeboard chiller. A mixture of solvent and water freezes on the freeboard chiller coils. From ice samples taken, the composition appeared to be approximately one part water to two parts solvent. This mixture should be removed from the system during the defrost cycle by the water separator. However, water removal was not being effected during the evaluation.

Conclusions

1. A refrigerated freeboard chiller can achieve a low level of emission control (16 percent) even with a cover in operation.

2. The emission control obtained by adding two systems to one solvent metal cleaning operation is not a sum of the expected emission control of each separately.

3. The refrigerated freeboard chiller studied in this evaluation was recovering 78.5% of the annual operating costs per year, including the capital.

4. A refrigerated chiller requires relatively low energy requirements compared to carbon adsorption but much greater energy than a powered cover.

5. A mixture of solvent and water freezes on the freeboard chiller coils. During the defrost cycle, the mixture enters the degreaser condensate trough. If not effectively removed, water can be a source of serious equipment corrosion.

Figure 2
DETREX 1 DVS-800-S NO 511854

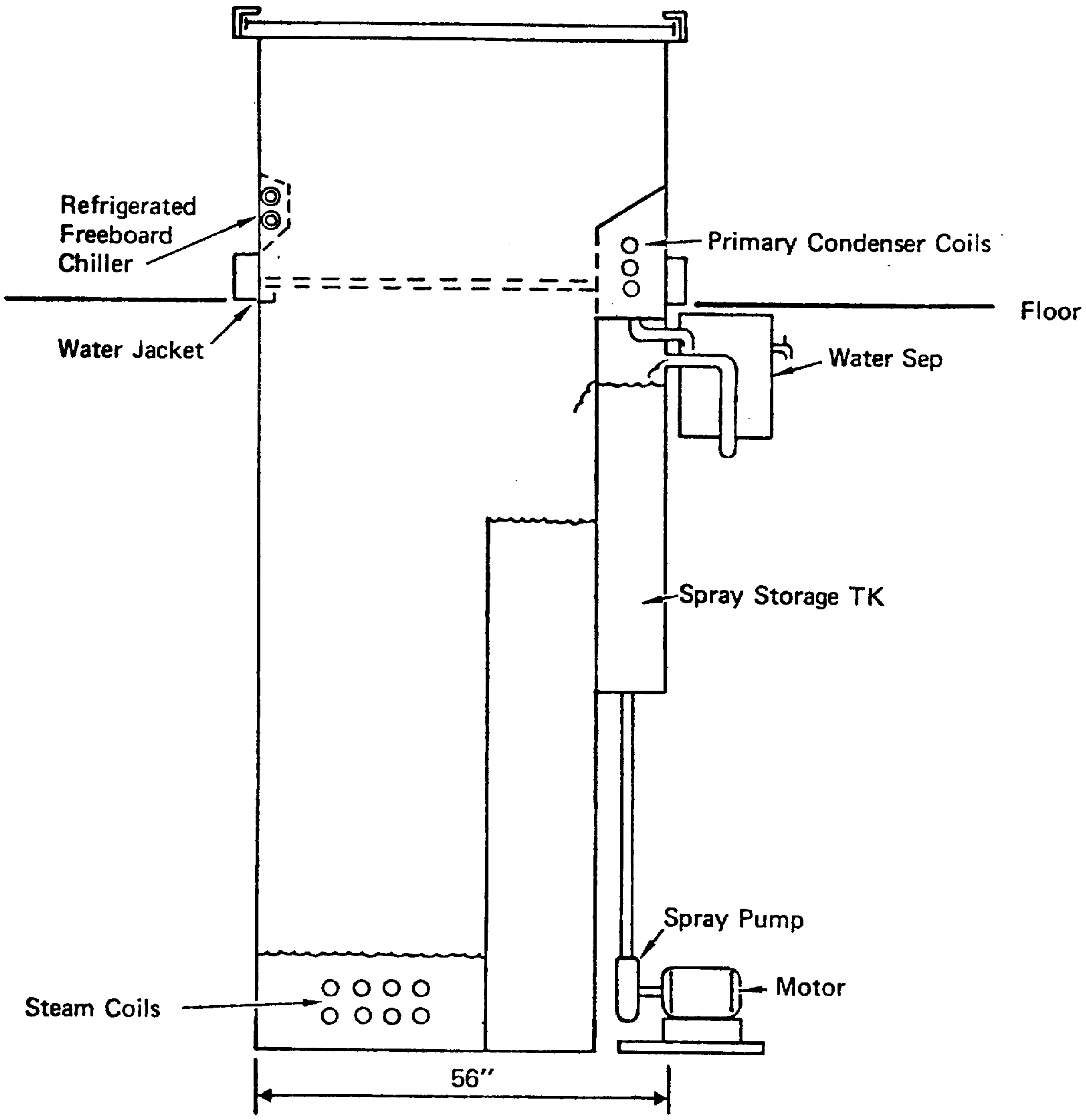


TABLE 3

Refrigerated Freeboard Chiller Records

Operational Mode	Week Beginning	Total Days	Work Days	Solvent Used	Parts Processed
Refrigeration On	June 20, 1975	6	4	48 Gals.	-
	June 26, 1975	7	3	41 Gals.	207
	July 3, 1975	7	4	40 Gals.	277
	July 10, 1975	7	5	57 Gals.	238
Refrigeration Off	July 17, 1975	7	5	52 Gals.	205
	July 24, 1975	7	5	63 Gals.	263

TABLE 4

Summary of Refrigerated Freeboard Chiller Data

Operational Mode	Weekly Average Solvent Used	Weekly Parts Processed
Refrigeration On	49 Gals.	258 Pieces
Refrigeration Off	58 Gals.	234 Pieces

$$\% \text{ Emission Control} = \frac{(58 - 49)}{58} 100 = 16\%$$

TABLE 5

Industrial Building*

Shell (M&L) Cost	$ 4.09/Ft.^{2}
Lighting and Electrical	1.75
Heating and Ventilating	1.50
Plumbing	1.70
Fire Prevention	1.10
	$10.14 (1968 Base)
	$17.3/Ft.^{2} (8%/Annum 1.71 Multiple in 1975)
Sub-Contract Cost (1.3)	$22.5/Ft.^{2}
Contingency (15%)	$25.9/Ft.^{2}

*Derived from "Modern Cost-Engineering Techniques" by H. Popper. The 8% inflation rate was estimated by the author.

TABLE 6

Capital Investment

Price	$4900
Installation (Included)	-
Floor Space*	256

Operating Costs

Capital*	
Equipment (15 Yr.)	644
Building (25 Yr.)	28
Insurance (2%)	
Equipment	98
Building	5
Maintenance (4%)	196
Utilities	
Electricity (1 1/2 Hp. Motor)***	245
Labor	Nil
Return on Investment	0
Total Cost/Year	$1216

*6.6 $Ft.^2$ of Direct Floor Space Times 1.5 for Indirect Space Times $25.9 Per Ft.

**The annual cost of capital was calculated at a 10% time value of money. The factor for a 15 year life is 0.13147, for a 25 year life is 0.11017.

***1 1/2 Horsepower x 0.746 KWH/Hp. x 365 Days x 24 Hrs. x $0.025/KWH

APPENDIX - C6

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Evaluation of Vapor Degreasing Vs. Cold Cleaning

Prestolite Corporation
Bay City, Michigan

Prepared By:

T. A. Vivian
W. C. Douglas
The Dow Chemical Company

Prepared For:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

The comparison of cold cleaning vs. vapor degreasing as sources of emission was conducted at the Prestolite Corporation. The particular cleaner used in the study had been under close observation by Mr. Ray Gittens of Prestolite for a period exceeding two years in order to ascertain the effect of vapor degreasing with 1,1,1-trichloroethane. Due to the thoroughness of Mr. Gitten's study, this data was not repeated but used as the reference on vapor degreasing, saving considerable time and money. The degreaser was used as a cold cleaner for five weeks after which the surface of the unit was covered with a layer of 1 1/2" diameter plastic balls. Cold cleaning with 1,1,1-trichloroethane as opposed to vapor degreasing with 1,1,1-trichloroethane reduced emissions 47%. The addition of the plastic balls to the cold cleaning operation reduced emission levels an additional 3% beyond the cold cleaning level, however, due to their hindrance of production, these balls had to be removed. Two additional points should be made in the 47% cold cleaning emission reduction. Not all operations can clean without vapor degreasing, which entails always clean solvent condensing on the part being cleaned. Second, the consumption figure for cold cleaning does not reflect distillation of contaminated solvent which would reduce losses even greater. The data does show that if cold cleaning can be used and the dirty solvent distilled or reclaimed, significant emission and cost savings can be recognized.

Objective

The objective of this test program was to compare energy and emission data via vapor degreasing vs. cold cleaning. The information sought for this test included the following:

1. Trichloroethane vapor degreasing

    Solvent consumption
    Heat requirements

2. Trichloroethane cold cleaning

    Solvent consumption

3. Cold cleaning with plastic balls as vapor depressants

Conclusions

At Prestolite:

A. Cold cleaning was 47% more efficient than vapor degreasing with 1,1,1-trichloroethane.

B. Plastic balls as vapor depressants in cold cleaning were only marginally effective and were too great a hindrance to production.

General Conclusions

Where feasible from a cleaning standpoint, cold cleaning offers a unique opportunity to reduce solvent costs, solvent emissions and energy. The addition of a still to a cold cleaning operation would further enhance the process.

Equipment

Prestolite has a Baron Blakeslee Model HD425 vapor degreaser (see sketch). The dimensions are: width 30.5 inches, height 59 inches, length 48.3 inches. The degreaser is equipped with a roll lid to prevent evaporation and a lip vent exhaust to prevent a concentration of vapors to the operator's breathing zone.

1. Trichloroethane vapor degreaser:

   The consumption for 1,1,1-trichloroethane for a one year period was 550 gallons. The work schedule for that year was 250 days, 16 hours per day. An estimated 211,200 Btu's per hour or $.42 per hour was the cost of the heat input. The consumption average was 1.5 lbs./hr.

2. Trichloroethane cold cleaning:

   On July 28, after being down for two weeks, the degreaser was filled with 1,1,1-trichloroethane to a height of 30 inches from the top; the overall height being 59 inches, with 185 gallons of 1,1,1-trichloroethane. This degreaser then was run as a cold cleaning operation with all heat turned off. On July 31, Halide measurements were taken around the top of the vapor degreaser and within the lip exhaust of the degreaser itself.

The Halide meter used was a Gas Tech Model 1192 calibrated 7-30-75. The recorder was a Rustrak Model P4002 with a chart speed of one inch per minute. On September 3, 1975, the solvent level was down three inches and 18 gallons of make-up solvent was added. This equates to an average consumption of 0.49 lbs./hr. of 1,1,1-trichloroethane. Halide measurements were again taken, this time from a set position at the lower right side of the degreaser. On September 18th, the solvent was too dirty and had to be disposed of.

This equates to an additional .3 lb./hr. consumption for a total consumption of .79 lb./hr. of 1,1,1-trichloroethane with cold cleaning. While it is not documented in this report, a common commerical still would be expected to get about 70% of the .3 lb./hr. back to give an estimated consumption of .59 lb./hr. of 1,1,1-trichloroethane cold cleaning with distillation.

3. Cold cleaning with plastic balls as vapor depressants:

After the readings, plastic balls were added to the degreaser to determine their effort as a vapor depressant and readings were again taken.

On September 10, 1975 we lost our balls at Prestolite. They interferred too much with production and had to be removed.

Summary: Consumption - All on same equipment

1,1,1-trichloroethane	1.5 Lb./Hr.	Degreasing
1,1,1-trichloroethane	.8 Lb./Hr.	Cold Cleaning

July 28, 1975

1. Fan Off Lower Right Corner

Meter	PPM
15	30
25	60
27	70
45	210
85	710
15	30
53	300
85	710

Range 30-710 PPM

Average 265 PPM

2. Top Right Corner

17	35
38	150
40	160
28	80
1	0
80	630
23	55
33	100
45	210

Range 0-630 PPM

Average 158 PPM

3. Lower Left Corner

100	1000
75	560
100	1000
58	360
70	490
75	560
70	490

Range 360-1000 PPM

Average 637 PPM

4. Upper Left Corner >1000 PPM

Fan On Air Speed Ft./Min. Lips 2" x 50"

	Lower Lip	Upper Lip
Left	260	260
Right	320	375
Middle	350	315

Average Air Velocity 313 Ft./Min. Facial Velocity

2/12 x 50/12 x 2 = Sq.^{1} Face 1.39

= 435 Cfm

5. Readings Taken At Inlet To Lip

	PPM
0	0
1	1
15	30
20	50
0	0
0	0
5	5
5	5

September 3, 1975

Prior Addition of
Plastic Balls

Readings	PPM
60	380
100	1000
65	430
75	560
45	210
60	380
55	320
40	160
60	380
100	1000
50	260
100	1000
55	320
	492 Av.

After Addition of
Plastic Balls

Readings	PPM
45	210
55	320
65	430
40	160
40	160
82	660
100	1000
100	1000
60	380
80	630
36	120
52	290
80	630
85	710
	479 Av. 3% Less

Evaporation Rate Calculations for Cold Cleaning (Based on Halide Meter Readings - Does Not Include Drag-out):

350 PPM = 1900 Mg./M^3

11.4 PPM = 62 Mg./M^3

62 Mg. x 10^{-3} G./Mg. x 10^{-3} Kg./G. = 62 x 10^{-6} Kg. = 1.37 x 10^{-4} Lb.

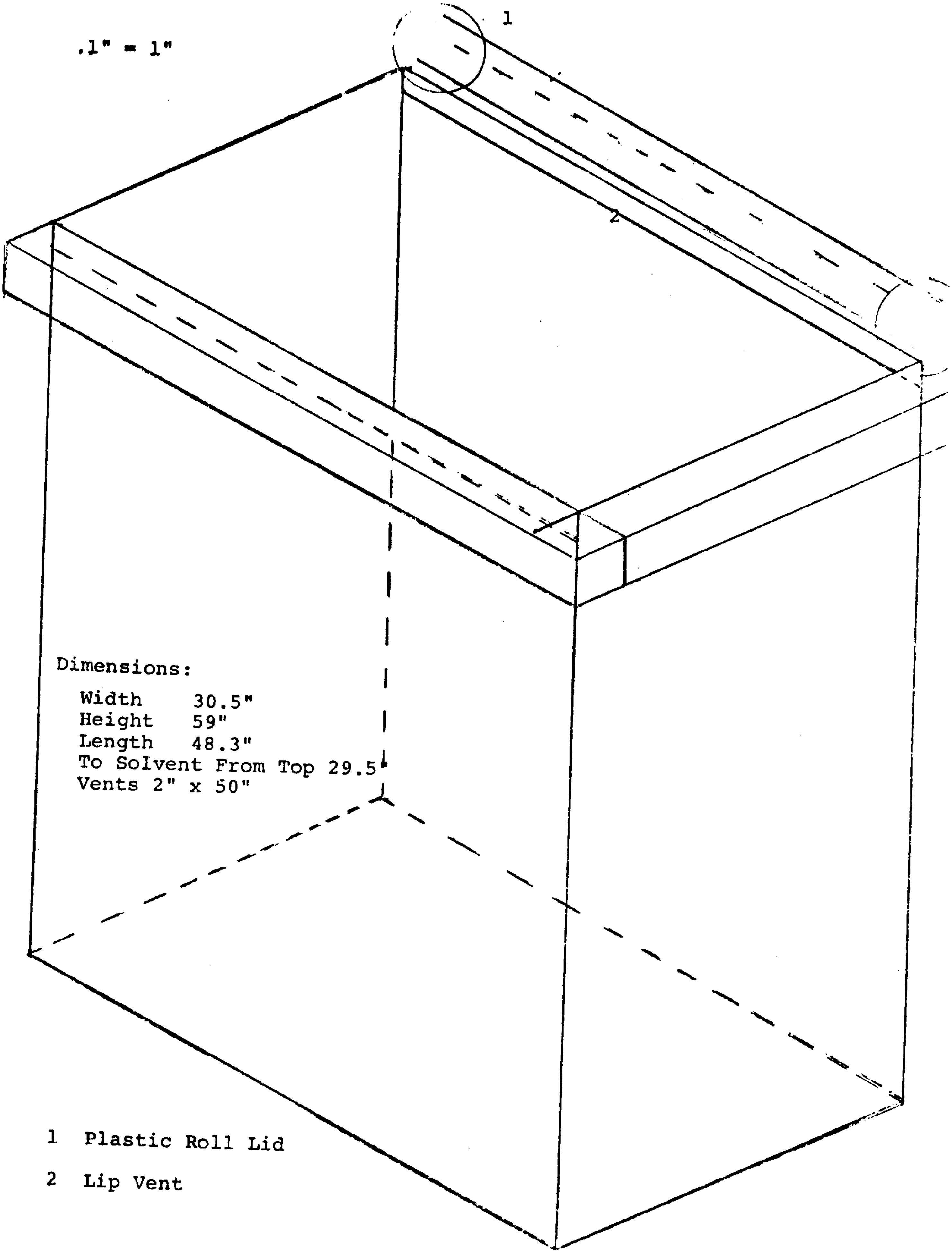
1 M^3 = 35.3 Ft.^{3}

1.37 x 10^4 - 35.3 = 3.88 x 10^6 Lb./Ft.^{3}

11.4 PPM = 3.88 x 10^{-6} Lb./Ft.^{3}

3.88 x 10^{-6} Lb./Ft.^{3} x 435 Ft.^{3}/Min. x 60 Min./Hr. = .101 Lb. Hr.
Due to Evaporation
Average Hourly
Consumption

.1" = 1"

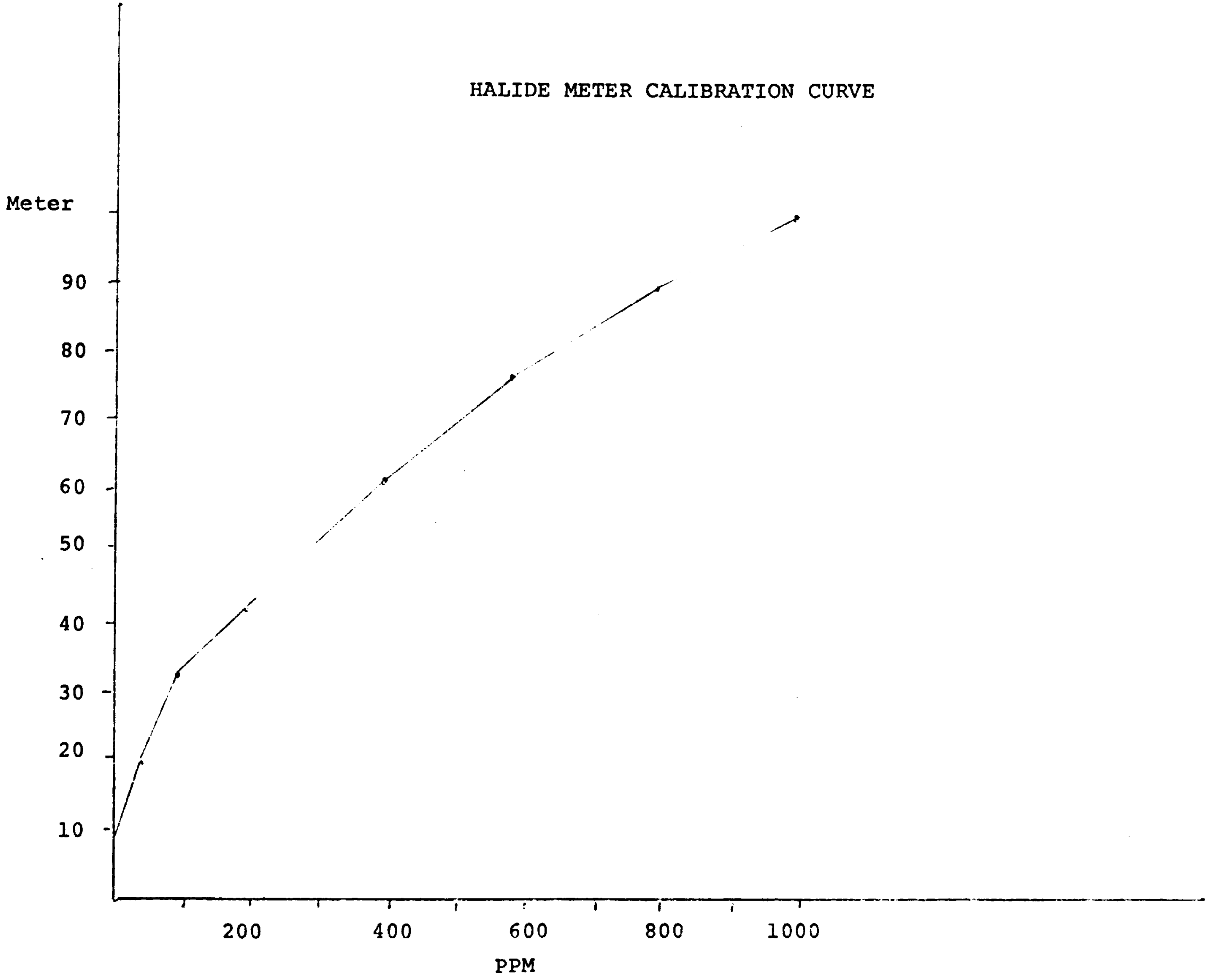


Dimensions:

Width 30.5"
Height 59"
Length 48.3"
To Solvent From Top 29.5"
Vents 2" x 50"

1 Plastic Roll Lid

2 Lip Vent



HALIDE METER CALIBRATION CURVE

APPENDIX - C7

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Evaluation of Refrigerated Freeboard Chillers

Schlage Lock
Rocky Mount, North Carolina

Prepared By:

J. C. Bollinger
U. S. Environmental Protection Agency

Prepared For:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

John Bollinger
May 1976

Test Report:

Refrigerated Freeboard Chiller
on a Monorail Vapor Degreaser

Test Site:

Schlage Lock Company
Rocky Mount, N.C.

Contents

I. Summary

This test measures the control efficiency of a refrigerated freeboard chiller on a conveyorized vapor degreaser using perchloroethylene. The refrigerated freeboard chiller uses finned coils with subfreezing coolant to establish a cold air "blanket" over the hot solvent vapor zone. (The cold air retards convection of hot solvent laden air and the diffusion of the vapors.) The vapor degreaser is a U-bent monorail. The emission rate was measured with and without the freeboard chiller.* Records of the solvent added indicate the solvent emitted. Operating the chiller and turning off the exhaust yielded a control efficiency of about 60 percent. Additional test runs could not be performed to confirm the results. Solvent purchase records showed a control efficiency of roughly 45 percent.

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*The degreaser exhaust was used only when the chiller was off. Employees complained of solvent smell when the exhaust was turned off without the chiller on. The exhaust increases the emission rate significantly.

II. Experiment Design

The method of measuring solvent emission is material balance. Basically, the solvent added to the degreaser during the test period, equals the solvent emitted. A solvent flow meter measures the gallons of solvent added into the degreaser. It is necessary that the solvent content of the degreaser at the beginning of the test period equals the content at the end. Plant personnel record the amount of workload each day in terms of number of trays. Thus the solvent emission rates can be adjusted to reflect equal workloads.

Complications arouse when equalizing the solvent content. When equalizing the solvent content, two procedures should be followed to avoid inaccuracies. First, overflow the two higher sumps and level the lowest sump to a constant depth. Note that sumps # 1 and # 2 overflow into # 3, which is the boiling sump. Second, fill the external still to the maximum level. This second step is an involved maneuver. Specifically, the operator shuts off the external still immediately after the still pump activates. He waits for the still condensate stream to cease. Then, he turns on the still so that the pump automatically fills up the still to the full level and shuts off. Next, he turns off the still again and proceeds to level the 3 sumps as previously described.

Previously given information led the measurement personnel to believe two incorrect premises: (1) sump # 2 was the lowest one and (2) the variation in solvent content of the still was negligible. Consequently, test periods # 1 to # 4 contain excess inaccuracy. Inconsistencies resulting from the previous information necessitated consulting the manufacturer of the degreaser, Detrex Inc. Thus, only test periods # 5 and # 6 are performed with the accurate sump leveling procedure, as described in the previous paragraph.

An upset which probably caused the biggest error in data is a malfunction of the primary condensing system. The thermostatically operated valve which automatically adjusts the flow of cooling water was stuck. During the hot summer months the cooling water was too warm because it was cooled by a tower. This augmented the cooling problem. Consequently, the vapor level rose up to the "Cold Trap" coils, or beyond it if it were turned off. The condensing system was not repaired until the end of test # 4. Thus the emission rates for tests # 1 to # 4 are all abnormally high.

III. Data Results

The results of the material balance measurements are present on the following table. Note that the workload is measured in number of trays. Thus the column to the farthest right can express an emission rate adjusted for a constant workload (500 trays per workday*).

Only test periods # 5 and 6 are reasonably accurate. The two periods included a proper sump leveling procedure and a repaired primary condensing system. Test periods # 1 to # 4 have less percision because the sumps were not leveled properly. More important though, the primary condensing system was deficient. This deficiency caused the emissions to be roughly twice the normal rate.

There are two approaches to calculating the emission control efficiency for the "Cold Trap." First, all six test periods could be considered. Second, tests # 1 to # 4 could be excluded, because they did not use the proper sump leveling procedure and had a defective primary condensing system

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*Only workdays are counted. The evaporation during shutdown days is comparatively insignificant. A plant engineer measured less than 1/32 inch loss in sump level during a 3 day weekend.

"Cold Trap" on Monorail Vapor Degreaser
Data Summary

								If 500 tray/ wrkdy
Test Period	Dates Spanned	Workday	No. of Trays (trays per wrkdy)	% Knobs	Cold Trap	Solvent Consumed (gal)	Emission Rate gal/wrkdy	Adjusted Em. Rate gal/500 trays
1*	7/10 to 7/21	6	2590 (432)	68%	ON	95	15.8	≈18.3
2*	7/22 to 8/4	8	3886 (486)	61%	OFF	307	38	≈39
3*	9/3 to 9/15	7	unknown	-	ON	110	15.7	≈16
4*	9/16 to 9/29	8	unknown	-	ON	140	17	≈17
5	10/14 to 10/26	10	6415 (640)	72%	OFF	250	25	19.5
5a	10/14 to 10/20	5	3615 (720)	73%	OFF	137	27.4	19.0
5b	10/21 to 10/26	5	2800 (560)	71%	OFF	113	22.6	20.2
6	10/28 to 11/10	11	-	-	ON	108	9.8	≈ 7.5
6a	10/28 to 11/3	5	-		ON	46	9.2	≈ 7.7
6b	11/3 to 11/10	6	4175 ≈ (700)		ON	62	10.3	7.4
6b	11/3 to 11/7	5	3479 (697)					

*Judge to be defective tests

Thus the second calculation should be the most accurate.

Calculations of Control Efficiencies:

1) Considering tests # 1 to # 6

Uncontrolled emission = $\frac{39 + 19.5}{2}$ = 29.3 gal/day*

Controlled emission = $\frac{18.3 + 16 + 17 + 7.5}{4}$ = 14.7 gal/day

Control efficiency = $\frac{\text{Emisison reduction}}{\text{Uncontrolled emission}} = \frac{29.3 - 14.7}{29.3} = 50\%$

2) Considering only tests # 5 and # 6

Uncontrolled = 19.5 gal/day

Controlled = 7.5 gal/day

Control Efficiency = 62% ≈ 60%

Let it be noted that omitting just test # 2 would not yield a reasonable result. At first it appears that only # 2 is far too high to be valid data. But actually tests # 1 to # 4 are all too high because of a defective primary condensing system.

IV. Equipment Description

Refrigerated Freeboard Chiller:

The refrigerated freeboard chiller uses subfreezing coolant. The subfreezing chiller design is patented by Auto Sonics Inc. (Chillers that use above freezing coolant are not patented.) Two levels of copper finned cooling coils run the inside perimeter of degreaser a few inches above the primary condensing coils. The cooling coils chill the air immediately above the air/vapor interface in order to retard convection of solvent laden air, diffusion and air/vapor mixing.

---

* All rates are adjusted to reflect a constant workload of 500 trays per workday.

Specifications:

- Manufacturer: Auto Sonics Co., Norriston, Pa.
- Model: Cold Trap # 302
- Compressor horsepower: 3 Hp
- Coils: total perimeter: 41 ft.
  number of levels: 3
  type coil: copper, finned
- Coolant temperature: 0 to -20 °F

Monorail Vapor Degreaser:

The monorail carries the parts in a U-bend pattern through the vapor zone and solvent sprays. The parts to be cleaned are door knobs and their base plates, which are loaded on tray racks. The parts experience a prewet spray, hot spray, cool rinse and then a final spray. The degreaser has three sumps: (#1) hot spray sump, (#2) cool rinse spray sump and (#3) boiling sump. The solvent overflows from sumps # 2 to # 1 to # 3. The solvent is perchloroethylene. There are two sets of primary condensing coils: one around the entrance and one around the exit. Both follow the perimeter of a rectangle 90" by 33". The degreaser is manufactured by Detrex Chemical Inc. and is a Chainveyor series 200 and Model VM 325-1S.

