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RECOVERY OF NAVY DISTILLATE FUEL FROM RECLAIMED PRODUCT

Technical Discussion

By
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November 1984
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RECOVERY OF NAVY DISTILLATE FUEL FROM RECLAIMED PRODUCT

Volume I
Technical Discussion

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PREFACE

In 1971, the Bartlesville Energy Research Center (BETC) (now the National Institute for Petroleum and Energy Research or NIPER) initiated a comprehensive research program with the objectives of developing efficient methods for reclaiming used lubricating oils and other waste hydrocarbon liquids; developing or adapting simple laboratory tests to evaluate the quality of reclaimed products; and providing specifications to help improve the marketability of recycled hydrocarbon liquids.

The U.S. Navy provided funds for this project to BETC through an Interagency agreement. During the first phase BETC became a not-for-profit research institute operated by IIT Research Institute for the Department of Energy. The work has been continued at NIPER under contract DE-AC19-84BC10823.

Phase I has revealed the potential for recycling Reclaimed Product (RP) into Naval Distillate Fuel (F-76). With the delineation of appropriate technology, potentially millions of gallons of Navy diesel fuel can be recycled rather than used as burner fuel.

Because of the large volume of information included in the report of our Phase I investigations, the report is being published in two volumes. Volume 1, Technical Discussion, includes the narrative and Appendices I and II. Appendix III, a detailed Literature Review, includes both a narrative portion and an annotated bibliography containing about 800 references and abstracts. For reader convenience, this appendix has been published separately as Volume 2.

ABSTRACT

In an effort to assist the Navy to better utilize its waste hydrocarbons, NIPER, with support from the U.S. Department of Energy, is conducting research designed to ultimately develop a practical technique for converting Reclaimed Product (RP) into specification Naval Distillate Fuel (F-76). This first phase of the project was focused on reviewing the literature and available information from equipment manufacturers. The literature survey has been carefully culled for methodology applicable to the conversion of RP into diesel fuel suitable for Navy use. Based upon the results of this study, a second phase has been developed and outlined in which experiments will be performed to determine the most practical recycling technologies. It is realized that the final selection of one particular technology may be site-specific due to vast differences in RP volume and available facilities. A final phase, if funded, would involve full-scale testing of one of the recommended techniques at a refueling depot.

The Phase I investigations are published in two volumes. Volume 1, Technical Discussion, includes the narrative and Appendices I and II. Appendix III, a detailed Literature Review, includes both a narrative portion and an annotated bibliography containing about 800 references and abstracts. This appendix, because of its volume, has been published separately as Volume 2.

ACKNOWLEDGEMENTS

It is impossible to put together a massive literature study like this without involving the time and effort of a large number of people. We would first like to thank Wayne Vreatt and Norm Schmokel, U.S. Navy, for constructive comments and suggestions throughout this project. They coordinated reviews by other offices within the Navy so that we could answer as many of the concerns as possible.

Dr. Marian Olson was a primary investigator at the initiation of this effort. Faye Cotton completed the literature search, abstract compilation, and the related narrative to the abstracts almost completely on her own. Marvin Whisman did much of the writing of the text of this Phase I final report.

Patsy Sears, Patty Hudson, Sharon Johnson, Stephanie Waits, and June Forbes put in many hours typing the initial drafts. Riley Wright provided editorial comments and his staff prepared the final copy. The Navy and Bill Good provided helpful technical comments. Through it all the library staff responded to our many requests for copies of reports and other literature with positive results.

We believe the result is a very useful compilation of information that will serve as a reference source as well as a planning document.

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1.1 PHASE I SUMMARY

Most of the Navy's fuel terminals have facilities to recover oil from oily wastes. This report identifies several processing techniques for converting the Reclaimed Product (RP) into a specification fuel. The next step is a laboratory comparison of each method to convert RP into specification Navy distillate fuel (F-76).

Phase I of this project is covered by this report and was designed to assess the problem and evaluate solutions found in the literature. Phase II will test the more attractive approaches in bench-scale experiments. Phase III could carry out a full-scale demonstration of the chosen technology.

In the first phase the problem was assessed through a literature survey, personal contacts with Navy personnel, and a review of Navy reclamation facilities. The literature survey was conducted to evaluate technologies for converting RP to F-76. About 800 literature references of varying degrees of applicability were found and abstracts for each are provided in Volume 2 along with a preliminary narrative section to provide an overview.

The candidates among the various technologies are discussed individually in this report. Each discussion includes a brief outline of the methodology, availability of equipment, cost, reliability, flexibility, simplicity, environmental impact and an assessment of applicability to the problem at hand. Table 1 contains a general summary of NIPER's evaluation of most of the processes that were reviewed, using the criteria just described.

Conventional re-refining, such as that applied to used lubricating oil, might represent the surest technology for a successful conversion of RP to F-76. But re-refining does not necessarily best fulfill the requirements of the Navy for low initial capital investment, readily available equipment, simplicity, flexibility and reliability of operation.

Table 1. - Summary of tentative processes for conversion of reclaimed product (RP) to F-76

Process	Equipment availability	Cost[1] Capital	Cost[1] Operational	Reliability	Flexibility	Simplicity	Environmental impact	Applicability[2]
Acid/Clay	Readily	<$2 million	$0.18-$0.39/gal	Fair	Fair	Good	Unacceptable	+
Distillation								
Thin-film	Readily	<$700K	$0.10	Good	Fair	Fair	Acceptable	+/-
Others	Readily	<$225K	$0.10	Good	Fair	Fair	Acceptable	-
Solvents								
BERC/MZF	Readily	<$400K	$0.15-0.20	Good	Good	Fair	Marginal	+/-
Supercritical Extraction	Customized	<$1 million	$0.10-0.25	Fair	Fair	Poor	Acceptable	+/-
Chemical Treat								
PROP	Customized	<$11 million	$0.35	Fair	Poor	Fair	Acceptable	+/-
Caustic	Readily	<$2 million	$0.20-0.40	Poor	Poor	Fair	Marginal	+/-
Sodium Borohydride	Customized	<$500K	$0.05	Unknown	Unknown	Fair	Acceptable	+/-
Hydrotreating	Customized	$600K	<$0.10	Good	Fair	Fair	Acceptable	+/-
Clay Contacting	Readily	>$100K	$0.15-0.20	Good	Good	Good	Marginal	+
Electromagnetic RF	Limited	>$100K	<$0.10	Unknown	Unknown	Unknown	Acceptable	+/-
Centrifuging	Readily	>$100K	<$0.02	Poor	Poor	Good	Acceptable	-
Ultrafiltration	Customized	>$100K	<$0.10	Unknown	Unknown	Unknown	Acceptable	+
Re-refinery								
Dist/Hydrotreat	Customized	<$4 million	$0.14-0.26	Good	Good	Poor	Acceptable	+
Chemical/Dist.	Readily	>$1.2 million	$0.20	Good	Good	Fair	Acceptable	+

[1] Generally based upon 5 million gallons per year throughput

[2] + indicates potentially sufficient without additional steps
+/- indicates probable need for additional step(s)
- indicates it has little applicability

Other techniques that appeared to have considerable potential include a combination of chemical treatment and distillation, clay treatment, supercritical extraction, ultrafiltration and electromagnetic energy.

Technical and economic analyses are provided in this report. Candidate technologies listed in Table 1 have a NIPER estimation for probable success using available Navy personnel and meeting the other requirements of cost and ease of operation. Phase II experimentation then will provide an opportunity to verify these ratings and converge on more accurate cost estimates.

1.2 PHASE II PROPOSAL

The objective of the next phase of this study is the experimental comparison of recycling technologies that seem most promising. This will permit selection of one or more methods based upon technical arguments while providing data for economic studies.

Several barrels of a representative and homogeneous contaminated fuel will be required for Phase II research. Portions will be doped with additional contaminants, identified in Phase I, such as sea water, used lubricating oil, and solvents.

The following technologies will be studied:

- Distillation
  - Simulated procedures
  - Bench-scale
- Chemical Treatment
- Clay Contacting
- Hydrotreating
- Filtration
- Solvent Treatment
  - Atmospheric
  - Supercritical Conditions
- Electromagnetic Radiation

The product of Phase II will be a technical report directly comparing the technologies that were investigated. The various factors will be combined into an overall rating of attractiveness to the Navy. A final recommendation will be made regarding steps the Navy could make to more effectively use their contaminated F-76.

1.3 PHASE III

A final option would involve the installation of the best technology as determined in Phase I and II as part of a full-scale demonstration at a Navy station. Some further technical study would be necessary during plant design, construction, start-up, and initial operation. However, the principal objective would be to devise a technique simple enough to eliminate the need for outside support. The decision on whether to proceed with Phase III will be based upon a combination of technical, economic and environmental considerations.

The general steps that would be followed for Phase III work would include:

a) Development of Implementation Program
b) Operation Evaluation
c) Technical Evaluation
d) Economics
e) Logistics

2. INTRODUCTION

2.1 BACKGROUND

2.1.1 The Problem

Large quantities of waste oils are continuously generated by Navy ships and Navy and Marine Corps shore activities. Based on recent data (200), these quantities are estimated at over 25 million gallons per year. Waste oils are predominantly generated from bilge and ballast water recovery and from fuel tank cleaning. The oils are composed primarily of the fuel used by the particular ship. Used lubricating oils and similar materials from vehicles and ships rank second highest in quantity. Contaminated high flash point fuels, such as JP-5, diesel, and kerosene, rank third highest in quantities generated. The smallest quantities produced consist of solvents and low flash point fuels.

Definitions provided by the Navy (NAVSUP Instruction 4100.3A, December 1983) include:

- Contaminated Product (CP) - A Product in which one or more grades or types of product have been mixed, or a product containing foreign matters such as dust, dirt, rust, water or emulsions.
- Reclaimed Product (RP) - The product of reclamation which is suitable for blending with Navy Special Fuel Oil (NSFO) (MIL-F-859E) or sale as Fuel Oil Reclaimed (FOR) (MIL-F-24951(SA)). Figure 1 shows the general scheme of waste hydrocarbon processing and disposition in the Navy. Reclaimed product can play at least three separate roles. First, it can be blended with Navy Special Fuel Oil (NSFO) with an end use as NSFO. Second, it can be designated as Fuel Oil Reclaimed if it meets the specification MIL-F-24951. Or alternately, with further processing, we propose to restore reclaimed product to Naval Distillate Fuel (F-76) meeting specification MIL-F-16884H.
- Fuel Oil Reclaimed (FOR) - A product of Navy reclamation operations meeting specification MIL-F-24951(SA) and destined for use as a boiler fuel.

