EGG-WTD--9297
IE91 006293

EGG-WTD-9297

SOLVENT RECYCLE/RECOVERY
PHASE I FINAL REPORT

M. W. Paffhausen
D. L. Smith
S. N. Ugaki

Published September 1990

EG&G Idaho, Inc.
Idaho Falls, Idaho 83415

Prepared for the
U.S. Department of Energy and
United States Air Force
Through the U.S. Department of Energy
Idaho Operations Office
Contract No. DE-AC07-76ID01570

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED
p2

ABSTRACT

This report describes Phase I of the Solvent Recycle/Recovery Task of the DOE Chlorinated Solvent Substitution Program for the U.S. Air Force by the Idaho National Engineering Laboratory, EG&G Idaho, Inc., through the U.S. Department of Energy, Idaho Operations Office. The purpose of the task is to identify and test recovery and recycling technologies for proposed substitution solvents identified by the Biodegradable Solvent Substitution Program and the Alternative Solvents/Technologies for Paint Stripping Program with the overall objective of minimizing hazardous wastes. A literature search to identify recycle/recovery technologies and initial distillation studies has been conducted.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

SUMMARY

Solvents are utilized for cleaning, stripping, and various other maintenance operations of aircraft parts and equipment. After use, many of the solvents can be recovered and reused. However, several of the solvents currently in use are chlorinated and emit volatile organic compounds, which are toxic to the environment and to operating personnel. Wastes generated from these solvents are regulated by the U.S. Environmental Protection Agency, and soon use and manufacture of the solvents may be restricted. Two programs that are a forerunner to this task and address substitution of these solvents with less toxic, non-chlorinated solvents are the Biodegradable Solvent Substitution Program and the Alternative Solvents/Technologies for Paint Stripping Program.

The objective of the Solvent Recycle/Recovery Task of the DOE Chlorinated Solvent Substitution Program is to minimize hazardous wastes by identifying recycle/recovery techniques for the proposed substitute solvents. The task is being conducted by EG&G Idaho, Inc., at Idaho National Engineering Laboratory and Montana State University through the U.S. Department of Energy, Idaho Operations Office.

Phase I objectives included conducting a literature search to identify techniques applicable to solvent recovery and recycle, including methods currently in use, and to initiate studies on the substitute solvents identified by the Biodegradable Solvent Substitution Program and the Alternative Solvents/Technologies For Paint Stripping Program. Phase II activities will include review of the literature obtained and review of solvent information, characterization of the solvents, distillation studies (continued from Phase I), collection of information on vendors, and initiation of laboratory studies of other techniques (i.e., membrane, extraction and ultrafiltration) etc. at INEL. Phase III of this task will include field scale verification studies of the techniques identified from Phase II.

The Phase I literature search conducted resulted in the gathering of patents, articles, reports, and conference papers on technologies applicable to solvent recovery and recycle. Several techniques, such as distillation, ultrafiltration, separation by organic and inorganic membranes, extraction, and molecular sieve technology, deserve further attention. As distillation is the most widely used method of solvent recovery, initial studies on the commercial grade solvents using distillation were conducted. These studies were conducted at Montana State University and provide baseline data to determine the feasibility of this technology for the substitute solvents. The replacement solvents are mostly multi-component and include terpenes, esters, glycols, alcohols, alkalis, and acids. Phase I tests indicate that distillation can accomplish recovery of key components of the solvents but not necessarily in the percentages reported by the manufacturer. Many of the solvents have high boiling components that will require vacuum distillation to prevent thermal decomposition.

CONTENTS

ABSTRACT	iii
SUMMARY	iv-v
1. INTRODUCTION	1
1.1 Objective	1
1.2 Background	2
1.3 Scope	3
1.3.1 Phase I: Data Gathering And Preliminary Screening	3
1.3.1.1 Activity 1: Literature Search	3
1.3.1.2 Activity 2: Information Gathering From Other Sources	3
1.3.1.3 Activity 3: Subcontract Agreement	3
1.3.1.4 Activity 4: Sample Accumulation	4
1.3.2 Phase II: 1.3.2.1 Activity 1: Literature Information/ Solvent Information Review	4
1.3.2.2 Activity 2: Solvent Characterization	4
1.3.2.3 Activity 3: Distillation Studies	4
1.3.2.4 Activity 4: Vendor Information	4
1.3.2.5 Activity 5: INEL Studies	4
1.3.3 Phase III: Field Scale Verification	5
2. TEST PROCEDURES: DISTILLATION STUDIES	6-8
3. PHASE I RESULTS	9
3.1 Activity 1: Literature Search	9
3.2 Activity 2: Information Gathering From Other Sources	9
3.3 Activity 3: Subcontract Agreement	9
3.4 Activity 4: Sample Accumulation	10-11
4. CONCLUSIONS	12
5. RECOMMENDATIONS	13
5.1 Laboratory Investigation of Spent Solvents	13
5.2 Bench Scale Field Verification Studies	13
5.3 Pilot Unit Proposal	13
5.4 Site Visits	14
5.5 Subcontracts	14
5.6 Economic Evaluation	14
5.7 Construction of Pilot Unit(s)	14
5.8 Data Input	14
6. REFERENCES	15
APPENDIX A LITERATURE REPORT	17-30
APPENDIX B MONTANA STATE UNIVERSITY DATA	31-60

1. INTRODUCTION

The utilization of solvents is integral in metal preparation and cleaning operations and is a part of many aircraft maintenance operations. Currently, chlorinated solvents are used in many of these processes. These solvents emit volatile organic compounds, which are hazardous to the environment and to operating personnel. The waste from cleaning operations such as degreasing, dewaxing, and paint stripping, contain toxic ingredients that require special handling and must be disposed of as hazardous waste.

Concerns for worker safety, environmental concerns, and impending imposition of stricter U.S. Environmental Protection Agency regulations has prompted investigation into replacing the solvents currently used with less toxic, non-chlorinated solvents. Economic factors necessitates the ability to treat, recover, and/or recycle these solvents.

This task is an outgrowth of the Department of Energy (DOE) Chlorinated Solvent Substitution Program and reports the initiation of a study of techniques applicable to solvent recovery and recycling. This work is being conducted at Idaho National Engineering Laboratory (INEL) and at Montana State University and is part of a collaborative effort by the Department of Energy and the U.S. Air Force Engineering and Services Center (AF-ESC).

1.1 OBJECTIVE

The objective of the Solvent Recycle/Recovery task is to identify and test available technologies to recover and recycle solvents proposed for use in production and maintenance operations. Solvent recovery techniques are applied in industry and at government facilities to single component

solvents such as methyl ethyl ketone, freon, and 1,1,1 trichloroethane. Distillation is the most common technique used. However, many of the potential solvent replacements are not single components and therefore might not be conducive to distillation. Also, some of the constituents may be consumed in the cleaning or paint stripping operations. Economic factors require that these solvents be recycled or recovered. Examples of the technologies that could be applicable are distillation, reverse osmosis, molecular sieves, and chemical separation. An investigation to determine the applicability of emerging technologies is also being conducted. A hybrid process employing several different technologies may have to be utilized to accomplish recovery and recycle.