Still:

The still is an integral part of the degreasing system. It consistently draws dirty solvent from the degreaser sumps and returns the solvent distillate to the spray sumps. Because it is very important to totally clean particles off the brass parts before laquering, the rinses must use the pure distillate. Detrex Inc. manufactured the still and the model is S 350 S.

V. Control Efficiency from Solvent Purchase Records

A. Emission rate without the refrigerated freeboard chiller (& with exhaust on)

1. Time Period: 2/15/73 to 11/18/74 = 610 days ÷ 7 = 87 weeks

2. Solvent bought: 14,130 gal : 87 - 3 vacation wks = 84 weeks

3. Emission rate = $\frac{14,130 \text{ gal}}{84 \text{ weeks}}$ = 168 gal/wk

B. Emission with Cold Trap

1. Time Periods

#1 3/25 to 7/9/75 = 14.2 wk; 1470 gal. (approximately consumed)*

#2 7/9 to 10/10 = 13.4 wk; 1500 gal.

#3 10/10 to 1/7/76 = 10.8 wk; 2040 gal.*

#4 1/7 to 3/25 = 12.0 wk; 1540 gal.

2. Workloads - (Very roughly estimated)

#1 Assume 40 hr/wk

#2 Assume 47 hr/wk: (45 hr/wk x 83 days + 60 hr/wk x 10 days) ÷ 93 days = 46.6

#3 Assume 72 hr/wk: (60 hr x 21 + 80 hr x 31) ÷ 52 = 72 hr/wk

#4 Assume 80 hr/wk

3. Gallons per week

#1 1470 gal/14.2 (40 hr.wk.) = 104 gal/40 hr.wk.

#2 150 gal/13.4 (40 hr.wk.) = 111 x 40/47 = 94 gal/40 hr.wk.

#3 2040 gal/10.8 (75 hr.wk.) = 189 x 40/72 = 105 gal/40 hr.wk.

#4 1540 gal/12.0 (80 hr.wk.) = 128 x 40/80 = 64 gal/40 hr.wk.

Average = $\frac{104 + 94 + 105 + 64}{4}$ = 92 gal/40 hr.wk.

---

*1 week vacation

C. Results

1. Calculated Control Efficiency

$$\frac{168-92}{168} = 45\%$$

2. Accuracy: The main cause of inaccuracy in the results is the differences in workloads before and after the Cold Trap installation. The number of hours per day and the number of workdays per week both varied greatly. The average workhours per week can only be roughly approximated.

3. Assumptions:

   a. Bill McPhillips estimated 40 hrs/wk up to June 1975, 45 hrs/wk for July to September, 60 hrs/wk for October and 80 hrs/wk for Nov. (1975) and on.

   b. One week shutdown in July and one week in December, each year.

   c. The solvent tank is filled to an equal level each filling.

VI. Conclusions

The control efficiency resulting from using refrigerated freeboard chiller on a monorail vapor degreaser and turning off the degreaser exhaust, was measured at approximately 45% to 60% emission reduction. This value is derived from solvent purchase data and from the test periods which used an accurate method of sump leveling and had satisfactory primary condensation. The uncontrolled emission rate averaged roughly 20 gallons of solvent for an 8 hour work workday,* or 35 lb/hr.

---

*This applies after the primary condensing system was repaired.

Appendix A - Energy Consumption

Refrigerated Freeboard Chiller

Averages about 2.8 kw ≈9,600 Btu/hr

(6.4 amp x 440 volt = 2820 watts)

Monorail Degreaser:

Steam for sump heat = 485,000 Btu/hr

Elect. for pumps = 11.8 kw = 40,000 Btu/hr

Still:

Steam = 450,000 Btu/hr

Elect. for pumps = 6 kw = 20,000 Btu/hr

Percent Energy Required for Chiller:

2% of Degreaser energy

1% of Degreaser and Still energy

Note: The energy requirement for the still is unusually high, because a high rate of distillate solvent is needed for this cleaning application.

Appendix B - Capital Cost Data

SCHLAGE
LOCK · COMPANY

EXECUTIVE OFFICES
AND FACTORY
BAYSHORE BOULEVARD
P.O. BOX 3324
SAN FRANCISCO 94119

ROCKY MOUNT
U. S. HIGHWAY 301 NORTH
P. O. BOX 552
ROCKY MOUNT, N. C. 27801

TELEPHONE
(919) 446-1905

977-0450

July 22, 1975

COST OF DEGREASER

Degreaser, Detrex Model No. VM-325-1S----------------- With S-350 Still	$40,884.37
Install Detrex Degreaser-------------------------------	591.00
Electrical Installation Detrex Degreaser-------------	1,227.00
Power Controls for Degreaser--------------------------	1,585.00
Total	$44,287.37
Cost of Cold Trap, Model 302-------------------------	7,944.00
Cost of Installation----------------------------------	350.00
Total	$8,294.00

Degreaser began operation in April , 1972, Cold Trap installed January, 1975.

Richard T. Wilcox

Richard T. Wilcox
Plant Engineer

Appendix C - Savings/Cost Ratios

High Control Efficiency = 60%

Savings: $60\% \times 20 \frac{\text{gal}}{\text{day}} \times 250 \frac{\text{day}}{\text{year}}{}^{*} \times 2.16 \frac{\$}{\text{gal}} = 6480 \ \$/\text{yr}.$

Annualized Cost:

Capital cost of "Cold Trap" = \$8,294 (Includes shipping & installation)

Assume 15 year life (x 0.1315)

Annualized capital cost =	1090 \$/yr
Electrical cost $2.8 \text{ kw} \times 3 \frac{¢}{\text{kw}} \times 8 \frac{\text{hr}}{\text{day}} \times 250 \text{ day} =$	170
Maintenance cost = 4% x \$8,300 =	330
Insurance = 2% x 8,300 =	165
	1775 \$/yr

Savings/Cost ratio = 6480/1755 = 3.7

Moderate Control Efficiency = 40%

Savings: $6480 \times \frac{40}{60} = 4320$ \$/yr

Savings/Cost ratio = 4320/1755 = 2.5

Breakeven Control Efficiency

$$\frac{E \cdot 10800}{1755} = 1 \rightarrow E = 16\% \text{ control efficiency}$$

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*Based on a 5 day workweek. Actually the plant tested, Schlage Lock, has been running only 4 days per week, but 5 days per week is more typical of degreasing plants.

APPENDIX - C8

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS OF SOLVENT METAL CLEANING OPERATIONS

EVALUATION OF CARBON ADSORPTION RECOVERY

Super Radiator Corporation
St. Louis Park, Minnesota

PREPARED BY:

D. W. Richards
The Dow Chemical Company

PREPARED FOR:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

An evaluation of a carbon adsorption unit in support of an open top degreaser was conducted at Super Radiator Corporation. Due to some unique circumstances, the use of the adsorber was found to cause a slight increase (~8%) in solvent emissions. Even though this situation was explained and corrected using the data obtained during the test period, it does point out what can occur in an actual industrial operation. The need to periodically monitor the performance of this control device is quite evident. Due to the manner in which the adsorber was operating, it was increasing plant operating costs by an estimated $8500 per year. Also, the solvent recovered by the carbon adsorber did not contain any stabilizers which means that it must be mixed with new solvent or restabilized before reuse.

Objective

The purpose of this test program is to evaluate carbon adsorption as a means of controlling solvent emissions from metal cleaning operations. The information needed for this evaluation includes determining:

1. the efficiency of this device in reducing solvent emissions to the atmosphere,
2. the cost/benefit relationship of this emission control system,
3. the energy requirement of the emission control system,
4. any alternate emissions created by the emission control system.

This data base is being developed to forecast the magnitude of emission reductions which can be achieved nationally and the effect on businesses involved. This information combined with the results of other testing will be used to design emission control regulations which effectively limit air pollution and are practical for industrial application.

Introduction

Carbon adsorption has been offered to solvent metal cleaning users since 1958. The efficiency of carbon beds to capture solvent vapor from low concentrations in air has been estimated to be greater than 95 percent. The typical efficiency of carbon adsorption in reducing solvent consumption (controlling solvent emissions) is commonly reported to be in the range of 50-60 percent. Inspite of this, only a small fraction (1.5 percent) of the industrial solvent metal cleaning users surveyed*, reported that they were using carbon adsorption.

Super Radiator was considered as an emission control test site on the recommendation of Vic Manufacturing Co. (carbon adsorption manufacturer). This operation was selected for evaluation because it was felt that it would demonstrate typical industrial use of carbon adsorption in support of an open top degreaser. The unit was installed in December, 1971.

Before the evaluation began, service personnel from Vic Manufacturing inspected the carbon adsorption equipment to be sure that it was functioning properly. Severe solvent "breakthrough" was discovered and two more desorption cycles were added to alleviate this problem.

*See Appendix A

Equipment

The basic metal cleaning process employed is a Detrex VS 800 (6' x 12') open top degreaser and a carbon adsorption unit operated with perchloroethylene. The degreaser is a gas-fired unit (See Figure 1 below).

The carbon adsorption unit is a model 554 AD. Solvent-laden air is drawn into the unit through a 14" duct. After passing through the carbon bed it is discharged outside through another 14" duct. When the carbon adsorption unit was installed, it was also necessary to install a 10 h.p. boiler to provide steam for the desorption cycles since no plant steam was available.

Behind the degreaser are two solvent storage tanks (2' diameter x 4' long) which are used during degreaser clean-outs. The degreaser contents are boiled down to the heating tube; pure solvent is condensed and transferred to the storage tanks; and any remaining "sludge" is removed from the degreaser. The solvent from the storage tanks is then returned to the degreaser and new solvent is added to bring the degreaser back to its operating level. A schematic of the metal cleaning system is presented in Figure 2.

Figure 1
OPEN TOP DEGREASER
SUPER RADIATOR CORPORATION

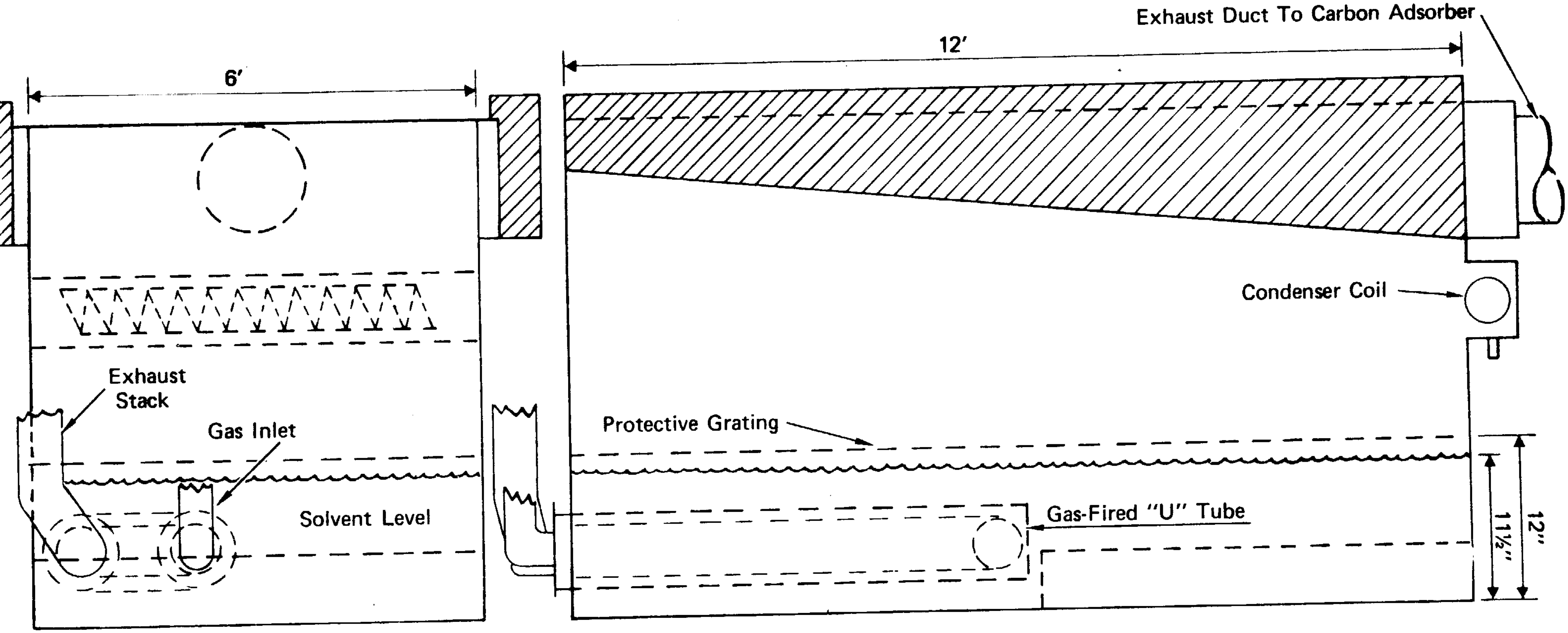
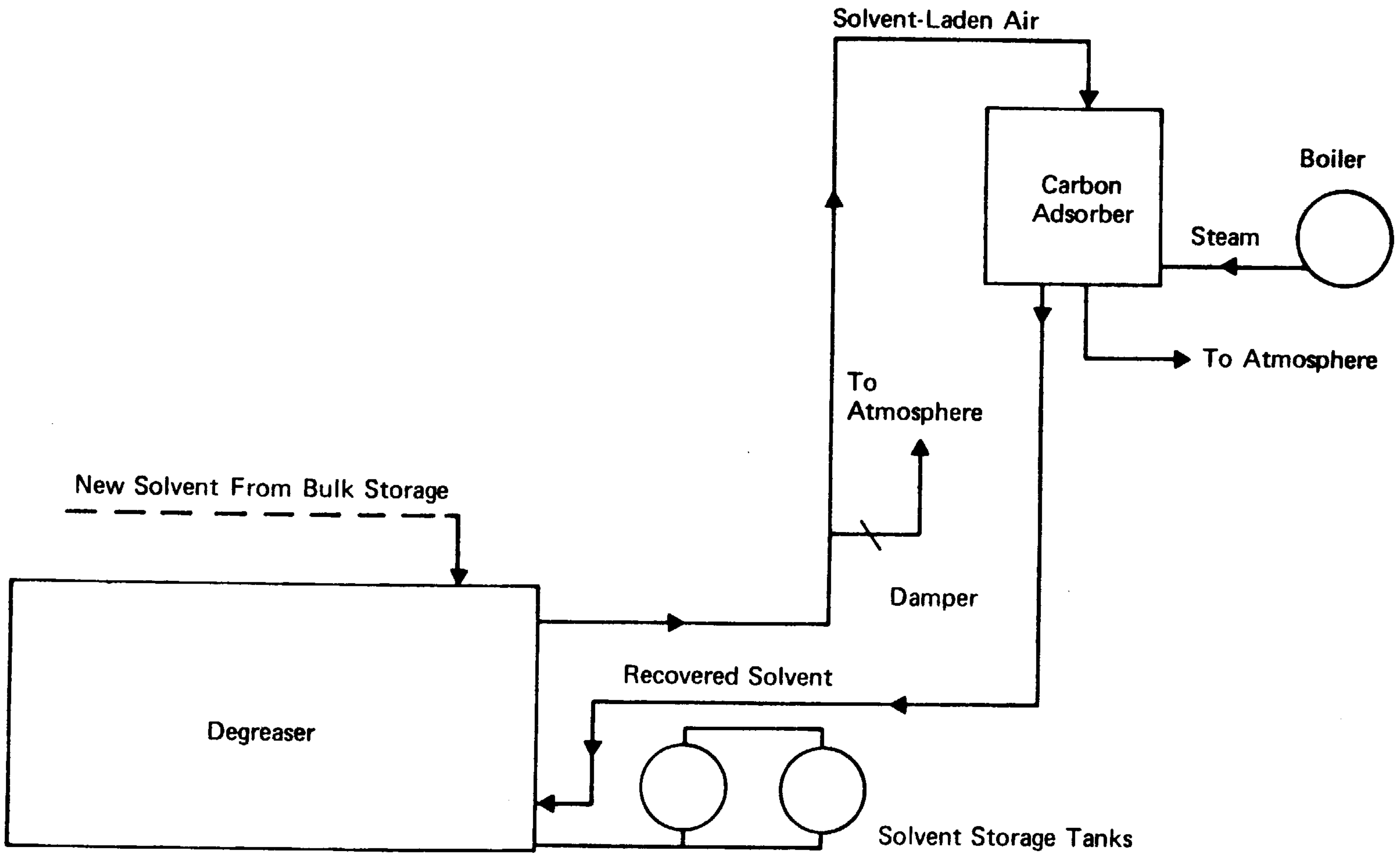


Figure 2
METAL CLEANING SYSTEM
SUPER RADIATOR CORPORATION



Experiment Design

This test was designed to obtain two weeks of data with the carbon adsorber operating and one or more weeks with it off. For reasons which will be discussed later, the carbon adsorber was operated for two weeks and turned off for three weeks during the actual test period. The test began on May 19, 1975 and concluded in August. Since all of the new solvent added to the degreaser comes from a bulk storage tank, a solvent meter was installed in the delivery line so that solvent additions could be measured accurately. A gas meter was also installed in the gas line to the degreaser so that the energy input could be determined. The difference in solvent inventory was determined by measuring the amount of solvent necessary to bring the level in the degreaser to a given mark on a "dip stick". The degreaser was leveled each morning before any work was processed. The amount of work processed was also recorded daily.

Data Discussion

During the initial three weeks of testing, the carbon adsorber was operated for two weeks and turned off for the final week. Since the solvent consumption decreased the week the carbon adsorber was turned off, two additional weeks of data were obtained with the adsorber off. Once again, a decrease in solvent consumption was observed. Several parameters were found to be responsible for this situation, some of which are unique to this metal cleaning system. Each will be discussed separately as will the method used to determine solvent consumption.

Solvent Consumption - As discussed previously, all new solvent added to the system was added through a solvent meter to the degreaser. Solvent was added each morning (before any heat was applied) to the same mark on a wooden "dip stick". The ability to fill to this mark was estimated to be ± 1/8". Since the mark was 11 1/2" from the bottom of the stick, this is equivalent to a possible error of ± 1.0%. The average new solvent additions to the degreaser when the carbon adsorber was operating was 49.0* gals./day and 36.6* gals./day when the adsorber was turned off. The average work processed by the degreaser when the adsorber was on was 8995 lbs./day and 7283 lbs./day when it was off. This is equivalent to

---

*New solvent additions to the degreaser were adjusted to account for the fact that varying amounts of kerosene were present in the degreaser during the test.

0.00545 gals./lb. with the adsorber "on" vs. 0.00503 gals./lb. with it "off". The emission control efficiency is therefore:

$$\frac{.00503 - .00545}{.00503} \times 100\% = -8.35\%$$

The negative efficiency calculated by this equation is obviously not representative of the degree of emission control possible with a carbon adsorber. For this system, however, this number is believed to be valid. Some of the key factors which are felt to be responsible for this negative percentage are as follows:

1. Minimal Freeboard Height - During the test period it was observed that the vapor level in the degreaser was allowed to continually rise to within 3" to 8" of the lip exhaust. Therefore, the effective freeboard height during the test period was 3" to 8". Standard degreasing practice calls for a desirable operating freeboard height of 0.5 to 0.6 times the inside width (72") of the degreaser, which in this case would be 36" - 43". If this cannot be practically achieved, a minimum freeboard height of 12" is recommended. Maintaining an adequate freeboard height in any open top degreaser

is important if solvent losses due to drafts in the plant are to be minimized. Another important reason for maintaining a greater freeboard height in this particular degreaser relates to the exhaust fan velocity which is discussed in paragraph No. 2.

2. Exhaust Fan Velocity - When the carbon adsorber is operated, both the degreaser lip exhaust fan and the adsorber fan are used. The inlet velocity to the adsorber which is developed with both fans operating (and both beds adsorbing) was found to be 2496 ft./min. (2671 cfm). The velocity dropped to 1460 ft./min. (1562 cfm) when one bed was desorbing. When the degreaser is exhausted to the atmosphere, only the degreaser fan is used. This fan was found to operate at only 557 ft./min. (741 cfm).

Since the vapor zone was allowed to rise to within 3" to 8" of the degreaser lip exhaust, large quantities of solvent were available to be drawn to the carbon adsorber at 1562-2671 cfm. Normally, this solvent would simply be recycled to the degreaser, but in this test much of it "broke through" (see para. 3) the beds and was lost to the atmosphere. This resulted in an abnormally high solvent consumption during the time the adsorber was operated. Also, the amount of solvent lost to the atmosphere when the adsorber was not operated was actually less because the exhaust velocity was only 741 cfm.

3. Vapor Concentration Measurements - The solvent vapor concentrations in the inlet and outlet ducts of the carbon adsorber were measured from 9:45 a.m. to 3:15 p.m. on August 19, 1975 with a Gas Tech halide meter. While much was learned from this data, no attempt will be made to plot concentrations in the outlet duct. The reason for this is that during the desorption of both beds, readings were obtained that were so high that they were off of the curve which is used to convert meter readings to parts per million of solvent. For the same reason, the average efficiency of the carbon beds could not be determined.

   Concentration measurements in the outlet duct did indicate, however, that "breakthrough" was occurring continuously. With both beds adsorbing, measurements varied from 160-770 ppm throughout the day. Also, when Bed "A" was desorbing, measurements in the outlet duct ranged from 35 ppm to greater than 2000* ppm. When Bed "B" was desorbing, readings ranged from 12 ppm to greater than 1500* ppm. All of these measurements were made at the end of the test period.

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*Estimated by extrapolating the curve for converting meter readings to ppm perchloroethylene.

As discussed earlier, the equipment manufacturer inspected the carbon adsorber before the test began, observed breakthrough, and added more desorption cycles to alleviate the problem. Therefore, no additional checks for breakthrough were deemed necessary until the solvent consumption data indicated negative adsorber efficiency.

Concentration measurements in the inlet duct were much higher than those in the outlet duct and therefore even further off of the conversion curve. Extrapolation estimates for inlet concentrations while the degreaser was idling range from 2,000 ppm to 5,000 ppm. Once again, it would be unrealistic to attempt to develop an average bed efficiency based on these estimates.

Solvent recovered by the carbon adsorber averaged 476.75 lbs./day (35.40 gals./day). This data was obtained by collecting solvent from all six of the one hour desorption cycles over a 24 hour period. The average solvent collected per desorption cycle is, therefore, 79.46 lbs. or 5.90 gals. These recovery rates are not indicative of the maximum capabilities of the carbon adsorber as predicted by the manufacturer. Manufacturer estimates for solvent recovery with this size adsorber are 200 lbs./hr. (14.85 gals./hr.) of perchloroethylene.

The utilities required to operate the carbon adsorber are steam, water, compressed air, and electricity. Steam condensate was collected and weighted for four separate one hour desorption cycles. The weights were: 262.75 lbs., 272.25 lbs., 244.25 lbs., and 288,50 lbs. (average = 266.94 lbs.). Using 1,000 Btu's per pound of steam, 267,000 Btu's are required per desorption cycle. Thus, 267,000 Btu's/desorption x 6 desorptions/day x 240 days/yr. = 384 x 10^6 Btu's/yr.

The carbon adsorption unit's condenser is estimated to use 16 G.P.M. by the manufacturer. Since the water only flows during the desorption cycles, the total water requirement is 16 gals./min. x 60 min./hr. x 6 hr./day x 240 days/yr. = 1,380,000 gals. The carbon adsorber fan is rated at 10 horsepower. This is equivalent to: 10 Hp x .746 KWH/Hp hour x 17 hrs./day x 240 days/yr. = 30,440 KWH/yr. An energy balance for one bed of the carbon adsorber (adsorption and desorption cycles) is summarized in Table No. 1. Radiation losses through the shell of the adsorber bed are not included in order to simplify the overall balance.

The replacement cost for the 554AD carbon adsorption unit studied during this test is estimated to be $13,990 by Vic Manufacturing. Since plant steam was not available at this test site, a boiler was also purchased with the carbon adsorber. The estimated replacement cost for this is $4000. A special platform was also constructed above ground level to house the adsorber and boiler and to save on floor space. The estimated cost of this is $3300, bringing the total replacement cost for this equipment to $21,290. This information was used to develop Tables 2 and 3. The assumptions involved in the development of these tables include a zero return on investment; charging the construction of the elevated platform as direct capital and therefore not charging any floor space against the carbon adsorber; the omission of costs for other facilities which are required, but already exist; and the omission of other minor costs such as heating, lighting, janitorial, etc. Based on these assumptions, the total operating cost per year for this model 554AD carbon adsorber is calculated to be $6293 (see Table 3). If a price of $0.16/lb.* ($2.16/gal.) is used for perchloroethylene, then 2913 gallons must be recovered per year by the carbon adsorber to offset the total annual operating cost. Based on plant purchasing records through July, 1975, a total of approximately

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*Chemical Marketing Reporter, July 7, 1975, Schnell Publishing Company

12,500 gallons of solvent will be used during 1975.

Using the emission control efficiency of -8.35% observed during the test, this would mean that approximately 1000 gallons of solvent would be saved per year by <u>not</u> using the adsorber. This would also mean that, under these operating conditions, the adsorber would actually add ~$8500/yr. to this company's operating costs [$6293 (from Table 3) + 1000 gals. x $2.16/gal.].

While this is obviously not a desirable situation, it can be remedied by making relatively minor changes in the operation of the degreaser and carbon adsorber.* This does demonstrate, however, the need to periodically check the performance of this type of emission control device using more sophisticated techniques than just measuring the amount of solvent recovered. One such technique would be to check the outlet duct of the adsorber to be sure that no significant solvent "breakthrough" is occurring.

Samples of solvent recovered from the carbon adsorber were obtained at various times during the test and analyzed for stabilizer content. Perchloroethylene is not as highly stabilized as most of the chlorinated solvents, but depletion

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*Changes were made in the operation of the adsorber after the "breakthrough" problem was discovered. Unfortunately there was not time to collect data for use in this study, but reports from Super Radiator indicate that the adsorber is now operating on a profitable basis.

of the stabilizers can result in considerable problems. No stabilizers were found in any of the samples of solvent recovered from the carbon adsorber. Before this solvent can be reused, therefore, it must be blended with an adequate amount of new solvent or replenished with a stabilizer concentrate.

The gas flow rate to the degreaser was checked several times during the test and averaged 51.87 ft.^{3}/min. Since the degreaser is operated approximately 14 hrs./day, the daily usage rate is: 51.87 ft.^{3}/min. x 1000 Btu's/ft.^{3} x 60 min./hr. x 14 hr./day = 43.6 x 10^6 Btu's day. The steam requirement for the carbon adsorber is 267,000 Btu's/desorption cycle x 6 cycles/day = 1.6 x 10^6 Btu's/day. Expressed in the same units, the energy required by the carbon adsorber fan is 10 hp. x 2545 Btu's/hp. x 6 cycles/day = 0.15 x 10^6 Btu/day. The total energy demand of the carbon adsorber is, therefore, 1.75 x 10^6 Btu's/day. This represents an energy increase of only about 4% over that required for the degreaser*.

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*The energy required for the small degreaser lip exhaust fan was considered to be negligible and not included in the calculation.

Conclusions

1. For this particular metal cleaning system, solvent emissions were actually reduced when the carbon adsorber was not operated, based on new solvent additions to the degreaser. It is important to recognize that this reflects the effects of several different parameters, many of which are unique to this cleaning system. This demonstrates, however, that this type of emission control device requires at least occasional checks to assure that it is functioning properly.

2. Since the solvent vapor level in the degreaser was maintained so near the lip exhaust, large quantities of solvent were drawn into the adsorber. This compounded the existing "breakthrough" problem and magnified the solvent losses considerably. As a result, operation of the adsorber in this manner will add ~$8500/yr. to the plant operating costs.

3. Since plant steam was not available at this test site, it was necessary to purchase a boiler to be used in conjunction with the carbon adsorber,

which demonstrates that additional costs may be incurred if this method of controlling emissions is selected.

4. Perchloroethylene recovered by the carbon adsorber contained no measurable amount of stabilzer and must be blended with new solvent or restabilized before it is used again.

TABLE 1

Energy Balance On Carbon Adsorber (1 Bed)

	Input	Output
Adsorption (Solvent Recovery)		
Solvent Latent Heat (79.46 Lbs.)	7,200 BTU's	
Air Flow At 2671 CFM		7,200 BTU's
Desorption		
Steam (267 Lbs.)	267,000 BTU's	
Heating Tank (~2000 Lbs.)		31,240 BTU's
Heating Carbon (1000 Lbs.)		28,400 BTU's
Condenser Water (16 GPM)		207,360 BTU's
Vaporizing Solvent*	7,200 BTU's	7,200 BTU's
Adsorption (Drying and Cooling Bed)		
Cooling Tank	31,240 BTU's	
Cooling Carbon	28,400 BTU's	
Air Flow At 2671 CFM		59,640 BTU's

*Vaporizing solvent from the bed requires heat from steam, but this same heat is given up to the condenser water when the vapor is condensed to liquid solvent.