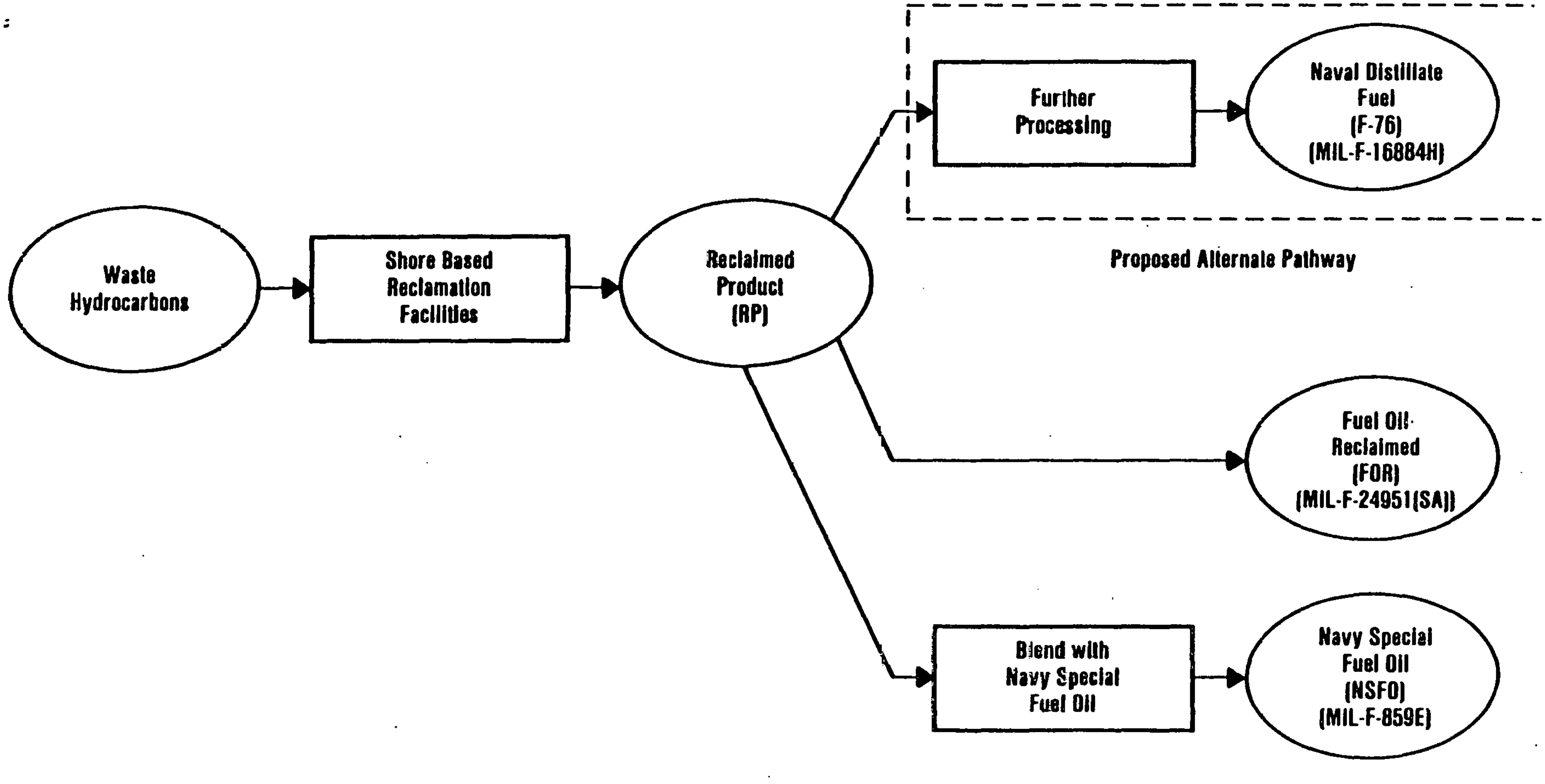


FIGURE1.— Schematic of waste hydrocarbon processing in US Navy

- Reclamation - The process which physically and/or chemically treats slop to a product suitable for blending into residual fuel or for burning in shoreside boilers.

Not included in this list of definitions is the specification grade Navy Distillate Fuel formerly designated as Diesel Fuel Marine (DFM) and currently designated as F-76. This specification can be found in Table I-37, Appendix I.

Navy waste oils may be grouped into three categories:

- Light Waste Oils - oils recovered from ship bilge and ballast water and contaminated high flash point fuels, representing an estimated 87 percent of the total waste oils generated throughout the Navy (200).
- Heavy Waste Oils - turbine engine drainings, shop facility waste, and tank cleanings, estimated to be 9 percent of the total waste oils generated (200).
- Low Flash Point Materials - used solvents and contaminated low flash point fuels, estimated at 4 percent of the total waste oils generated (200).

The Navy, in keeping with the National concern for future energy resources, has among its objectives the substitution of more plentiful energy sources for natural petroleum. In line with that objective, many Navy boilers have been and are being converted to the use of coal rather than oil. In a related area, the Navy has also been concerned with management of its oily wastes.

Most of the Navy's major fuel terminals have facilities to recover oil from the products which are generated from ship's bilges and waste oil tanks, tank bottoms, line cleaning, and other contaminated oil turn-in that cannot be downgraded (438). After a primary oil/water separation, most of the recovered oil is given secondary treatment consisting of settling. Sometimes additional treatment using heat or chemical addition or both removes the remaining water and sludge. The end product from these processes is suitable for blending with Naval Special Fuel Oil (NSF) or sale as Fuel Oil Reclaimed (FOR) for use in shore boilers.

Over a two year period (1980-82) the Naval Civil Engineering Laboratory conducted a survey of waste oil generation and disposition by Navy and Marine Corps shore activities. Approximately 19 million gallons per year of waste oil are generated by 325 Naval activities. The Navy reclaimed 15.5 million gallons of oil during FY-81 (Data supplied by Norm Schmokel, Navy). Of this, 30 percent was blended into NSF stocks, and 70 percent was sold as FOR. The ratio favoring FOR is expected to increase over the next several years.

Others estimate the total amount of waste fuel (oil) generated by the Navy to be as much as 70 million gallons per year. Although this is a rough estimation, there is every reason to believe that the volume being recovered has been increasing at a rate of approximately one million gallons per year.

About 3 to 4 percent of the Navy's total F-76 requirement could be met with an estimated savings of around $10 million per year if even half of the reclaimed oil could be processed for use as F-76.

2.1.2 NIPER/Navy Objectives

The current cooperative project between the Navy and NIPER proposes to systematically assess the problem, evaluate the solutions, test the more attractive approaches, and finally to implement full-scale demonstration programs to convert Reclaimed Product (RP) into F-76. Investigation of technologies that can be installed on site or contracted at commercial facilities must include investigations of the by-products that might be formed as a precaution against ecological problems.

2.1.3 Scope of Work

The proposed study has been designed to be performed in three sequential phases. This report represents completion of Phase I in which about 800 literature references were identified, tabulated, abstracted, and evaluated for potential and viability in transforming RP into a marine diesel fuel of a specific grade and quality. The second phase will involve testing of techniques that seem to have promise. This testing will be performed in bench- and pilot-scale facilities. Some of the work will be done at NIPER; some might more economically be performed at vendor laboratories. It is proposed that NIPER would provide feedstocks to these vendor labs, including RP that had been purposely contaminated. Suspect contaminants will be identified through analytical data provided from the Southwest Research Institute, test data generated internally, from those knowledgeable in the Navy oily waste programs, and from literature citation.

If required, the third phase of this study will involve full scale tests, probably at one of the refueling depots. This does not mean that only one answer will serve in all cases. In fact, it may be that different size depots will require different equipment/technologies. Phase III will permit testing of proposals resulting from the first two phases of the study and will resolve such questions as whether one technology will serve multi-sized Navy applications. The decision to proceed with Phase III will be based upon a combination of considerations including technical, economical, and environmental. The general steps that would be followed in preceeding with a third phase would include:

a) Development of the Implementation Program
b) Evaluation of operational procedures
c) Evaluation of technical parameters
d) A thorough consideration of economics
e) Logistics of reclamation.

2.1.4 Duration of the Project

Phase I of this study has been completed. Phase II should be completed before the end of FY 85. Phase III could extend into FY 87 or beyond dependent upon the technology selected and developed for full-scale demonstration.

2.2 APPROACH TO OVERALL PROBLEM

2.2.1 Information Compilation

A literature search for technology applicable to the conversion of RP to specification grade marine diesel fuel was initiated through the library facilities of NIPER. In addition to extensive in-house literature files, the library facility has computerized access to several retrieval systems developed to provide rapid and easy access to energy-related data bases. One such retrieval system that was used extensively in this program was DOE/RECON which contains information obtained or produced by the Technical Information Center (TIC) of the Department of Energy.

A second system that was used in this literature search was the ORBIT System which is an online interactive bibliographic retrieval system designed and implemented by the SDC Search Service division of System Development Corporation, Santa Monica, California.

In any systematic literature search it is necessary to establish and provide key words as a basis for reference discrimination. Key words used for this literature search are as follows:

waste oil
diesel fuel marine
contamination
sea water
reclamation
processes
equipment
oil
purifiers
re-refining
reprocessing
salt
middle distillates
filtration
fuel recovery
oily waste
oil reclamation
reclamation of oily wastes
oily waste uses
uses of oily wastes

This search led to the accumulation of about 800 abstracts of publications related to this study. These are provided as an annotated bibliography in Appendix III.

2.2.1.1 Patents

About 25 percent of the relevant literature found were patents, both domestic and foreign. No patent was directly related to a technology specifically designed to convert RP to F-76. However, many of the patents were related to waste oil recovery and therefore had some relevance to the problems being addressed in this study.

2.2.1.2 Literature References

The remaining 75 percent of the 800 citations documented in Appendix III were found in journals, company and government publications, and in proceedings from conferences, seminars and meetings. Generally, these sources were considerably more explicit and to the point in describing technical procedures than were patents.

The journal articles dealt with a host of subjects from oil/water separation to refining procedures for petroleum products. The processes for purification of used oil were, mostly, directly applicable to used lubricating oil and only indirectly applicable to fuel oil processing. However, such techniques as acid/clay treatment, caustic treatment, chemical reaction of contaminants, solvent precipitation of sludges and contaminants, distillation, clay contacting, ultrafiltration, supercritical extraction and electromagnetic radiation appeared to merit further consideration.

A later section of this report will deal with each of the applicable processes in depth, including the potential for the conversion of RP to F-76.

2.2.1.3 Southwest Research Institute (SwRI) Data

In conjunction with this program for the Navy, the Southwest Research Institute in San Antonio, working as a subcontractor to NIPER, collected F-76 and RP samples from six refueling stations. Two F-76 samples were requested from each depot and three monthly RP samples, in order to develop good data on starting material for continuing phases of this study.

Table 2 is a summary of the data generated by SwRI on 12 F-76 (or DFM) samples and 18 RP samples that were obtained from six refueling stations. The table includes current F-76 and FOR requirements along with the range for each of several properties found among the diesel fuels and the average value for each property. The individual analyses for each of the 12 F-76 fuels and 18 RP samples are included in Tables I-1 through I-5 in Appendix I. Note that the fuels fall consistently within the requirements for F-76 and FOR and that the average is as good as or better than the MIL specifications. Physical properties, however, do not necessarily define the quality of RP because of the trace contaminants that are not reflected in gross physical properties.

To have better insight into the overall quality of RP, a hazardous materials investigation was made in search of contaminants that might have been generated during use. This sort of contamination is possible due to the inclusion of slop oil and ballast water oils in the feed streams. A vastly more complex handling procedure will be necessary if the RP must be treated as a hazardous waste.