1.2 BACKGROUND

Recovery and recycle is being performed at Air Force installations on solvents such as trichloroethane, Freon-113 (trade name for 1,1,2 trichloro-1,2,2, trifluoroethane), and isopropyl alcohol. Typically, the trichloroethane is contaminated with wax, oil and grease, and dirt. Freon-113 is contaminated with sealant and oil; isopropyl alcohol, with oil and dirt. These different solvents can be recycled in the same atmospheric pressure still on a batch basis. Benefits include cost savings, manpower savings, and reduced environmental impact, as recycling eliminates the need for disposal and future environmental liability that could be associated with disposal. Recovery percentages of 70, 95 and 95, for trichloroethane, Freon 113, and isopropyl alcohol, respectively, have been reported.

Distillation, as in the above example, is the most widely used process for solvent recovery and recycle. Applications involve atmospheric distillation, vacuum distillation, and solar distillation. Another process currently used involves the use of extraction techniques such as with acetone and water, using a dessicant column to remove the water.

1.3 SCOPE

1.3.1. Phase I: Data Gathering and Preliminary Screening

Phase I goals of this task were to identify techniques potentially applicable to solvent recovery and recycle, including methods currently in use, and to initiate studies on the substitute solvents identified by the Biodegradable Solvent Substitution Program and the Alternative Solvents/Technologies For Paint Stripping Program. Several activities contributed to accomplishing these goals.

1.3.1.1. Activity 1: Literature Search. A literature search was conducted to find information on applicable technologies. Information was gathered on established technologies, innovations of these technologies, and emerging technologies.

1.3.1.2. Activity 2: Information Gathering From Other Sources. Valuable information was gathered from sources such as the Final Reports from Phase I and Phase II of the Biodegradable Solvent Substitution Program, and the Final Report from the Alternative Solvents/Technologies For Paint Stripping Program. Information was also gathered through consultation with other researchers who provided technical information as well as DOE and DOD reports and operations information.

1.3.1.3 Activity 3: Subcontract Agreement A subcontract agreement was made with Montana State University for initiation of solvent recovery studies. This work includes an evaluation of commercial solvents to establish the feasibility of their recovery by distillation. This data was gathered to establish baseline data for the distillation studies to be conducted on the spent solvents.

1.3.1.4. Activity 4: Sample Accumulation Fifteen commercial solvents have been identified for the recycle/recovery testing. Ten solvents were identified from the Alternative Solvents/Technologies For Paint Stripping Program and the remaining five were identified from the Biodegradable Solvent Substitution Program. The replacement solvents are mostly multi-component and include terpenes, esters, glycols, alcohols, alkalis, and acids. Vendors of these solvents were requested to send one-gallon samples of the pure solvents to Montana State University for initial distillation studies.

1.3.2. Phase II

Phase II will involve a continuation or extension of Phase I activities as well as other activities.

1.3.2.1. Literature Solvent Information Review Further review of the literature gathered on the emerging technologies will be conducted, and the components in each individual solvent will be studied.

1.3.2.2. Solvent Characterization The spent solvents obtained from extended performance tests conducted at Tinker Air Force Base and INEL will be characterized.

1.3.2.3. Distillation Studies Montana State University will continue solvent evaluation and will assess distillation techniques that can be utilized to recover the spent solvents.

1.3.2.4. Vendor Information Vendors of system for products that are to be utilized in solvent recovery processes will be identified and contacted.

1.3.2.5. INEL Studies Tentatively, studies to be conducted at INEL include tests using membrane systems being developed at INEL, testing of commercial membrane systems, and extraction studies.

1.3.3. Phase III

Phase III of this task will include field scale verification tests of the technologies identified and construction of a demonstration unit. Specific tasks will be addressed in a later report.

2. TEST PROCEDURES:

Distillation studies on commercial grade solvents were identified for Phase I testing. Distillation is a mass transfer operation that separates the components of liquid mixtures containing miscible and volatile substances by vaporization into individual components or in some cases into groups of components. Some examples of distillations include the separation of: a mixture of alcohol and water into its components, liquid air into nitrogen, oxygen, and argon; and crude petroleum into gasoline, kerosene, fuel oil, and lubricating stock.

Simple fractional distillation is applicable when the liquid feed mixture to be separated forms no azeotrope(s) and the difference in the volatilities of its components is significant (i.e., usually meaning relative volatility values of 1.05 or greater). The components must be capable of vaporizing at feasible temperature and pressure. Also, no reaction can occur between components, nor can one or more of the components decompose or polymerize.

In some cases, distillation techniques other than simple fractional distillation must be used due to its ineffectiveness. Those cases are when (a) two or more of the components to be separated have only a slight, if any, difference in volatility (i.e., relative volatility is approximately equal to unity) (b) two or more of the components form homogeneous azeotrope(s) that are not pressure sensitive or are pressure sensitive but will not provide sufficient relative volatility change; and (c) one or more of the components will decompose or change chemically at temperatures and pressures well within economic distillation ranges. Fortunately, the technology of distillation has developed two modified forms that are now available for use: azeotropic and extractive distillation techniques. These methods utilize distillation with a third liquid, an entrainer (also called a withdrawing agent). The added liquid in an extractive distillation lowers the relative volatility of one of the constituents so as to lower the boiling point and bring it out at the base. The added liquid boils higher than any of the components being separated and comes off at the bottom with the higher boiling component. In azeotropic distillation, the added liquid increases the relative volatility of one constituent and brings it over the

head of the column. The third liquid comes off overhead with the constituents being separated at a slightly lower temperature.

The column used by Montana State University is packed with stainless steel helices and contains eight theoretical plates. The test procedure used in these studies is as follows:

1. Obtain one-gallon commercial grade samples from suppliers.
2. Visually observe samples on arrival. If they contain solids or sediment, filter off the solids. If they comprise two liquid phases, decant to separate the phases (each phase should then be distilled separately).
3. Rectify liquid samples in batch mode in rectification column. Have 8, 15, and 30 theoretical plate helices packed columns available. The column used will be determined based on the difference in the relative volatility encountered. If components of a solvent have a low relative volatility, more theoretical plates (increasing the surface area of the column) will be necessary to affect separation. Record and plot data as temperature of overhead percent of charge distilled. This will indicate the quality and boiling point of each constituent.
4. Repeat rectification and collect samples of the pure material as they come off. Check purity with a gas chromatograph and integrator.
5. Knowing the several compounds in the mixture, determine the relative volatility in the vapor-liquid equilibrium still between each adjacent pair.
6. On the adjacent pairs from #5, if the relative volatility is above 1.4, demonstrate separation in the rectification column of known theoretical plates in order to precisely determine the true relative volatility.

7. From all the samples received, identify which can be separated by straight rectification and which will require more intensive separation methods such as azeotropic or extractive distillation.

3. PHASE I RESULTS:

3.1. Activity 1: Literature Search

A literature search was conducted to gather information regarding techniques that have potential application to the recovery and/or recycle of solvents. Patents describing distillation techniques were found. Also, articles, reports, and conference papers on the emerging technologies of the large-scale use of organic and inorganic membranes were gathered; at least one article addressed the use of molecular sieves. The report on the literature is contained in Appendix A and will describe the technologies and the information found in each article relating to the technology. Summaries and abstracts are provided where applicable.

3.2. Activity 2: Information Gathering From Other Sources

The information gathered from other sources include the programs that this task supports and research consultants. This information is being used to help determine the thrust of this work. DOE and DOD documents used as a source of information will be included in the reference section of this document. Ideas generated or expounded upon in discussions with others will be considered in the conclusions section.