TABLE 2

Carbon Adsorber

Building Space	Not Applicable (None is assigned since the carbon adsorber and boiler are housed on an elevated platform and do not occupy any previously existing floor space.)

Direct Capital

Carbon Adsorber (13,990) Boiler (4,000)	$17,990	(Vic Manufacturing)
Platform	$ 3,300	
Shipping and Installation	$ 2,700	(At 15% of Selling Price)
Total Capital	$23,990	
Cost/Annum	= $3,154	(At 15 Yrs. Depreciation Rate)*

*10% Interest Rate On Investment

TABLE 3

MODEL 554 AD CARBON ADSORBER
OPERATING COST PER ANNUM

Capital		
Equipment	$3154	
Building	N.A.	
Insurance (2%)		
Equipment	$ 480	
Building	N.A.	
Maintenance (4%)	$ 960	
Utilities		
Steam	$ 883	(384 M̄ BTU's at $2.30/M̄ BTU's)
Electricity	$ 761	(30,440 KWH at $0.025/KWH)
Water	$ 55	(1380 M Gals. at $0.04/M Gals.)
Compressed Air	Nil	
Labor	Nil	
Return on Investment	0	
Total Cost/Annum	$6293	

APPENDIX - C9

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Evaluation of Carbon Adsorption Recovery

at J. L. Thompson Co.
Waltham, Mass.

Prepared by:

K. S. Surprenant
The Dow Chemical Company

Prepared for:

Emission Standards and Engineering Div.
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

The evaluation of a carbon adsorption system as an emission control for a cross-rod degreaser was conducted at J. L. Thompson Co. This solvent recovery system had been installed several years ago. After minor repairs, it was found to be recovering 23 percent of the solvent supplied to the degreaser. The major costs of operation and the capital invested exceeded the value of the reclaimed solvent annually by $817.

Solvent recovery data from earlier operation (when the process volume was higher) indicated a greater volume of solvent recovered. This solvent recovery rate would provide a return of 2.58 times the annual costs of the recovery system. However, all samples of recovered solvent were unstabilized and unsuitable for reuse without restabilization or dilution with fresh solvent.

Objective

The purpose of this test program is to evaluate carbon adsorption as a means of controlling solvent emissions from metal cleaning operations. The information needed for this evaluation includes determining:

1. the efficiency of this device in reducing solvent emissions to the atmosphere,
2. the cost/benefit relationship of this emission control system,
3. the energy requirement of the emission control system,
4. any alternate emissions created by the emission control system.

This data base is being developed to forecast the magnitude of emission reductions which can be achieved nationally and the effect on businesses involved. This information combined with the results of other testing will be used to design emission control regulations which effectively limit air pollution and are practical for industrial application.

Introduction

Carbon adsorption has been offered to the solvent metal cleaning users since 1958. The efficiency of carbon beds to capture solvent vapor from low concentrations in air has been estimated to be greater than 95 percent. The typical efficiency of carbon adsorption in reducing solvent consumption (controlling solvent emissions) is commonly reported to be in the range of 50-60 percent. Inspite of this, only a small fraction (1.5 percent) of the industrial solvent metal cleaning users surveyed*, reported that they were using carbon adsorption.

J. L. Thompson was selected as an emission control test site on the recommendation of the carbon adsorption supplier, Hoyt Manufacturing. During the preliminary inspection of the equipment, a weekly solvent recovery rate of 50 gallons was reported along with a solvent consumption rate of about 50 gallons per week. This level of solvent recovery or emission control was judged to be worthy of evaluation.

The initial study time revealed an equipment failure. This failure caused the inlet damper to carbon Bed B to remain closed. Bed B was recovering no solvent. Although some data was lost, the efficient efforts of J. L. Thompson Co. and Hoyt Manufacturing restored the carbon adsorber to operating condition in time to permit brief testing.

In April 1968, this equipment was installed. Carbon adsorption equipment has been reported to have an operating life of 15 or more years. Thus, data on seven year old equipment is both valid and needed. This combined with the excellent records and cooperation of J. L. Thompson, the metal cleaning process equipment (a cross-rod degreaser) and the process solvent (trichloroethylene) continued to make this test site a valuable source of emission control testing.

*See Appendix A

Equipment

The basic metal cleaning process employed is a three sump cross-rod degreaser and still operated with trichloroethylene. The equipment is steam heated. Every other fixture is a rotating basket designed to handle small metal parts, fasteners in this case. The alternate fixtures process the tote pans for the parts. Rotation is obtained by rack and pinion design. See Figure 1.

The carbon adsorption system is a Model 536AD. A three horsepower fan draws air through a 12 inch duct from the left side of the operator loading station at the degreaser. After adsorbing the solvent vapor contained in the air in its passage down through the carbon bed, the air is discharged by another 12 inch diameter duct.

Experiment Design

The plan for testing this emission control system called for running the degreaser system for two weeks with the carbon adsorption system operating and two weeks without the carbon adsorber. The workload, solvent consumption and heat input data would be recorded weekly and compared. Additional weeks of testing would be used if the data under similar operating conditions did not compare favorably.

This plan had to be abandoned due to two factors. First, the carbon adsorber was found to be mal-functioning. Second, the firm had arranged to convert the system to methylene chloride during plant shut-down July 12 thru July 21, 1975. This change was being made to comply with local air pollution codes and required considerable equipment changes. These equipment changes could not be done when the plant was operating.

Some valuable data was obtained after repair of the carbon adsorber. However, time did not permit data collection without the carbon adsorber. For purposes of evaluation, it will be assumed that the solvent losses from the degreaser would be the same with or without the carbon adsorption ventilation system. This assumption should be essentially valid. The air collection plenum to the carbon adsorber is sufficiently removed from the degreaser vapor zone that it would be unlikely to cause increased losses of solvent vapor. This assumption is not valid when the adsorber vent system is close enough to disturb the vapor zone which is often the case with open top degreasing systems.

Data Discussion

Solvent Consumption

Production records were kept from May 19, 1975 to July 11, 1975. During this time, an average of 3.50 tons of work was processed per day. Workload variations ranged from 3,338 pounds to 10,081 pounds per day. The parts are transferred in tote boxes which are cleaned in the same degreaser. An average of 81.1 boxes were processed per day. Their estimated weight is about 22 pounds, thus, they contribute to another 0.89 tons per day to the work processed by the vapor degreaser. The total metal cleaned (including the tote boxes) per day was 4.39 tons per day.

A solvent meter was located between the main storage tank and the degreasing operation. Meter readings between May 19 and July 11 reported 374 gallons of solvent consumed. Thirty-seven days of operation occurred during this interval. Based on this information, the average solvent consumption rate was 10.1 gallons per day or 50.5 gallons per week.

Solvent purchase records for the months of January, February, March and April 1975 totaled 842 gallons.

Thus, the solvent consumption rate based on that 17 week period was 49.5 gallons per week. Note that both figures for solvent consumption make no adjustment for solvent lost from the system as still residues or solvent recovered by the carbon adsorption and used again.

Solvent Recovery

The total solvent available for recovery is the sum of all solvent additions needed to maintain a constant inventory in the system. Although only 50.5 gallons of new solvent was required per week in this degreasing operation, an additional 5 gallons per week, on an average basis, was added to the degreaser from the carbon adsorption system. This includes both that portion of the operating time when poor solvent recovery was being obtained and after the carbon adsorption system had been repaired.

The quantity of solvent removed from the system with the still residues would not be available for solvent recovery. A total of 1,987 pounds of still residues were removed from the system during the same test period. The average solvent content of this material was 23 percent by volume. Consequently, 46 gallons of trichloroethylene were removed from the system with these still residues and were unavailable to be recovered by the carbon adsorption system. On a daily basis, this amounts to 1.2 gallons which were unavailable to be recovered by the carbon adsorption system. Therefore, only 9.9 gallons of solvent were available for recovery per day. This was determined as follows: 10.1 gallons of fresh solvent per day plus 1.0 gallons/day from the carbon adsorber minus 1.2 gallons/day removed from oil residues. It is also valuable to note that the 46 gallons of solvent lost in the still residues represent 12 percent of the new solvent added to the system. This percentile of solvent could not have been recovered even if the emission control had performed at 100 percent efficiency.

After repairing the carbon adsorber, the solvent recovery was determined. From July 1, 1975 to July 11, 1975, the quantity of solvent recovered ranged from 2.01 to 3.52 gallons per day. The solvent was measured by the level change which occurred in the solvent collection tank (14 inches in diameter). A factor of 0.667 gallons per inch was used to convert to gallons. Two calibration checks were made by weighing the solvent and comparing the level change in the tank. Both were within the ± 0.008 gallons expected from measurements in error by ± 1/8 inch. The average solvent collected per day was 2.53 gallons (30.4 pounds/day). An overall recovery efficiency of 25.5 percent is obtained when this is compared to the 9.9 gallons available for recovery.

Solvent Use Per Ton of Work

The total quantity of metal cleaned including both tote boxes and the work pieces amounted to 4.39 tons per day. When this is divided into the 374 gallons consumed during the test (10.1 gallons per day), 2.30 gallons per ton of metal cleaned is found to be the consumption rate on a tonnage basis. Although not demonstrated in this testing, the solvent consumption rate per ton is expected to diminish as the work processed increases. In terms of the capacity of the vapor degreasing machine, only about 25 percent of the total work capacity of this degreaser was being used during the test interval. It is not uncommon to find metal cleaning equipment being used at much lower than machine capcity. Over-design of this equipment is common in order to avoid having it become the limiting factor in plant production.

Bed Efficiency

The solvent vapor concentrations in the exhaust duct from the carbon adsorption system for a single day of the test, July 11, 1975, are plotted on Figure 2. The average vapor concentration in the exhaust was 10.6 ppm.

The elevated plateaus between 11:00 - 12:00 and 1:30 - 2:30 correspond to the desorption cycle for Bed B and Bed A respectively. During the time that one bed is desorbing, the other bed is trying to do the work of both. Consequently, the higher concentrations during the desorption cycles are expected.

The concentrations of vapor being carried from the vapor degreaser to the carbon adsorption beds were measured several times during the course of the day and varied only with whether the machine was processing work or being maintained at idle. During the work process periods the vapor concentrations varied from 37 to 145 ppm and had an average of 74 ppm. The solvent vapor concentrations when no work was being processed (idling) ranged between 37 and 50 ppm with an average of 41 ppm. It is valuable to know that no variations in the exhaust vapor concentration from the carbon adsorber were observed to correspond to variations in the inlet composition or the work activity in the vapor degreasing operation.

Although the vapor degreaser was processing work on and off throughout the day, the hours of actual vapor degreaser operation were estimated at two hours per day. When vapor concentrations in the inlet duct to the carbon adsorber are time-weighted for the hours of operation and idling, the average daily concentration was found to be 49 ppm. The bed efficiency was determined as follows:

$$\frac{\text{Aver. Inlet Conc. (49 ppm)} - \text{Aver. Exhaust Conc. (10.6 ppm)}}{\text{Aver. Inlet Conc. (49 ppm)}} = 78\%$$

The bed efficiency can be expect to increase as the vapor concentrations in the inlet to the carbon adsorption system increase. This is supported by examining the vapor concentrations obtained earlier in the workday when the inlet vapor concentration was 102 ppm and the average exhaust concentration was 5 ppm. The bed recovery efficiency during this time interval was 95 percent. Of course, the recovery efficiency of the bed diminishes as the bed approaches its saturation capacity for the solvent. The bed recovery efficiency can also be expected to decline with time over years of use due to the adsorption of materials on the bed which cannot be desorbed. This diminishes the capacity of the bed for solvent. This carbon adsorption system is seven years old, as noted earlier.

Vapor concentrations in the inlet duct of the carbon adsorber can be expected to vary greatly with the type of work processed. Some work parts carry decidely more solvent out of the degreaser in spite of the rotary action of the fixtures. For instance, on May 28, 1975, vapor concentrations going to the carbon adsorber varied between 50-86 ppm without work being processed (average 65 ppm). The concentrations with work processed from 80 ppm to ~1500 ppm (average 480 ppm).

The average inlet concentration on a daily basis was estimated to be 169 ppm. The exhaust concentration was ~5 ppm. In this case a bed recovery efficiency of ~97 percent was observed.

On the same day, the vapor concentrations in the operator breathing zone were determined. This was done to establish whether or not safe conditions could be maintained without the carbon adsorption system operating. These concentrations were found to average 15 ppm trichloroethylene (ranged 0-40 ppm over 16 minutes) without the carbon adsorber vent fan or the standing operator fan. Safe levels for trichloroethylene are 100 ppm for an average daily exposure.

Solvent samples representing five separate desorption cycles were analyzed for stabilizer content. In every case, the stabilizers were almost entirely lost. If this solvent were used by itself or in larger proportion to the volume of fresh solvent, the solvent would be unprotected and would be likely to decompose. Samples of solvent taken from all portions of the equipment were found to contain only marginal stabilizer levels.

Steam Requirement

The carbon adsorption system uses steam and water for desorption, compressed air to activate the valves and dampers and a 3 horsepower electric motor for the fan. The steam condensate from two desorption cycles was collected and measured (120 pounds, 106 pounds) for an average of 113 pounds per desorption cycle. At 1,000 btu's per pound of steam, 113,000 btu's were used per desorption. With two desorptions per day and 240 days per year, the annual steam requirement is estimated at 54.2 million btu's.

Water Requirement

Water used by the carbon adsorption system is estimated by the manufacturer at 8 gallons per minute. The water flows only during the desorption cycles which last 60 minutes each. Thus, the water per year amounts to 230,000 gallons. The electric 3 horsepower motor demand can be estimated as follows: 3 horsepower x .746 kilowatt hours per horsepower hour x 8 hours per day x 240 days per year = 4,300 kilowatt hours per year.

Cost of Operation

Hoyt Manufacturing estimated a replacement purchase price of $8,000 for an equivalent model to that sold to J. L. Thompson in 1968. This information along with the utility consumption data allows the estimation of the cost of this operation summarized on Tables 1, 2 and 3. The total operating cost per year was estimated to be $2,256 (see Table 3). The 50 percent extra floor space assigned in Table 2 provides for aisleways, restrooms, etc., in proportion to the equipment direct space consumption. No costs have been included to account for numerous other facilities which are needed but already present. Some of these would include a portion of the investment capital for electrical transformer equipment, a steam boiler, an air compressor, roads and grounds. Also, minor routine costs have been omitted such as roads, grounds and building maintenance, heating and lighting operating costs for the work area and janitorial services.

If the operating cost per year of $2,256 and a solvent price of $2.37/gallon ($0.195/pound) are taken 952 gallons of solvent would be needed to recover these costs. Based on the 2.53 gallons/day recovery rate, only 607 gallons/year (2.53 gallons x 240 days/year) would actually be recovered. A net loss of about $817 per year was being experienced during the evaluation period.

In contrast, customer records for seven months beginning December 1970 show a recovery average of 205 gallons/month. This time period was chosen at random and was roughly two years after initial installation. At this rate of recovery, an estimated 2,460 gallons would be saved per year. The value of this solvent (at today's prices) would be $5,830 per year. At this recovery rate a profit of $3,574 per year was being created by the carbon adsorber. During 1971, 2453 gallons of trichloroethylene were purchased. Thus, 50 percent of the total solvent supplied to the degreaser was being recycled by the adsorption system.

The ventilation rate through the carbon adsorption system was determined to be 1,190 feet per minute. The duct diameter is 12 inches. Thus, the average ventilation rate was found to be 935 cubic feet per minute (cfm) when both beds were adsorbing. When one bed was in the desorption phase of its cycle, the back pressure created by only one bed reduced the ventilation velocity to about 850 feet per minute and the volume of air to about 670 cfm . The solvent recovery rate reported earlier was 2.53 gallons per day or 30.4 pounds per day. This quanity of solvent is recovered from both beds desorbing once per day. The average solvent yield per desorption cycle was 15.2 pounds.

Since this process has two distinctly different phases of operation, a material balance for each phase is represented separately. Figure No. 3 describes the adsorption phase while Figure No. 4 summarizes the desorption phase. In both cases, the data represented is the average of results obtained during testing. The carbon adsorption system, like the degreasing operation, is operated only eight hours per day. Each bed is desorbed once per day for one hour. Thus, each bed operates on the adsorption phase of the cycle seven hours per day. Table No. 4 summarizes the energy exchanges which take place in the various portions of the adsorption-desorption cycle. For simplicity, heat loss through the wall of the carbon adsorption bed is not considered.

Essentially all of the heat energy transferred to a vapor degreaser or still results in the generation of solvent vapors. If it were not for the need to replace heat lost through the walls of the equipment and to heat condensed solvent back up to the boiling temperature, the heat input could be measured directly by measuring the volume of solvent condensate when no work was being processed through the equipment. Three separate measurements of the solvent condensate rate in the degreaser were found to have an average of 95.4 gallons per hour. Three determinations of the distillation rate from the still averaged 16.8 gallons per hour. As in the case of the degreaser, the still experiences a heat loss through the walls of the equipment but it does not require a heat input to bring the solvent to boiling temperature. The solvent pumped to the still is drawn from the boiling sump of the vapor degreaser. This information permits the following estimates of heat requirements for the degreaser and still.

Degreaser Heat Requirement

Heat for Solvent Vapor Generation

95.4 Gal./Hr. x 12 Lb./Gal. x 102 Btu*/Lb. $\cong$ 117,000 Btu/Hr.

Heat Loss Through Walls

173 Ft.^{2} x 266 Btu**Ft.^{2} $\cong$ 46,000 Btu/Hr.

Heat Required for Solvent Condensate

94.4 Gph. from Degreaser

16.8 Gph. from Still

112. 2 Gph. Total

112 Gph. x 12 Lb./Gal. x 0.22***Btu/Lb.-Hr. x
(189-149°F) ≅ 11.800 Btu/Hr.

Total ≅ 175,000 Btu/Hr.

Still Heat Requirement

Heat for Solvent Vapor Generation

16.8 Gal./Hr. x 12 Lb. x 102 Btu*/Lb. ≅ 20,600 Btu/Hr.

Heat Loss Through Walls

25 Ft.^{2} x 266 Btu**/Ft.^{2} ≅ 6,700 Btu/Hr.

Total ≅ 27,300 Btu/Hr.

The combined heat requirement for the metal cleaning equipment is approximately 202,000 Btu's per hour. By comparison, the steam required for two desorption cycles per day (113,000 Btu's each) is 226,000 Btu's per day or 28,300 Btu's per hour. The energy demand by the 3 horsepower electric motor expressed in Btu's per hour is 7,600 (3 horsepower x 2,545 Btu's per horsepower). The increase energy demanded by the carbon adsorption system represents about 18 percent of the energy required for the metal cleaning equipment alone. If more desorption cycles were needed, this energy demand would increase rapidly as a percentile of the direct metal cleaning heat requirement.

An overall flow diagram for solvent, showing the various process operations, is shown in Figure 5. This information is simplified on Figure 6 and reduced to percentiles in Figure 7. Most of this information has been developed earlier in the report. No method was available during this testing to measure the amount of solvent loss from this system during down-time versus that lost during operating hours. In order to account for this an estimate of between 15-20 percent was made and an arbitrary 18 percent was chosen. The solvent loss during non-operating hours was expressed on a per hour basis for the operating time to be consistant with the system presented. All of the solvent not accounted for by losses through the still or estimated as losses taking place during non-operative time were assumed to be lost to the atmosphere through the operator loading station. The quantity of solvent captured by the ventilation system was calculated using a 95 percent efficiency of the carbon bed to recover solvent and the known solvent (2.53 gallons per day) recovered. Again, the quantity of solvent loss in the exhaust air was calculated based on a 95 percent carbon bed efficiency. Of the total solvent feed to the system only 23% is captured and returned by the carbon adsorption system. As is generally the case, the overall recovery efficiency is largely controlled by the low percentage capture of solvent vapors by the ventilation system to the carbon adsorber beds.

Conclusions

1. The efficiency of emission control by carbon adsorption of solvents from this metal cleaning process is largely a function of the effectiveness of the vent system in capturing the air containing the solvent.

2. The control of solvent emissions by carbon adsorption can be profitable to the manufacturer.

3. The solvent recovered (neutrally stabilized trichloroethylene) is not suitably stabilized for reuse as is. If blended with sufficient fresh unused solvent or if restabilized, it can be reused.

4. The capital investment for carbon adsorption is sufficiently large that solvent metal cleaning process must be relatively large to justify such an expenditure. A recovery of 952 gallons (at $2.37/gal.) per year would be needed to support the investment for a Model 536AD carbon adsorber or equivalent on a break-even basis.

5. A solvent emission control of 25 percent was measured based on the quantity of solvent required by the metal cleaning system.

TABLE I

Industrial Building*

Shell (M&L) Cost	\$ 4.09/Ft.^{2}
Lighting and Electrical	1.75
Heating and Ventilating	1.50
Plumbing	1.70
Fire Prevention	1.10
	\$10.14 (1968 Base)
	\$17.3/Ft.^{2} (8%/Annum 1.71 Multiple in 1975)
Sub-Contract Cost (1.3)	\$22.5/Ft.^{2}
Contingency (15%)	\$25.9/Ft.^{2}

*Derived from "Modern Cost-Engineering Techniques by H. Popper. The 8% inflation rate was estimated by the author.

TABLE 2

Carbon Adsorber

Building Space

Direct	50 Ft.^{2}	
Indirect (50%)	25 Ft.^{2}	
TOTAL	75 Ft.^{2}	
Value	\$1,943	(At \$25.9/Ft.^{2} - Table I)
Cost/Annum	\$214	(At 25 Yrs. Life @ 10% Time Value of Money)

Direct Capital

Price	\$8,000	(Hoyt Manufacturing)
Shipping and Installation	\$1,200	(At 15% of Selling Price)
Total Capital	\$9,200	
Cost/Annum	\$1,200	(At 15 Yrs. Life @ 10% Time Value of Money)

TABLE 3

Model 536AD Carbon Adsorber

Operating Cost Per Annum

Capital		
Equipment	$1,210	
Building	214	
Insurance (2%)		
Equipment	184	
Building	39	
Maintenance (4%)	368	
Utilities		
Steam	125	(54.2×10^6 Btu @ $\$2.30/10^6$ Btu)
Electricity	107	(4,300 KWH @ $0.025/KWH)
Water	9	(230×10^3 Gal. @ $\$0.04/10^3$ Gal.)
Compressed Air	Nil	
Labor	Nil	
Return on Investment	0	
Total Cost/Annum	$2,256	

TABLE 4

Energy Balance on Carbon Adsorber (1 Bed)

Adsorption (Solvent Recovery)	Input	Output
Solvent Latent Heat 15.2 Lbs.	1,550 Btu's	
Air Flow @ 935 Cfm		1,550 Btu's
Desorption		
Steam 113 Lbs.	113,000 Btu's	
Heating Tank (~900 Lbs.)		13,860 Btu's
Heating Carbon (~300 Lbs.)		10,500 Btu's
Condenser Water (8 Gph)		88,640 Btu's
Vaporizing Solvent*	1,550 Btu's	1,550 Btu's
Adsorption (Drying and Cooling Bed)		
Cooling Tank	13,860 Btu's	
Cooling Carbon	10,500 Btu's	
Air Flow		24,360 Btu's

*Vaporizing solvent from bed requires heat from steam but this same heat is given up to the condenser water when the vapor is condensed to liquid solvent.

Figure 1

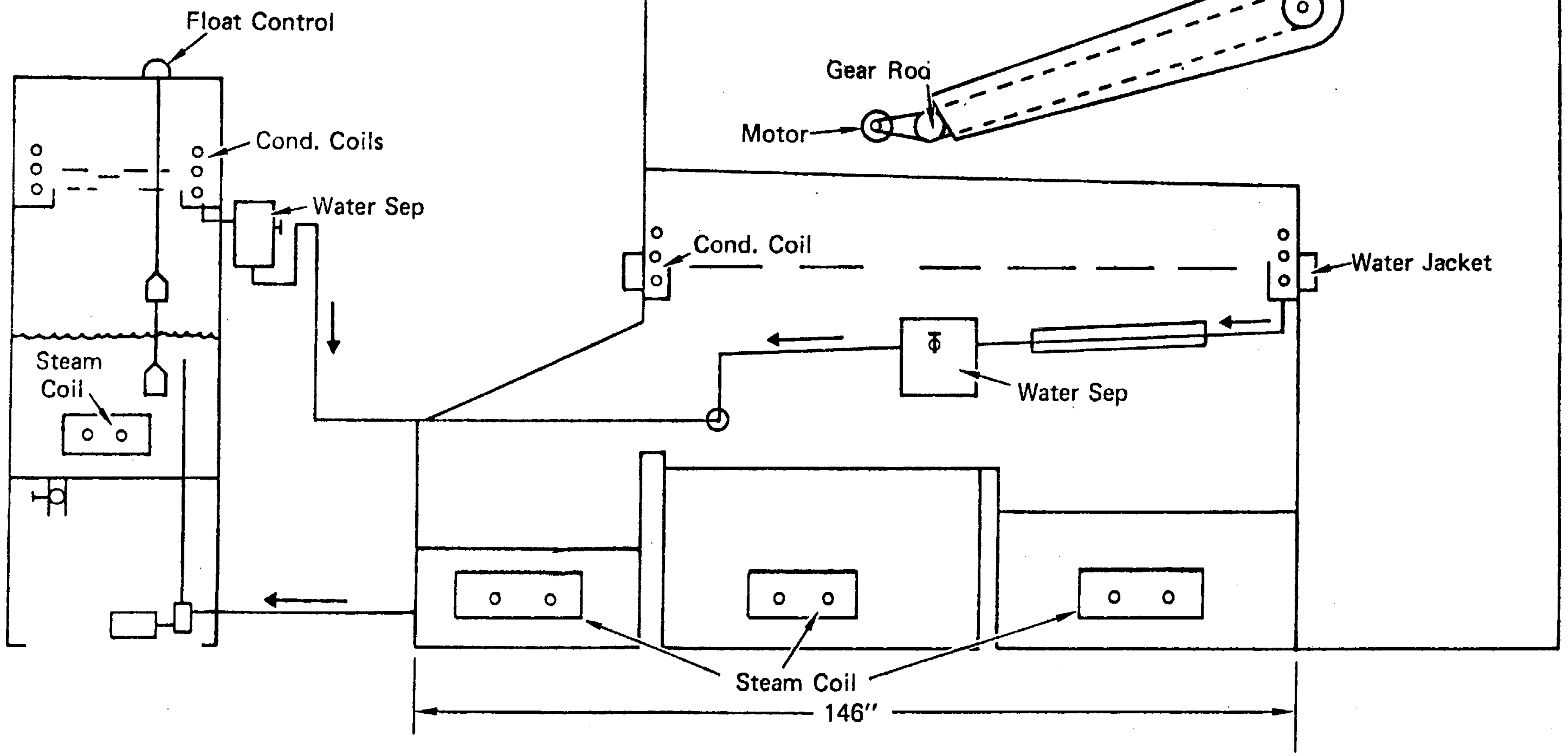

Float Control
Cond. Coils
Water Sep
Steam Coil
Gear Rod
Motor
Cond. Coil
Water Jacket
Water Sep
Steam Coil
146″