Table 2. - Summary of analyses of MIL-G-16884H (F-76) and Reclaimed Product (RP) samples

Properties	Requirements		Range found among		Average value for	
	F-76	FOR	12 samples F-76	18 samples RP	12 samples F-76	18 samples RP
Appearance	Clear, bright and free of particles		C&B [1]		C&B	
Distillation, °C(°F)						
IBP	----		193(380)-227(441)	174(346)-206(402)	208(406)	189(372)
10% point	----		227(440)-253(488)	208(407)-236(456)	240(464)	220(428)
50% point	Record		273(525)-289(552)	250(483)-297(567)	281(538)	271(520)
90% point	357(675)max		317(603)-335(635)	347(657)-384(723)	327(620)	364(687)
EP	385(725)max		338(641)-367(693)	364(688)-432(809)	356(672)	385(725)
Residue and loss, v%	3.0 max		1-1.5	0-4.8	1.04	1.6
Flash point, °C(°F)	60(140)min	55(130)min	72(162)-102(216)	72(162)-92(198)[2]	83(181)	79(174)[2]
Pour point, °C(°F)	-6(20)max	-6.7(20)max	-18(0)to-9(16)	-45(-49) to -18(0)	-12(10)	-35(-31)
Cloud point, °C(°F)	-1(30)max		-14(7)to-5(23)		-10(14)	
Viscosity @40° C(104° F)	1.7-4.3 cSt	2.0-15.0 cSt	3.04 - 4.04	2.95 - 7.55	3.47	4.03
Viscosity @50° C(122° F)		30-29 SUS		34.1 - 45.9		37.3
Carbon residue, 10% bottoms, wt%	0.20 max	NR	0.12 - 0.17	0.70 - 10.8	0.14	3.41
Sulfur, wt%	1.00 max	2.0 max	0.28 - 0.75	0.27 - 0.58	0.45	0.41
Cu corrosion @ 100°C (212° F)	1 max	NR	1a	3b - 4c	1a	
Color	3 max		1.5 - 4.0		2.8	
Ash, wt%	0.005 max	0.15 max	0 - <0.005	0.002-0.18[3]	<0.0025	0.04[3]
Gravity, API	Record	24-40	31.9 - 34.8	30.4 - 35.6	33.3	33.9
Cetane No.	45 min	NR	46 - 53	46 - 51	49	38
Demulsification @ 25° C (77° F) min	10 max		2 - 5		3.2	
Acid No, MgKOH/g	0.30	NR	0.07 - 0.24	0.13 - 1.36	0.13	0.39
Neutrality	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral
Aniline point, °C(°F)	Record	NR	63(145)-69(150)	64(147)-83(181)	64.9(148.8)	71.5(161)
Acc. stability, mg/100ml	1.5 max	NR	0.1 - 3.6	0.5 - 12.33[4]	1.0	2.45[4]
Trace elements, ppm	NR	NR	none - 21[5]	52 - 2536[6]	15[5]	780
Aromatics, v%	NR		32 - 41		36	
Heat of comb., Btu/lb	NR	NR	18,145-18,423	17,177-18,343	18,248	18,555
Water and sed, v%	0.01	2.0 max	-- to 0.02	0.02 - 8.0	0.002	1.4
Sediment, wt%		0.5 max		0 - 0.14		0.025
Bromine number		NR		0.73 - 1.72		1.20
Explosiveness, %		50 max		5 - 25		12
Chlorinated material		no green		none		none
Water, Karl Fischer wt%		NR		0.012 - 4.17		0.64

NR - No Requirement

[1] One sample was cloudy and hazy.
[2] These values represent 14 samples. Four samples did not flash due to water vapor.
[3] Sulfated ash.
[4] These values represent 15 samples. Three samples caused filter plugging.
[5] These values do not include phosphorus (reported for 2 samples to be 0.04% and 0.06%) or chlorine (reported for 1 sample to be 0.01%).
[6] These values represent total trace metals in a single sample.

The same F-76 and RP samples analyzed by SwRI for bulk properties were used for determination of hazardous species. The F-76 sample data provide a background level demonstrating which compounds are inherent in petroleum products. Table 3 lists the volatile and semivolatile pollutants, pesticides and polychlorinated biphenyls results for the 12 F-76 samples and 18 RP samples. As expected, the F-76 is relatively clean, with only low levels of benzene of any interest.

The 18 RP samples consistently contained significant levels of halogenated compounds. When the concentrations (some as high as 395 ppm for a single component) are viewed in light of the very large dilution that has occurred, it becomes obvious that significant sources of chlorinated compounds exist in the current collection system. Some states (e.g. New York) consider any fuel with a total halogen content of over 1,000 ppm a hazardous waste. The RP from Puget Sound exceeds this level without evaluating any other factors.

The RP from Charleston is just under the maximum level allowed. In addition to environmental problems, such levels of halogens will affect the technologies and equipment that can be used. Corrosion is a definite probability. Very little other contamination has been identified so far. Thus, the Phase II experiments can focus on the halogens problem unless other problems are uncovered in subsequent samples.

The detailed analyses for these F-76 and RP samples are included in Appendix I.

2.2.1.4 Vendor Contacts

In mid-1983 a letter of inquiry was sent to a number of vendors selected for their potential in providing processes and/or related equipment applicable to the problem of converting RP into F-76.

The vendors were selected by reviewing appropriate categories in the Thomas Register and from personal experience and contacts made in conducting this project and earlier related studies of re-refining of used lubricating oil. A list of vendors to whom the letter was sent is included in Appendix II.

Table 3. - Summary of contaminants found in MIL-G-16884H (F-76) and Reclaimed Product (RP) samples

	Range found among		Average value for	
Contaminant or pollutant, ppm	12 samples F-76	18 samples RP	12 samples F-76	18 samples RP
Methylene chloride	ND	ND - 33	ND	5.8
Trichlorofluoromethane	ND - 0.5	ND - 100	ND	22
1,1-Dichloroethane	ND	ND - 3.8	ND	1.1
trans -1,2-Dichloroethylene	ND	ND - 15	ND	3.0
1,1,1-Trichloroethane	ND	ND - 520	ND	109
1,2-Dichloropropane	ND	ND - 0.3	ND	0.03
Trichloroethylene	ND	ND - 190	ND	28
1,1,2-Trichloroethane	ND	ND - 0.3	ND	0.02
Benzene	4.4 - 53	4.9 - 23	29	14
Tetrachloroethylene	ND	ND - 180	ND	56
Toluene	47 - 190	89 - 290	132	180
Ethylbenzene	38 - 180	80 - 210	118	146
1,1,2-Trichlorotrifluoroethane	ND	ND - 940	ND	219
Naphthalene	160 - 1000	520 - 2600	595	1264
Fluorene	240 - 1100	150 - 670	405	267
Phenanthrene	420 - 960	ND - 540	617	359
Anthracene	ND - 41		3	
Di-n-butyl phthalate	ND - 25	ND - 18	6	1
Pyrene	Trace - 50	ND - 110	22	39
Benzo(a)anthracene	ND	ND	ND	ND
Chrysene	ND	ND - 20	ND	2
bis-2-Ethylhexylphthalate	ND - 690	ND - 61	59	19
1,2,4-Trimethylbenzene	ND - 1000	38 - 1500	438	757
Tetramethylbenzene	ND - 350	160 - 640	145	360
Tetralin	ND - 360	ND - 1800	166	661
2-Methylnaphthalene	63 - 2200	840 - 1500	1107	1155
Biphenyl	ND - 910	180 - 420	409	284
2-Ethylnaphthalene	120 - 930	160 - 790	548	554
2,3-Dimethylnaphthalene	410 - 1900	430 - 1100	913	702
2,6-Dibutyl methyl phenol	ND - 12	ND - 340	1	123
Dibenzofuran	ND - 190	ND - 96	88	60
Triphenyl phosphate	ND - Trace	ND - 240	ND	63
Diethyl hexyl sebacate	ND	ND - 93	ND	19

ND - None detected

A copy of this letter of inquiry and its attachments, which included chemical and physical requirements for both FOR and DFM, is included in Appendix II. Subsequent to that mailing, the marine diesel fuel designation has changed from DFM to F-76. However, the properties are essentially identical.

The response to the letter which briefly described the goals of this research program and requested information and brochures describing processes, related equipment, laboratory facilities, and commercial-scale installations of such equipment was neither overwhelming nor disappointing. Of 52 letters, responses were received from 25 companies. Five of these 25 responses were negative indicating no equipment, process, or expertise applicable to the problem as stated.

Of the positive vendor responses, two companies offered centrifugation equipment, six offered filtration devices, two offered ultrafiltration, two companies incorporated clay and filtration, one company offered chemical demulsification, one company promoted electrostatic separation for oil/water mixtures, and two companies offered portable devices that included filtration and/or dehydration. Three laboratories responded positively to the inquiry about pilot-scale test facilities.

One important product of these vendor inquiries was the knowledge that a turn-key, off-the-shelf process (machine) for converting RP to F-76 may not exist in the market place. It does seem likely, however, that some of the vendors contacted can make valuable contributions to the ultimate solution of this project, and that we can probably take advantage of vendor pilot-scale test facilities--if not for performing a complete process, at least for testing a part of the developed technology.

2.2.1.5 Personal Contacts

Most of the direct Navy input to this project has come from Wayne Vreatt, Naval Material Command Headquarters, and Norm Schmokel, Navy Petroleum Office. They have provided guidance as to the specific objectives and have reviewed the progress of our work.

We visited two of the seven refueling depots for an in-depth survey of existing facilities and capabilities, augmenting information available from a recent Navy report (533). Both Point Loma (San Diego, CA) and Norfolk, VA are large fueling ports, so they were considered prime candidates for a recycling facility, if that was the final recommendation of this project. Both were found to have clean, efficient systems for handling oily waste water and waste oils. Both had some equipment which could be made available. On the negative side, both were minimally staffed. Point Loma had only one person to handle all operations. Thus, any on-site recycling would require additional personnel, which will have to be factored into cost estimates. Also, it was obvious that segregation of hydrocarbon materials coming off the ships was essentially non-existent. That was further confirmed by the halogenated solvent levels found in the RP samples taken during this study.

An important factor was the strong expressions of support for the idea of recycling received during both site visits. All personnel with whom we visited believed the concept to be feasible and offered their assistance.

In addition to the in-depth discussions and visits described above, considerable input has been received during Navy briefings at various stages of this first phase. Representatives of a number of commands, such as NAVSEA and NAVSUP, provided information concerning the data they needed to properly evaluate the proposed recycling technologies and scenarios. It is with all this in mind that the recommendations of this report are made.

2.2.2 Organization and Utilization of Information

A large amount of literature was collected, studied and tabulated early in Phase I. The raw product of this search has been compiled in Volume 2, Literature Review and is designated Appendix III.

A large number of literature references marginally applicable to the final solution of this project involved the separation of oil and water. Actually, the separation of oil and water is not a major problem in converting RP to F-76 since they must be separated before the bilge and slop accumulations can be processed to a fuel. As with most such separations, there will be low levels of water left in the oil even as it is being used as a boiler fuel. A good estimate of the range after processing to RP would be from 0.2 to 2.0 percent water. This amount should not represent a major problem in the final processing technology aimed at producing a specification diesel fuel.

The references relating to the separation of oil and water were categorized according to the following headings:

settling, flotation, and flocculation
filtration
coalescence
centrifuging
demulsification

A number of literature references dealt with processes, although the majority of these were intended for reclaiming used lubricating oils rather than fuel oil. These references were grouped according to the following generic technologies:

sulfuric acid treatment
caustic treatment
chemical treatment
solvent treatment/supercritical
  extraction
distillation and/or evaporation
clay contacting
ultrafiltration
electromagnetic radiation
combinations of the above
miscellaneous methods

A number of publications appeared to have some degree of relevance but were difficult to categorize. Therefore, a general category entitled "Miscellaneous" was used to list each of these patents and publications. Included under this heading were:

burning
pollution
analysis
management and economics
potential
microbial contamination
other

2.2.2.1 Information Screening

Copies of publications related to the subject were obtained when such was accessible. First priority was given to publications that dealt directly with the processing of fuel oil. Since there were few such articles, attention was then directed to publications that dealt with subject matter that was closely related to the re-refining or reclamation of fuel oil. Finally, articles were divided into groups based upon subject matter including oil/water separation with a number of sub-headings under that general category. Another major category was processes, and under this heading many separate technologies were listed as sub-headings.

2.2.2.2 Narrative to Annotated Bibliography

A narrative forward to the bibliography has also been included in Appendix III. This narrative is actually a further condensation of the abstracts. The various references within each subject area are tied together and discussed to give an overview of the information available.