3.3. Activity 3: Subcontract Agreement

The results of the initial testing of the commercial grade solvents conducted as of this writing have been received from Montana State University and are found in Appendix B. Results show that some of the components can be separated by distillation and that many of the components have high boiling points and are better separated using vacuum rectification to prevent decomposition. The pressure associated with the system was measured at the still head. Volume percentage were collected as overhead at their specific critical boiling temperature. It is necessary to maintain 15-25% of the original charge in the still pot as bottoms when running the

column. The volume percentages remaining as bottoms are considered residue. A vacuum pump was used for the last runs to keep pressure at about 1 mm Hg pressure. Prior to this, at 640 mm Hg, the temperatures were too high and thermal degradation was occurring. The graphs show the critical temperatures and the volume percent of each component. The curve connecting these components indicates the "mid-fraction," during which a mixture of the two components are codistilled. Unknowns are indicated because of the limited amount of information given by the manufacturer. Verification of known components was obtained by gas chromatographic analyses.

3.4. Activity 4: Sample Accumulation

The samples identified and acquired for Phase I distillation tests are included in the following table:

<u>Company</u>	<u>Product</u>
<u>Paint Stripper Solvents</u>	
1. Chemical Methods	CM-3707
2. Chemical Solvents	SP-800
3. Fine Organics	FO 606
4. Frederick Gumm	Clepo Envirostrip 222
5. GAF	M-Pyrol
6. McGean-Rohco	Cee-Bee A245
7. McGean-Rohco	Cee-Bee A477
8. Patclin	126 Hot Stripper
9. Rochester Midland	PSS 600
10. Turco	T-5668

COMPANY
Biodegradable Solvents

- 11. Exxon
- 12. Bio-Tek
- 13. Orange-Sol
- 14. 3D
- 15. Fremont

PRODUCT

- Exxate 1000
- 140 Saf-Solv
- De-Solv-It
- 3D Supreme
- Fremont 776

4. CONCLUSIONS

As a result of Phase I activities, it can be concluded that there are many interrelated factors to be considered when determining the thrust of this work. This task depends on the results of testing in both the Biodegradable Solvent Substitution Program and the Alternative Solvent/Technologies Paint Stripping Program. It depends on the specific solvent identified, its particular solvent characteristics, and applications that determine specific solvent contaminants.

Phase I distillation testing indicates that for the commercial grade solvents, distillation can accomplish recovery of key components but not necessarily in the percentages reported by the manufacturer. Many of the solvents would require the use of vacuum rectification if these components are still intact after use in the various cleaning and paint stripping operations.

Results of the literature search and consultation with several researchers indicate that emerging membrane technologies, such as pervaporation, may be applicable to some solvents. Other solvents may be suitable for distillation while others may be conducive to extraction with subsequent use of a hybrid ultrafiltration/distillation process.

5. RECOMMENDATIONS

The results of the work to date show that there are several technologies that deserve attention in order to properly address this task of solvent recovery and/or recycle. Extensive review of the literature obtained, further laboratory investigation, contact with vendors, researchers and industrial experts, and other research will be needed to establish satisfactory data to determine technologies for full-scale implementation. The following subsections are recommendations for future work.

5.1. Laboratory Investigation of Spent Solvents

This task will involve laboratory investigations of the spent solvents generated in the paint stripping and parts cleaning operations. These tests will be used to determine the feasibility and effectiveness of the various technologies identified.

5.2. Bench Scale Field Verification Studies

These tests would be on-site verification of tests conducted in the laboratories at INEL and at Montana State University. This would include scaled-up performance testing of viable techniques for the individual solvents identified.

5.3. Pilot Unit Proposal

This task will include determination of a pilot scale site and the requirements for design and construction of large module unit(s). The unit will be capable of separating contaminants from different cleaning operations as well as recovering solvents or their key components. The proposal will contain complete background descriptions on the separation and recovery methods, results of previous laboratory testing, and a pilot model proposal including all technical and economic information.

5.4. Site Visits

Site visits will be made to obtain an exact location for set-up and to establish contacts necessary to facilitate module unit installation and start-up of operation. It may be necessary to visit industrial facilities that use these types of systems on a large scale.

5.5. Subcontracts

In association with the membrane studies, if there is a need to establish subcontracts with companies that develop and manufacture materials for polymer casting and housing and that are involved in the construction of module systems, it will be addressed in this task if such a need is identified.

5.6. Economic Evaluation

An economic evaluation will be conducted to gauge the feasibility and attractiveness associated with each technology.

5.7. Construction of Pilot Unit(s)

It is maintained that solvent recovery and recycle might involve several different technologies. One particular technology might work to recover a particular solvent or the combination of several different technologies may be the solution. This task will involve construction of demonstration units of the technology identified for the recovery/recycle of the different types of solvents.

5.8. Data Input

Results of testing should be included in the Biodegradable Solvent Substitution Handbook to increase awareness of recovery and recycle techniques for these solvents.

REFERENCES

J.M. Beller, G.S. Carpenter, R. E. McAtee, P.A. Pryfogle, D.F. Suci, P.M. Wikoff, T.L. Harris, R.K. Schober, Substitution of Wax and Grease Cleaners With Biodegradable Solvents Phase I Report, November 1988.

P.E. Boubel, W. Richard, Recovery, Reuse and Recycle of Solvents, December 1985.

A.A. Chavez, S.N. Ugaki, P.M. Wikoff, et al., Substitution of Cleaners with Biodegradable Solvents, Phase II, Extended Performance Testing, Final Report, November 1989.

M.N. Tsang, T.L. Harris, E.J. Mommens, B.D. Lee, P.M. Wikoff, and D. F. Suci, Alternatives Solvents/Technologies For Paint Stripping Phase I Draft Final Report

APPENDIX A
LITERATURE REPORT

APPENDIX A LITERATURE REPORT

Distillation

Distillation is broadly defined as the separation of more volatile materials from less volatile materials by a process of vaporization and condensation. In engineering terminology, the term distillation is reserved for the separation of two or more liquids by vaporization and condensation. It is the most widely used method of separating liquids\mixtures and is at the heart of the separation processes in many chemical and petroleum plants.

Basic distillation involves heating a liquid mixture, vaporizing part of the mixture, and removing the heat from the vaporized portion. The resulting condensed liquid, the distillate, is richer in the more volatile components. Simple distillation utilizes a single equilibrium stage to obtain separation. This stage may either batch (also called differential) or continuous (also called flash) distillation.

Other types of distillation include steam, azeotropic, and extractive. Steam distillation is used to lower the distillation temperatures of high boiling organic compounds that are essentially incapable of mixing or blending with water. Azeotropic distillation is a technique used to separate solutions of two or more liquids that are difficult to separate because of the closeness of boiling points. Extractive distillation is the use of distillation with a third, liquid referred to as an entrainer or a withdrawing agent.

Distillation columns (also called towers) furnish the contact area between streams of descending liquids and ascending vapor and thereby provide an approach toward physical vapor/liquid equilibrium. The contact may occur in discrete steps, called plates or trays, or in a continuous differential contacting on the surface of packing. The fundamental requirement of the column is to provide the mass transfer contacting at a required rate and at a minimum overall cost. Individual column requirements vary from light vacuum to high pressure, from low liquid to high liquid rates, from clean to dirty systems, etc. As a result, a large variety of equipment has been developed to fill these needs.