Figure 2

SOLVENT VAPOR CONCENTRATION IN CARBON ADSORPTION EXHAUST DUCT

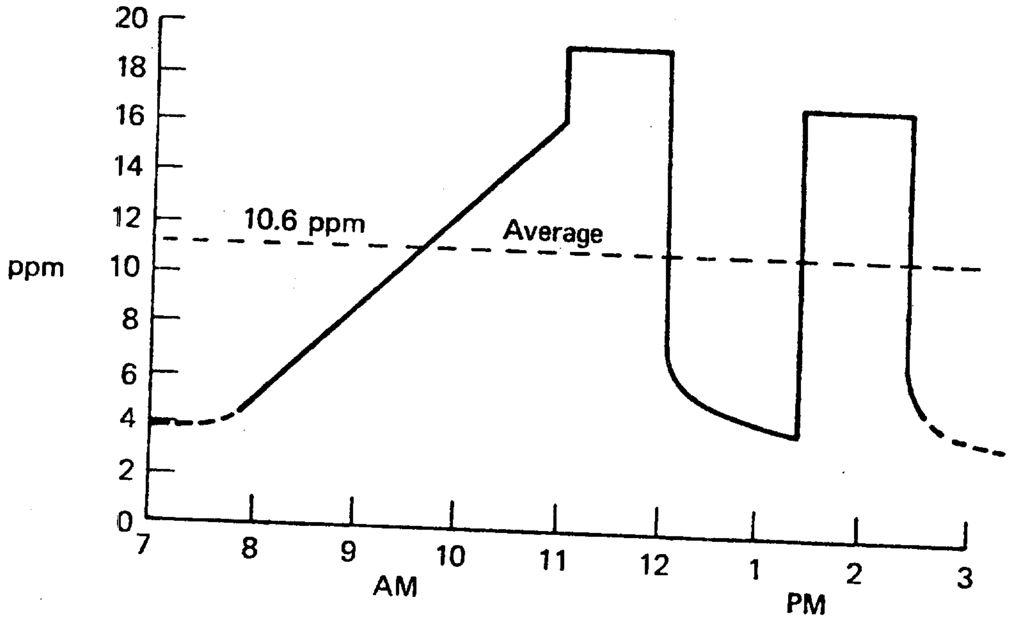
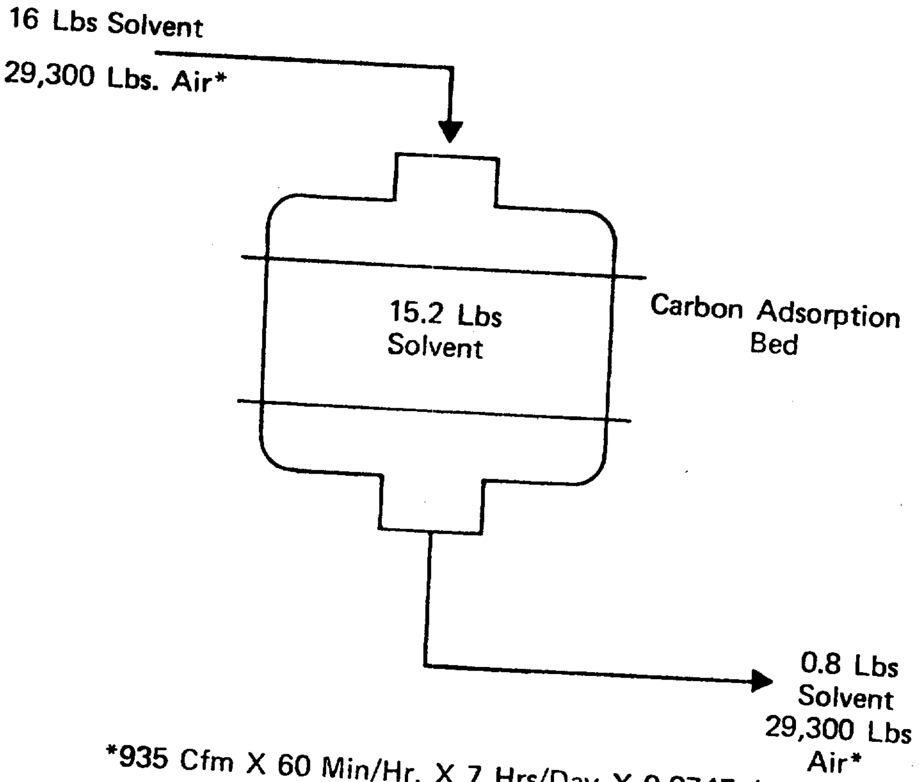
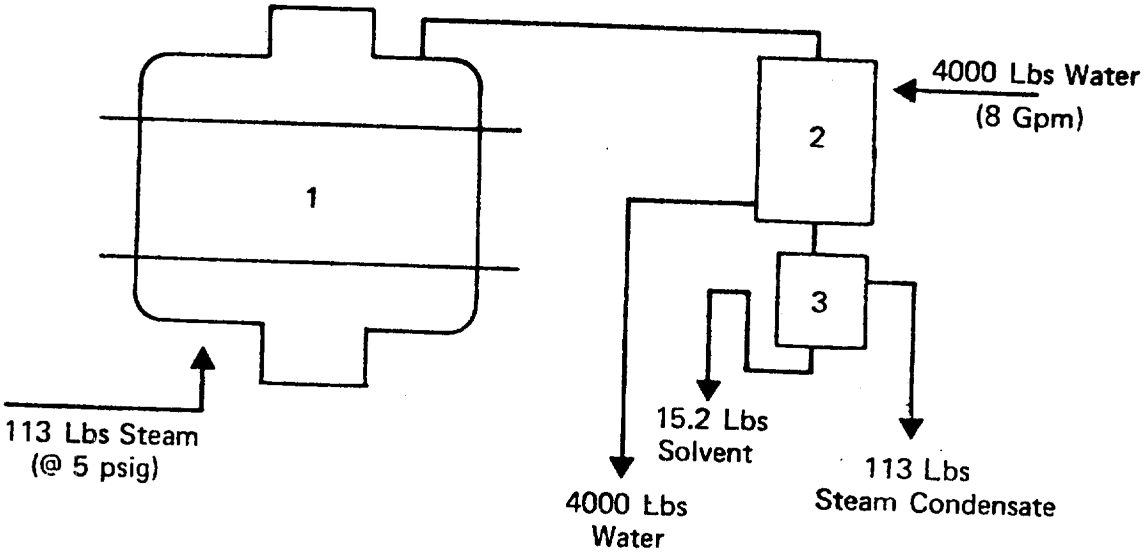


Figure 3
ADSORPTION CYCLE PHASE (Seven Hours Per Day)



*935 Cfm X 60 Min/Hr. X 7 Hrs/Day X 0.0745 Lb/Cu Ft

Figure 4
DESORPTION CYCLE PHASE (ONE HOUR PER DAY)



1	Carbon Adsorption Bed
2	Shell And Tube Condenser
3	Solvent/Water Separator

Figure 5
SOLVENT MATERIAL BALANCE FOR TOTAL SYSTEM

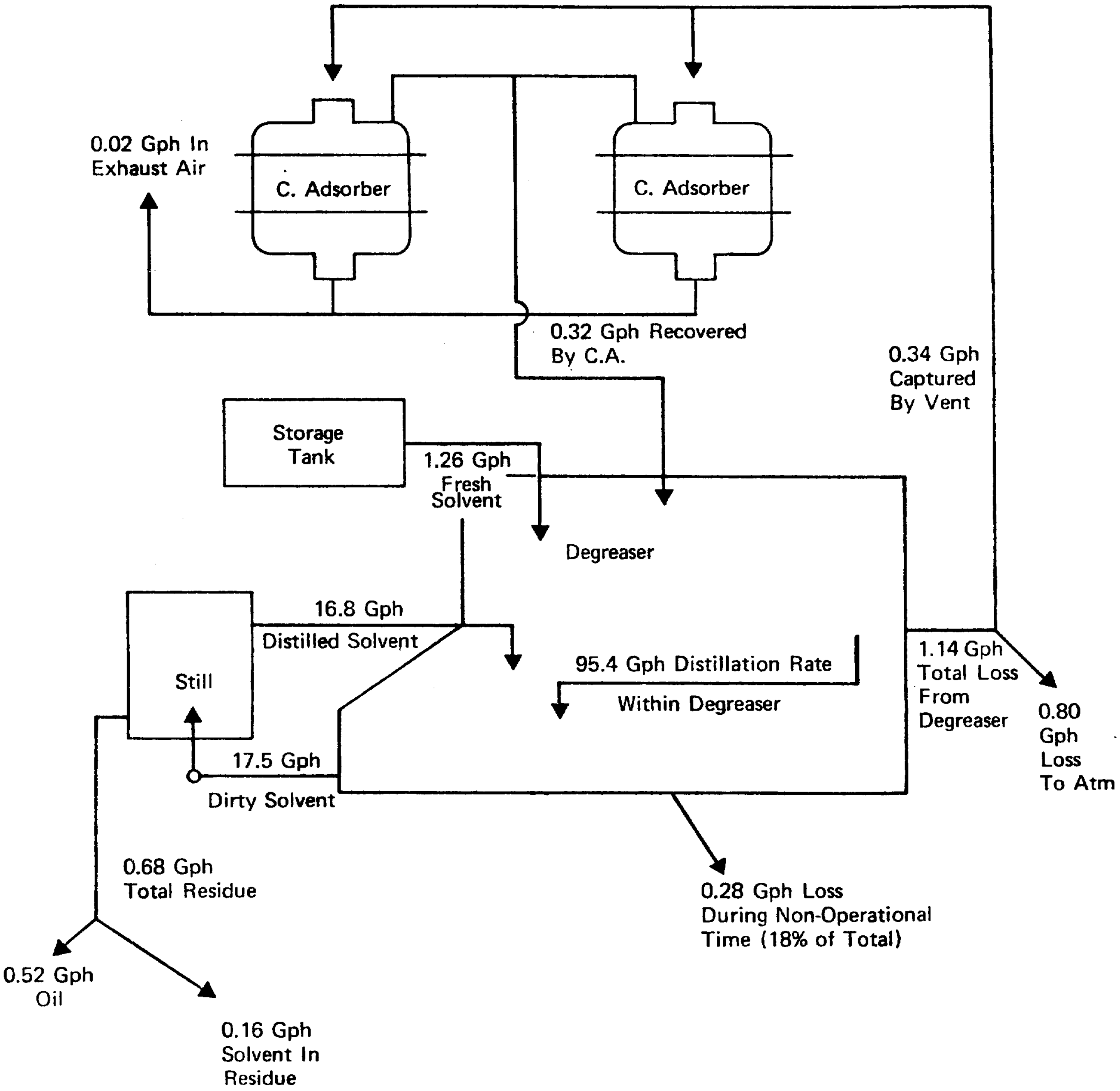
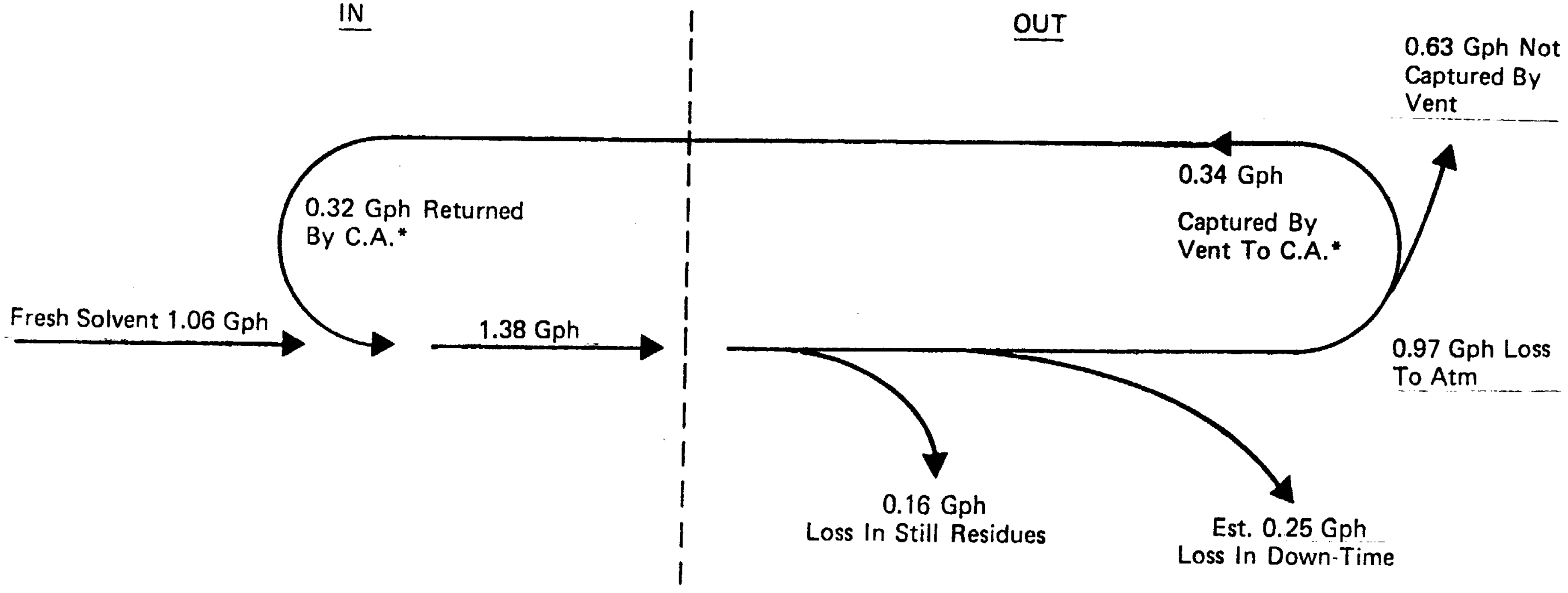


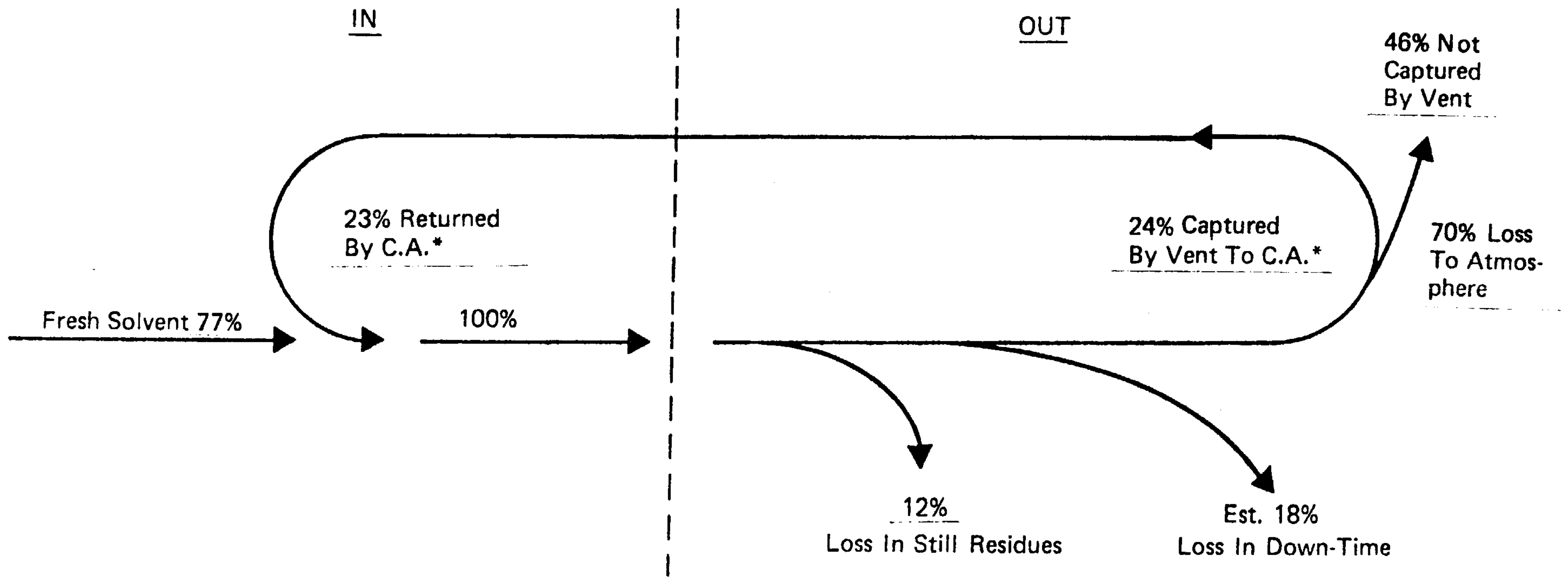
Figure 6
TOTAL METAL CLEANING AND RECOVERY SYSTEM SOLVENT BALANCE



*Carbon Adsorption

Figure 7

TOTAL METAL CLEANING AND RECOVERY SYSTEM SOLVENT BALANCE



*Carbon Adsorption

APPENDIX - C10

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS OF SOLVENT METAL CLEANING OPERATIONS

EVALUATION OF CARBON ADSORPTION RECOVERY

Vic Manufacturing Company
Minneapolis, Minnesota

PREPARED BY:

D. W. Richards
The Dow Chemical Company

PREPARED FOR:

Emission Standards and Engineering Division
Office of Air Quality Planning
U.S. Environmental Protection Agency

Summary

A test was conducted at Vic Manufacturing Company to evaluate the use of a carbon adsorption unit to control solvent emissions from an open top degreaser. Since recorded additions of new solvent to the degreaser fluctuated erratically during the time the adsorber was operated, the new solvent additions required with the adsorber "on" had to be estimated from plant purchasing records. New solvent additions with the adsorber "off" were then measured. A comparison of the new solvent additions with the adsorber "on" to the new solvent additions with it "off" show an emission control efficiency of 65 percent. It was also determined that the adsorber had significantly more capacity than was required based on the amount of solvent available for adsorption. In spite of this, the adsorber was still shown to be recovering 1.08 times the amount of solvent required to offset its annual operating cost. The stabilizers in the recovered solvent, however, were almost entirely depleted. The recovered solvent must be blended with sufficient quantities of new solvent or restabilized before it is used again.

Emission testing at this location was conducted by George W. Scheil of the Midwest Research Institute. This work is reported in EPA Project Report No. 76-DEG-1.

Objective

The purpose of this test program is to evaluate carbon adsorption as a means of controlling solvent emissions from metal cleaning operations. The information needed for this evaluation includes determining:

1. the efficiency of this device in reducing solvent emissions to the atmosphere,

2. the cost/benefit relationship of this emission control system,

3. the energy requirement of the emission control system,

4. any alternate emissions created by the emission control system.

This data base is being developed to forecast the magnitude of emission reductions which can be achieved nationally and the effect on businesses involved. This information combined with the results of other testing will be used to design emission control regulations which effectively limit air pollution and are practical for industrial application.

Introduction

Carbon adsorption has been offered to solvent metal cleaning users since 1958. The efficiency of carbon beds to capture solvent vapor from low concentrations in air has been estimated to be greater than 95 percent. The typical efficiency of carbon adsorption in reducing solvent consumption (controlling solvent emissions) is commonly reported to be in the range of 50-60 percent. Inspite of this, only a small fraction (1.5 percent) of the industrial solvent metal cleaning users surveyed*, reported that they were using carbon adsorption.

Vic Manufacturing was selected as an emission control test site primarily because their open top degreaser supported by a carbon adsorption unit was reported to be extremely efficient. Since open top degreasers account for approximately 85 percent of the degreasers now in use*, the potential applicability of the data from this test was judged to be quite high.

The carbon adsorber was installed in 1968.

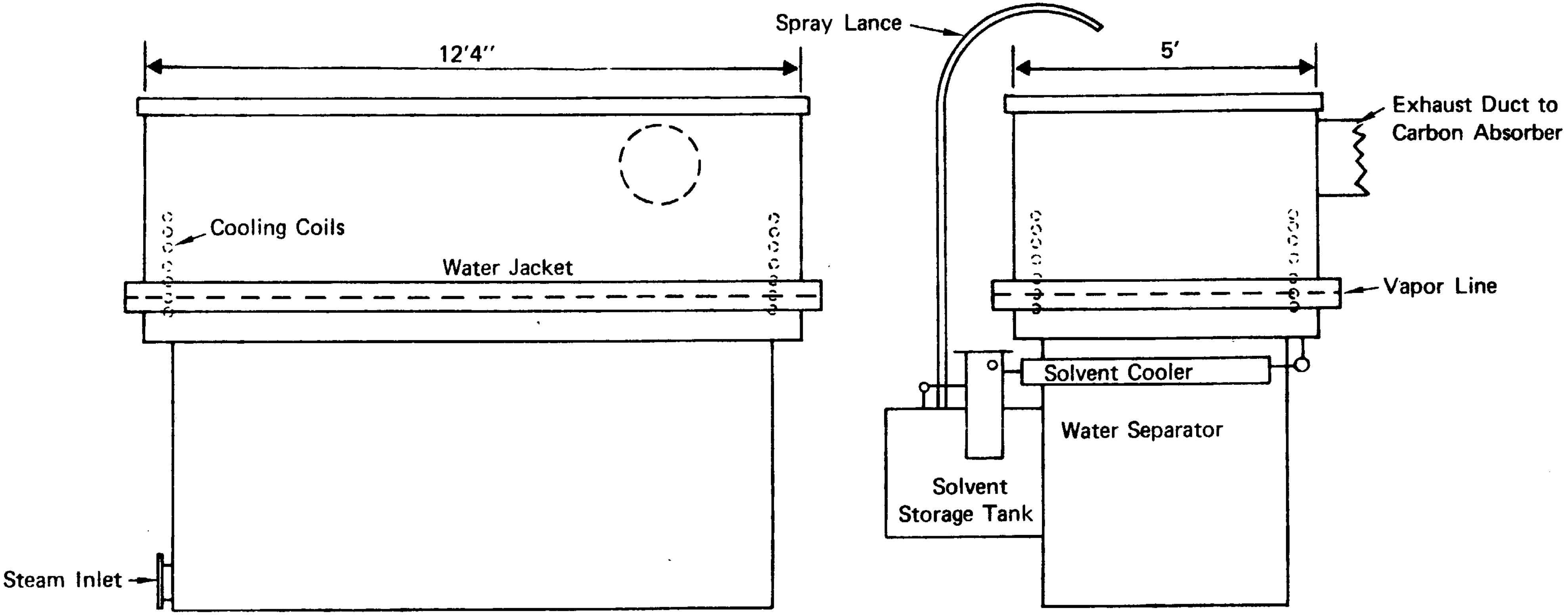
*See Appendix A

Equipment

The basic metal cleaning process employed is a G. S. Blakeslee D-95-P (12' x 4 1/2') open top degreaser and carbon adsorption unit operated with trichloroethylene. The degreaser is steam heated. See Figure 1 below.

The carbon adsorption unit is a model 572 AD. Solvent-laden air is drawn into the unit through an 18" duct. After passing through the carbon bed, it is discharged outside through another 18" duct.

OPEN TOP DEGREASER
(Vic Manufacturing Company)



Experiment Design

This test was designed to obtain several weeks of data with the carbon adsorber operating and one to two weeks of data with it off. The work processed and solvent consumption were to be recorded daily and compared. The actual test period ran from May 19, 1975 to August 15, 1975. Due to problems encountered during the test (which will be reviewed in the Data Discussion section), solvent consumption data obtained while the adsorber was operating could not be used. Also, solvent consumption data could be obtained for only four days with the adsorber off.

Data Discussion

Several weeks of data were obtained with the carbon adsorber operating. The recorded additions of new solvent to the degreaser, however, fluctuated widely. At different times during this period, average new solvent additions varied from ~0.5 gal./day to ~11 gals./day. Also, there were periods of several consecutive days when no new solvent was added. Discussions with operating personnel produced several possible explanations for these discrepancies, but the accuracy of the data was still in doubt. Therefore, the emission control efficiency of the carbon adsorber could not be determined directly. An approximation can be made, however, by using plant purchasing records to determine the average solvent consumption of the degreaser while the carbon adsorber is operating. This number can then be compared to the measured degreaser solvent consumption while the adsorber was turned off. Purchasing records were available from January 1 through August 22, 1975 and indicated that ~38 gals./week of new solvent were added to the degreaser while the adsorber was operating. During the four days the adsorber was off, 1047 lbs. (86.5 gals.) of new solvent was added. This is equivalent to: 86.5 gals. x 5/4 = 108 gals./week. On this basis, the adsorber emission control efficiency is:

$$\frac{108 - 38}{108} \times 100\% = 65\%$$

As mentioned, this number is only an approximation, but it does indicate that the adsorber is very efficient.

The average bed efficiency also indicates the effectiveness of the adsorber as an emission control device. This value is determined by measuring and comparing the time-weighted average solvent vapor concentrations in the inlet and outlet ducts of the adsorber. On August 20, 1975, these measurements were made from 10:00 a.m. to 1:30 p.m. using a Gas Tech Halide meter. The average solvent concentration in the inlet duct while the degreaser was idling was 21 ppm and 29 ppm while work was being processed. Since the amount of idling time was about equal to the amount of work time for this particular day, the time-weighted average solvent concentration in the inlet duct was 25 ppm.

The solvent concentration in the outlet duct of the adsorber was extremely low at all times and averaged only 1.6 ppm. Therefore, the average bed efficiency of the carbon adsorber for this day was:

$$\frac{25 - 1.6}{25} \times 100\% = 93.6\%$$

Obviously, the solvent concentration in the inlet has a great effect on the efficiency of this unit. If the solvent concentration increases significantly above the 25 ppm level that was observed, the efficiency should approach 100%. It is reasonable to expect this since the adsorber is larger than is required for this system and is capable of handling much greater inlet concentrations. This was verified during the test period since the average amount of solvent collected during the two daily desorption cycles (of ~1 hr. each) was only 167.25 lbs. (13.81 gals.). Vic Manufacturing estimates that the model 572 AD adsorber is capable of recovering at least 450 lbs. (37.16 gals.) of trichloroethylene during the same time (two desorption cycles). Separate material balances for the adsorption and desorption phases of the carbon adsorption unit are represented in Figures 2 and 3 respectively. The numbers shown in each figure are average values obtained during testing except for water values which are manufacturer's estimates. The carbon adsorption unit is operated eight hours per day. Each bed is desorbed once per day for one hour. This means that each bed must adsorb for seven hours per day.

The inlet velocity to the carbon adsorber was also measured on August 22, 1975 and found to be 3133 ft./min. when both beds were adsorbing. Since the duct diameter is 18", this is equivalent to 5545 cfm. The back pressure created when one bed was desorbing dropped the velocity to an average of 2225 ft./min. (3938 cfm).

The utilities required for the carbon adsorption system are steam, water, compressed air, and electricity. Steam condensate was collected and weighted for four separate desorption cycles. The weights were: 669.5 lbs., 600.0 lbs., 606.0 lbs., and 586.0 lbs. (average = 615 lbs.). Using 1000 Btu's per pound of steam, 615,000 Btu's are required per desorption cycle. Thus, 615,000 Btu's/desorption x 2 desorptions/day x 250 days/yr. = 308 x 10^6 Btu's/yr.

The carbon adsorption unit's condenser is estimated to use 21 G.P.M. by Vic Manufacturing. Since the water only flows during the desorption cycles, the total water requirement is 21 gals./min. x 60 min./hr. x 2 hr./day x 250 days/yr. = 630,000 gals. The carbon adsorber fan is rated at 20 horsepower. This is equivalent to: 20 hp. x .746 Kwh/hp. x 8 hrs./day x 250 days/yr. = 29,840 Kwh/yr. An energy balance for one bed of the carbon adsorber (adsorption and desorption cycles) is summarized in Table 1. Radiation losses through the shell of the adsorber bed are not included in order to simplify the overall balance.

The replacement cost for the 572 AD carbon adsorption unit studied during this test is estimated to be $22,085 by Vic Manufacturing. This information and Table 2 were used to develop Tables 3 and 4. The assumptions involved in the development of these Tables include a zero return on investment; the assignment of 50 percent extra floor space for aisleways, etc.; the omission of costs for other facilities which are required, but already exist; and the omission of other minor costs such as heating, lighting, janitorial services, etc. Based on these assumptions, the total operating cost per year for the model 572 AD carbon adsorber is calculated to be $6903 (see Table 4). If a price of $2.15/gal.* is used for trichloroethylene, then 3211 gallons must be recovered by the carbon adsorber per year to offset the total annual operating cost. Based on the measured 13.81 gals./day recovery rate, a total of 3453 gallons will be recovered yearly, or 1.08 times more than is required to offset the adsorber's annual operating cost.

Due to the relatively small quantity of work processed by the degreaser, the 572AD adsorber has quite a bit more capacity than is required for this system. Substitution of a smaller 554AD adsorber, for example, would still allow the same amount of work to be processed and would also increase the profitability of the system. The total yearly operating cost for a 554AD (calculated in the same manner as the 572AD) would be ~$4410. Using the same price of $2.15/gal. for trichloroethylene, only 2051 gallons must be recovered to offset the annual operating cost. Comparing this to the

3453 gallon yearly recovery developed earlier, it is found that it would be possible to recover 1.68 times more than the amount of solvent required to offset the annual operating cost instead of only 1.08 times more.

The affect of carbon adsorption on solvent stabilizer levels was somewhat difficult to ascertain at this test site. Several impurities (such as methyl chloroform, perchloroethylene, and Fluorocarbon 113) were identified in the adsorber samples. Most of these impurities are introduced into the degreaser on the various parts which are cleaned; however, some are also present in the new solvent that is added to the degreaser. Analyses showed that the stabilizers were severely depleted by the carbon adsorber. This means that any solvent recovered by the adsorber must be restabilized (by blending with adequate amounts of new solvent or adding a stabilizer concentrate) before it is used again.

The solvent condensation rate in the degreaser was found to be 198.2 gals./hr. while no work was being processed. Using this information and the knowledge that some heat is lost via radiation through the degreaser **walls**, the degreaser heat requirement can be calculated.

Degreaser Heat Requirement

Heat for Solvent Vapor Generation

198.2 Gals./Hr. x 12.11 Lbs./Gal. x (189°-70°F) x .22* Btu/Lb./°F

= 62,387 Btu/Hr.

198.2 Gals./Hr. x 12.11 Lbs./Gal. x 101.6** Btu/Lb.

= 243,860 Btu/Hr.

Heat Loss Through Walls

208 Ft.^2 x 266*** Btu/Hr./Ft.^2 = 55,328 Btu/Hr.

361,575 Btu/Hr.

The energy required for two desorption cycles per day is 615,000 Btu's x 2 = 1.23 x 10^6 Btu's/Day. The energy required by the carbon adsorber's 20 horsepower fan (expressed in equivalent units) is 20 Hp. x 2545 Btu/Hp./Hr. x 8 Hrs./Day = 407,200 Btu's/Day. Therefore, the total energy demand of the carbon adsorption unit is 1.64 x 10^6 Btu's/Day. Since the degreaser represents the total heat input for the metal cleaning system, the total heat requirement for the system is 361,575 Btu's/Hr. x 8 Hrs./Day = 2.89 x 10^6 Btu's/Day. Thus, the energy required for the carbon adsorber represents an increase of 57 percent of that required for the degreaser alone.