2.2.2.3 Compilation of Abstracts

After the articles, abstracts, and patents had been assembled and subdivided as indicated above, an abstract was prepared for each article. These abstracts were taken unchanged, directly from the article, if such a summation was available. Otherwise, the article was condensed to its essentials by a member of the team preparing the literature survey. These abstracts were tabulated alphabetically based upon the principal author's last name and is included in Appendix III (Volume 2).

3. DISCUSSION OF AVAILABLE TECHNOLOGY

3.1 DESCRIPTION OF METHODS

Figure 2 is a general schematic diagram showing steps that might be pertinent to the processing of Reclaimed Product (RP) to F-76. Current Navy technology encompasses steps A and B; the separation of water and oil and the final dehydration of the oil to RP. The technology that is required to convert RP to F-76 could involve the addition of one or more succeeding steps. For example, it might be found that clay contacting would be sufficient to reclaim F-76. On the other hand, a process that included distillation might be required with the incorporation of a chemical or solvent pretreatment step ahead of E. This figure is used only to demonstrate some of the possibilities that can be addressed in arriving at a good technology for converting RP to F-76.

The methods found in the literature generally described applications to used lubricating oil or waste oil, which is generally identified as one or more of the following:

hydraulic oil
automotive lubricating oil
quench oil
tramp oil
cutting oil
metal-working oil
diesel engine oil
gear oils
railway diesel oil
marine cylinder oil
marine diesel oil
gas engine oil
turbine oil
machine oil
spindle oil
transformer oil

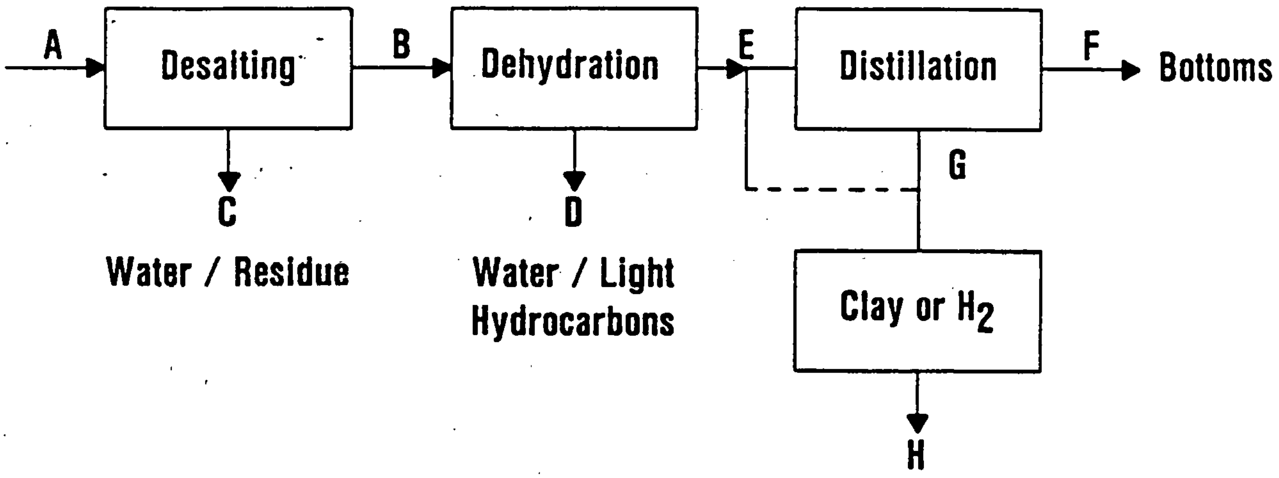


Figure 2.- General schematic showing potential processing steps

Most often the methodologies refer to complex mixtures of the above oils. Processors encourage segregation of the various kinds and types of oils that are recyclable, but only a small percentage of the used oil that is generated is actually segregated. Some lubricating oil service such as railroad locomotives demands the use of a naphthenic-based formula, and companies usually will segregate these oils from contamination with paraffin-based lubes in order to preserve the integrity of their product for re-refining.

Processes such as chemical treatment, clay contacting, distillation, solvent treatment, supercritical extraction, and hydrotreatment plus combinations of these were found for removing gross contaminants from used oil. These will each be discussed in more detail in the following subsections.

3.1.1 Sulfuric Acid/Clay Treatment

Until a decade or two ago, the re-refining industry throughout the world used acid-clay technology to reclaim used lubricating oil. The process is nearly extinct today because of damage to the environment caused by by-products from this process.

The former dominance of acid/clay technology for re-refining of used lube oil actually can be attributed to the petroleum industry that used acid extensively prior to WW II to desludge, decolorize, and deodorize petroleum lubricating oil basestocks. It is reported that one or more of the older crude oil refineries still use acid technology for clarification of lube oil basestocks (592). In addition, it was used as a dewaxing/deasphalting agent for crude oils.

While the literature references to acid technology (31,59,150,346,564, 766) contain minor differences, the process scheme is essentially as shown in Figure 3. It involves removal of debris from the feedstock and settling of the free water. The decanted oil is pumped through a heat exchanger to a flash dehydrator at 300° F and atmospheric pressure. The steam/light hydrocarbon overhead is condensed and separated. The organic phase is sent to the light end storage to be used for fuel, and the water is routed to the wastewater disposal system.

The resulting dehydrated oil is generally pumped directly to dry oil tanks where it is stored and cooled. It can be stored for 2 to 4 days before it picks up appreciable moisture, which tends to increase acid requirements during the following step. After 48 hours storage, the oil temperature has dropped to about 100° F. The dry oil is then pumped to one of the several acid treating units. These units are steam-jacketed and are agitated with plant air. Sulfuric acid (92 percent) is added to a reactor maintained at about 100° F. The amount of acid added ranges between 4 and 6 percent of the oil depending upon water and contaminants. The oxidized products contained in the oil are usually coagulated within 24 hours. The acid sludge, containing oil contaminants and ash, separates from the oil and is drawn off from the reactor bottom. Acid sludge disposal, which formerly was done in landfills or lagoons, is the problem which contributed to the abandonment of this process.

The acid-treated, dehydrated oil is transferred to a steam stripping/clay treatment operation. This is usually a batch operation. The temperature of the batch is brought up to 500-600° F by circulating through a heater. Simultaneously, live steam is introduced into the batch. The purpose of

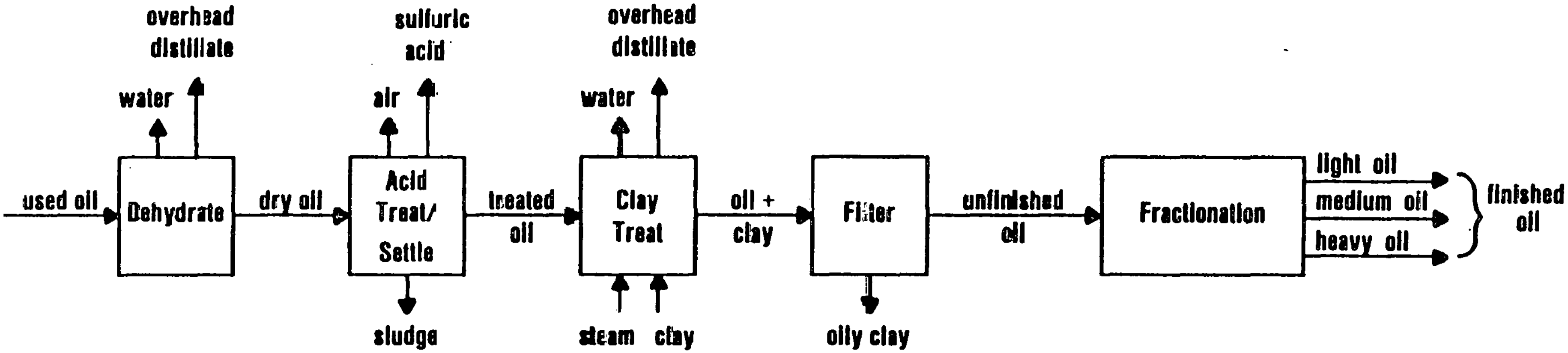


Figure 3.- Schematic of an acid-clay process

stripping is to remove remaining light fuel fractions and any mercaptans which may be present. This operation normally takes 12-15 hours to complete. The steam-stripped materials are condensed, and the oil is separated from the water.

The hot oil containing the clay is filtered through a plate and frame filter press, sometimes followed by a second filter. The clarified oil is then stored prior to sale and/or formulation with additives.

3.1.1.1 Availability of Equipment

The equipment used for acid/clay treatment is not complex, but, as far as these investigations could determine, there is no off-the-shelf acid/clay treater available as a turn-key machine. The equipment generally consists of pumps, tanks, mixers, a circulating heater and one or more filters. Although plate and frame filters are still used by some processors there are filters available commercially today that are as effective and much less labor intensive.

3.1.1.2 Cost (Capital and Operational)

Based upon earlier studies (62,161) the capital cost for a complete acid/clay refinery would probably exceed $2 million and operational costs might range from $0.18 to $0.39 per gallon of Reclaimed Product (RP) converted to F-76.

Current costs for an acid/clay treatment facility are not available. Most of the plants process used lubricating oil and were assembled over a period of many years without a blueprint or master plan. One exception to the evolutionary development of acid/clay was Bernd Meinken of Haltern, West Germany, who designed and built acid/clay treating facilities and was once considered the world's foremost advocate and expert in such technology. Meinken designed plants were built in nearly all parts of the world (479).

A 1974 study (161) sponsored by the Environmental Protection Agency (EPA) estimated the capital investment for an acid/clay plant with a 5 million gallon per year throughput at $1.176 million. A later study published in 1980 by Bigda and Cowan (62) estimated the capital costs for a plant with a nominal 10 million gallon per year throughput capacity at slightly less than $1 million. The correspondence between these estimates is not good and even worse when inflation during the period from 1974 to 1980 is considered. The EPA estimate included land and site improvement while the Bigda estimate did not and this factor would tend to equalize the estimates. Reducing the EPA estimate by $94K for land, the capital investment becomes $1.1 million. Applying an estimated 100 percent inflation rate for the period since 1974, one would arrive at a capital cost very close to $2 million for a 5 million gallon per year acid/clay re-refining facility exclusive of land and site improvement.

Operating costs for the acid/clay technology as defined by the EPA study were $0.18 per gallon, exclusive of feedstock cost, while the Bigda estimate, exclusive of feedstock, was $0.39 per gallon. These differences more nearly fit the pattern of inflation during the 9 or 10 year spread represented by the two estimates.

The question of cost appears somewhat academic since the acid/clay technology is no longer a viable process due to environmental problems associated with that technique. The acid-sludge produced is a hazardous waste that must be neutralized at considerable cost before conventional disposition, and the oily-clay represents a disposal problem in many areas.

3.1.1.3 Reliability of Acid/Clay Technology

Despite the many problems and weaknesses of the acid/clay technology, it is reliable. The process was almost universally conducted in a batch-wise mode and the final product from any given plant was consistent, and the product from one acid/clay plant to another was also similar.

3.1.1.4 Flexibility of Acid/Clay Technology

The flexibility of the acid/clay process is somewhat limited. Since the technology includes only a flash evaporation to remove water and light solvents, there is no way to accurately control boiling range and related properties, such as flash point and freeze point.

3.1.1.5 Simplicity of Acid/Clay Technology

Acid/clay must rank close to the top re-refining process in simplicity. Little is involved except the judgment of the plant operator with regard to acid required, settling time, amount of clay, temperature of clay contacting and duration of the heating cycle. Most of these judgments were obtained by experience and carefully guarded by the proprietor of the plant, although there was little difference in techniques used by the various acid/clay plants that have existed over the past 30 or 40 years.