Distillation Patents

Burkhardt et al., Method and Apparatus For Reclaiming Solvents From Solvent-Bearing Sludge Material, U. S. Patent 4,157,281 (June 5, 1979).

A solvent-bearing sludge material is pumped by a variable speed positive displacement pump from a preparation mixing tank through a pressure

responsive valve, which distributes the material into the upper portion of an insulated distilling or processing vessel. Steam is introduced into the lower portion of the vessel and mixes with the material to volatilize the solvents, which are directed through a condenser and pump to a storage tank. Water and suspended solid particles are continuously pumped from the bottom of the vessel to an insulated settling tank where the solid particles settle and are continuously removed by a drag-out conveyor. The hot water in the upper portion of the settling tank is pumped back into the vessel and may have sufficient velocity to increase agitation within the vessel, or a mechanical agitator may be installed within the bottom portion of the vessel. The continuous feed of material into the vessel and the continuous removal of hot water and suspended particles from the vessel cooperate with continuous separation and removal of the solid particles and the return of the hot water to the vessel. This will eliminate agglomeration of the solids within the bottom portion of the vessel and provide for an effective and efficient solvent reclaiming process.

Claunch et al., Small Solvent Recovery Unit, U. S. Patent 4,488,833 (Dec. 18, 1985).

Disclosed herein is a process and apparatus for safely distilling relatively small batches of organic compounds such as paint solvents by directly and safely heating the compounds in a vessel. The vessel has a relatively thick bottom with electrical heating units cast in it and a relatively thick side wall integrally attached to the bottom. The side wall may be integrally attached to the bottom by a weld that penetrates the joint between the side wall and bottom preferably by 100% in order to provide a complete heat transfer from the bottom to the side wall. The vessel is designed to provide a relatively uniform temperature throughout the inner periphery of the vessel, thereby avoiding hot spots and ensuring that the hottest spot of the inner periphery of the vessel does not exceed the auto ignition temperature of the compound being processed. The process utilizes the thermal conductivity and thickness of the bottom and side wall of the vessel to avoid hot spots and to increase the area for heat transfer from the vessel to the organic compound being distilled. The vessel may be sealed and a vacuum provided in the vessel to enhance the evaporation. The vessel may be provided with a bag inside to remove residual material.

David Finlay-Maxwell, Distillation Method for Solvent Recovery, U. S. Patent 4,233,120 (Nov. 11, 1980).

An improved solvent recovery process distills soiled solvent in a main still and conducts hot solvent vapor through a heat exchanger associated with a tank in which an azeotropic mixture of water and soiled solvent is maintained, which boils at a temperature lower than the boiling point of the solvent alone.

A series of tanks feed soiled solvent to the azeotropic mixture tank and then to the main still, and heat exchangers fed by the vapor from the main still and the vapor from the azeotropic mixture tank pre-heat the soiled solvent. Automatic control of water injection into the azeotropic mixture tank depending on the temperature of conductivity of the tank contents maintains the composition of the azeotropic mixture, the injected water being taken from separators that separate water from condensate of the main still, and azeotropic mixture tank vapors.

Berth U. Gustafsson, Method for Controlling the Flow Through A Distillation Apparatus And A Device For Carrying Out The Method, U. S. Patent 4,201,631 (May 6, 1980).

A method and a device is invented for controlling the flow through a distillation apparatus for separating components of a mixture, particularly a dissolved substance, such as an oil, or a wax, a polishing agent, from a liquid mixture, particularly containing a chlorinated solvent, such as tri- or perchloroethylene. According to the present invention, the feeding of new liquid mixture or solution into the boiling vessel of the distillation apparatus is controlled in response to the liquid level in the boiling vessel, whereas the draining of substance-enriched liquid or separated substance from the boiling vessel is controlled in response to the distillation temperature, or to a co-variant variable, so as to maintain the distillation temperature within predetermined limits and the flow through the apparatus at an optimal, high level.

Frank Lucas, Waste Effluent Treatment and Solvent Recovery System, U. S. Patent 4,495,034 (Jan. 22, 1985).

This describes a system for treating waste effluent and recovering solids and solvent material. It requires a reservoir mounted on a stand connected to a solvent migrator which includes an adjustable trough for receiving new solution, a secondary reservoir with a cloth or absorbent material designed to contact the fluid in the secondary reservoir, while parallel to the sun's rays, an automatic leveling means and apparatus for recirculation of waste effluent including a leveling ball, and suction tubing for the recirculation of fluid. A solar unit is used comprising of glass panels to form a collecting dome with collecting troughs, a bonnet to shade the top of the pyramid formed by the dome panels, and mirrors for the reflection of sunlight. The whole system is connected to a chemical trap and subsequently a collecting means for the pure product.

Manual G. Pastor, Solvent Recovery Apparatus and Method, U. S. Patent 4,457,805 (July 3, 1984).

This describes an apparatus and a method for recovering clean solvent from a mixture of solvent and contaminants. The apparatus includes an open top tank mounted in a housing below a sink in which articles are cleaned with solvent. The sink has a tap through which solvent is directed so that the articles can be cleaned by hand in the sink itself. A disposable, plastic bag is receivable in the tank and the tank is closed by a cover. Contaminated solvent can flow from the sink, through the cover, and into a bag in the tank. Contaminated solvent in the bag is heated by heating a liquid surrounding the side wall of the tank and below the bottom of the tank so that the solvent in the bag will vaporize. A tube leading out of the tank carries solvent vapor into a heat exchanger where the vapor is condensed to form clean, liquid solvent, and the liquid solvent is collected in a reservoir. A pump is coupled to the reservoir for pumping liquid solvent to the tap at the sink. The heating means has an expansion chamber for allowing expansion of the heating liquid. The apparatus provides a solvent reclaiming and cleaning station near a location where articles to be cleaned are used.

Shenoy et al., Split-Column Extractive Distillation U. S. Patent 4,556,404 (Dec. 3, 1985).

The present invention is directed to an extractive distillation separatory technique wherein the improvement comprises a two stage column with a condenser only for the high pressure section and a reboiler only for the low pressure section, as well as a flash separation of a stream communicating between the sections.

Membrane Technology

In membrane technology, a membrane is a thin barrier that separates two fluids and permits selective transport of solutes from one fluid to the other fluid. The material can be organic polymer, metal, or ceramic; layer of chemicals; or a liquid or gas.

Membrane separation processes involve applying a driving force, such as pressure, concentration, or an electromagnetic field, across the membrane. This driving force allows specific components to pass through or permeate the membrane, and the fluid that is retained is the retentate, retentant, or concentrate.

Membrane separation unit systems can be distinguished according to type of driving force, molecular size of the retentate, or type of operation. The following are the types of basic membrane separation processes: reverse osmosis or hyperfiltration, ultrafiltration, diafiltration, microfiltration,

gas separation, pervaporation, dialysis, electrodialysis, electrosmosis, and membrane extraction.

Membrane Literature

Gary L. Amy, Bruce C. Alleman, Brent C. Cluff, "Removal of Dissolved Organic Matter by Nanofiltration," Journal of Environmental Engineering, Volume 116, Number 1, February 1990, pp. 200-205.