*Specific Heat For Trichloroethylene
**Heat of Vaporization For Trichloroethylene
***Radiation Heat Loss For Trichloroethylene

} Obtained From "Modern Vapor Degreasing", The Dow Chemical Co.

Conclusions

1. Carbon adsorption can provide significant emission control for an open top degreaser. In this case, the reduction in emissions was estimated to be ~65 percent by comparing solvent usage with the adsorber turned off to solvent usage with the adsorber operating (calculated from plant purchasing records).

2. Controlling emissions with carbon adsorption can be profitable. At the solvent recovery rate measured during this test, 1.08 times the amount of solvent required to offset the model 572AD adsorber's annual operating cost can be recovered. This can be theoretically increased to 1.68 times by substituting a model 554AD adsorber for the model 572AD.

3. Trichloroethylene recovered by the adsorber is not suitable for reuse unless it is either restabilized with a concentrate, or blended with a sufficient amount of new solvent.

TABLE 1

Energy Balance on Carbon Adsorber (1 Bed)

Adsorption (Solvent Recovery)	Input	Output
Solvent Latent Heat (83.63 Lbs.)	8500 BTU'S	
Air Flow At 5545 CFM		8500 BTU'S
Desorption		
Steam (615 Lbs.)	615,000 BTU'S	
Heating Tank (2800 Lbs.)		43,740 BTU'S
Heating Carbon (1500 Lbs.)		42,600 BTU'S
Condenser Water (21 GPH)		528,660 BTU'S
Vaporizing Solvent*	8500 BTU'S	8500 BTU'S
Adsorption (Drying and Cooling Bed)		
Cooling Tank	43,740 BTU'S	
Cooling Carbon	42,600 BTU'S	
Air Flow At 5545 CFM		86,340 BTU'S

*Vaporizing solvent from the bed requires heat from steam, but this same heat is given up to the condenser water when the vapor is condensed to liquid solvent.

TABLE 2

Industrial Building*

Shell (M&L) Cost	\$ 4.09/Ft.^{2}
Lighting and Electrical	1.75
Heating and Ventilating	1.50
Plumbing	1.70
Fire Prevention	1.10
	\$10.14/Ft.^{2} (1968 Base)
	\$17.3/Ft.^{2} (8%/Annum 1.71 Multiple in 1975)
Sub-Contract Cost (1.3)	\$22.5/Ft.^{2}
Contingency (15%)	\$25.9/Ft.^{2}

*Derived from "Modern Cost-Engineering Techniques by H. Popper (pg. 103). The 8% inflation rate was estimated by the author.

TABLE 3

Carbon Adsorber

Building Space		
Direct	111 Ft.^{2}	
Indirect (50%)	55.5 Ft.^{2}	
TOTAL	166.5 Ft.^{2}	
Value	$4312	(At $25.9/Ft.^{2} - Table 2)
Cost/Annum	$ 475	(At 25 Years Depreciation Rate)*
Direct Capital		
Price	$22,085	(Vic Manufacturing)
Shipping and Installation	$ 3,313	(At 15% of Selling Price)
TOTAL CAPITAL	$25,398	
Cost/Annum	$ 3,339	(At 15 Years Depreciation Rate)*

*10% Interest Rate On Investment

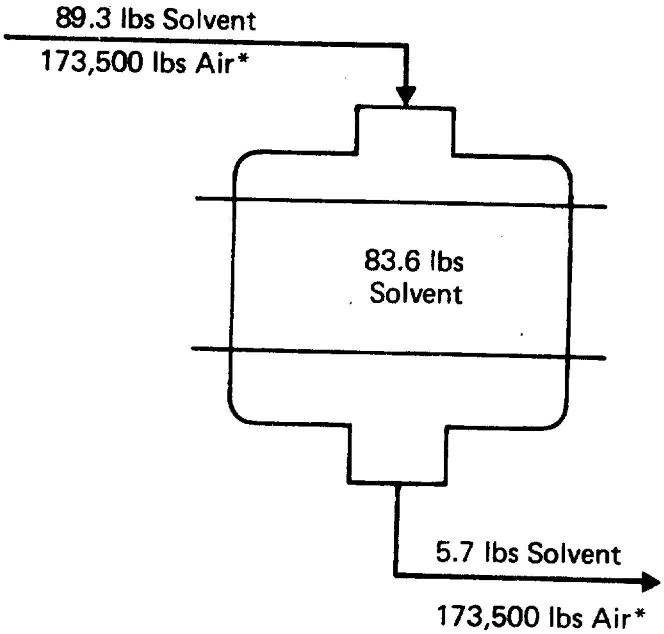
TABLE 4

Model 572 AD Carbon Adsorber

Operating Cost Per Annum

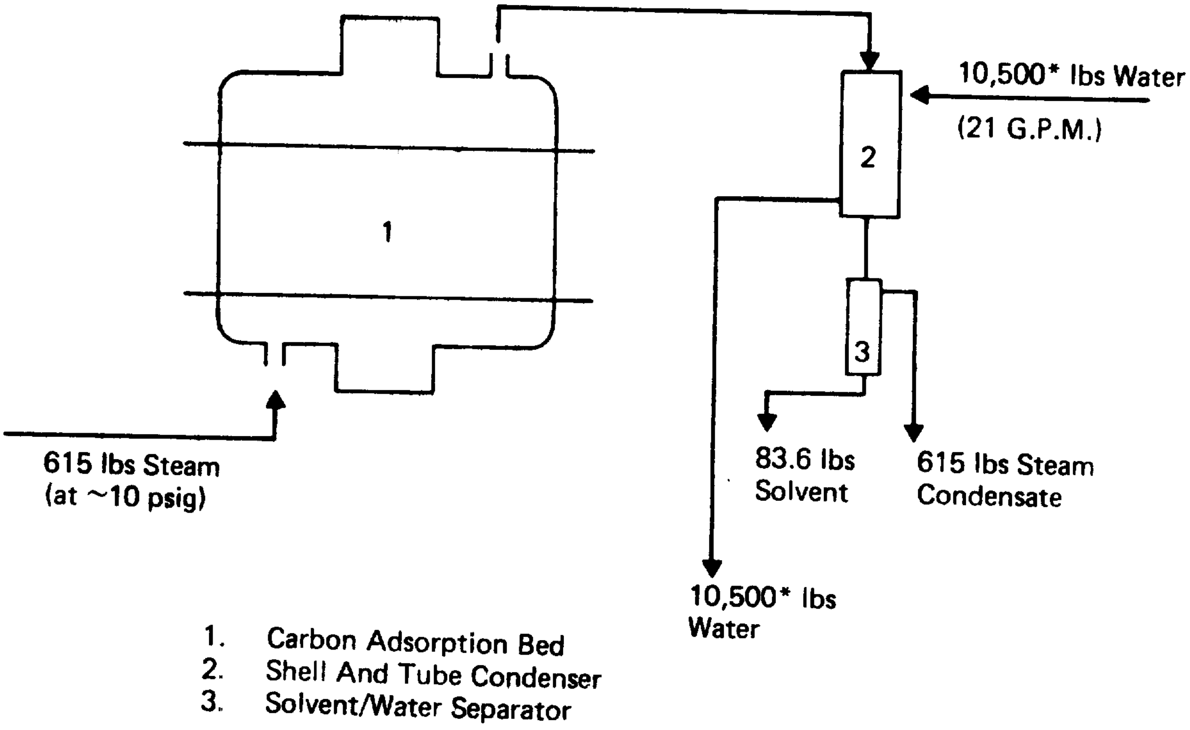
Capital		
Equipment	$3339	
Building	$ 475	
Insurance (2%)		
Equipment	$ 508	
Building	$ 86	
Maintenance (4%)	$1016	
Utilities		
Steam	$ 708	(308 M̄ BTU's At $2.30/M̄ BTU's)
Electricity	$ 746	(29,840 KWH At $0.025/KWH
Water	$ 25	(630 M Gals. At $0.04/M Gals.)
Compressed Air	Nil	
Labor	Nil	
Return on Investment	0	
Total Cost/Annum	$6903	

Figure 2
ADSORPTION CYCLE PHASE
(7 HOURS PER DAY)



*5545 CPM x 60 min/hr x 7 hrs/day x 0.0745 lbs/cu ft

Figure 3
DESORPTION CYCLE PHASE
(ONE HOUR PER DAY)



GPM x 60 min/hr x 8.33 lbs/gal x 1 hr/day

cc: K. S. Surprenant, 2020
CRC/PF 2192008
Centi l Files

December 15, 1975

Mr. J. W. Barber
Research Director
Vic Manufacturing Company
1620 Central Ave. N.E.
Minneapolis, Minnesota 55413

REPORT PREPARED FOR ENVIRONMENTAL PROTECTION AGENCY COVERING VIC CARBON ADSORBER STUDY

Dear Joe:

This is to confirm our telephone conversation of this morning during which we discussed my report and your December 31, 1975 letter commenting on the report. As I mentioned, I feel that many of the comments you made concerning Vic's costs, etc. are quite valid. By using your actual cost experience, I would have arrived at an annual operating cost for the Model 572AD adsorber of $5052 (as you stated) instead of the $6903 that appears in my report. The problem that this creates, however, is one of comparing "apples to oranges". The assumptions that were used to develop the adsorber operating cost for the Vic report were also used for all of our other studies. This was done deliberate so that the EPA can compare the operating costs of the various emission control devices tested on an equal basis. It is important to note that the cost information we have developed applies to the use of adsorbers and other control devices in new facilities. Using the actual costs for floor space, maintenance, insurance, etc. for an existing facility, therefore, would not be consistent with this approach.

Because of this methodology, therefore, I am going to submit the Vic report to the FPA without changing the development of the adsorber annual operating cost. I will also submit a copy of your December 3, 1975 letter for their inspection.

Thanks again for all of your assistance during this study.

Best regards,

Dave

David W. Richards
Inorganic Chemicals Department
Chlorinated Solvents Section
Phone: (517) 636-6640

cc: Mr. John C. Bollinger
Industrial Studies Branch
U. S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

kw

VIC MANUFACTURING COMPANY
1620 CENTRAL AVENUE N.E., MINNEAPOLIS, MINNESOTA 55413/PHONE (612) 781-6601

AIR POLLUTION CONTROL SYSTEMS

December 3, 1975

Dave Richards
Inorganic Chemicals Department
2020 Dow Center
Midland, Michigan 48640

RE: EPA Report

Dear Dave:

We have received your draft copy and find it well-organized and quite complete. We do, however, have some comments regarding the treatment of various costs.

1. We believe that the area costs assigned to the equipment to be excessive. The costs detailed in the report refer to new building costs. Since the report is concerned with the VIC Manufacturing Company unit, we believe that the cost should be related to the present market value of $6.00/S.F.

   We suggest that in new buildings the cost/S.F. may dictate that an adsorption system be mounted on an elevated platform or on the roof and thus reduce or eliminate the S.F. charges against the equipment.

   While we support the application of all reasonable costs to provide a true costing picture, we do not agree that new building costs should be so applied.

2. If we apply $6.00/S.F. to the VIC installation and use the 166.5 S.F. space allocation, then the value would be $999.00 and cost/annum would be $110.00. Then in Table 4 the following changes are suggested to reflect the actual costs based on our rates and charges.

Capitol:	Equipment	3340	
	Building	110	
Insurance (our FIA Premium is 0.7% of dollar value)			
	Equipment	178	
	Building	7	
Maintenance		200	(Based on our experience with the installed system).
Utilities (our gas costs 1.16/Million BTU's)			
	Using your 308$\overline{M}$ input to the adsorber we would need 385$\overline{M}$ at the boiler (w/80% efficiency) and that would cost	446	
	Electricity	746	
	Water	25	
		5052/Annum	

3. If we apply this annual cost to the valve of the solvent reclaimed, their 2350 gallons would need to be recovered and the measured 3453 gallons reclaimed become 1.47 times the breakeven quantity.

   Applying these same values to the 554 assumptions would probably raise the factor to 2X or more.

4. We also note that there are available two tax credits that may be applied to Air Pollution Control equipment. One is the Investment Tax Credit of 10% on new equipment applied to the Federal Income Tax, and the second is the Air Pollution Control Equipment Tax Credit that is applicable to many State Income Tax assessments.

We have appreciated this opportunity to review your excellent report and hope that you will find our comments helpful.

Best regards,

J.W. Barber
Research Director

JB:mek
cc/IVictor
CGorman

APPENDIX - C11

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS OF SOLVENT METAL CLEANING OPERATIONS

EVALUATION OF CARBON ADSORPTION RECOVERY

Western Electric Company
Hawthorne Station
Chicago, Illinois

PREPARED BY:

D. W. Richards
The Dow Chemical Company

PREPARED FOR:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

The use of a carbon adsorption unit to control solvent emissions from an enclosed "cold cleaning" system (circuit board cleaner) was studied at Western Electric Company. It was determined that the carbon adsorber recovered 60% of the new solvent added to the system and that the solvent recovery rate was 2.5 times that required to offset the annual adsorber operating costs. The stabilizers in the recovered solvent, however, were totally depleted. Before the recovered solvent can be reused it must be mixed with adequate quantities of new solvent or restabilized.

Objective

The purpose of this test program is to evaluate carbon adsorption as a means of controlling solvent emissions from metal cleaning operations. The information needed for this evaluation includes determining:

1. the efficiency of this device in reducing solvent emissions to the atmosphere,
2. the cost/benefit relationship of this emission control system,
3. the energy requirement of the emission control system,
4. any alternate emissions created by the emission control system.

This data base is being developed to forecast the magnitude of emission reductions which can be achieved nationally and the effect on businesses involved. This information combined with the results of other testing will be used to design emission control regulations which effectively limit air pollution and are practical for industrial application.

Introduction

Carbon adsorption has been offered to solvent metal cleaning users since 1958. The efficiency of carbon beds to capture solvent vapor from low concentrations in air has been estimated to be greater than 95 percent. The typical efficiency of carbon adsorption in reducing solvent consumption (controlling solvent emissions) is commonly reported to be in the range of 50-60 percent. Inspite of this, only a small fraction (1.5 percent) of the industrial solvent metal cleaning users surveyed*, reported that they were using carbon adsorption.

Western Electric was considered as an emission control test site on the recommendation of Baron-Blakeslee (equipment manufacturer). Western Electric has extensive experience with solvent metal cleaning and their personnel have excellent reputations in this area. Also, the carbon adsorption system was reported to reduce solvent consumption from 76 pounds per hour to 1.5 pounds per hour (98% recovery). This operation was selected for evaluation because it was felt that it could demonstrate the highest level of efficiency possible through the use of carbon adsorption in support of an enclosed cold cleaning system.

Just prior to the first week of the study it was noticed that the steam-solvent vapor valve on bed "A" was cracked. Fine

*See Appendix A

efforts by both Vic Manufacturing and Western Electric resulted in the replacement of this valve before the first week of the study was completed.

The carbon adsorption unit was installed in July, 1973. Since these units are reported to have operating lives of 15 years or more, this unit must be considered to be relatively new.

Equipment

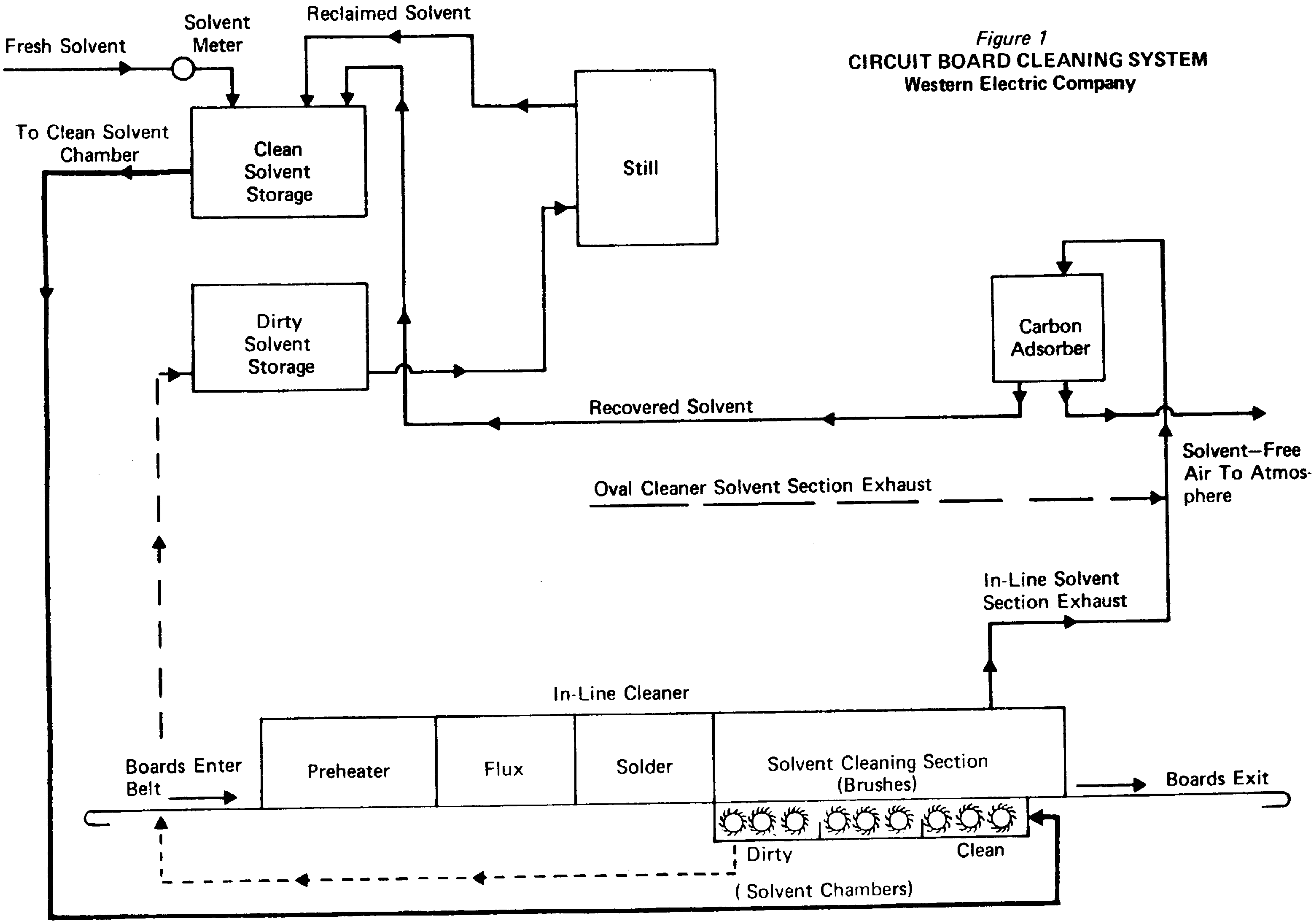
The metal cleaning process employed consists of two circuit board flux cleaners, a still, and a carbon adsorption unit operated with trichloroethylene. One of the flux cleaners is oval in shape while the other one is a straight line (in-line) unit. Both are room temperature cleaners and both are exhausted to the carbon adsorption unit. During the test period, however, approximately 75-90% of the work was processed through the in-line cleaner. Therefore, the oval cleaner is not shown below in Figure 1 (which is a schematic of the entire system).

The carbon adsorption unit is a Model 536AD. Solvent-laden air from both flux cleaners is drawn into the carbon adsorber through a 12 inch duct. After passing through the carbon bed, the essentially solvent-free air is then discharged outside through a 10 inch duct.

The clean solvent storage tank receives solvent from three different sources: 1) reclaimed solvent from the still, 2) recovered solvent from the carbon adsorber, and 3) new solvent pumped from 55 gallon drums. All of the new solvent is pumped through a meter.

The dirty solvent storage tank receives contaminated solvent from both flux cleaners. When enough dirty solvent is collected, it is then transferred automatically to the still to be reclaimed.

Figure 1
CIRCUIT BOARD CLEANING SYSTEM
Western Electric Company



Experiment Design

The original plan for testing was designed to obtain two weeks of data with the carbon adsorption system operating and one to two weeks with it off. The test was scheduled to begin on May 22, 1975, but due to unforeseeable circumstances did not begin until June 16, 1975. As a result of the early delays, the testing ran into Western Electric's scheduled shutdown (July 13 - July 26) and had to be extended into August. The total workload and solvent consumption for both cleaners were to be recorded weekly and compared. Since the cleaners' solvent chambers are kept at a constant level by a float control system, the difference in solvent inventory could be measured by manually filling the still and measuring the solvent levels in the clean and dirty solvent storage tanks. These quantities could then be added to or subtracted from the total new solvent added to the system (which is measured by the solvent meter). Small quantities of solvent are withdrawn from the clean storage tank by the operator for miscellaneous cleaning operations. These withdrawals were recorded during the entire period of the test and were subtracted from solvent consumption figures.

Data Discussion

Two weeks of data were obtained with the carbon adsorption unit "on" and two weeks with it "off". An additional two weeks of data had to be discarded. During one of these weeks solvent was lost through a crack in the housing of the steam-vapor valve on bed "A" of the carbon adsorber. During the other week a leak developed in the carbon adsorber condenser coil and the system was contaminated with water.

Solvent emission rates were determined by differences in solvent inventory within the clean and dirty storage tanks as discussed previously. The clean and dirty storage tanks are each 2' high x 4' wide x 5' long. Therefore, an inch of solvent in either tank is equivalent to 12.5 gallons. Each tank is equipped with a sight glass, and the solvent depth can be accurately measured to within 1/8 inch. Thus, the error in measuring solvent in either tank is ± 1.5 gallons.

The average solvent consumption for the two weeks the carbon adsorber was operating was 10.4 gal./day. The average work processed during this two week period was 417.7 $ft.^2$/day. The daily solvent consumption per square foot of work processed is therefore, 10.4/417.7 or 0.025 $gal./ft^2$. Similarly, the daily solvent consumption per square foot of work processed for the two weeks the carbon adsorber was off is 23.8/375.8 or 0.063 gal./ft^2. On this basis, the emission control efficiency is determined to be:

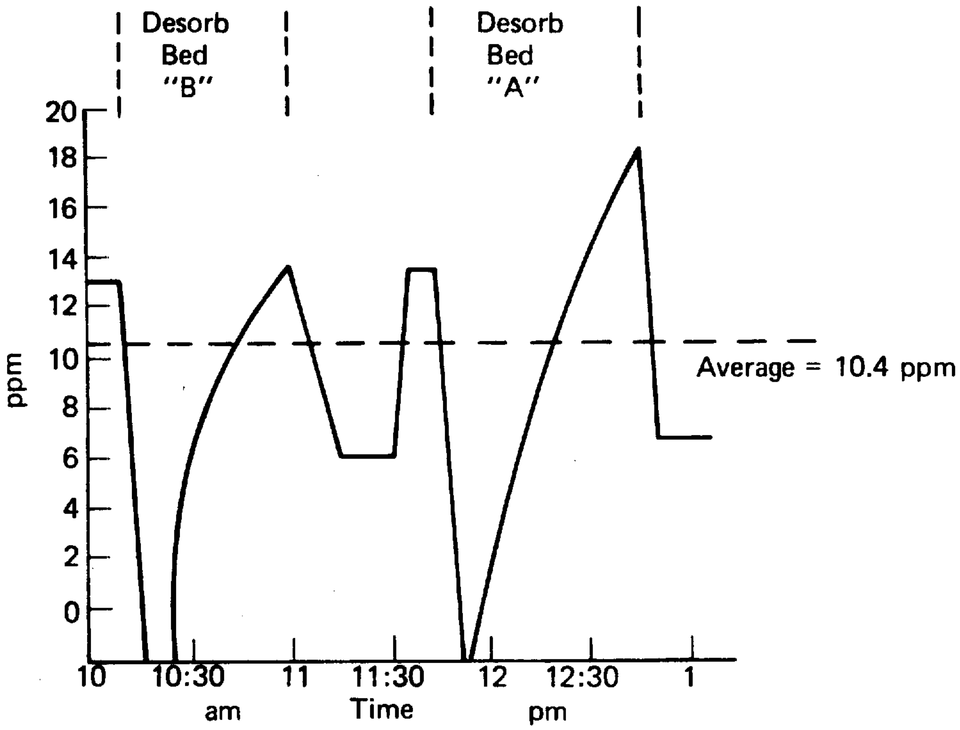
$$\frac{0.063 - 0.025}{0.063} \times 100\% = 60.3\%$$

The solvent removed from the system as still residue was insignificant during the test period and was not considered in this calculation.

The solvent vapor concentrations in the outlet duct of the carbon adsorber were measured from 10:00 A.M. to 1:00 P.M. on August 21, 1975 and are plotted below in Figure 2. The average vapor concentration in the outlet duct was 10.4 ppm.

Figure 2

SOLVENT VAPOR CONCENTRATION IN CARBON ADSORPTION OUTLET DUCT (August 21, 1975)



As shown in Figure 2, the portion of the curve between 10:10-11:10 corresponds to the desorption of bed "B" and the portion of the curve between 11:45-12:45 corresponds to the desorption of bed "A". At the beginning of both desorption cycles the vapor concentration decreases rapidly indicating that the bed being desorbed was saturated and losing solvent to the atmosphere. As the desorption cycles continue, the vapor concentration gradually increases since only one bed is adsorbing. At the end of the desorption cycle, the concentration decreases to an equilibrium level since both beds are adsorbing again.

Vapor concentration measurements were also made in the inlet duct to the carbon adsorber. The readings ranged from 132 to 177 ppm with an average of 165 ppm. On this particular day, only the in-line circuit board cleaner was being operated. This was typical throughout the test period. No significant differences in vapor concentrations were observed when work was being processed compared to the time the machine was idling. This was anticipated since the brushes in the solvent cleaning section continue to rotate whether or not work is being processed, and the entire section is covered by metal "pans" to reduce splashing and solvent loss to the exhaust duct. The only additional loss of solvent that occurs while work is being processed is that which is retained on the circuit boards and carried outside of the machine. The average inlet vapor concentration (165 ppm), therefore, was simply determined by averaging the readings and was not time-weighted for hours of working operation and idling. The average bed efficiency for this particular day was:

$$\frac{165 - 10.4}{165} \times 100\% = 93.7\%$$

Samples of solvent were obtained during three different desorption cycles and analyzed for stabilizer content.

Although the depletion of stabilizers in a room temperature cleaning operation is not as critical as it is in a vapor degreasing operation, carbon adsorbers are used in conjunction with both types of systems. Therefore, the affect of carbon adsorption on solvent stabilizer levels was studied during this test. No stabilizers were found in any of the samples. This indicates that solvent recovered from the carbon adsorber is not suitable for reuse unless it is mixed with a greater quantity of fresh solvent, or replenished with a stabilizer concentrate.

The utilities required for the carbon adsorption system are steam, water, compressed air, and electricity. Steam condensate was collected and weighed for three separate one hour desorption cycles. The weights were: 395 lbs., 406 lbs., and 402 lbs. (average = 401 lbs.). While the weights were consistent, they were extremely high for this carbon adsorber (manufacturer's estimate for the unit is 180 lbs./hr.). Near the end of the test period a leak was detected in the condenser coil of the carbon adsorber. Since there is no way to determine how much additional water this added to the steam condensate being measured, the manufacturer's estimate of 180 lbs./hr. will be used for calculations. Using 1000 Btu's per pound of steam, 180,000 Btu's are required per desorption cycle. Thus, 180,000 Btu's per desorption x 12 desorptions per day x 240 days per year = 518 x 10^6 Btu's/year.

The carbon adsorption unit's condenser is estimated to use eight gallons per minute by the manufacturer. Since the water only flows during the desorption cycles, the total water requirement per year is 1,380,000 gallons. The carbon adsorber fan motor is rated at 3 horsepower. This is equivalent to: 3 hp. x .746 KWH/hp. hour x 24 hrs./day x 240 days/yr. = 12,890 KWH/yr.

The replacement cost for the 536AD carbon adsorber unit studied during this test was estimated to be $9320 by Vic Manufacturing.

This information and Table 2 were used to develop Tables 3 and 4. The assumption involved in the development of these Tables include a zero return on investment; the assignment of 50 percent extra floor space for aisleways, etc.; the omission of costs for other facilities which are required, but already exist; and the omission of other minor costs such as heating, lighting, janitorial services, etc. Based on these assumptions, the total operating cost per year for the model 536AD carbon adsorber is calculated to be $3874 (see Table 4). If a price of $2.15/gal. ($0.1775/lb.)* is used for trichloroethylene, then 1802 gallons must be recovered by the carbon adsorber per year to offset the total annual operating cost. Based on the measured 17.3 gals./day recovery rate, a total of 4152 gallons will be recovered yearly. Thus, a net savings of about $5050/yr. will be realized at this recovery rate.

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*Chemical Marketing Reporter, July 7, 1975, Schnell Publishing Company

The inlet velocity through the carbon adsorption unit was found to be 1458 ft./min. when both beds were adsorbing. Since the inlet duct diameter is 12 inches, this is equivalent to an air volume of 1145 cfm. When one bed was desorbing, the back pressure reduced the velocity to an average of 804 ft./min. and the air volume to 631 cfm. The average solvent recovery rate was determined to be 209 lbs./day or 17.28 gals./day. This data was obtained by collecting solvent from all twelve of the desorption cycles during a 24 hour period. Therefore, the average solvent collected per desorption cycle was 17.4 lbs. (1.4 gals.).