3.1.1.6 Environmental Impact of Acid/Clay Process

The acid/clay process has become essentially extinct in the U.S. because of environmental stress created by the technology. The acid-sludge must be disposed either through incineration (which is practiced in Europe but produces too much sulfurous vapors and heavy metal products to be acceptable in the U.S.) or it must be disposed of by neutralization and landfilling. Disposal is costly and generally a hazardous landfill must be used rather than a conventional municipal landfill. Oily-clay has not found a large number of useful roles although there have been reports of incineration to recover the caloric value of absorbed oil and there have been some attempts of landfarming oily clay (698). However, oily-clay could represent an environmentally objectionable by-product. These major factors are in addition to other environmental problems attendant to many re-refining operations such as odor and corrosive non-condensable vapors. Acid/clay plants are reported to have worse than normal odors.

3.1.2 Distillation

Distillation has not been documented extensively as the sole process for re-refining but it is conceivable that distillation could serve as the primary process for converting RP to F-76. The literature contains extensive references to distillation (23,57,59,63,64,98,101,120,134,140,159,171,173,183, 204,222,230,243,257,259,287,341,347,421,424,478,479,534,535,541,566,663,673, 704,705,723). While gross oil/water separations are best accomplished by physical separators, distillation is probably the most effective technique for removing suspended and dissolved water and for separating fuel from sludge, particulates, and some contaminants.

Dehydration of waste oils in re-refining operations often employs flash distillation equipment. Some re-refiners consider that heat (100° F), settling, and chemical treatment, in combination or singly will reduce suspended and dissolved water to a 2 percent by volume level. The chemicals most commonly used are demulsifiers and caustic. The dehydration distillation is usually a batch operation and the water content is lowered to a range between 0.2 and 0.9 percent by volume.

Distillation to remove hydrocarbons boiling between 300 and 550° F is another routine procedure in most existing re-refining operations. As in the dehydration step, the variety of distillation equipment available for separating fuel oil from lubricating oil is extensive. Generally a simple flash evaporator operated below atmospheric pressure is selected because this is not considered a critical step in the re-refining of lubricating oil. The importance of this distillation takes on a new dimension, however, when thought of in relationship to the restoration of specification F-76 from RP. It is not uncommon for an under-designed flash evaporator to leave 5 to 15 percent of fuel in the lubricating oil and an equal amount of lubricating oil in the fuel fraction. The fractionation efficiency of flash evaporators can be very poor, depending upon design. A thin-film or wiped-film evaporator would accomplish better separation of fuel oil and lube oil than would a flash tank with a spray-type feed but the cost of the former is much greater. Thus, less sophisticated distillation equipment is usually the choice among re-refiners of lube oils.

Fractionating towers, as shown in Figure 4, are used extensively in many industries where good separation of materials with differing boiling points is required. The petroleum refining industry is one that depends upon such equipment. Although operation is complex, good separation can be achieved and products of differing boiling points and molecular weight can be separated from a single distillation tower. A disadvantage of fractionation towers is that they depend upon a reboiler to maintain reflux equilibrium. This implies that the material being distilled remains at an elevated temperature over a long period of time. The extended thermal stress is often quoted as the cause of coking and fouling in the re-refining of used lubricating oil. This objection may not be valid in the consideration of converting RP to F-76, but there would not seem to be any reason for requiring the complexity of a fractionation tower.

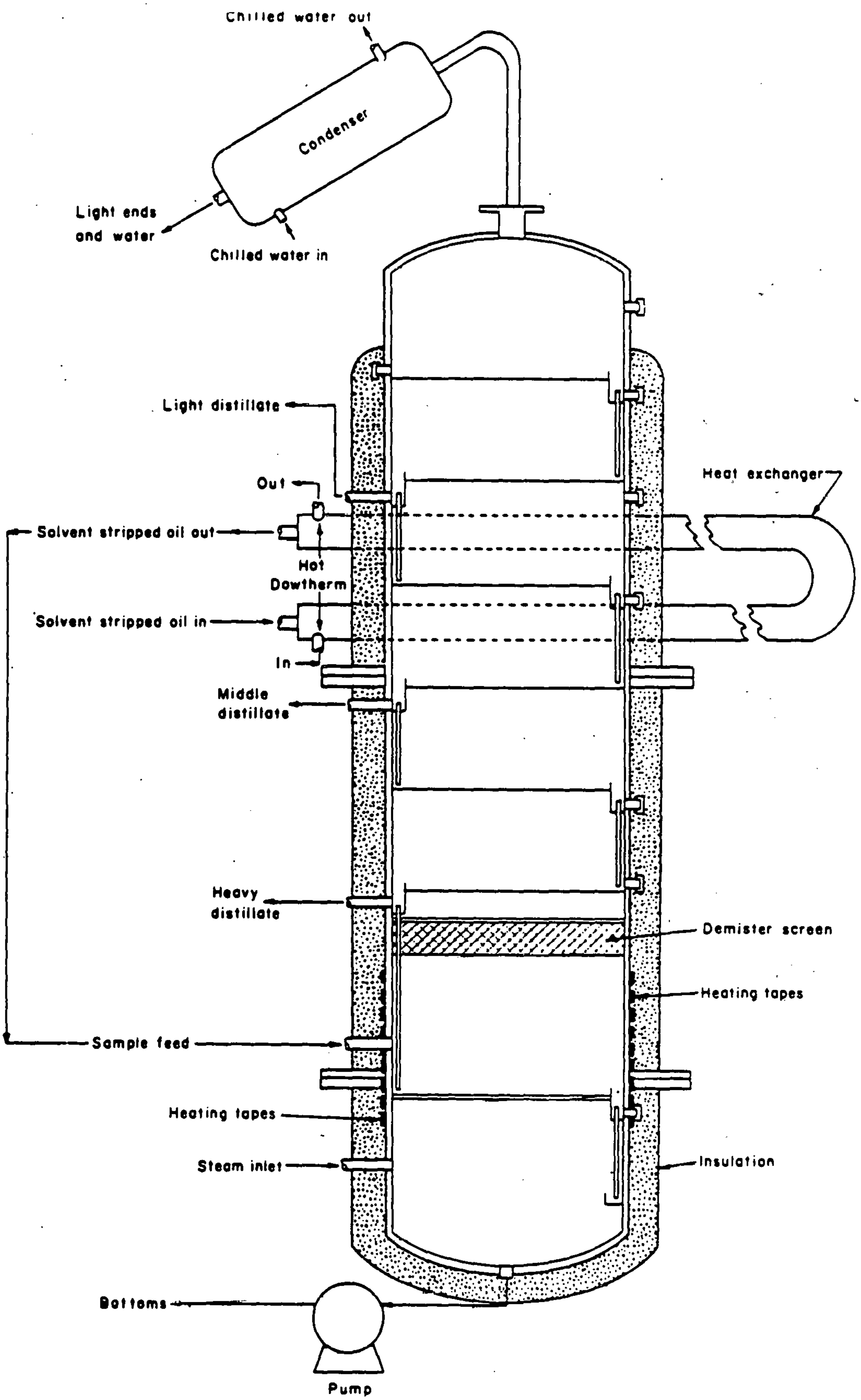


Figure 4.- Schematic of seven - plate distillation tower

A demonstration that distillation represents a potentially powerful tool for the conversion of RP to F-76 was made by Mohawk Lubricants Ltd. of North Vancouver, B.C. They processed 15,000 gallons of RP for the Navy. The treatment included distillation in a thin-film evaporator at 500° F and 100 mm pressure. This was followed by a hydrotreatment at 400° F and 450 psig. A typical schematic for such a distillation/hydrotreating technology is shown in Figure 5.

Tests were performed on the final products from distillation and hydrogenation. Results of these tests are presented in some detail in section 4.2 of this report.

3.1.2.1 Availablility of Equipment

Distillation equipment is readily obtainable commercially. There are several manufacturers of thin-film and wiped-film evaporators in the U.S. and in Europe. Among the companies that manufacture such equipment are Pfaudler (23,63,64), Luwa (259,287,566), Artisan (120), and Leybold-Heraeus (594). Figure 6 shows a general schematic diagram of typical thin-film equipment as manufactured by Luwa. One of the advantages of all the thin-film evaporators is the short residence time of the sample in the heated zone, thus minimizing thermal degradation processes. Another factor in favor of this equipment is that there are a variety of sizes that can be purchased to fit particular applications. Although thin-film evaporators are considered an off-the-shelf item, delivery is based upon fabrication time after receipt of order.

Distillation towers are available new or used. Those for refinery service are usually of extremely large throughput volumes and are not directly applicable to smaller roles such as converting RP to F-76. However, distillation is such an integral part of the chemical, food, and petroleum industries one should readily find a used tower that would be applicable with minor revisions. Distillation towers are generally built to the specifications of the buyer and therefore could not be considered as strictly an off-the-shelf item even though it is an off-the-shelf technology.

There are some smaller skid-mount crude oil distillation units that are specifically designed for small-scale field use and which may be applicable to the problem of decontaminating RP. The availability and applicability of such equipment could be investigated as a part of Phase II research.