Nanofiltration (NF) represents a recent development in membrane technology. The principal objective in this study was to evaluate the organic matter rejection properties of the nanofilter. It examined the ability of the NF module to remove dissolved organic matter (DOM). Particular emphasis was placed on the removal of trihalomethane (THM) precursors since each of the candidate waters is considered a possible source of drinking water. The study showed that NF was effective in removing DOM from both a surface-water and a ground water source. The process represents a potential THM control strategy providing excellent rejection of high- and medium-molecular weight precursors (removal of divalent cations). An added benefit is partial demineralization and softening. Presently, a key constraint is pretreatment requirements.

Anderson, Bryce P., "Ultrafiltration Polyamide Membrane and its use for Recovery of Dewaxing Aid," U. S. Patent Number: 4, 908, 134 (March 13, 1990).

Polyimide ultrafiltration membranes made using a single solvent and containing a pore forming agent have been produced and have been found to be useful for recovering dewaxing aid used in a dewaxing process.

Bailey, P. A., Abcor Environmental Systems Ltd., Surbiton, Surrey, "The Treatment of Waste Emulsified Oils by Ultrafiltration," Filtration & Separation, January/February, 1977, pp. 53- 55.

Ultrafiltration involves the use of semi-permeable membranes, generally fabricated from polymeric materials containing micropores of only tens of thousands of a micron in diameter. When emulsified oil is applied to the membrane under slight pressure, water molecules pass easily through the pores but the much larger emulsified oil particles are retained. As well as describing the principle of the process, the paper outlines practical applications, factors affecting performance, and advantages of ultrafiltration.

Behling, R.D., Ohlrogge, K., Peinemann K.V. Kyburz, E., "The Separation of Hydrocarbons From Waste Vapor Streams," AICHE Symposium Series, Volume 85, Number 272, 1989, pp. 68-73.

Hydrocarbon vapors generated dispersed into the air from industrial processes contribute to photochemical smog. The separation of hydrocarbon vapor by means of membranes can be a technically simple and economic process. A membrane vapor separation process with a following treatment of the retentate by catalytic incineration is introduced in this paper.

Bitter, J. G. A., J. P. Huan, J. P., Rijkens, "Solvent Recovery Using Membranes In The Lubcoil Dewaxing Process," AICHE Symposium Series, Volume 85, Number 272, 1989, pp. 98-100.

A novel membrane has been developed for separating solvents from the oil/solvent mixture of the lubcoil dewaxing process. The membrane, which consists of a modified silicone rubber, shows a high flux (more than $1 \text{ m}^3/(\text{m}^2\text{-day})$ at 40 bar) and a selectivity varying between 50 and 100, depending on solvent dilution and grade of lubcoil. The membrane has now been in continuous operation on a laboratory scale for about four years, without showing a significant change of its properties. Economic evaluations indicate that the operation costs (including capital charges) of a membrane unit based on the use of this membrane are lower than the energy costs of the conventional separation by flashing.

Bitter, Johan George Albert, Shell Internationale Research Maatschappij B.V., Process for the Separation of Solvents from Hydrocarbons Dissolved in Said Solvents, European Patent Application Number: 86201362.0 (01.08.86).

This describes a process for the separation of solvents from hydrocarbons dissolved in the solvents by contacting a feed solution (e.g., obtained from a dewaxing unit) comprising hydrocarbons dissolved in an aromatic solvent and a polar aliphatic solvent (e.g., toluene and methyl ethyl ketone) at elevated pressure with one side of a dense membrane which is substantially impermeable to said hydrocarbons and which membrane comprises a layer of a halogen-substituted silicone compound comprising units according to the general formula:

where R_1 and R_2 represent units chosen from the group consisting of hydrogen, halogen, alkyl, aryl, aralkyl, and halogen-substituted alkyl, aryl and aralkyl units, and recovering said solvents from the other side of the membrane.

Cartwright, Peter S., "Reverse Osmosis and Ultrafiltration in the Plating Shop," Plating and Surface Finishing, April 1981, pp. 40-45.

The mechanisms, present applications, and potential uses of reverse osmosis (RO) and ultrafiltration (UF) for waste treatment in the plating shop are addressed in the paper. Commonly used membrane materials (including operating pH and temperature ranges) and chemical resistance properties for RO and UF are listed. Membrane configurations for RO and UF processes are described. The paper mentions potential applications for these processes in metal finishing.

Crampton, Peter and Wilmath, Roger, "Reverse Osmosis in the Metal Finishing Industry," Metal Finishing, March, 1982, pp 21-27.

Reverse osmosis (RO) technology has been used in the metal finishing industry to recover plating chemicals from rinse water as well as to purify mixed wastewater to allow its reuse. In plating chemical recovery applications, RO units separate the valuable metal salts from rinse solutions, which yields a concentrated metal solution that can be recycled to the plating bath and water that can be reused for rinsing.

The capacity of RO units used in electroplating applications ranges from a few thousand gallons per day to several hundred thousand gallons per day. The attractive features of RO systems include relatively low initial and operating costs compared to alternative separation processes, capability for continuous operation, simplicity of operation and maintenance, and minimal floor space requirements for the system installation.

Funk et.al., Membrane Separation of Hydrocarbons, U. S. Patent Number 4,617,126. (October 14, 1986).

Mixtures of heavy oils and light hydrocarbons may be separated by passing the mixture over a polymeric membrane that comprises a polymer capable of maintaining its integrity in the presence of hydrocarbon compounds at temperature ranging from about ambient to about 100°C, and pressures ranging from about 50 to about 1000 psi. The membranes possess pore sizes ranging from about 10 to about 500 angstroms, are cast from a solvent solution and recovered.

Hazlett, J. D., Kutowy, O., Tweddle, T. A., National Research Council of Canada, Ottawa, Ontario, Canada, "Processing Of Crude Oils With Polymeric Ultrafiltration Membranes," AIChE Symposium Series, Volume 85, Number 272, 1989, pp. 101-107.

Hydrocarbon vapors generated from industrial processes dispersed into air are contributing factors for the creation of photochemical smog. The separation of hydrocarbon vapor by means of membranes is in case of some applications a technically simple and economic process. A membrane vapor separation process with a following treatment of the retentate by catalytic incineration is introduced in this paper.

Isoonka, Y., Imamura, Y., Sakamoto, Y., "Recovery and Resue of Organic Solvent Solutions," Metal Finishing, June 1984, pp. 113-118.

This article describes the use of ultrafiltration membranes to recover solvent. The recent development of solvent-resistant ultrafiltration membranes offers another solution to the solvent recovery problem. It describes several ultrafiltration systems and operating parameters associated with ultrafiltration. Also provided are histories of the use of ultrafiltration technology in top-coat paint solvent reclamation, recovery of paint solvents, and treatment of printing ink waste streams.

Iwama, A. and Kazuse, Y., Nitto Electric Industrial Co. Ltd., Ibaraki, Japan, "New Polyimide Ultrafiltration Membranes for Organic Use," Journal of Membrane Science, Volume 11, 1982, pp. 297-309.

A solvent-resistant membrane for use in ultrafiltration processes has recently been developed by Nitto. The membrane is made from a new polyimide that can be synthesized from 1,2,3,4-butane tetracarboxylic acid and an aromatic diamine. This membrane exhibits excellent stability and high fluxes with most common organic solvents, even when tested at elevated temperatures. These membranes are very stable in a variety of organic solvents and may be used to separate, purify, or concentrate organic solutions that contain solutes of relatively large molecular sizes.