Separate material balances for the adsorption and desorption phases of the carbon adsorption unit are represented in Figures 3 and 4 respectively. The numbers shown in each figure are average values obtained during testing except for steam and water values which are manufacturer's estimates. The carbon adsorption unit is operated 24 hours per day. Each bed is desorbed six times per day for one hour each time. This means that each bed must adsorb for 18 hours per day. An energy balance for one bed of the carbon adsorber (adsorption and desorption cycles) is summarized in Table No. 1. Radiation losses through the shell of the adsorber bed are not included in order to simplify the overall balance.

The still distillation rate was checked several times during the test period and averaged 95.6 gallons per hour. Dirty solvent from the circuit board cleaner is collected in the dirty solvent storage tank and is automatically pumped to the still when enough is collected for distillation. Therefore, heat must be added to bring the dirty solvent from room temperature to the boiling point as well as additional heat to maintain the distillation. Also, some of the heat can be lost by radiation through the still walls. Using this information, the still heat requirements can be calculated.

Still Heat Requirement

Heat for Solvent Vapor Generation

95.6 Gal./Hr. x 12.11 Lbs./Gal. x (189° - 70°F) x .22* Btu/Lb./°F
= 30,300 Btu/Hr.

95.6 Gal./Hr. x 12.11 Lbs./Gal. x 101.6** Btu/Lb.

= 117,600 Btu/Hr.

Heat Loss Through Walls

62 Ft.^{2} x 266*** Btu/Hr./Ft.^{2} = 16,500 Btu/Hr.

164,400 Btu/Hr.

*Specific Heat For Trichloroethylene
**Heat of Vaporization For Trichloroethylene
***Radiation Heat Loss For Trichloroethylene

Obtained From "Modern Vapor Degreasing", The Dow Chemical Co.

The energy required for 12 desorption cycles per day is 180,000 Btu's x 12 = 2.16 x 10^6 Btu's/Day. The total energy required by the carbon adsorber 3 horsepower fan (expressed in equivalent units) is 3 hp. x 2545 Btu/hp./Hr. x 24 Hrs./Day = 183,240 Btu's/Day. Thus, the total energy demand of the carbon adsorption unit is 2.3 x 10^6 Btu's/Day. Since the still represents the total heat input for the metal cleaning system and only operates ~2 Hrs./Day, the total heat requirement for the cleaning system is 164,400 Btu's/Hr. x 2 Hrs./Day = 328,800 Btu's/Day. Therefore, the energy required for the carbon adsorber represents an increase of .7 times that required for the metal cleaning operation alone. This is not nearly as significant as it appears at first glance, since (as discussed earlier) the carbon adsorber recovers more than enough solvent to offset its annual operating cost.

A simplified overall flow diagram which traces the flow of solvent through the cleaning system is presented in Figure 5. The same information is also presented in Figure 6 in percentile form. As discussed earlier, solvent losses in the still bottoms are omitted. During the test period it was estimated that less than 0.2 Gpd of solvent added to the system was lost in the still bottoms. Therefore, all of the losses of liquid solvent were assumed to occur as carryout on the circuit boards. The losses of solvent vapor to the atmosphere were determined using a 93.7% carbon adsorber bed efficiency and the measured amount of solvent recovered (17.28 Gpd).

Conclusions

1. For an enclosed cold cleaning system, carbon adsorption can provide significant emission control. In this case a 60% reduction in emissions was measured based on the amount of new solvent added to the system.

2. Controlling emissions with carbon adsorption can be profitable (at the solvent recovery rate measured during this test, 2.5 times the amount of solvent required to offset the adsorber yearly operating cost can be recovered).

3. Trichloroethylene recovered by the the adsorber is not suitable for reuse unless it is: a) blended with an adequate amount of new solvent or b) restabilized.

TABLE 1

Energy Balance on Carbon Adsorber (1 Bed)

	Input	Output
Adsorption (Solvent Recovery)		
Solvent Latent Heat (17.4 Lbs.)	1770 Btu's	
Air Flow At 1145 cfm		1770 Btu's
Desorption		
Steam (180 Lbs.)	180,000 Btu's	
Heating Tank (~900 Lbs.)		14,050 Btu's
Heating Carbon (~350 Lbs.)		9940 Btu's
Condenser Water (8 gpm)		156,010 Btu's
Vaporizing Solvent*	1770 Btu's	1770 Btu's
Adsorption (Drying and Cooling Bed)		
Cooling Tank	14,050 Btu's	
Cooling Carbon	9940 Btu's	
Air Flow At 1145 cfm		23,990 Btu's

*Vaporizing solvent from the bed requires heat from steam, but this same heat is given up to the condenser water when the vapor is condensed to liquid solvent.

TABLE 2

Industrial Building*

Shell (M&L) Cost	\$ 4.09/Ft.^{2}
Lighting and Electrical	1.75
Heating and Ventilating	1.50
Plumbing	1.70
Fire Prevention	1.10
	\$10.14/Ft.^{2} (1968 Base)
	\$17.3/Ft.^{2} (8%/Annum 1.71 Multiple in 1975)
Sub-Contract Cost (1.3)	\$22.5/Ft.^{2}
Contingency (15%)	\$25.9/Ft.^{2}

*Derived from "Modern Cost-Engineering Techniques by H. Popper (pg. 103). The 8% inflation rate was estimated by the author.

TABLE 3

Carbon Adsorber

Building Space	
Direct	50 $Ft.^2$
Indirect (50%)	25 $Ft.^2$
Total	75 $Ft.^2$
Value	\$1940 (At \$25.9/$Ft.^2$ - Table I)
Cost/Annum	\$ 214 (At 25 Years Depreciation Rate)*
Direct Capital	
Price	\$9320 (Vic Manufacturing)
Shipping and Installation	\$1400 (At 15% of Selling Price)
Total Capital	\$10,720
Cost/Annum	\$1410 (At 15 Yrs. Depreciation Rate)*

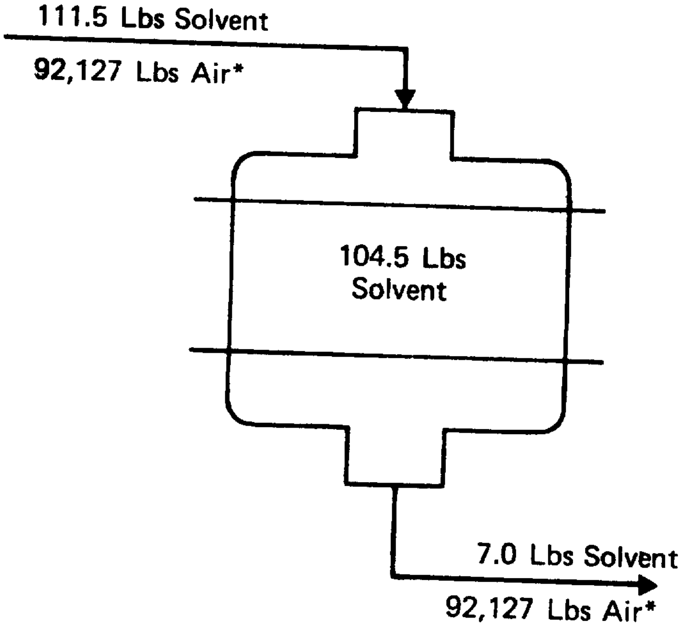
*10% Interest Rate on Investment

TABLE 4

MODEL 536AD CARBON ADSORBER
OPERATING COST PER ANNUM

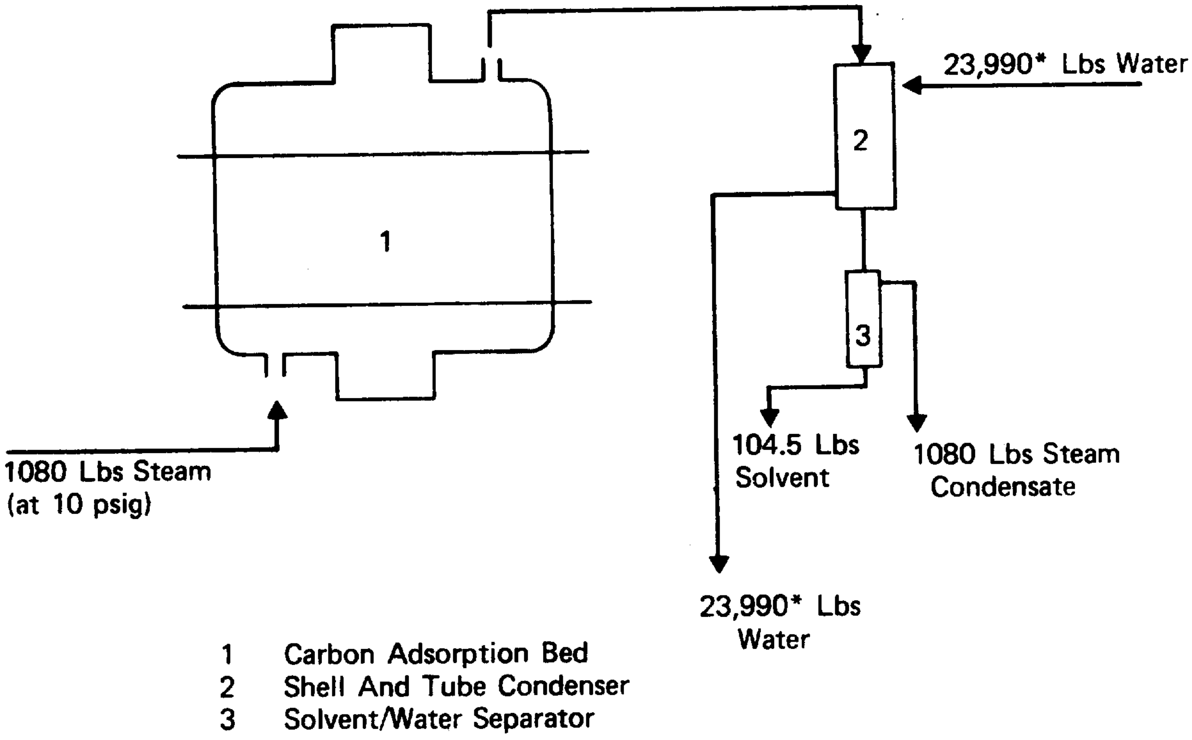
Capital		
Equipment	$1410	
Building	$ 214	
Insurance (2%)		
Equipment	$ 214	
Building	$ 39	
Maintenance (4%)	$ 429	
Utilities		
Steam	$1191	(518 M̄ Btu's At $2.30/M̄ Btu)
Electricity	$ 322	(12,890 KWH At $0.025/KWH
Water	$ 55	(1380 M Gal. At $0.04/M Gal.)
Compressed Air	Nil	
Labor	Nil	
Return on Investment	0	
Total Cost/Annum	$3874	

Figure 3
ADSORPTION CYCLE PHASE (18 Hours Per Day)



***1145 CFM X 60 min/hr X 18 hrs/day X 0.0745 lbs/cubic foot**

Figure 4
DESORPTION CYCLE PHASE (6 Hrs. Per Day)



*8 GPM X 60 min/hr X 8.33 lbs/gal X 6 hrs/day

Figure 5

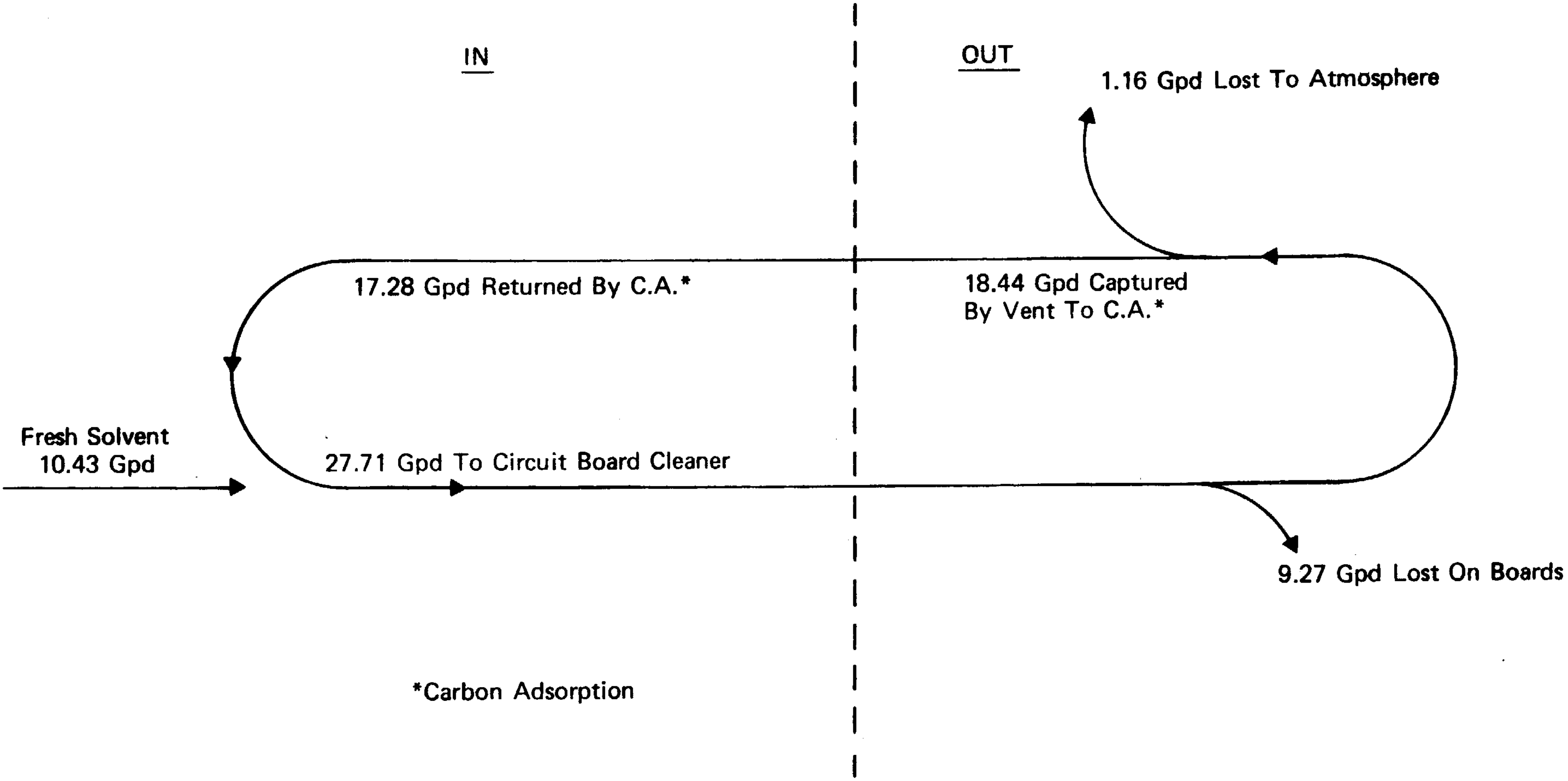
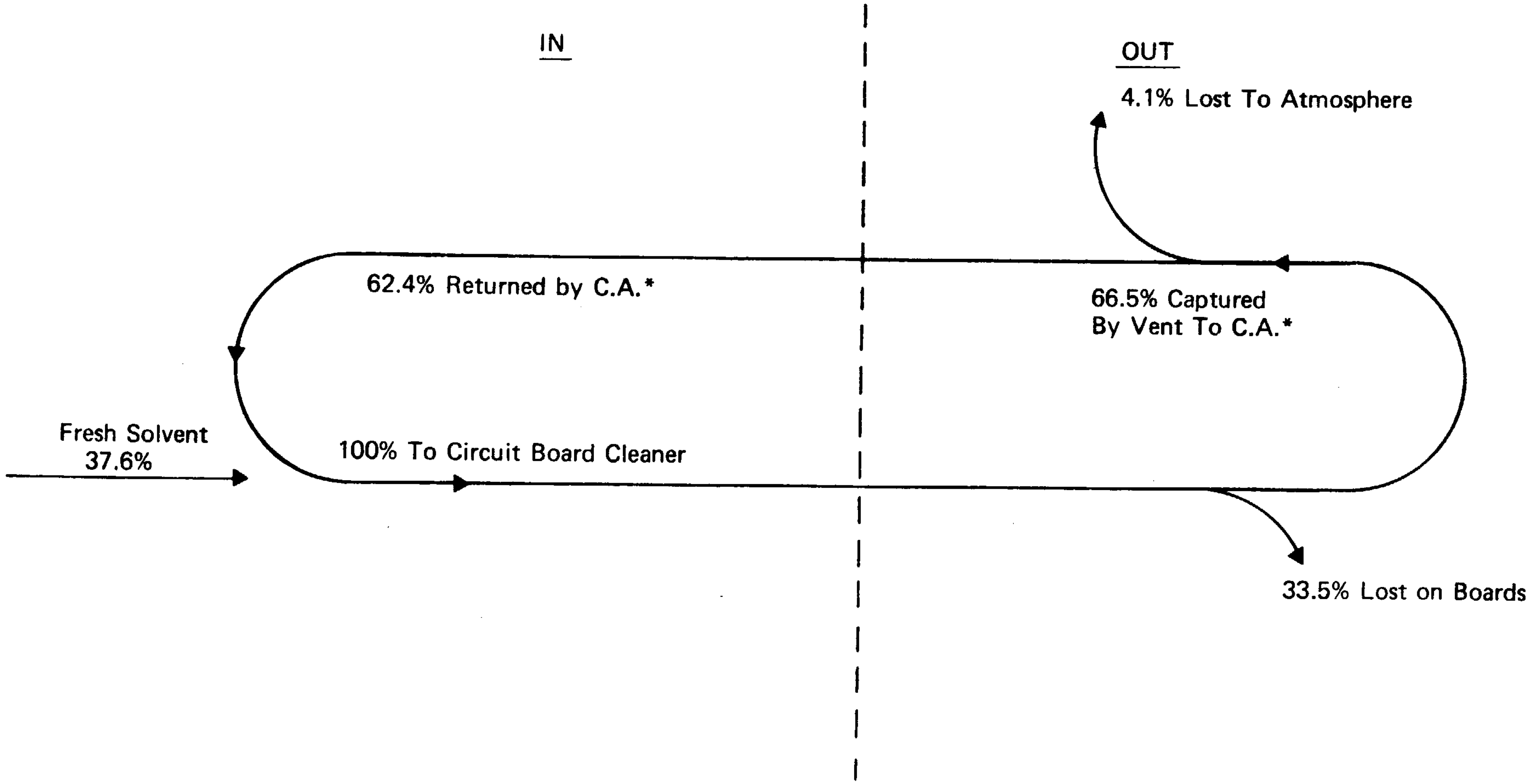

IN
OUT
1.16 Gpd Lost To Atmosphere
17.28 Gpd Returned By C.A.*
18.44 Gpd Captured By Vent To C.A.*
Fresh Solvent 10.43 Gpd
27.71 Gpd To Circuit Board Cleaner
9.27 Gpd Lost On Boards
*Carbon Adsorption

Figure 6



*Carbon Adsorption

APPENDIX - C12

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS OF SOLVENT METAL CLEANING OPERATIONS

STUDY OF THE EMISSION CONTROL EFFECTIVENESS OF INCREASED FREEBOARD ON OPEN TOP VAPOR DEGREASERS

PREPARED BY:

K. S. Surprenant
The Dow Chemical Company

PREPARED FOR:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

Summary

A series of laboratory tests were made to determine 1) the emission control effect of increasing freeboard on open top degreasers, and 2) the relative solvent loss rates with and without lip exhaust ventilation. In quiet air, increasing the freeboard to width ratio from 0.50 to 0.75 causes a 46% reduced emission rate. A 55% emission reduction (in turbulent air) was achieved by increasing the freeboard to equal to the width of the degreaser. Solvent losses to the environment are likely to be doubled by the addition of lip exhaust ventilation.

Objective

This study was conducted to determine how effectively solvent emissions from vapor degreasing operations could be controlled by increased freeboard height. Portions of this testing were also designed to determine the effect of lip exhaust ventilation on the solvent emission rate, the idling degreaser loss rate while heated and the shut down degreaser loss rate.

The aim of this work is to provide basic background data to be used in conjunction with emission control evaluations of actual metal cleaning operations. This background information will be used to develop New Source Performance Standards which effectively limit air pollution and are practical for industrial application.

Introduction

Industrial vapor degreasing operations do not lend themselves to the determination of some basic information. Specifically, working vapor degreasers can not be modified conveniently to examine solvent loss rates vs. freeboard height without work interruption. Again, it is of value to know the solvent emission rates of vapor degreasers during shut down time and during idling (heated but not cleaning metal parts). In each of these cases, the work capacity of the degreaser would have to be lost for periods of time to determine this data. According to the survey reported in Appendix A, 85% of the vapor degreasers are open top degreasers. Both experience and in-plant emission control testing has shown that open top degreasers are used for actual metal cleaning only a relatively small percent (25%) of the time. Typically, open top degreasers are left heated and open during the balance of the work shifts. This idling operation represents a significant portion of the total solvent emissions. Yet, it is difficult to define this emission rate at in-plant locations. For this reason, the evaluations made in this report were conducted under laboratory conditions.

Compliance with the Occupational Safety and Health Act requirements and concern for employee safety are causing manufacturing concerns to consider ventilation of vapor degreasing operations. Although ventilation systems have been shown to provide limited reductions in exposures of workers to solvent vapors, ventilation has also been known to cause higher solvent emission rates. This also was studied on a laboratory basis to avoid variations caused by workloads.

Equipment

The equipment used for this testing was a Model 2D500 E size D20. This equipment was supplied as a standard model of the Detrex Chemical Industries Co. and was operated with 1,1,1-trichloroethane throughout all the tests described. A copy of the Detrex specification sheet for this degreaser is attached. The test degreaser was heated electrically with 15 kilowatt heaters. This degreaser was located in a room with exceptionally quiet air having dimensions of approximately 20' x 35'.

Experiment Design

The valve from the immersion chamber of the degreaser to the boiling chamber was left open throughout testing so that this chamber was maintained in an empty state. The water separator and solvent spray reservoir were maintained at an overflow level. Thus, all solvent losses from the equipment were reflected in a change in the level of the boiling chamber. The solvent level in the degreaser was adjusted to a specific elevation before and after each test with the heat turned "off". The dimensions of the boiling chamber are roughly 24 1/4" x 22". Every inch of elevation is equal to approximately 2.31 gallons. Since the level could be measured within ± 1/8 inch, the error in estimating solvent consumption was controlled to within ± 0.298 gallons or 1.09 liters. The solvent loss was determined in liters of solvent necessary to return the solvent level to the starting level.

Data Discussion

Solvent Emission Rate Vs. Freeboard Height (Quiet Air)

The roll top cover provided with this degreaser was removed for this testing, exposing the full open top area of the degreaser (2' x 4'). A sleeve was constructed to fit just inside the walls of the degreaser and designed so that the existing freeboard height of 12 inches could be increased by 6 inches or by another 12 inches. The original freeboard height to degreaser width ratio was 0.50, that is, 12 inches of freeboard over 24 inches of degreaser width. The increased freeboard heights increased this ratio to 0.75 and 1.0. The degreaser was operated for one week each at the 12 inch freeboard and 24 inch freeboard and two weeks at the 18 inch freeboard height. During each seven day test, the degreaser was held at a boil continuously and no work was processed. The results of this testing are summarized in Table 1.

TABLE 1

Emission Rate Versus Freeboard Height (Quiet Air)

Freeboard	Solvent Used	Lbs./Ft.^{2}-Hr.	Emission Control
12"	44 L./7 Days	0.095	
18"	23 L./7 Days 24 L./7 Days	0.051	46% ± 3%
24"	25 L./7 Days	0.054	43% ± 3%

Due to the workloads processed in industrial degreasers and the lack of totally quiet air, emission rates from operating vapor degreasers can not be expected to achieve these levels. However, most of the emissions which occur while degreasers are idling can be prevented through the use of a cover. Thus, a degreasing operation with an automatic cover could be expected to conserve almost one-tenth of a pound per square foot-hour of operation if it has a normal 0.50 freeboard design. Increasing the freeboard height to a ratio of 0.75 reduces the solvent emissions in a quiet area by approximately 46% without a cover. Again, in a quiet area, no further decrease in solvent emission rate occurs by increasing the freeboard height to equal to the width of the degreaser.

Solvent Emission Rate Vs. Freeboard Height (With Air Movement)

To determine solvent emission rates on a more realistic basis, a fan was located 16 feet from the degreaser and turned on low. The air movement created by this fan was barely noticeable when standing at the end of the degreaser closest to the fan. Velometer measurements of the air velocity near the lip of the vapor degreaser were found to fluctuate from a low of about 30 feet per minute to slightly less than 100 feet per minute. Most of the air velocity measurements were found in the range of 50 to 100 feet per minute or less than one mile per hour. With the normal freeboard on the equipment, the vapor zone was not seriously disturbed but wave motions having an amplitude of one to two inches were observed. When the six inch freeboard addition was added to provide 18 inches of freeboard the wave motion of the vapors was almost eliminated. The vapor zone was essentially quiet with the 24 inch freeboard.

Due to the larger volumes of solvent being consumed measureable losses would occur in 24 hours and data was taken on that basis. This data is summarized in Table 2. It should be noted that the solvent emission rate varied considerably with the original 12 inch freeboard as would be expected of a system not being entirely controlled. However, the solvent loss rate experienced under these conditions (0.373 pounds per square foot hour) is not as great as the commonly used industry value of 0.50 pound per square foot hour for an operating industrial degreaser. The emission rate was reduced by approximately 27% with an 18 inch or 0.75 freeboard ratio. The consistency of the results also demonstrate the better control experienced by the system. The 24 inch or 1.0 ratio freeboard reduced solvent emissions by 55%. The last two results reported for the 12 inch freeboard were measured after the testing with the 18 inch and the 24 inch freeboard testing, whereas the first three measurements with the 12 inch freeboard were made before. This was done to assure that uncontrolled variables had not influenced the test results. The average of the three results before the increased freeboard testing was 25.7 liters per 24 hours and that after the increased freeboard testing was 23.0 liters per 24 hours.

TABLE 2

Emission Rate Versus Freeboard Height
(With Air Movement)

Freeboard	Solvent Used	Lbs./Ft.^{2}-Hr.	Emission Control
12"	20 L./24 Hrs. 30 L./24 Hrs. 27 L./24 Hrs. 21 L./24 Hrs. 25 L./24 Hrs.	0.373	
18"	18 L./24 Hrs. 18 L./24 Hrs.	0.273	27%
24"	10 L./24 Hrs. 12 L./24 Hrs.	0.167	55%

As noted before, the quiet air testing indicated no improvement in solvent emission rate when the freeboard was increased from 18 inches to 24 inches. The function of the freeboard is to prevent the vapor zone from being disturbed by air movements in the room. When the freeboard height becomes high enough to isolate the vapor zone from the air movements in the room, further increases in the freeboard would not be expected to produce any improved control of the vapor zone. However, when the air in the room is more greatly disturbed as in the testing summarized in Table 2, the isolation of the vapor zone would not occur until higher freeboard ratios were employed. This is well illustrated by the results obtained.

Using this information the graph on Figure 1 can be prepared. The emission rates for the 12, 18 and 24 inch freeboard heights construct a straight line. The dotted line extension of this data is based on the quiet room testing which indicates this system can be expected to loose approximately 0.05 pound per square foot hour when it is totally isolated from the room air movements.

Lip Exhaust Ventilation Effect

In this testing, the degreaser was operated with the roll top cover in-place but open, seven days per week and 24 hours per day. One week of operation without the lip exhaust ventilation consumed 33 1/4 liters. When the lip exhaust was operated for similar period, 71 liters were consumed. Allowing for the slightly decreased open top surface area of the degreaser due to the roll cover fixture, the solvent loss rate without ventilation was 0.083 pounds per square foot hour and with ventilation was 0.177 pounds per square foot hour. Solvent emissions increased slightly over 210% with the lip exhaust vent operating. The ventilation rate is described on the equipment specification sheet.

Shut Down Loss Rate

The solvent loss rate with a standard 12 inch freeboard was found to be 0.023 pounds per square foot hour. Solvent emission rates with the 18 inch freeboard of 0.020 and 0.023 during two 64 hour periods. Therefore, the added freeboard has little effect on the solvent loss rate experienced during shut down time when the degreaser is left open. This data also suggests that it is much less important to cover a degreaser when it is completely shut down than when it is idling.

Conclusions

1. Solvent emission rates from open top degreasers can be reduced by at least 27% by increasing the freeboard to degreaser width ratio to 0.75.

2. Emission reductions of between 43% and 55% can be expected when degreaser freeboard ratios are increased to equal to the width of the degreaser.

3. Automatic covers for open top vapor degreasers which would maintain the degreaser covered when idling or during shut down can be expected to reduce emissions to the atmosphere between 0.1 and 0.4 pounds per square foot per hour of idling vapor degreaser operation.