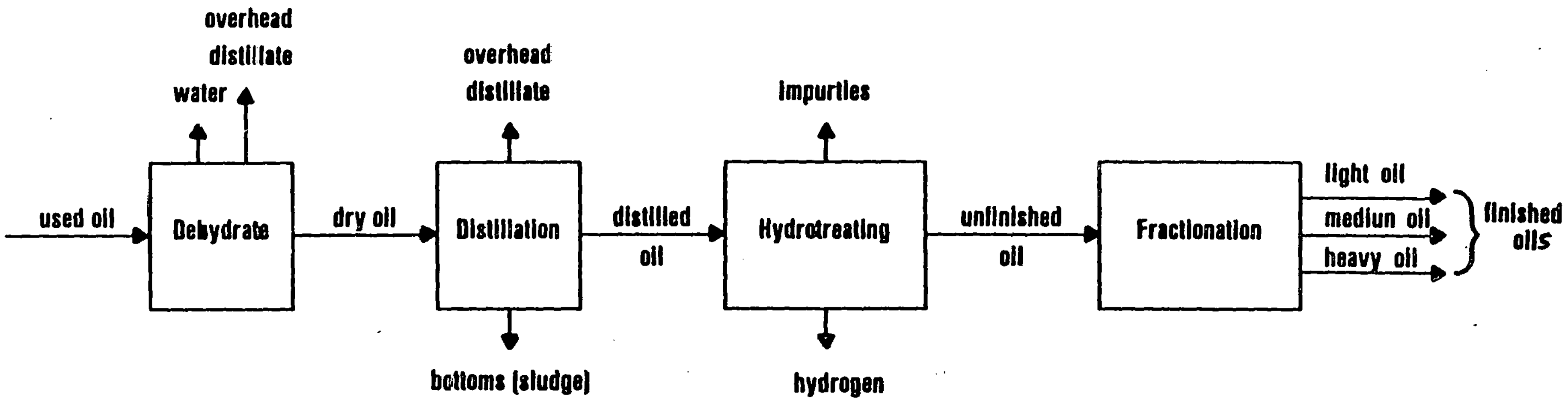


Figure 5.- Schematic of a distillation-hydrotreating process

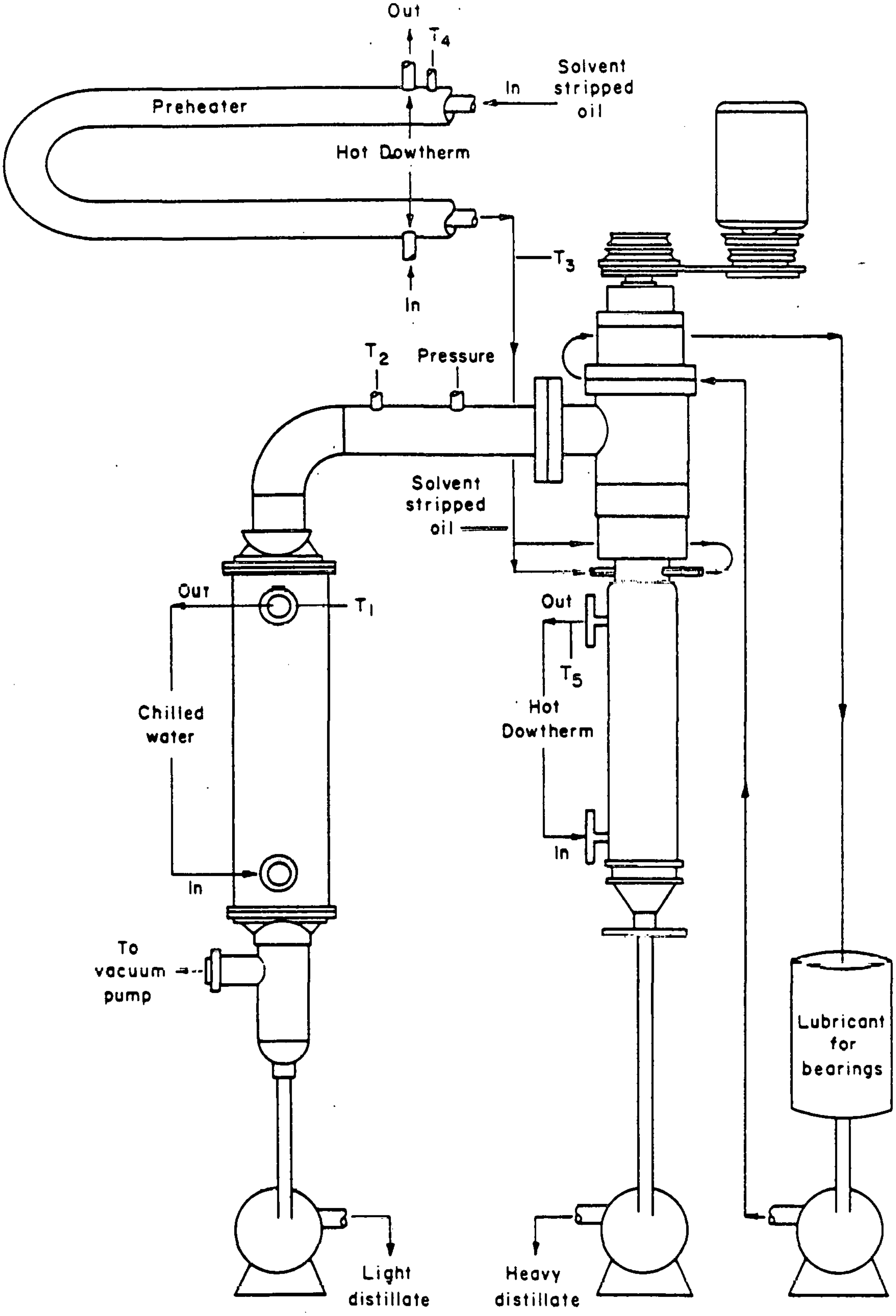


Figure 6.- Schematic of Luwa thin - film evaporator

Flash evaporators are more often than not constructed on-site from such simple components as tanks, pipe, pumps, heaters, valves, and such. These custom built stills can be moderately efficient to very inefficient depending upon the design. Without reflux, as in the distillation tower, or without the thin-film concept, a flash evaporator is generally less efficient and desirable than other techniques.

3.1.2.2 Cost (Capital and Operational)

The costs for essential equipment to dehydrate and flash light hydrocarbons from waste oil were estimated by Bigda and Cowan in a 1980 publication (62) and in their 1977 study (59) for a plant with 10 million gallon per year throughput. Their 1980 estimates did not include engineering design, and certain installation costs. The capital costs were reported at $55K. One could increase this estimate by perhaps 15 percent to take into consideration the inflation rate over the past few years giving a gross estimate of about $65K.

In the 1977 report (59) the cost for a conventional distillation tower, erected, including such peripheral equipment as vacuum ejectors, feed pump, and a series of five additional lube condensers, accumulators, and pumps was $170K. Increasing this by the estimated inflation rate during the past 6 or 7 years would raise the capital equipment cost for a conventional vacuum distillation tower to about $225K. Note that such a still would produce as many as four or five basestocks simultaneously but this would not be an advantage to the Navy.

Thin-film evaporators have advantages and disadvantages over vacuum towers. One primary disadvantage in reclaiming lube oil is the ability to produce only one overhead fraction and distillation bottoms from a single evaporator. This is not viewed as a problem in converting RP to F-76 and only one unit would probably be required for the fuel recycling envisioned for the Navy operation. Thin-film evaporators are sized based upon the heat exchange area of the interior of the unit (23,63,64).

A thin-film evaporator sized to process about 10 million gallons per year would have a heat exchange area of about 32 square meters and the unit constructed from common steel would cost in the range of $350K to $375K. The installed price including peripherals such as pumps, vacuum systems, tanks, piping, and a condensing tower would raise this to between $1 million and $1.2 million. The condenser that would be a part of this system has the capability of producing two or more fractions through a partial condensation system based upon zone temperatures within the condenser. A smaller thin-film evaporator sized to process 5 million gallons per year would be about 14 square meters of heat exchange area and the cost would be about $220K for the unit itself. Installation and peripherals would raise this overall cost to between $600K and $700K.

3.1.2.3 Reliability of Distillation Technology

Distillation is reliable and in its simplest forms consists of a retort in which the liquid is heated, a condenser to cool the vapors, and a receiver to collect the distillate. Fractional distillation was developed because simple distillation is not efficient in the separation of liquids whose boiling points lie close to one another. The aim is to achieve the closest possible contact between rising vapors and descending liquid, and so to allow only the most volatile vapor to proceed to the receiver while returning the less volatile material as liquid toward the still pot. The descending liquid is known as reflux.

3.1.2.4 Flexibility of Distillation

Some distillation equipment can be quite flexible while other configurations are quite rigid in their scope of application. Probably the thin-film evaporator represents an intermediate range of flexibility, for it can produce only one overhead fraction and the residue. However, by altering the temperature of the interior vaporizing surfaces, the boiling range of a fraction taken overhead can be altered.

3.1.2.5 Simplicity of Distillation Technology

The theory of distillation is well understood and the equipment operation ranges from simple to complex.

Distillation represents a technology for separating one or more liquids with differing physical properties. The type of equipment available and applications are so varied that one cannot make a generalized statement regarding simplicity. Probably the thin film concept is simpler from an operational standpoint than a vacuum tower. And a skid-mount crude still is probably simpler to operate than a thin-film evaporator.

3.1.2.6 Environmental Impact of Distillation Process

Distillation, like most other techniques for processing of RP, will produce by-products but it is an environmentally acceptable process. These by-products can be classified in the following primary categories:

light vapors and gases (non-condensable)
light vapors and gases (condensable)
distillation bottoms (residue)

Non-condensable vapors can contain odoriferous sulfur compounds of varying degrees of toxicity. Additionally, halogens--primarily organic chlorides--can be present resulting in highly corrosive gases. Non-condensable gases rarely represent an insoluble problem environmentally speaking. A simple flare to combust the vapors to a less objectional form is often the simplest solution. Otherwise, scrubber technology has been developed for practically all non-condensable gases that are not amenable to flaring. Scrubber technology most often employs caustic but often uses strong oxidizing agents in series with caustic to protect the environment from toxic or objectionable vapors.

The condensable vapors represent a problem only in their final disposition. Combustion is a tool often used to dispose of these materials while chemical reactions represent a viable technique for converting toxic liquids to non-toxic liquids, solids, and gases.

3.1.3 Solvent Treatment

The one important commonality among solvent treatment procedures is the probable requirement for additional process steps. Solvents, alone, have never been demonstrated in tests to be sufficent for the re-refining of used oil, but that is not to say that solvents would not be effective in reclaiming RP. The advantage of solvent treatment in reducing the requirements for additional processing steps or materials has never been well documented and the ability for solvents to reduce coking and fouling precursors in used lubricating oil is not an advantage in RP/F-76 processing. The nature of RP contaminants make supercritical extraction a candidate as a primary process step and should be investigated in Phase II.

A comprehensive and significant addition to the literature on re-refining has resulted from DOE/BETC (NIPER) studies (119). A process was developed by BERC[1/] which was covered by two patents (754,755). The major steps of this process, as shown in Figure 7, were dehydration to remove water and light ends; solvent extraction, including solvent recovery from the extract and raffinate; fractional vacuum distillation for additional contaminant removal and base stock production; and clay treatment for color improvement, although hydrofinishing was also investigated.

The solvent treatment (75) of this process, which may have some application to RP/F-76 processing, consists of using a mixture of alcohols and a ketone to precipitate certain contaminants from the used hydrocarbon prior to vacuum distillation. The claims made for the process are that solvent treatment reduces the tendency for used lube oil to coke and foul upon heating in subsequent distillation. This particular claim may have no application to the Navy project and was never completely substantiated. Nor was the process ever used in a full-scale plant. However, pilot-scale studies (120) produced enough material to obtain engine performance data and to conduct fleet tests (119). The testing substantiated the quality of product produced by the BERC solvent process but did not establish that coking and fouling tendencies during heating of used oil were diminished.

[1/] This method was developed when the Bartlesville Center was designated the Bartlesville Energy Research Center (BERC) and thus the technology has been called the BERC process. Subsequently, the Center designation was changed to the Bartlesville Energy Technology Center (BETC) and currently is known as NIPER.

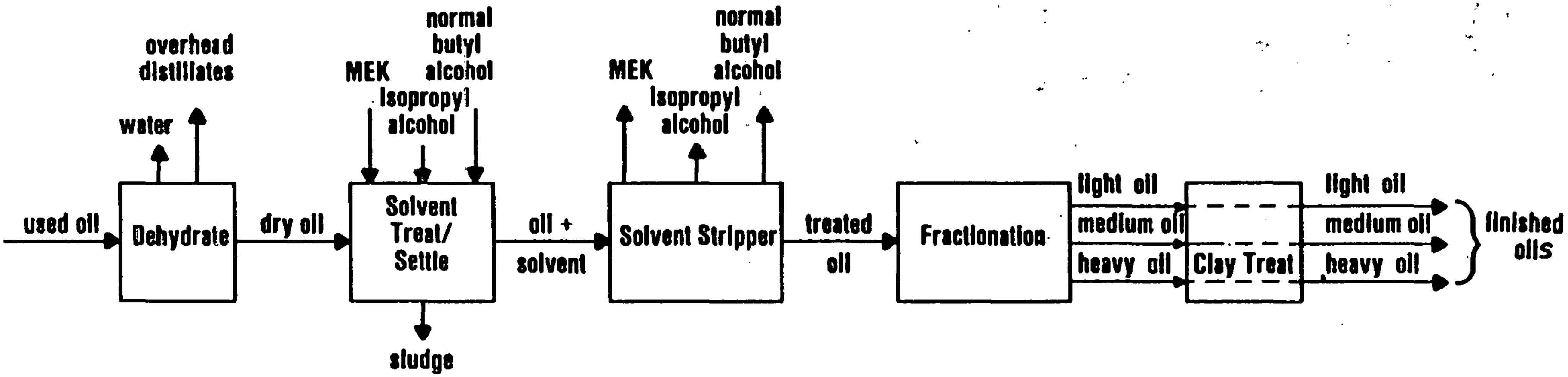


Figure 7.- Schematic of the BERC Solvent process

Another solvent extraction process developed on the West coast was called the MZF process (165). This technique, shown in Figure 8, requires aqueous isopropyl alcohol solutions in conjunction with small amounts of alkali or acid to remove contaminants from the used oil. The alkali technique was preferred and patents were obtained for the process. Claims for the process were never verified in a full-scale plant.