Kaschemekat, J., Wijmans, J. G., Baker, R. W., Blume, I., "Separation of Organics from Water Using Pervaporation," Proceedings of the International Conference on Pervaporation Process: Chemical Ind., 3rd, Edited by Bakish, Robert A., 1988, pp. 405-12.

For the past four years Membrane Technology and Research, Inc. (MTR), has been developing pervaporation membranes and systems to recover organic solvents from aqueous feed streams. Thin-film composite membranes are used to perform the separation. The membranes are incorporated into spiral-wound modules with a membrane area of 5 to 8 m². The standard units contain one to six PerVap modules and are equipped with feed stream heating systems and chillers for the condensers. Depending on the conditions, permeate fluxes from 0.5 to 5 kg/m²hr are obtained. The enrichment of polar solvents are 5- to 20-fold. Nonpolar solvents, such as ethyl acetate, 1,1,2-trichloroethane, or chloroform, are concentrated from 50- to 3000-fold. The MTR PerVap Systems have been demonstrated in the laboratory, and data from these experiments are used to calculate the economic feasibility of the technology. It offers significant advantages for streams containing 0.1--10% organic solvent, especially where recovery of the solvent is important.

Kulkarne, S. S., Funk, E. W., Li Norman, N. "Hydrocarbon Separations with Polymeric Membranes," AICHE Symposium Series, Volume 82, Number 250, 1986, pp. 78-84.

This paper presents a potentially new use of polymeric membranes for the separation of hydrocarbon mixtures. The focus is on the separation of hydrocarbons that are chemically similar but differ significantly in molecular weight. Specifically, new permeation data are presented on the use of ultrafiltration membranes for the energy-efficient recovery of low-molecular weight paraffinic solvents from high-molecular-weight oils. The study shows that ultrafiltration membranes can be stable in the hydrocarbon environment at least up to 50°C and can give fluxes of 10 to 15 gal/ft²-D) with solvent/oil selectivities of 3 to 10.

Leeper, Stephen A., Stevenson, Daniel H., Chiu Peter Y.-C. Stephen J., Sanchez, Herbert F. and Wikoff, Penny M., Membrane Technology and Applications: An Assessment, February 1984

EG&G Idaho, Inc., has reviewed and assessed the literature on membrane technology and applications. This is a report of that assessment and has two purposes: to support current and future efforts of the Department of Energy, Industrial Programs (in Conservation and Renewable Energy), in membrane technology development for energy conservation and productivity improvement; and to provide a resource document on membrane technology and applications to persons interested in supporting, studying, or applying this technology. The report provides an overview of membrane technology, reviews potential applications of membrane technology in several industries, identifies technological deficiencies, and recommends areas for future research and development (R&D). Applications and R&D that will contribute to energy conservation and productivity improvements are emphasized.

In the overview of membrane technology, the basic concepts of membrane separations are discussed and the various membrane unit operations are described. Operations, membrane materials and equipment, and engineering aspects are also discussed.

The industries considered in the review of membrane applications include mining, primary metal recovery, and fabricated metal products; food processing; textile and leather products; pulp and paper products; hydrocarbon (including synfuels) extraction and refining; the chemical process industries (inorganic chemicals processing, organic chemicals processing, pharmaceutical/biotechnology products); and medical and health care industry. In addition, application in domestic, municipal, and commercial water treatment and energy conversion are viewed.

The assessment identifies promising applications for energy conservation and productivity improvement in various industries. Six generic process applications are identified: displacement of energy-intensive separations, recycle of sensible heat, recycle of materials, recovery of materials, chemical conversion, and energy conversion. In addition, seven areas of technology deficiency are

identified: insufficient standard materials limitations; operation limitations; lack of well-developed engineering methods for manufacturing, scale-up, and process monitoring; insufficient data base; and high costs for new materials and new applications. Each of these areas is discussed. Finally, the following R&D areas are recommended and discussed: standardization of terms and methods, basic science and engineering, materials development, engineering development, and applications development.

The report concludes that many of the problems presently inhibiting expanded use of membrane technology can be overcome.

Paul, Helmut, Daimler-Benz AG, Mercedesstr. 136, 7000 Stuttgart 60, W. Germany, "Recycling of Detergents After Ultrafiltration of Cleaning Solutions," Desalination, Volume 41, 1989, pp. 313-325.

The experiments have shown that recycling detergents after separation by an UF process is possible without any risks to production. Considerable savings in operating costs as well as a simultaneous discharge of the wastewater can be expected.

As a result of the experience gained, the range of application of membrane technology, in this case UF, has been extended to an important range of application, which is of major importance for the future.

Tests with respect to optimizing the recovery of detergent substances by changing the hydraulic values of UF systems are being conducted out in collaboration with the Institute for Chemical Engineering at Aachen.

Peter, S. and Stefan, R., "The Separation of Organic Solvents and Phenols from Aqueous Solutions By Means of Chemically Stable Asymmetric Membranes Derived From PVA," Proceedings of the 7th International Symposium on Fresh Water from the Sea, Volume 2, 1980, pp. 197-206.

Asymmetric membranes were made of polyvinylalcohols of a suitable molecular weight by the phase-inversion procedure. The precipitated membranes were then made insoluble in water and resistant to solvents by cross-linking, adding compounds with one or two carbonyl groups in the molecule as well as dicarboxyl compounds. During this process it was possible to retain the "primary" asymmetry entirely. The cross-linked membranes exhibit good chemical, mechanical, and thermal resistance in a pH range of 1 to 14. The membranes were tested in reverse-osmosis experiments in aqueous solutions of phenols, methyl isobutyl ketone, and acetic acid esters. The retention and permeability properties in these solvents are reported.

Rautenbach, R., Herion, C., Franke, M., Dehydration of Multicomponent Organic Systems by a Reverse Osmosis Pervaporation - Hybrid Process .
Module-, Process Design and Economics Elsevier Science Publishers, B. V.
Amsterdam, pp. 445-453.

Presently the commonly applied technique for the removal of solvents from polluted air streams is adsorption on active coal, for example paint drying ovens. The necessary regeneration of the active coal by steam stripping results, after condensation and phase separation, in an almost pure solvent mixture, which can be recycled, and in a water-rich phase, which has to be treated further before discharge.

Since the solvents are low-boilers as well as high-boilers with respect to water, and some, in addition, form azeotropes, distillation of this desorbate is complex and expensive. Membrane processes could be advantageous in this case. In cooperation with a German car manufacturer, such a process has been developed, essentially consisting of a combination of RO and PV.

After a brief description of the whole process, the reverse osmosis and the pervaporation stage will be discussed in detail. The economics of this process will be discussed and compared with the costs for alternative disposal concepts.

Roy, Kimberly A., "Increasing Environmental Consciousness," Hazmat World, November, 1989, pp. 32-33.

This article describes how Lockheed Aeronautical Systems Co. (LASC), one of the world's largest producer of military aircraft, initiated a broad environmental protection effort, which included on-site solvent recycling. The lubricant is recycled at three points at the Burbank operations with a portable system supplied by Fluid Recycling of Santa Ana, California. The portable system uses filtration and centrifugation to separate the lubricant from small metal pieces and dirty oil collected by the fluid as it lubricates the cutting machines. The system allows LASC to re-use about 90% of its coolant (10% evaporates) and to realize a return on the tramp oil and metal pieces while eliminating the risk of a spill or accident during transportation. The article also describes an Aqua-Detox facility used in clean-up of contaminated groundwater. The system was designed by San Francisco-based AWD Technologies, an environmental services contractor, to treat groundwater and soil gases without releasing volatile organic compounds (VOCs). Instead, the VOCs are contained and recycled.