4. Lip exhaust ventilation can increase solvent emission rates by over 200%.

DETREX MODEL 2D500S, G and E, TWO-DIP, IMMERSION SPRAY, SIZE D20 DEGREASERS

Applications and Advantages

1. The Model 2D500 Detrex degreasers provide maximum flexibility of cleaning cycles. Work can be cleaned:

(a) By immersion in boiling solvent, then in a cool solvent rinse that is continuously replenished with clean solvent distillate.

(b) By immersion in solvent vapors, then in a cool solvent rinse and finally in pure solvent vapors. Optionally a spray pump and lance can be purchased to provide a distillate spray prior to the final vapor rinse and drying cycle.

2. The Model 2D500 degreasers rapidly remove greases, oils, wax and other soils from all kinds of parts.

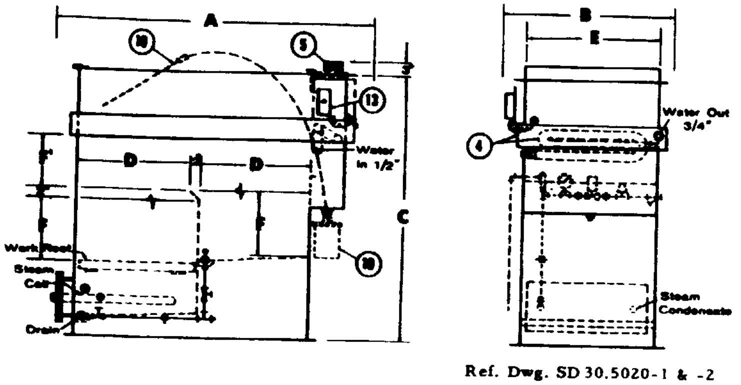
3. Available from stock.

Standard Features

4. Water jacket and auxiliary condenser coil.
5. Spring loaded retractable roll-up cover.
6. Interior sidewall unobstructed.
7. All welded construction - Detrex FF-1 coated interior.
8. All units of material of construction to allow choice of solvents - trichloroethylene, perchloroethylene, or 1,1,1-trichloroethane.
9. Lower safety temperature and liquid level control for gas and electric units.

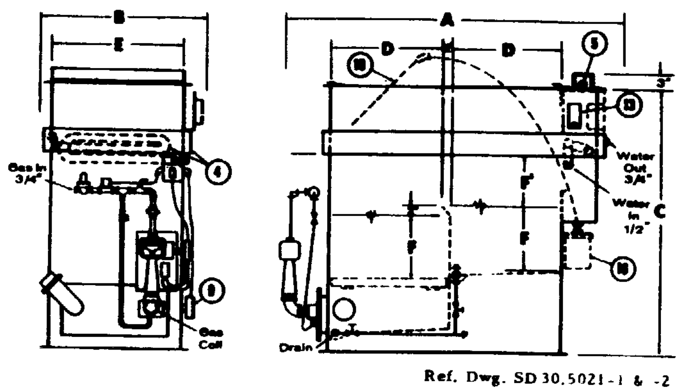
Optional Accessories Available

10. Solvent spray pump and spray lance for distillate rinse.
11. Water separator.
12. Lip exhaust system
13. Vapor level control.
14. Water temperature controls.



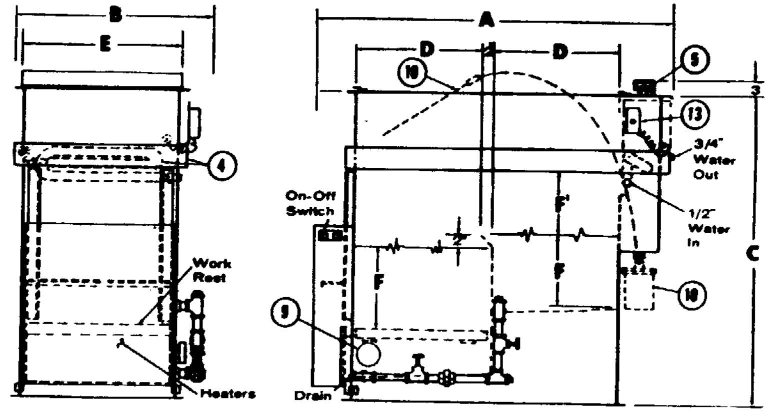
Ref. Dwg. SD 30.5020-1 & -2

Steam-Heated



Ref. Dwg. SD 30.5021-1 & -2

Gas-Heated



Ref. Dwg. SD 30.5022

Electric-Heated

Optional Accessories (Cont'd)

15. Detrex Freeboard Chiller.
16. Degreaser body fabricated of type 304 stainless steel.

EXHAUST SYSTEM SPECIFICATIONS:

O.S.H. 1445 cfm @ 1" S.P. - 1 hp, 1750 rpm motor. (Increases "A" dimension approx. 22 inches).

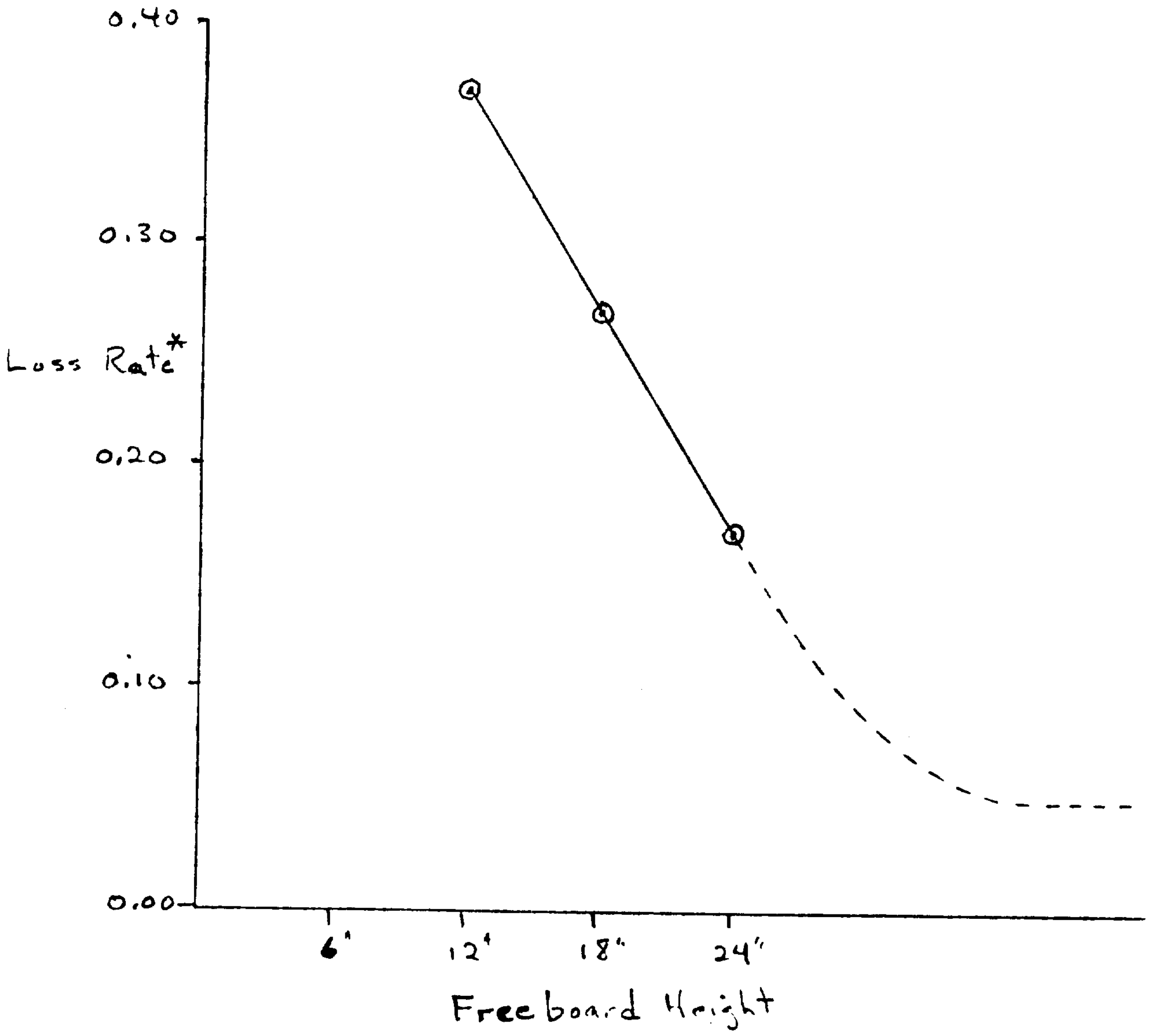
SPECIFICATIONS

SIZE	OVERALL DIMENSIONS			MAX. WORK CLEARANCE				RATED PRODUCTION	HEAT INPUT			SOLVENT CAPACITY	WATER @ 50°F RISE
									STEAM	GAS	ELECTRIC		
	A	B	C**	D	E	F	F'	Lb Steel/Hr	Lb/Hr	cf/hr @ 1000 btu	kw	Gallons	gph
D 20	60"	33"	48"	20"	24"	12"	10"	1500	55	80	15	70	100

**Add 3" for Cover or Exhaust.

Litho in U.S.A.

Solvent Loss Rate Versus Freeboard Height
(with air movement)



* Pounds solvent per square foot - hour

APPENDIX D

STUDY TO SUPPORT NEW SOURCE PERFORMANCE STANDARDS FOR SOLVENT METAL CLEANING OPERATIONS

Review of Existing Hydrocarbon Emission Regulations Applicable to Stationary Sources

Prepared By:

Angela J. Williams
The Dow Chemical Company

Prepared For:

Emission Standards and Engineering Division
Office of Air Quality Planning
U. S. Environmental Protection Agency

TITLE REVIEW OF EXISTING HYDROCARBON EMISSION REGULATIONS APPLICABLE TO STATIONARY SOURCES - SUB-TASK 6 OF PROJECT PLAN FOR EPA CONTRACT NO. 68-02-1329, TASK NO. 9

INFORMATIVE SUMMARY WITH CONCLUSIONS

As part of the EPA contract "Study to Support New Source Performance Standards for Organic Metal Cleaning Operations", a review of existing state and local emission regulations applicable to metal cleaning has been conducted.

This report categorizes and compares the emission control regulations currently in operation, the effects of controls on the degreasing solvent industry and future control guidelines.

LAB. CODE LETTERS AND REPORT NUMBER

REVIEW OF EXISTING HYDROCARBON EMISSION REGULATIONS APPLICABLE TO STATIONARY SOURCES

INTRODUCTION

Since 1966, when Los Angeles County developed regulations to control the emission of hydrocarbons into the atmosphere, many State and local areas have followed by developing and implementing control regulations applicable to their own areas.

The Clean Air Act was created in 1963 with the goal of establishing uniform State and local laws relative to the prevention and control of air pollution, while recognizing the need for practicable controls in the light of varying conditions and requirements.

In a major revision of the Act in 1970, many amendments occurred and the Environmental Protection Agency was formed and made responsible for air pollution control.

In 1971, the EPA published their criteria for "------National Primary and Secondary Ambient Air Quality Standards for Photochemical Oxidants and Hydrocarbons" (Federal Register Vol. 36, No. 84, April 30, 1971).

In Appendix B of this document, emission guidelines and controls were promulgated in accordance with EPA's estimation of the degree of emission control that could be attained using available control technology.

Los Angeles County - Air Pollution Control District developed and implemented "Rule 66" in mid-1966 and subsequently amended it in 1971. It was developed as a result of close consultation of the industries most affected by the regulations and ultimately it was accepted as a workable approach to reduce the formation of photochemical smog.

Most overall emission control plans which have been implemented are based on either the Rule 66 or Appendix B approach.

DEFINITIONS OF CONTROL

RULE 66

The original rule defines a photochemically reactive solvent as any solvent with more than 20% of its total volume composed of the following compounds, or which exceeds any of the individual total composition limitations stated:

1. Combination of hydrocarbons, alcohols, aldehydes esters or ketones having an olefinic or cyclo-olefinic type of unsaturation: 5 percent.
2. Combination of aromatic compounds with eight or more carbon atoms per molecule, except ethylbenzene: 8 percent.
3. Combination of ethylbenzene, ketones having branched hydrocarbon structures, toluene or trichloroethylene: 20 percent.

Whenever solvents may be described by the above classifications, they are regarded as reactive and subject to the following restrictions:

No more than 40 lbs./day or 8 lbs. in any one hour may be discharged, unless that discharge has been reduced 85%.

The 1971 amendment to Rule 66 exerts the following controls of total solvent emissions after August 31, 1974:

No more than 3,000 lbs./day or 450 lbs. in any one hour of organic material may be discharged from a single source unless that discharge has been reduced by 85%.

APPENDIX B

This regulation exerts more stringent and controversial limitations on solvent emission controls. It states that:

No more than 15 lbs. in any one day or 3 lbs. in any one hour of organic compounds may be emitted unless the emissions have been reduced at least 85% by: a) incineration - provided that 90% or more of the carbon is oxidized to carbon dioxide; b) carbon adsorption.

The EPA regulations have identified hydrocarbon solvents as pollutants as they are pre-cursors of photochemical oxidants. However, some organic solvents have been shown to be virtually photochemically unreactive and may be considered exempt from the regulations. These include saturated halogenated hydrocarbons, perchloroethylene, benzene, acetone and $C_1 - C_5$ paraffins.

Solvents in this group may be considered for exemption only if sources are not major contributors to hydrocarbon emissions.

The following table summarizes the state and local district regulations currently in operation.

STATE AND LOCAL AREA REGULATIONS AFFECTING
METAL CLEANING OPERATIONS

State or Local Area	Type of Regulation	Date of Implementation
Alabama	Rule 66	January 1973
Arizona--	Rule 66	August 1972
Maricopa County	*Rule 34A (Rule 66)	August 1972
Puma County	Rule 5 (App. B)	August 1972
California--		
Los Angeles APCD	*Rule 66	August 1971
San Francisco Bay Area	Reg. 3 (Revised Rule 66)	November 1974
Sacramento	EPA	
San Diego	Imposed	March 1974
San Bernadino	Regulation	
Orange County	Rule 66	August 1967
Riverside		
Santa Barbara		
Ventura		
Colorado--	Rule 66	September 1973
Denver AQCR	Rule 66 - EPA Imposed	May 1975
Connecticut--	Rule 66 and App. B	March 1973 - Jan. 197
D.C.	App. B	June 1974
Illinois--	*Rule 66	December 1973
Chicago	Rule 66	June 1974
Indiana	APC-15 (App. B)	October 1974
Kentucky	AP-5 (Rule 66)	April 1972
Louisiana	App. B	February 1974
Maryland	Rule 66	January 1973
Massachusetts--	Rule 66 - EPA Imposed	February 1974
Boston AQCR	Rule 66	February 1974
New York		
New York City	Parts 204 and 205	August 1972
Metropolitan Area	*(Rule 66 Type With Exception)	January 1974
New Jersey	Original (Rule 66 Type)	Open
North Carolina	Rule 66	January 1972
Ohio	Rule 66	February 1972
Oklahoma	Rule 66	January 1972
Philadelphia	Rule 66	July 1971
Rhode Island	AP-15 (Rule 66 Type)	Open
Texas--	Reg V (Rule 66 Type)	
Houston Galveston	*Rule 66 and	January 1974
San Antonio AQCR	App. B	May 1975
Virginia	Rule 66	June 1974
Wisconsin	Rule 66	April 1972

The Clean Air Act has scheduled State implementation plans for attaining ambient air standards be adopted no later than May 31, 1975. However, in those cases where implementation in 1975 is not economically or technically feasible, extensions up to two years are being granted by EPA.

Regulations affecting metal cleaning operations have so far been classified under "Organic Solvent Control" or "Control of Emissions from Stationary Sources". There are only a few promulgations actually stated for degreasing operations. These include:

*Maricopa County--Rule 34 A

1. No person shall use or conduct any vapor phase degreasing operation without minimizing organic solvent vapor diffusion emissions by good modern practices such as, but not limited to, the use of a freeboard chiller or other effective device operated and maintained in accordance with solvent and equipment manufacturers specifications.
2. Spray degreasing shall be conducted in an enclosure equipped with controls which will minimize the emission of organic solvents.

*Houston, Galveston, San Antonio A.Q.C.R.

Under sub-heading "Control of Degreasing Operations", the basic requirements are modeled upon Rule 66. Some exemptions to to the Rules' restrictions are defined as follows:

1. Degreasing operations which emit less than 3 lbs./hr. or 15 lbs./day of controlled organic materials.
2. Degreasing operations using perchloroethylene 1,1,1-trichloroethane or saturated halogenated hydrocarbons as an organic solvent.

Under sub-heading "Regulations for Limitations of New Sources", Appendix B has been adopted and becomes effective May 31, 1975.

*Los Angeles, San Diego, Sacramento Valley, San Joaquin Valley and San Francisco Bay Area Intrastate Regions - January 1975

"Control of Degreasing Operations"

(a) Degreasing means any operaton using an organic solvent as a surface cleaning agent prior to fabricating, surface coating, electroplating, or any other process.

(c) Any organic emissions must be reduced by 85% or solvent must be classfied non-photochemically reactive---Degreasing operations using perchloroethylene or saturated halogenated hydrocarbons shall be exempt.

EFFECT OF REGULATIONS ON THE METAL CLEANING INDUSTRY

The largest change since legislation was first introduced is the decrease in use of trichloroethylene in vapor degreasers. With two exceptions (*New York City Metropolitan Area - Part 205, January 28, 1974 and Illinois, December 1973) trichloroethylene is classified as a photochemical reactant and therefore subject to severe emission limitations. Since, for most operations, 85% reduction of emissions is impossible to meet, the alternative is to change solvents to using a non-photochemically-reactive classified solvent, which would then be exempt from severe restrictions. The solvents most used from this category are 1,1,1-trichloroethane and perchloroethylene.

These solvents are used in degreasing operations within the workable, less stringent emission controls by moderate equipment modifications and recommended improved work habits.

SUMMARY

Future legislation specifically for metal cleaning should consider the large quantity of miscellaneous hydrocarbons used in cold cleaning applications. Generally, small amounts of solvent are involved for each individual operation, but collectively the solvent loss from these add up to a large volume of emissions without any form of control.

Overall regulations, whether modeled on Rule 66 or Appendix B type of control, as applied to stationary sources are not wholly viable when applied to vapor degreasing.

In this extremely varied industry, the design and type of degreasing equipment influence the volume of solvent vapors emitted.

Workable controls could be postulated taking into account the following:

- Areas of solvent vapor/air interface
- Surface area of metal cleaned
- Installation of equipment to limit vapor loss (covers, free board chillers, etc.)

- Consideration of solvent vapor recovery systems (carbon adsorption)
- Improvement of degreaser operating habits (manual spraying, work racking, hoist speeds, drainage time, etc.)
- Disposal of solvent residues

The solvent industry is cognizant of the necessity for controls to limit solvent vapor emissions from both health and economic viewpoints.

Regulations applicable to this industry should take into consideration the energy and economic impact of emission controls, as recognized by the Environmental Protection Agency.

Appendix E-1

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals

COPIES TO

E. J. Fort, Chicago
S. J. Nolan, Chicago
R. R. Lapine, 2020
G. E. Forrest, 2020
C. R. Crabb, 2020

R. T. Gerard, 2020
KOG/PF 2192008

NAMES

FIRM NAME (In full, do not abbreviate)
Safety Clean Corporation

STREET	CITY	STATE	ZIP CODE
655 Big Timber Road	Elgin	Illinois	60120

PERSONS INTERVIEWED AND TITLES
Mr. Harry Logue, Sales Manager
Mr. Allen Manteuffel

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant KS			X	X	3/25/75	3/26/75

OTHER DOW PERSONNEL PRESENT
None

SUBJECT
EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

Discussions with Graymills Corporation and Kleer-Flo both indicated that Safety Clean Corporation makes parts washers and provides them to customers on a lease basis. This leasing arrangement involves both the supply of solvent for metal cleaning as well as the equipment.

Mr. Manteuffel indicated that Safety Clean Corporation has about 140,000 units under lease at the present time. These metal washers come in two sizes, 16 gallon capacity and 30 gallon capacity. The average fill is approximately 12 gallons. Customers are set up on a solvent replacement cycle from two to six weeks. The annual replacement need for solvent to service these customers amounts to 16 - 17 million gallons. Mr. Manteuffel estimated that the solvent use of these customers would be in the range of 40 million gallons per year if they were disposing of this solvent in landfills rather than using Safety Clean Corporation's service which recovers the solvent for reuse.

REMARKS

Appendix E-2

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals Department

COPIES TO

J. C. Carlaw, Sarnia
D. R. Heinz, 2020
H. R. Krimbill, 2020
R. R. Lapine, 2020
S. J. Nolan, Chicago
D. W. Richards, 2020
A. J. Williams, 2020
KOG/PF 2192008

NAMES

FIRM NAME (In full, do not abbreviate): Graymills Corporation

STREET	CITY	STATE	ZIP CODE
3759 N. Lincoln St.	Chicago	Illinois	60613

PERSONS INTERVIEWED AND TITLES: Mr. Ed Roels

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant KSS	x			x	1-15-75	1-29-75

OTHER DOW PERSONNEL PRESENT: A. J. Williams

SUBJECT: EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

REMARKS

The purpose of this visit was to obtain additional background on solvent room temperature cleaning or "cold cleaning". Graymills Corporation is a leading manufacturer of tanks for this cleaning method which they refer to as parts washers. They also offer a line of solvents under the trademark Agitene Solvents which are both straight petroleum solvents and solvent blends made with chlorinated hydrocarbons and other chemicals. Our discussion brought out the facts that cold cleaning requires the least capital investment, is completely portable and requires no energy consumption. Graymills have been making parts wahsers since 1945 and have marketed as many as 15,000 units per year. They estimate that between 750,000 to 1,000,000 units are in operation in the U. S. today. The parts washers require five gallons upwards to 100 or 200 gallons for an initial fill. The most common model holds about 30 gallons. In the automotive after-market, it was estimated that this volume of solvent is changed twice per year whereas the industrial users change the solvent from every two weeks to four times per year. The waste solvent was reported to be returned to drums and sold as scrap. Make-up solvent between solvent changes probably equals the volume of solvent contained in the parts washer. In addition, it was estimated that as many as an equal number of units are "homemade" from solvent drums or pails. Kleer-Flo was reported to be their major competitor and located in Eden Prairie, Minnesota. Safety Clean Corporation of New Berlin, Wisconsin leases similar solvent cleaning equipment but does not sell it. Lesser competition is received from Phillips Manufacturing and Gunk Incorporated. The life of this equipment is very long since it experiences little or no corrosion. Ed Roels has no doubt that some of their equipment is still in operation from their first year of manufacture. Safety Clean Corporation reportedly has 120,000 units under lease in the field right now.

Appendix E-3

DOW CHEMICAL U.S.A

EPARTMENT: Inorganic Chemicals

COPIES TO

J. C. Carlaw, Sarnia
D. R. Heinz, 2020
H. R. Krimbill, 2020
R. R. Lapine, 2020
S. J. Nolan, Chicago
D. W. Richards, 2020
A. J. Williams, 2020
KOG/PF

NAMES

FIRM NAME (In full, do not abbreviate): Kleer-Flo Corp.

STREET	CITY	STATE	ZIP CODE
6600 Washington Ave., S.	Eden Prairie	Minnesota	55346

PERSONS INTERVIEWED AND TITLES: Mr. Graham Pendelton

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant			x	x	1/23/75	2/17/75

OTHER DOW PERSONNEL PRESENT: None

SUBJECT: EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

REMARKS

Mr. Ed Roels of Graymills Corp. indicated that Kleer-Flo Corp. is their main competitor and would be able to offer additional information on the cold cleaning market. After reviewing both companies literature and having discussions with both, they obviously compete head-to-head.

Kleer-Flo has been marketing parts washers (cold cleaning tanks), petroleum solvents and safety blends since 1936. Graham Pendelton estimated the total number of parts washers manufactured by Kleer-Flo or other competitors to be between the one half to one million units. Truck and car dealers and independent garages as well as industrial accounts are major consumers. The automotive dealers probably average two to three units each whereas garages average one to two units. The volume of an average parts washer would be in the twenty-five to thirty gallon range. Operational instructions as provided by Kleer-Flo suggest that the solvent be changed every two to three months. However, in practice many firms drain the system only once every six months. Graham indicated the evaporation loss to be 5 to 10 gallons of solvent between changes. Again as in the case of Graymills Corp., their prime profit center is in the equipment manufacturer. Graham noted that Safety Clean Corp. of Wisconsin has greater than 100,000 units on lease, primarily in automotive after market applications.

sm

Appendix E-4

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals Dept.

COPIES TO

L. W. Stump, Detroit
J. W. Hennington, 2020
C. R. Crabb, 2020
D. W. Richards, 2020
KOG/PF 2192008

NAMES

FIRM NAME (In full, do not abbreviate)
Horton Co.

STREET	CITY	STATE	ZIP CODE
2070 Brooklyn Rd.	Jackson,	Michigan	49203

PERSONS INTERVIEWED AND TITLES
Mr. Robert Ziegler, President

WRITTEN BY	FIELD DOW PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant KSS	x	x	7/1/75	7/30/75

OTHER DOW PERSONNEL PRESENT
None

SUBJECT
EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

Summary

The Horton Company replaced a small open top degreaser with a Phillips Manufacturing Rotomatic degreaser. Solvent records show that the larger more enclosed degreaser system is consuming at least 40% less solvent on a work load basis and less than a third as much solvent loss on a sq. ft. of exposed vapor zone basis. The newer equipment demonstrates two generally recognized facts:

1. Enclosed conveyorized equipment is much more efficient in controlling solvent losses than a number of open top degreasers having equal work capacity.

2. The sale of enclosed conveyorized vapor degreasers often reduces the volume of solvent used in the market rather than increasing it.

REMARKS

Discussion

In earlier conversations with vapor degreasing equipment manufacturers, reports were received indicating that the sale of some large cross-rod or monorail type vapor degreasing systems can result in less solvent usage by the customer rather than more. The reason offered for this was that large conveyorized vapor degreasers often replace several open top vapor degreasers. Because of the greater control over solvent losses in the conveyorized equipment, the solvent requirement for the firm involved is actually reduced and sometimes substantially (see Baron Blakeslee report on January 16, 1975 trip).

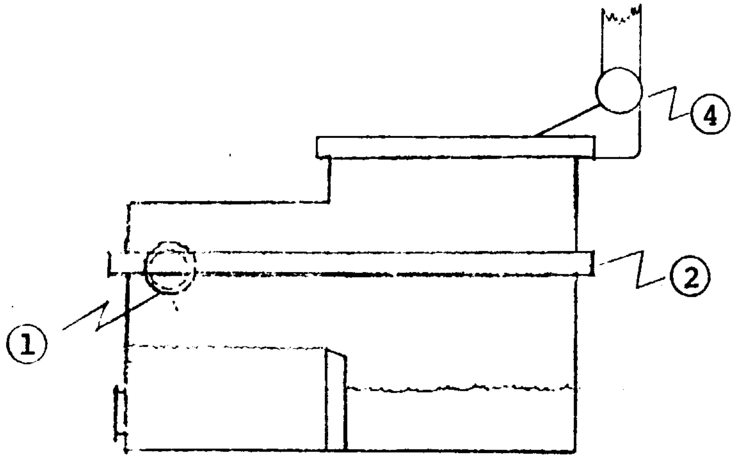
Prior to January 1974, Horton Manufacturing Co. had a gas heated open top degreaser. The open top dimensions of this degreaser were 36"x24". This degreaser was replaced by a Rotomatic vapor degreaser manufactured by Phillips Manufacturing Co., Model 60TRMD. This degreaser is electrically heated with a 24kw rating the Rotomatic is supported with a still (Model RS-30) having a 15kw heating capacity. These pieces of equipment are sketched on the attached sheet with no scale intended. The left figure represents the open top degreaser while that on the right represents the Rotomatic and still. The Rotomatic degreaser is hooded over a substantial proportion of the degreaser system and has a ferris wheel type of material handling system. This design is similar to a cross-rod vapor degreaser in that the handling system causes the parts to rotate providing improved solvent drainage from the parts. However, the Rotomatic design is less enclosed then a cross-rod degreaser.

Both the open top and the Rotomatic vapor degreasers have been operated on a one shift per day five days per week basis. The open top degreaser was closed during non-operating shifts on weekends and was equipped with a manually rotatable parts fixture. This degreaser was not equipped with a still. The Rotomatic degreaser is equipped with a still, as mentioned, which provides much greater capability of recovering solvent from the oil removed from parts. The open top surface area of this degreaser including that which is located under the hood is 100"x33". These dimensions in addition to the ferris wheel design make the use of a cover during non-operating hours difficult. Horton Co. solved this problem by pumping all of the solvent in the degreaser into storage tanks during down shifts and weekends. Of course, this is a highly efficient wasy to prevent solvent evaporative losses during non-working hours.