Although solvent extraction in re-refining was studied for many years, only one process was ever put into operation on a commercial level. This supercritical extraction process was developed by the Institute Francais du Petrole and was known as the IFP or Selectopropane process (291,428,585). See Figure 9. At least three plants were constructed using the technology. Unfortunately, the IFP process did not totally replace acid/clay treatment but was intended to reduce the quantity of these materials required and consequently the amount of waste by-products to be disposed.

Cutler (129) also developed a similar supercritical extraction process which he called the PVH process. This was an acronym for propane-vacuum-hydrogen. Cutler's method looked very attractive in bench-scale testing but was never put into full-scale production.

Still others have used propane in supercritical extraction processes for used lubricating oil including Crowley (127), Wielezynski (757), and Zosel (781). These latter processes are primarily applicable to the deasphalting of distillation residues but may have other applications.

A supercritical fluid extraction method, developed by Coenen (113), can use carbon dioxide, ethane, or propane equally effectively. In this methodology the water is first removed by an atmospheric distillation. The dry oil is subsequently raised to the temperature of an autoclave reactor and enters the top of a packed column in the autoclave where it is met by a counter-current of supercritical gas of equal temperature and pressure. The gas becomes loaded with volatile hydrocarbons and is then subjected to a stepwise reduction in pressure so that the gas gradually losses its ability to absorb the organic material. The extracted materials are drawn off and the expanded and essentially pure gas leaves the final separator at the top to be recycled as the extractor solvent. Almost all oxidation products, low-volatile and nonvolatile hydrocarbons, and solids remain in the autoclave and are discharged as residue.

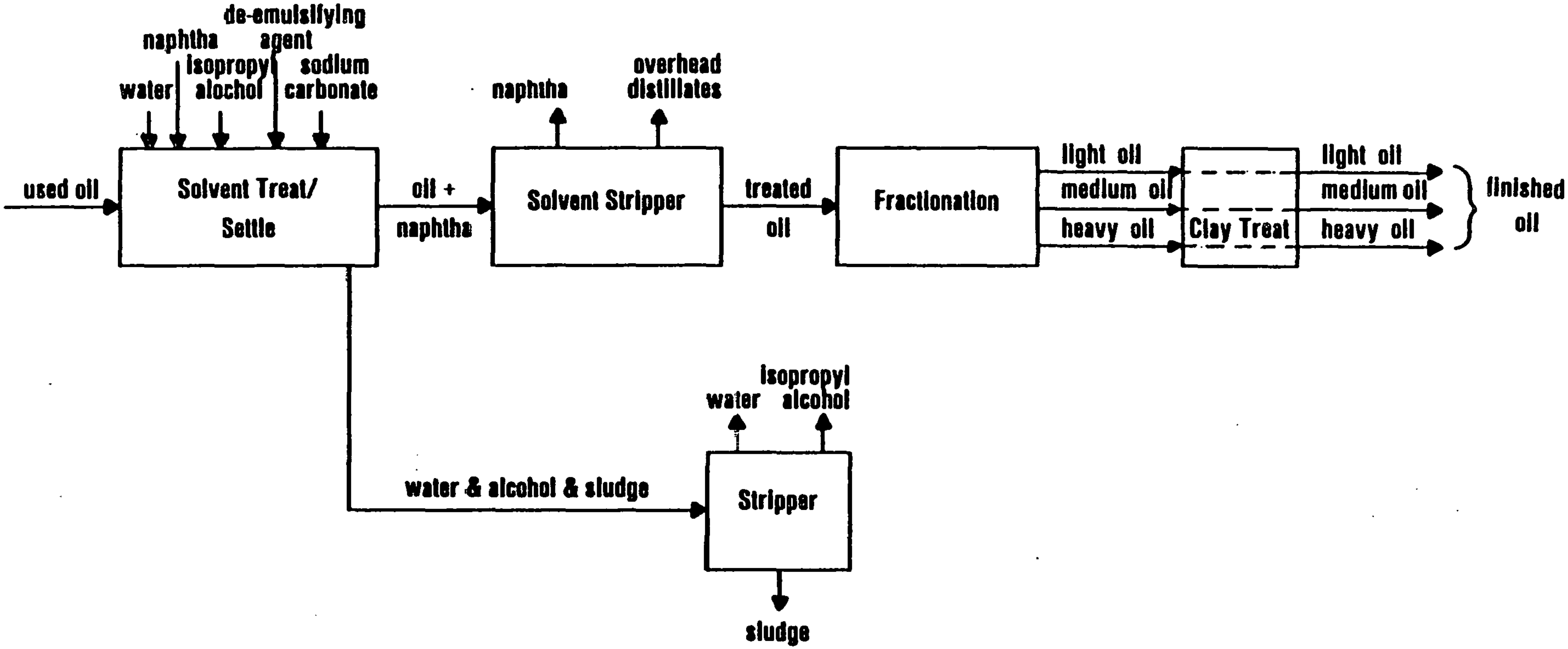


Figure 8.- Schematic of the MZF Solvent process

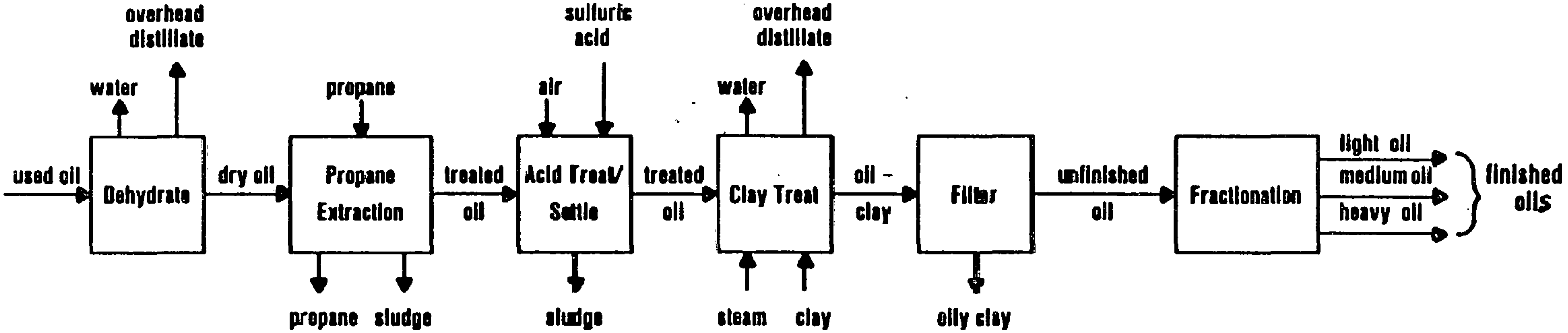


Figure 9.- Schematic of a propane extraction process similar to the IFP

For comparison, in the propane extraction step of Quang (585), dehydrated oil is mixed with recycled liquid propane and sent to a reactor operating at supercritical conditions. The propane and oil mixture are taken off the top, while the insoluble residue is drawn from the reactor bottom. The propane and oil solution is flashed to recover propane which is recycled. The clarified oil is sent to acid/clay treating equipment.

3.1.3.1 Availability of Equipment for Solvent Treatment

Equipment for solvent treatment using either the BERC or MZF process is quite simple and requires nothing extraordinary. The essential equipment includes such items as tanks, mixers, pumps, a stripping still, and valves, and all of these items are off-the-shelf variety. Figure 10 shows a simple example of a solvent treatment tank with four 1-inch ball valves at various levels on the side of the tank and a 2-inch gate valve at the bottom of the cone for removing sludge.

The exception to good availability of equipment is that for propane extraction which requires keeping the propane at its critical point. The temperature and pressure required to keep propane and/or CO_2 in a dense gas state requires specially engineered equipment with appropriate controls. This equipment would require pre-design and custom construction, whereas, equipment for most other solvent processes might be salvaged from defunct chemical plants or purchased without the expense of engineering design costs.

3.1.3.2 Cost (Capital and Operational)

The process equipment costs to solvent treat dehydrated motor oil as defined by the BERC process were estimated by Bigda and Cowan (59) in 1977 at $100.4K. Adjustment for the rise in the consumer price index raises this estimate to near $160K. The equipment to recover the solvent was estimated at $154.9K or an adjusted $245K. The total equipment cost to solvent treat and recover the solvent, based upon this earlier study, is above $405K.

Equipment for the MZF solvent treatment technique has been less well defined in the literature, but it is reasonable to assume that the equipment costs would approximate those proposed by Bigda et al for the BERC solvent treatment process.

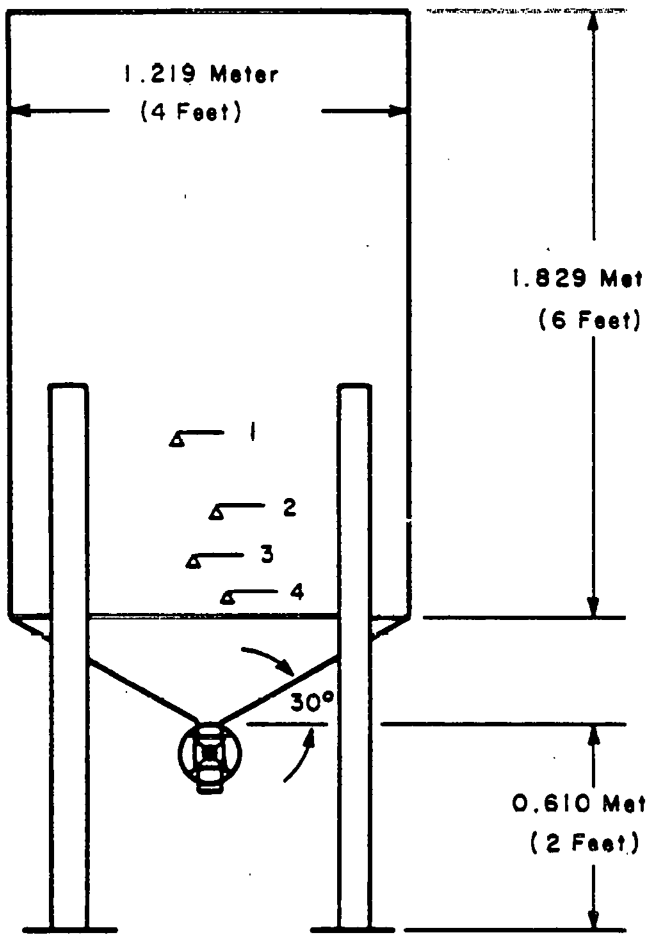


Figure 10.- Twenty - one hundred liter (550 gallon) solvent treatment tank

The capital costs of the IFP propane supercritical extraction process are also not well defined. However, Quang (585,586) provided an erected cost, battery limits (French basis) for a 12 million gallon per year plant at $700K. Adjusting by 100 percent for the U.S. increase in the consumer price index since that estimate would make the plant cost in the range of $1.4 million. Considering, the various studies that have been done on processes less complex than the Selectopropane technology and the estimates of plant capital investment, this value is probably considerably understated. Note this estimate is for a complete plant rather than just the solvent treatment section.