Thompson, John A. Selective Extraction Solvent Recovery Using Regenerated Cellulose Membrane Under Reverse Osmosis Conditions, U. S Patent Number 4,510,047 (April 9, 1985).

Selective extraction solvents such as NMP, phenol, or furfural employed for the extraction of specialty oils (i.e., lubricating,

transformer, and insulating oils) to remove undesirable aromatic components, is itself recovered from the extract stream and/or raffinate streams by preferential ultrafiltration through selective membranes of regenerated cellulose. The extract stream, because of its higher solvent content, is the preferred stream for solvent recovery treatment by the reverse osmosis membrane permeation technique. Solvent recovery using membrane permeation exhibits the advantage of not being as energy intensive as distillation or stripping. The solvent is recovered at a high enough flux rate and at a high enough level of purity to be introduced back into the solvent extraction process optionally at some point in the process where the composition of the recovered solvent approximately matches the composition of the original solvent.

A variety of commercial and NRC developed polysulfone membranes were evaluated for the processing of crude oil feedstocks. The products of the process are a permeate, which is the equivalent of a gas oil, and a concentrate enriched in asphaltenes. A systematic study of the effects of temperature, pressure and circulation flow has been completed for Mydale crude using a plate and frame membrane module. The establishment of a gel layer on the membrane surface has been demonstrated. The influence of this dynamic gel-polysulfone composite membrane on the flux and separation characteristics is discussed.

Wijmans, J. G. and Helm, V. D., "A Membrane System For the Separation and Recovery of Organic Vapors From Gas Streams," AICHE Symposium Series, Volume 85, Number 272, 1989, pp. 74-79.

A newly developed membrane process to treat airstreams contaminated with volatile organic compounds is described. The process uses multilayer composite membranes that are selective for organic vapors over nitrogen and oxygen. The membranes are assembled into spiral-wound modules specifically designed for this particular membrane application. A pilot plant containing 15 m² of membrane area has been constructed and is being operated at Membrane Technology Research's facilities. The membrane removes organic vapors from air streams and produces a secondary stream enriched in the organic. The organic is recovered from this stream by condensation. The system is best suited for applications where the organic vapor content is too high to make carbon adsorption economically feasible, but too low to permit incineration. The membrane process is also capable of removing organic vapors from gas streams other than air, such as natural gas streams.

Molecular Sieves

Molecular sieve zeolites are crystalline aluminosilicates of Group IA and Group IIA elements, such as sodium, potassium, magnesium, and calcium. Structurally, they are complex, crystalline inorganic polymers based on an infinitely extending framework of AlO_4 and SiO_4 tetrahedra linked to each other by the sharing of oxygen ions. The specific pore sizes of the crystals enable purification of gases by permitting passage of small gas molecules while blocking and/or absorbing others such as hydrocarbons, which are too large to pass through the pores. In other applications, the material may be used to absorb the small molecules that enter the pores while rejecting the ones that are too large.

Mathews, Golde, and Schweitzer, Stefan, Procedure and Device for the Separation and Recovery of Volatile Solvents, Australian Patent Office, Document No. AU-B-78321/87 (March 8, 1989).

Procedure for the separation and recovery of volatile solvents from water-vapor containing exhaust air from dry-cleaning machines, metal degreasing installations, installations for the cleaning of electronic conductor plate-, solvent baths, and similar installations. The exhaust air is led through regeneratable molecular sieves and then discharged into the atmosphere. The exhaust air is first let through a molecular sieve packing, which adsorbs only the water vapor, and subsequently through a molecular sieve packing, which only absorbs the solvent vapors, and both molecular sieve packings are regenerated independent of each other by means of air or inert gas. The exclusively water vapor adsorbing molecular sieve packing is regenerated to the atmosphere, and the exclusively solvent adsorbing molecular sieve packing is a closed circuit. The solvent vapors are condensed within the closed circuit in a condensation heat exchanger and, after collection, and returned to be reused.

APPENDIX B
MONTANA STATE UNIVERSITY DATA

TEST CONDITIONS:

Condition: 8 T. P. Helices packed
column. 400 ml charged

Distillation Procedure Simple Batch x Extractive Azeotropic

<u>Water</u>	<u>30</u>	<u>°C</u>	<u>4</u>	<u>%</u>
<u>N-Methyl pyrrolidone</u>	<u>73</u>	<u>°C</u>	<u>34</u>	<u>%</u>
<u>Tetrahydrofurfuryl alcohol</u>	<u>76</u>	<u>°C</u>	<u>6</u>	<u>%</u>
<u>Diethanolamine</u>	<u>141</u>	<u>°C</u>	<u>25</u>	<u>%</u>
		<u>°C</u>		<u>%</u>

32

0-8130

0-8131

SOLVENT RECYCLE/RECOVERY TASK

<u>N-Methyl pyrrolidone</u>	<u>194</u>	<u>°C</u>	<u>62</u>	<u>%</u>
		°C		%
		°C		%
		°C		%
		°C		%

0-8129

TEST CONDITIONS:

Condition: 8 T. P. Helices packed
column. 500 ml charged

Distillation Procedure Simple Batch x Extractive Azeotropic

Recovered Component, Critical Temperature, and Volume % from GC Analysis

	°C	%
1	100	100
2	100	100
3	100	100
4	100	100
5	100	100
6	100	100
7	100	100
8	100	100
9	100	100
10	100	100
11	100	100
12	100	100
13	100	100
14	100	100
15	100	100
16	100	100
17	100	100
18	100	100
19	100	100
20	100	100
21	100	100
22	100	100
23	100	100
24	100	100
25	100	100
26	100	100
27	100	100
28	100	100
29	100	100
30	100	100
31	100	100
32	100	100
33	100	100
34	100	100
35	100	100
36	100	100
37	100	100
38	100	100
39	100	100
40	100	100
41	100	100
42	100	100
43	100	100
44	100	100
45	100	100
46	100	100
47	100	100
48	100	100
49	100	100
50	100	100
51	100	100
52	100	100
53	100	100
54	100	100
55	100	100
56	100	100
57	100	100
58	100	100
59	100	100
60	100	100
61	100	100
62	100	100
63	100	100
64	100	100
65	100	100
66	100	100
67	100	100
68	100	100
69	100	100
70	100	100
71	100	100
72	100	100
73	100	100
74	100	100
75	100	100
76	100	100
77	100	100
78	100	100
79	100	100
80	100	100
81	100	100
82	100	100
83	100	100
84	100	100
85	100	100
86	100	100
87	100	100
88	100	100
89	100	100
90	100	100
91	100	100
92	100	100
93	100	100
94	100	100
95	100	100
96	100	100
97	100	100
98	100	100
99	100	100
100	100	100

0-8138

DATA ACCUMULATION SHEET
CHLORINATED SOLVENT SUBSTITUTION PROGRAM
SOLVENT RECYCLE/RECOVERY TASK

TEST CONDITIONS:

Parameter: 640 mm Hg

Condition: 8 T. P. Helices packed
column.