Solvent records were available for over a year before the new equipment was installed and over half a year after the conversion to the new equipment. These records indicated 37 drums of 1,1,1-trichloroethane were consumed during a 58 week period prior to the new equipment installation or 0.64 drums per week. The solvent consumption for a 29 week interval with the Rotomatic degreaser consumed 22 drums of 1,1,1-trichloroethane. This amounts to 0.76 drums per week. Although this indicates a solvent consumption of 18% greater then that experienced with the small open top degreaser, the work load being processed was estimated to be two to three times greater after the installation of the Rotomatic degreaser. Thus, on a work load basis, the solvent consumption had been reduced by at least 40%. Using 600 pounds/drum, 384 pounds of 1,1,1-trichloroethane were consumed per week in the open top degreaser and 456 pounds of solvent were consumed per week in the Rotomatic degreaser. The solvent consumption rate can be determined on a pound per hour per sq. ft. basis. Expressed in this way, values of 0.50 and 1.60 lbs/hour/sq. ft. are found for the Rotomatic and the open top respectively. This calculation assumes that no solvent is lost during non-operating time. On this basis the Rotomatic degreaser is three times more efficient then the open top.

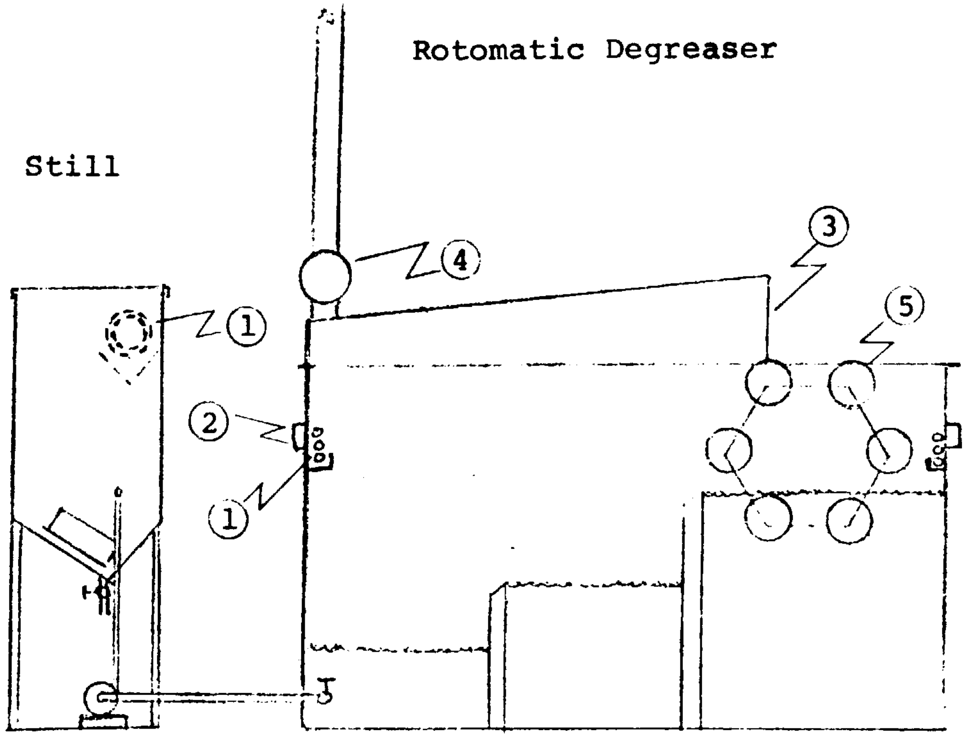
sm

Open Top Degreaser



Still

Rotomatic Degreaser



1. Condenser Coils
2. Freeboard Chiller
3. Hood
4. Fan
5. Rotating Baskets and
   Rotating Fixture

Appendix E-5

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals

COPIES TO

J. C. Carlaw, Sarnia	D. W. Richards, 2020
D. R. Heinz, 2020	A. J. Williams, 2020
H. R. Krimbill, 2020	KOG/PF 2192008
R. R. Lapine, 2020	Central Files
S. J. Nolan, Chicago	

NAMES

FIRM NAME (In full, do not abbreviate): BARON BLAKESLEE

STREET	CITY	STATE	ZIP CODE
1620 South Laramie Ave.	Chicago	Illinois	

PERSONS INTERVIEWED AND TITLES:
Mr. R. A. Kullerstrand, Vice-President
Mr. Parker Johnson, National Sales Manager

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant	x			x	1/16/75	1/29/75

OTHER DOW PERSONNEL PRESENT: A. J. Williams (Dow), W. Johnson (EPA)

SUBJECT: EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

Baron Blakeslee is the leading manufacturer of vapor degreasing equipment. This discussion was arranged to provide further background on solvent metal cleaning operations, to determine any new emission control technology for controlling solvent emissions and to provide Baron Blakeslee with the opportunity to suggest sites to evaluate emission control methods.

REMARKS

As a major supplier of vapor degreasing equipment and solvents used in this process, the Baron people felt that they could be of help in discussing vapor degreasing but could offer little information on solvent room temperature or cold cleaning. However, they agreed that the volume of solvent used in room temperature or cold cleaning is very large. Mr. Kullerstrand and Mr. Johnson agreed that there are approximately 25,000 vapor degreasing systems operating in the U.S. It was estimated that 70% of these systems are of the open top design. Approximately 2,500 vapor degreasers are manufactured per year with a life expectance of 15 years or more. Less than one half of these units were estimated to be replacement of existing systems. In some cases the sale of a new vapor degreaser can result in less solvent use rather than more. This occurs where a conveyorized degreaser replaces one or more open top degreasers.

Such was the case at Tillotson, Toledo, Ohio where one cross-rod vapor degreaser reduced the solvent consumption from 25 drums per month to about 5 drums per month by replacing two open top vapor degreasers. Mr. Johnson emphasized the point that vapor degreasing equipment is sold to all kinds of industries not only metal fabricating plants. These would include maintenance operations at paper mills or textile mills, automotive repair and rebuilding shops, heavy duty construction equipment maintenance and agricultural equipment maintenance locations as well. This diverse use of vapor degreasing was illustrated by the vapor degreasing of candelabra at St. Patrick's Cathedral and by degreasing skeletons for laboratory and educational use.

Parker Johnson observed that vapor degreasing is different from cold cleaning by its ability to distill and recover pure and reusable solvent. When a still is attached to a vapor degreaser he pointed out that the oils removed from metal parts can be concentrated up to 90% or better in the case of perchloroethylene and trichloroethylene, by steam distillation. With 1,1,1-trichloroethane, steam distillation is not recommended and he noted as much as 30% by volume of solvent may be residual in the concentrated still residues. In the Baron Blakeslee solvent distillation recovery service portion of their business, the dirty solvent received for reclaiming usually contains between 50 and 90% solvent most of this solvent can be recovered by distillation and is returned to the customer as a service or sold to new customers.

Mr. Johnson reported that Baron Blakeslee also sells carbon adsorption recovery equipment and the Econ-O-coil. The latter is a refrigerated water system to reduce solvent vapor emissions from vapor degreasing operations. It supplements the existing condenser coils used to control the vapor zone within the vapor degreaser, these two techniques were the only two techniques which had been observed in the U.S. market place. No solvent recovery or emission control devices were reported to be used in connection with cold cleaning. The Baron people reported that 40% solvent recovery can be expected on open top vapor degreasing equipment but on specially designed and conveyorized equipment recoveries up to 85% are possible. However, no known company installations are performing this well.

Mr. Kullerstrand and Mr. Johnson agreed that vapor degreasing is regarded as a conservation device vs. alkaline washing or a cold cleaning. In addition to the water pollution created by alkaline washing, an alkaline washing system reportedly consumes 8 times as much energy as a comparable vapor degreasing system. As many as 30 to 40% of the vapor degreasing operations do not require ventilation whereas all alkaline washing equipment does which requires additional heat to condition the replenishment air for the factory. They agreed that locating a single plant where both vapor degreasing and alkaline washing could be compared on a direct basis would be very difficult if not impossible. It was suggested that a real vapor degreasing operation could be studied and compared to a paper analysis of a comparable alkaline washing operation to develop comparable data on the two systems. Western Electrics Hawthorne Plant was suggested as an excellent location to evaluate various vapor emission control systems due to their large number of installations and the knowledgability of Mr. James Weston of these systems. (Mr. Weston will be contacted as an evaluation cite).

sm

Appendix E-6

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals Dept.

COPIES TO

D. W. Richards, 2020
R. R. Lapine, 2020
R. C. Ormiston, Philadelphia
J. Carlaw, Sarnia

Metal Cleaning Group
Non-Metal Cleaning Group
KOG/PF 2192008

NAMES

FIRM NAME (In full, do not abbreviate): Auto Sonics Co. (215) 828-9090

STREET	CITY	STATE	ZIP CODE
P. O. Box 300	Conshohocken ,	Pennsylvania	19428

PERSONS INTERVIEWED AND TITLES:

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant			x	x	5/9/75	5/13/75

OTHER DOW PERSONNEL PRESENT: None

SUBJECT: SOLVENT METAL CLEANING CONTRACT FOR EPA

SUMMARY

This phone conversation was made to determine current pricing of the cold trap. This pricing data will be used to determine the cost benefit relationship of the cold trap installations to be tested under the Dow- EPA contract on solvent metal cleaning.

The compressor size is determined by the peripheral footage of an open top degreaser. The peripheral footage is calculated as follows:

- Less than 42" width -- 2 (width plus length)
- 42" to 72" width -- 2 (width plus length) times 1.5
- Greater than 72" width -- 2 (width plus length) times 2.0

The footage corrections for degreasers having widths of 42" to 72" or greater than 72" is done because an extra refrigeration coil is added for the 42" to 72" width and two extra refrigeration coils for degreasers with greater than 6' width.

REMARKS

The size of compressor needed and its cost can be obtained from the following table:

Horsepower	Peripheral Footage	Dollars
1/2	10'	$2,510
3/4	16'	2,595
1	21'	3,662
1-1/2	35'	4,795
2	47'	5,575
3	70'	6,265

To obtain the total purchase price a value for the peripheral footage should be added to the compressor cost. Peripheral footage cost can be obtained by adding the degreaser length and width and multiplying by two. The cost is $32.00 per foot for a degreaser having a width of less than 42". $41.00 per foot is the cost for degreasers 42" to 72" in width. Degreasers 6' wide and larger cost $50.00 per foot.

Appendix E-7

DOW CHEMICAL U.S.A

DEPARTMENT: Inorganic Chemicals

COPIES TO

D. N. DeMott - 2020
R. R. Lapine - 2020
D. W. Richards - 2020
A. J. Williams - 2020
KOG/PF 2192008
H. A. Pastor - 2020
J. C. Copus - 2020
T. A. Vivian - 2020
R. T. Gerard - 2020

NAMES

FIRM NAME (In full, do not abbreviate): Vic Manufacturing Company

STREET	CITY	STATE	ZIP CODE
1620 Central Ave.	Minneapolis	Minnesota	

PERSONS INTERVIEWED AND TITLES
Mr. Irving Victor, Executive Vice-President
Mr. Oscar Victor, President
Mr. Joe Barber, Chief Chemist
Charles Gorman, Sales Manager

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant	X			X	12/13/74	12/20/74

OTHER DOW PERSONNEL PRESENT: K. O. Groves

SUBJECT: CARBON ADSORPTION

SUMMARY

REMARKS

Non-Dow Personnel

Mr. David Patrick - EPA
Mr. William Johnson - EPA
Mr. Billy McCoy - TRW Corp.

This call was made to provide an indepth background and understanding of the process of carbon adsorption as it can be applied to both chlorinate and flammable solvents used in metal cleaning and drycleaning. Historically, Vic Manufacturing began marketing carbon adsorption recovery equipment to the drycleaning market in 1957 and to the metal cleaning (vapor degreasing) market place in 1958. They have maintained a dominate position in both markets ever since. The basic Vic carbon adsorption patents will expire in 1976, however, they should maintain their position in the market even without patent protection. The typical efficiency of carbon adsorption on open-top or cross-rod degreasers is estimated to be in the range of 50 to 60%. The recovery efficiency on a monorail degreaser is estimated to be about 70%. With special design considerations maximum efficiency are estimated to be in the range of 90% under expertly controlled conditions. With best design techniques, the solvent recovery efficiency for typical vapor degreasing operations could be improved to about 70%. In drycleaning, the recovery efficiency is estimated at 50% savings as a general rule. Vic Manufacturing will guarantee solvent recovery efficiencies up to 50% in both market places. A crude estimate of the cost of carbon adsorption equipment can be made from the volume of air which needs to be processed. The equipment can cost as low as $2.00 per cfm. Common equipment used to recover solvent from vapor degreasing operations cost between $4.00 and $4.5 per cfm based on the maximum bed gas flow rates.

Carbon Adsorption - Metal Cleaning Processes

Vic Manufacturing estimates that they have sold about 1,000 carbon adsorption systems to metal cleaning operations and that they have supplied 90 to 95% of the carbon adsorption systems to this use. All sales to date have been motivated by the dollar savings the customer could obtain. Some interest is being noted by Vic Manufacturing of customers wanting adsorption to comply with air pollution regulations.

The typical open-top degreaser ventilated at a rate of 60 cfm per square foot will experience a recovery efficiency of 50 to 60%. They believe that an open-top degreaser with an optimum design and top notch maintenance and operation of carbon adsorption can recover 90 to 95% of the solvent which would escape the system without recovery equipment. The ventilation rate used in this best design system is 100 cfm per square foot. Using 100 cfm per square foot in a typical open-top degreasing operation, they expect that a 70% recovery would be common. Cross-rod degreasers are ventilated on the basis of the vertical section open area by the loading station. Ventilation rates of 35 to 50 cfm per square foot are used and recovery efficiencies of 50 to 60% are typical. Monorail degreasers are ventilated at the rate of 35 cfm or greater per square foot of an open area at both inlet and outlet and obtain 70% recovery. The carbon beds are designed to retain the processed air for a minimum of 1/100 of a minute. Bed depths run from a minimum of 9 inches to a maximum of 32 inches and the volume flow is calculated based on a 100 ft. per minute per square foot of bed, the working bed capacities are shown below:

Trichloroethylene -- 15% by weight of the carbon bed
Perchloroethylene -- 20% " " "
Methylene Chloride -- 10% " " "
1,1,1-trichloroethane -- 12% " " "
Toluene/Xylene -- 6% " " "
Stoddard Solvent -- 6-7% " " "

In general carbon adsorption beds are designed to operate on the adsorption cycle for at least 1 hour. For vapor degreasing operations the typical bed would have a four hour capacity. Detection systems are available to determine breakthrough or saturation of the bed for combustable solvents, but a similar system is not available for halogenated solvents.

Typical equipment is mild steel with baked phenolic as a protective coating. Equipment life is expected to exceed 10 years and some units have been operating for 15 years. Again, in the case of bed life the expected life is 10 years or more. 1,1,1-trichloroethane equipment is designed of Hastaloy and is accompanied with sodium carbonate neutralization and calcium chloride drying beds. Carbon adsorption equipment prices change annually or semi-annually and have been inflating at a rate of 5 to 11% per annum. The equipment is sold f.o.b. plant and freight and installation costs are estimated to be between 15 and 25%. Vic Manufacturing estimated the cost of maintenance and operation to be 3 to 5% of the equipment cost.

Solvent recovered from adsorbers has reduced levels of stabilizers but can be reused when recovery efficiencies are 50 - 60% or less. Higher recovery efficiencies require restabilization of the solvent for reuse.

Carbon Adsorption - Drycleaning

Vic Manufacturing estimates the number of perchloroethylene drycleaning plants to be about 16,000. Of this number Vic has sold 7,000 carbon adsorption units and estimates that Hoyt Manufacturing (a licensee of Vic Manufacturing) has sold another 3,500. Other carbon adsorption manufacturers account for about 10% more bringing the total to about 11.6 thousand carbon adsorbers. Of this total, the Vic Manufacturing share of market is about 70%. This agrees with their own estimate of share of market. Model 128 is used for drycleaners loosing one drum of solvent per month or less and is a most commonly sold machine (about 3,000 machines). The next most common model is Model 118 for one to two drum customers (about 2,600 machines have been sold). Model 108 is designed for customers using more than two drums of solvent per month (about 900 machines sold). Again, a 50% effiency and saving of solvent is estimated for these units. Model 415 is designed for coin operated drycleaners and about 100 machines have been sold.

Vic estimates that there are about 12,000 Stoddard drycleaning plants and that they process approximately 60% of the total drycleaning market. Perchloroethylene is estimated to process the 40% balance. 3.5% of the the drycleaning was estimated to be done by Valclene (trichloro-trifluoroethane). Almost none of the Stoddard plants have carbon adsorption. Carbon adsorption equipment for Stoddard plants cost more because of the lower capacity (6 to 7% by weight of bed) than that of perchloroethylene (20% by weight) and because vapor concentrations must be held to less than 25% of the lower explosive limit. It is estimated that the equipment pay off for carbon adsorption at Stoddard plants will require 2 1/2 to 8 years.

No mileage rates were offered for Stoddard solvent. Perchloroethylene mileage on dry to dry or transfer machines without carbon adsorption was estimated to be 5,000 pounds per drum. With carbon adsorption the mileage was estimated to be about 10,000 pounds per drum and the best systems with carbon adsorption were reported to achieve 20,000 pounds per drum. The average income per drycleaning plant was estimated to be about $800. per week. Income can be converted to pounds of clothes cleaned using a rate of $1.00 to $1.10 of income per pound of clothes.

New drycleaning equipment sales have been slow for the past year or so. Current equipment sales in drycleaning are about 60% for Valclene and 40% for perchloroethylene.

Large numbers of used perchloroethylene machine are on the market reducing the demand for new equipment of this type. Refrigeration is being used instead of carbon adsorption for the new Valclene equipment. Two-ton refrigeration capacity units are used for this system and the heat exchange temperature is set for about 0°F. Vic estimates the cost per pound of clothes for Valclene at 2¢ and for perchloroethylene at 1¢. The Valclene equipment is not vented to the world but uses a plastic lung or bag to collect the final drying air. The cost of Valclene was estimated to be about $8.00 per gallon in 5 - 20 gallon quantities.

12/74

SPECIFICATIONS
Vic Air Pollution Control System

(Contact factory for solvents not listed.)

MODEL	CFM		LBS OF CARBON Per Tank	SOLVENT	LBS SOLVENT PER TANK/HR	LBS STEAM PER HOUR	CONDENSING WATER - GPM			
	Single	Double					@65°	@85°	Primary 85°-120°	Secondary 50°-60°
534	700	-	150	Trichloroethylene	23	67	2.7	4	-	-
				Perchloroethylene	30	90	3.5	5	-	-
				Toluene	9	32	-	2	-	-
				Freon TF	12	42	-	-	2.5	1
				Methylene Chloride	15	45	-	-	2.8	1
#3000 automatic 536 #1550 manual	800	#2.3/cfm 1300	350	Trichloroethylene	53	158	6.5	10	-	-
				Perchloroethylene	70	210	8.5	13.5	-	-
				Toluene	21	84	-	5.5	-	-
				Freon TF	28	98	-	-	6	1.5
				Methylene Chloride	35	105	-	-	6.5	1.7
548	1400	-	400	Trichloroethylene	60	180	7	11.5	-	-
				Perchloroethylene	80	240	9	15.5	-	-
				Toluene	24	96	-	6.5	-	-
				Freon TF	32	112	-	-	7	2
				Methylene Chloride	40	120	-	-	8	2.5
554 #13 m	1700	#4.3/cfm 3000	1000	Trichloroethylene	150	450	18.5	29	-	-
				Perchloroethylene	180	540	22	35	-	-
				Toluene	60	240	-	15.5	-	-
				Freon TF	80	280	-	-	17	4.5
				Methylene Chloride	100	300	-	-	19.5	5
572 #21 m	3000	#3.8/cfm 5500	1500	Trichloroethylene	225	675	27.5	43.5	-	-
				Perchloroethylene	300	900	37	58	-	-
				Toluene	90	360	-	23.5	-	-
				Freon TF	120	420	-	-	25.5	7
				Methylene Chloride	150	450	-	-	28	8
584	3800	7500	3000	Trichloroethylene	450	1350	55	87	-	-
				Perchloroethylene	600	1800	73.5	115	-	-
				Toluene	180	720	-	47	-	-
				Freon TF	240	840	-	-	51	14
				Methylene Chloride	300	900	-	-	56	16
596	5000	10.000	4500	Trichloroethylene	675	2025	82	130	-	-
				Perchloroethylene	900	2700	110	173	-	-
				Toluene	270	1080	-	70	-	-
				Freon TF	360	1260	-	-	76	20
				Methylene Chloride	450	1350	-	-	84	24

Appendix E-8

DOW CHEMICAL U.S.A

DEPARTMENT: Inorganic Chemicals Department

COPIES TO

C. R. Crabb, 2020   KOG/PF 2192008
R. R. Lapine, 2020
K. P. Schultz, Stamford
M. Meglio, Boston
D. W. Richards, 2020

NAMES

FIRM NAME (In full, do not abbreviate): HOYT MANUFACTURING

STREET	CITY	STATE	ZIP CODE
	Westport	Massachusetts	02790

PERSONS INTERVIEWED AND TITLES: Mr. Derek Oakes, Vice President

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant	X			X	5-28-75	6-11-75

OTHER DOW PERSONNEL PRESENT: Mr. John Bollinger, EPA

SUBJECT: EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

Hoyt Manufacturing was visited to advise them of the low recovery efficiency being obtained from the carbon adsorption system at J. L. Thompson, Waltham, Massachusetts. The J. L. Thompson location as a test site for carbon adsorption recovery was suggested by Hoyt Manufacturing and the equipment was supplied by them. Mr. Oakes agreed to have one of his engineers reinspect the operation as soon as possible after hearing that our recovery efficiency had not improved over the earlier report. Since then, Mr. Oakes has reported that his engineer found the air inlet damper to bed "B" broken and in the closed position so that bed "B" could not effectively adsorb solvent vapors. This agrees well with the observation that no solvent was recovered from the desorption cycle of bed "B" during the test evaluation.

REMARKS

The model located at J. L. Thompson was Model 536AD. This model is now redefined as Model ABRS15F and is constructed of mild steel and Heresite coated. The system contains 300 pounds of carbon per bed and has the capability of recovering 45 to 50 pounds of trichloroethylene per desorption cycle. Mr. Oakes indicated that the normal ratio of steam condensate to pounds of solvent recovered is three to four pounds of steam to one pound of solvent. This model now costs approximately $8,000. The installation costs can vary considerably depending upon the amount of vent ducting and the availability of utilities (compressed air, steam, electricity) in the immediate area of installation. However, 15% of the purchase price was thought to be a reasonable estimate for installation.

Mr. Oakes indicated that about 200 Hoyt units have been sold for solvent metal cleaning operations. Bed efficiency is over 95%. Hoyt Manufacturing use to guarantee 50% overall recovery on a total solvent consumption basis and never had a unit returned to him on this basis. Normal maintenance costs were estimated to be about $200 per year. Titanium clad or Hastaloy C are recommended for solvent recovery of 1,1,1-trichloroethane although they have limited experience in recovery of this solvent.

Appendix E-9

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals

COPIES TO

C. R. Crabb, 2020
R. T. Gerard, 2020
J. W. Hennington, 2020
R. R. Lapine, 2020
D. W. Richards, 2020

L. W. Stump, Detroit
KOG/PF 2192008
Central Files

NAMES

FIRM NAME (In full, do not abbreviate)
Detrex Chemical Industries Inc.

STREET	CITY	STATE	ZIP CODE
14331 Woodrow Wilson	Detroit	Michigan	48232

PERSONS INTERVIEWED AND TITLES
Mr. Tom Kearney, Engineer
Mr. Robert Clark, General Sales Mgr.
Mr. Robert White, Manager Gold Shield Sales

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. S. Surprenant KS	x			x	3/7/75	3/21/75

OTHER DOW PERSONNEL PRESENT
None

SUBJECT
EPA CONTRACT ON SOLVENT METAL CLEANING

SUMMARY

The primary purpose of this call was to discuss the EPA Contract with Mr. Tom Kearney, one of the most experienced men in solvent metal cleaning equipment. An earlier letter as a result of a phone conversation is included with this call report discussing solvent losses from various kinds of metal cleaning operations and comparing alkaline cleaning costs vs. vapor degreasing costs.

Mr. Kearney reported that no new technology was available to control solvent emissions from metal cleaning operations. Freeboard chillers including the "cold trap" and carbon adsorption represent essentially all the commercial technology available for solvent recovery or loss control. He emphasized the importance of good metal cleaning practices and the use of covers as a means of conserving solvent, and noted that these techniques are nearly always economically practical.

The ratio of freeboard height to degreaser width has been known to effect solvent losses from solvent cleaning. Two graphs showing this relationship we are provided. Copies are attached.

REMARKS

As discussed in his letter, he felt that vapor degreasing results in less solvent emissions on a general basis than cold cleaning. He will arrange for the use of a small open top degreaser on a loan or consignment basis to develop information to confirm or deny this conclusion. Mr. Kearney agreed to help us locate a manufacturing plant where the comparative cost of alkaline cleaning and vapor degreasing could be developed.

Appendix E-10

DOW CHEMICAL U.S.A.

DEPARTMENT: Inorganic Chemicals Department

COPIES TO

J. C. Carlaw, Sarnia
D. R. Heinz, 2020
H. R. Krimbill, 2020
R. R. Lapine, 2020
S. J. Nolan, Chicago

D. W. Richards, 2020
A. J. Williams, 2020
KOG/PF 2192008

NAMES

FIRM NAME (In full, do not abbreviate)
Phillips Manufacturing Company

STREET	CITY	STATE	ZIP CODE
7334 N. Clark Street	Chicago	Illinois	60626

PERSONS INTERVIEWED AND TITLES
Mr. Donald L. Racquet
Mr. Oskar Franz

WRITTEN BY	FIELD	DOW	PHONE	DEVELOP. SERV. OTHER	DATE CALLED	DATE WRITTEN
K. Surprenant	x			x	1-16-75	1-29-75

OTHER DOW PERSONNEL PRESENT
A. J. Williams (Dow), Mr. William Johnson (EPA)

SUBJECT
ORGANIC SOLVENT METAL CLEANING

SUMMARY

This discussion was held to develop a further general background in solvent metal cleaning for the Environmental Protection Agency Contract. Further, it was our purpose to solicit Phillips Manufacturing cooperation in locating industrial sites which could be used to evaluate various emission control technology. Much of the background information on solvent metal cleaning technology and emission control technology supported that provided by various Dow personnel in the initial orientation program provided in the Dow laboratories. Phillips agreed to help select emission control evaluation sites.

REMARKS

Don Racquet of Phillips Manufacturing discussed the wide use of "cold cleaning" with Petroleum Solvents particularly with Stoddard solvent. No estimate could be made of the volume of cold cleaning solvents in use today in the U. S., however, the volume of petroleum solvent used in this manner was estimated to be very large. Vapor degreasing was observed to be a more sophisticated solvent cleaning method providing much greater quality in cleaning and some capability of separating the solvent from the accumulated soils. It was noted that well-designed and well-operated open top degreasers could function with losses of only 0.25-0.50 pounds per hour per square foot of open top area. Enclosed vapor degreasers were reported to be more economical in the use of solvent on a tonnage of parts basis, although they often consume more solvent on an individual equipment basis. The enclosed and conveyorized vapor degreaser designs often solve the problem of handling parts which would ordinarily trap solvent and cause large solvent drag-out losses.

Recent concern by customers for preventing pollution has resulted in an increased ratio of stills to vapor degreasers so that the ratio is about 1:1 at present. Phillips reported that they manufacture 200 medium or larger vapor degreasers per year. Approximately 60% of these are of the open top design. Four different vapor control methods are used on their vapor degreasing equipment. They are: 1) thermostat control; 2) direct refrigeration; 3) a freeboard chiller (water over 32°F), and, 4) standard condenser water coils with refrigerated coils above them ("the cold trap"). Of these methods they reported that "the cold trap" method is the most effective in controlling solvent losses but causes problems with excessive water contamination. Don Racquet also observed that the OSHA guidelines for ventilation require excessive ventilation rates (75 to 250 cfm) resulting in higher solvent losses than necessary.

Both carbon adsorbtion and refrigerated coils are employed on vapor degreasing equipment to control solvent emissions. Phillips knew of no practical applications of liquid absorption or incineration being used on either petroleum solvents or halogenated solvents. In fact, no known emission control techniques are being employed in connection with cold cleaning operations. Phillips Manufacturing sells six to ten carbon adsorption units per year. They forecast that a 50% solvent recovery can be expected and that 60% solvent recovery can be obtained at some locations. Stabilizers would be expected to be lost when the solvent recovery efficiency reached 80% or greater, even in the case of trichloroethylene. It was noted that 1,1,1-trichloroethane has stabilizer difficulty at much lower solvent recovery rates by carbon adsorption.

Phillips indicated that their customers lean toward vapor degreasing as opposed to alkaline washing. The cause for this tendency was reported to be economics as well as pollution considerations. Sources for additional information were reported to be available in: 1) Electroplating Engineering, published by Van Nostrum; 2) The Handbook of Industrial Loss Prevention, published by McGraw Hill, and, 3) The Welding Handbook - Sixth Addition, Section Four, published by The American Welding Society.