Company Name McGEAN-ROHCO

Solvent Name CEE BEE A-477

Distillation Procedure Simple Batch x Extractive Azeotropic

Recovered Component, Critical Temperature, and Volume % from GC Analysis

<u>Ethanolamine</u>	<u>163</u> °C	<u>50</u> %
<u>Unknown</u>	<u>187</u> °C	<u>16</u> %
<u> </u>	<u> </u> °C	<u> </u> %
<u> </u>	<u> </u> °C	<u> </u> %
<u> </u>	<u> </u> °C	<u> </u> %

0-8136

TEST CONDITIONS:

Condition: 8 T. P. Helices packed column.

Solvent Name 126 HOT STRIPPER

Distillation Procedure Simple Batch x Extractive Azeotropic

Recovered Component, Critical Temperature, and Volume % from GC Analysis

Water	30 °C	20 %
2-Butoxyethoxy-2-ethanol	107 °C	51 %
	°C	%
	°C	%
	°C	%

0-8132

CHLORINATED SOLVENT SUBSTITUTION PROGRAM

TEST CONDITIONS:

Condition: 8 T. P. Helices packed column.

Solvent Name PSS-600

Distillation Procedure Simple Batch x Extractive Azeotropic

Recovered Component, Critical Temperature, and Volume % from GC Analysis

N-Methyl pyrrolidone	77 °C	80 %
	°C	%
	°C	%
	°C	%
	°C	%

0-8128

0-8133

DATA ACCUMULATION SHEET

CHLORINATED SOLVENT SUBSTITUTION PROGRAM

SOLVENT RECYCLE/RECOVERY TASK

TEST CONDITIONS:

Parameter: 1 mm Hg

Condition: 8 T. P. Helices packed column.

Company Name EXXON

Solvent Name EXXATE 1000

Distillation Procedure Simple Batch x Extractive Azeotropic

Recovered Component, Critical Temperature, and Volume % from GC Analysis

Nonyl acetate	101 °C	49 %
---------------	--------	------

Undecyl acetate	103 °C	34 %
-----------------	--------	------

0C %

_____ °C _____ %

_____ °C _____ %

0-8137

0-8135

CHLORINATED SOLVENT SUBSTITUTION PROGRAM

TEST CONDITIONS:

Condition: 8 T. P. Helices packed column.

Solvent Name DE-SOLV-IT

Recovered Component, Critical Temperature, and Volume % from GC Analysis

Mineral oil - hydrocarbons	90-120 °C	90 %
----------------------------	-----------	------

	°C	%
--	----	---

_____ °C _____ %

0C %

0-8134

DATA ACCUMULATION SHEET

CHLORINATED SOLVENT SUBSTITUTION PROGRAM

SOLVENT RECYCLE/RECOVERY TASK

TEST CONDITIONS:

Parameter: 1 mm Hg

Condition: 8 T. P. Helices packed column.

Company Name 3-D

Solvent Name 3-D SUPREME

Distillation Procedure Simple Batch x Extractive Azeotropic

Recovered Component, Critical Temperature, and Volume % from GC Analysis

Water	30 °C	86 %
-------	-------	------

_____ °C _____ %

_____ °C _____ %

	0C	%
1. 1. 1. 1. 1.		
2. 2. 2. 2. 2.		
3. 3. 3. 3. 3.		
4. 4. 4. 4. 4.		
5. 5. 5. 5. 5.		
6. 6. 6. 6. 6.		
7. 7. 7. 7. 7.		
8. 8. 8. 8. 8.		
9. 9. 9. 9. 9.		
10. 10. 10. 10. 10.		
11. 11. 11. 11. 11.		
12. 12. 12. 12. 12.		
13. 13. 13. 13. 13.		
14. 14. 14. 14. 14.		
15. 15. 15. 15. 15.		
16. 16. 16. 16. 16.		
17. 17. 17. 17. 17.		
18. 18. 18. 18. 18.		
19. 19. 19. 19. 19.		
20. 20. 20. 20. 20.		
21. 21. 21. 21. 21.		
22. 22. 22. 22. 22.		
23. 23. 23. 23. 23.		
24. 24. 24. 24. 24.		
25. 25. 25. 25. 25.		
26. 26. 26. 26. 26.		
27. 27. 27. 27. 27.		
28. 28. 28. 28. 28.		
29. 29. 29. 29. 29.		
30. 30. 30. 30. 30.		
31. 31. 31. 31. 31.		
32. 32. 32. 32. 32.		
33. 33. 33. 33. 33.		
34. 34. 34. 34. 34.		
35. 35. 35. 35. 35.		
36. 36. 36. 36. 36.		
37. 37. 37. 37. 37.		
38. 38. 38. 38. 38.		
39. 39. 39. 39. 39.		
40. 40. 40. 40. 40.		
41. 41. 41. 41. 41.		
42. 42. 42. 42. 42.		
43. 43. 43. 43. 43.		
44. 44. 44. 44. 44.		
45. 45. 45. 45. 45.		
46. 46. 46. 46. 46.		
47. 47. 47. 47. 47.		
48. 48. 48. 48. 48.		
49. 49. 49. 49. 49.		
50. 50. 50. 50. 50.		
51. 51. 51. 51. 51.		
52. 52. 52. 52. 52.		
53. 53. 53. 53. 53.		
54. 54. 54. 54. 54.		
55. 55. 55. 55. 55.		
56. 56. 56. 56. 56.		
57. 57. 57. 57. 57.		
58. 58. 58. 58. 58.		
59. 59. 59. 59. 59.		
60. 60. 60. 60. 60.		
61. 61. 61. 61. 61.		
62. 62. 62. 62. 62.		
63. 63. 63. 63. 63.		
64. 64. 64. 64. 64.		
65. 65. 65. 65. 65.		
66. 66. 66. 66. 66.		
67. 67. 67. 67. 67.		
68. 68. 68. 68. 68.		
69. 69. 69. 69. 69.		
70. 70. 70. 70. 70.		
71. 71. 71. 71. 71.		
72. 72. 72. 72. 72.		
73. 73. 73. 73. 73.		
74. 74. 74. 74. 74.		
75. 75. 75. 75. 75.		
76. 76. 76. 76. 76.		
77. 77. 77. 77. 77.		
78. 78. 78. 78. 78.		
79. 79. 79. 79. 79.		
80. 80. 80. 80. 80.		
81. 81. 81. 81. 81.		
82. 82. 82. 82. 82.		
83. 83. 83. 83. 83.		
84. 84. 84. 84. 84.		
85. 85. 85. 85. 85.		
86. 86. 86. 86. 86.		
87. 87. 87. 87. 87.		
88. 88. 88. 88. 88.		
89. 89. 89. 89. 89.		
90. 90. 90. 90. 90.		
91. 91. 91. 91. 91.		
92. 92. 92. 92. 92.		
93. 93. 93. 93. 93.		
94. 94. 94. 94. 94.		
95. 95. 95. 95. 95.		
96. 96. 96. 96. 96.		
97. 97. 97. 97. 97.		
98. 98. 98. 98. 98.		
99. 99. 99. 99. 99.		
100. 100. 100. 100. 100.		

_____ °C _____ %

Remarks: Active ingredients contained in the remaining 14% residual charge.

END

DATE FILMED

02 / 04 / 91