Operational costs for the various solvent treatment techniques can, at best, only be estimated within the scope of this study. A study by Aerospace in 1978 (394) made comparisons of process energy requirements for several processes and these are tabulated below:

	Process Energy	
Process	Million Btu per Barrel Used Oil Feedstock	Million Btu per Barrel Fractionated Product
BERC Solvents	0.97	1.37
MZF Solvents	0.79	1.03
Propane Solvent	0.99	1.21

Bigda and Cowan (59) estimated the manufacturing or operational costs for the BERC solvent process incorporating cost of the feedstock. Since the conversion of RP to F-76 eliminates the factor of feedstock cost, the Bigda numbers have been adjusted for both the cost of feedstock and for the increase in the consumer price index since 1977. With these corrections, operating expenses for the BERC solvent treatment process would be $0.15 to $0.20 per gallon. If, one includes the other process steps of the BERC technology which are dehydration, fractional distillation, and a polishing step such as clay-contacting, the cost would escalate to about $0.39.

Assuming the above estimate to be somewhat reasonable, one could project the operational costs of the MZF solvent treatment technology at \$0.10 to \$0.25 per gallon and the propane solvent treatment at \$0.10 to \$0.25 per gallon.

3.1.3.3 Reliability of Solvent Treatment

The reliability of solvent treatment in terms of product acceptability cannot be addressed without data from further testing. As discussed in an earlier part of this section, solvents alone do not necessarily represent a viable technology for the conversion of RP to F-76. Bench-scale and pilot-scale studies probably would have to be conducted to determine the effectiveness of solvents and/or supercritical extraction as an effective tool for converting RP to F-76. Reliability in terms of day-to-day dependability of equipment should be another matter, and it is estimated that the unsophisticated process equipment required to solvent treat would lend itself to a high degree of equipment reliability. The IFP propane solvent equipment and other supercritical extraction apparatus is somewhat more complex requiring pressurization and elevated temperatures and therefore could be less reliable.

3.1.3.4 Flexibility of Solvent Treatment

Flexibility could be addressed in terms of ability to alter solvent-to-fuel ratios, change mixing and stirring rates, extend or shorten contact times, etc. In these terms the solvent technology represents a very flexible tool for the treatment of fuels and/or oils for the removal of certain suspended particulates and sludges. However, the application of critical extraction techniques would narrow the limits of parameter flexibility to a degree.

3.1.3.5 Simplicity of Solvent Treatment

In relationship to many re-refining schemes and processes, solvent treatment represents a very simple technique. Hydrogenation, fractional distillation, and chemical treatments generally are more complex. The supercritical solvent system represents the most complex technology of those discussed in this section.

3.1.3.6 Environmental Impact of Solvent Treatment

Solvent treatment produces two primary by-products that require control to prevent adverse environmental impact. These include:

residual solvent and vapors
precipitated sludge

Technology developed by the petroleum industry for the dewaxing and deasphalting steps in crude oil refining (292-330) include the use of both propane and other solvents such as MEK and/or furfural. The industry has been able to develop engineering concepts that cope with the problems of solvent recovery and with vapor control. These problems are therefore felt to be a matter of applying engineering design principles that have already been developed.

The disposal of sludge derived from solvent treatment of used lubricating oil is not as well researched and the little work that has been performed (749) indicates that the sludge from the BERC solvent technology does not have great potential as an asphalt extender. Sludge from supercritical extraction is also an unknown environmental factor that should be investigated in Phase II.

3.1.4 Chemical Treatment

Perhaps the most prominent of the chemical treatment technologies for re-refining applications is that proposed by the Phillips Re-refined Oil Process (PROP) (362,363,364,431,432,433,530). It was developed, patented, marketed, and licensed by the Phillips Petroleum Company of Bartlesville, OK. Very briefly, this process as shown in Figure 11 involves the precipitation of metal contaminants from the used oil as ammonium salts. The resultant oil is flash distilled to remove water and light hydrocarbons. Solid absorbents are then contacted with the oil to remove the major polar compounds such as sulfates and oxides of metals. Reaction with hydrogen gas (hydrogenation) over a catalyst bed completes the removal of impurities in the oil. The process was initially designed to process only feedstock from normal crankcase drainings and excluded other types of used oil such as industrial oils and fuel oils. The original marketing approach for the process was to offer a prefabricated plant, sized to meet specific volume requirements.

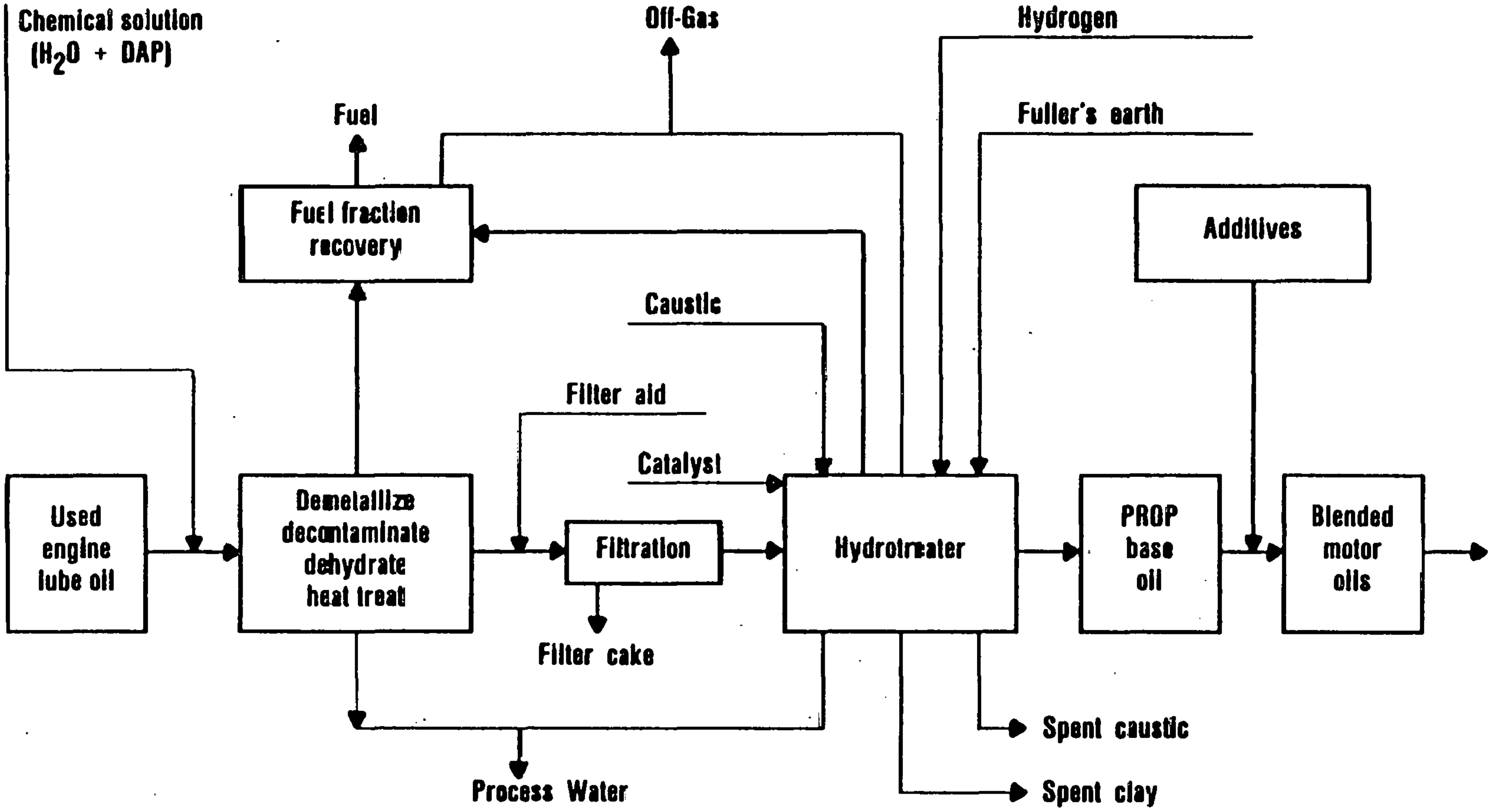


FIGURE 11. - Schematic of the PROP process

The PROP process, may have some application to the re-refining of lower boiling material such as RP. Phillips has found that the ammonium salt requires a soak temperature in the range of 550° F in order to effectively precipitate sludge as a salt. This temperature is near the 50 percent point of RP and, therefore, one would expect to lose the large part of the sample. However, it might be possible, through the use of pressurized equipment, to achieve the reaction temperature required to precipitate the additives and sludges with the diammonium phosphate without losing the product. Therefore, the PROP process is not eliminated from consideration. The PROP process, of course, contains several other steps some of which might be required to treat RP and others that may not be applicable. In summary, the PROP, may be applicable, either in part or in its entirety, to the processing of RP to F-76.

There are other methods that use chemicals to re-refine used lubricating oil, but most of the processes include steps other than the chemical treatment such as dehydration, filtering, heating, distillation, clay and/or combinations of these.

In addition to having developed and patented chemical-based polishing (finishing) steps for re-refining processes (<u>172</u>,<u>535</u>), Richard O'Blasny of Delta Central Refining has also developed a chemical pretreatment step that appears to have promise. ("Re-refining Used Lubricating Oils with Hydride Reducing Agents", U.S. Patent Application Number 336900). In this treatment, about 150 ppm of sodium borohydride in a caustic solution is combined with the waste hydrocarbon. O'Blasny has found that this pretreatment has several beneficial effects; 1) the acid number of the waste hydrocarbon is substantially reduced; 2) the corrosiveness of the waste hydrocarbon is vastly improved as indicated by copper strip corrosion tests (typically from a 3c to a 1a or 1b; 3) samples that tend to coke or foul heat exchangers and distillation equipment have improved thermal stability characteristics; and, 4) metallic and elemental contaminants such as lead, zinc, and phosphorous are reduced to a greater extent after distillation by thin-film technology than untreated samples.

Borenstein (68) has a method of treating waste oil using ammonium persulfate and a nonionic surfactant. Anhydrous zinc chloride was used by Clark (108). Eberle (153) used aluminum oxide powder and Friel (181) used an anionic surfactant in combination with polyalklene polyamine. Other such methods are documented in Volume 2, Appendix III of this report.

One alternative chemical treatment uses caustic. This methodology has been used as a re-refining process (162,578,594) both by itself and in conjunction with clay contacting. Figure 12 shows a typical schematic diagram of caustic/clay methodology. At least one technique employing caustic (sodium hydroxide) to precipitate sludges from used oil first diluted the lubricating oil with a low-boiling (160 to 250° F) petroleum fraction. The diluted oil was then combined with caustic and heated with refluxing for four hours at about 250° F. After settling or filtering, the oil was distilled or clay contacted. Distillation was performed to recover the light petroleum fraction and may not be essential in the application of caustic treatment to RP.

Caustic has the advantage of neutralizing acidic components in the treated oil or fuel and it has been suggested by users of the technology that preliminary caustic treatment helps reduce suspended water during a heated settling period. The presence of caustic also has the potential advantage of reacting with acids produced through thermal degradation of chlorinated or halogenated solvents that may be present in RP.

None of the additional chemical treatments listed have had the testing and investigation that the caustic and PROP chemical treatment processes have had. The possibility of direct application of these latter processes to a hydrocarbon mixture with similar contaminants but a lower boiling range appears to have more promise than techniques that lack even pilot scale applications to any form of contaminated hydrocarbons.

3.1.4.1 Availability of Equipment for Chemical Treatment

Equipment required to chemical treat RP or FOR using sodium borohydride or caustic is probably readily available. Until recently, the equipment to utilize the PROP chemical process was less readily available. Phillips Petroleum Company has indicated a change of policy regarding its marketing procedures for the PROP process. They are expected to discontinue the manufacture of a "turn-key" plant in favor of licensing the process. In light

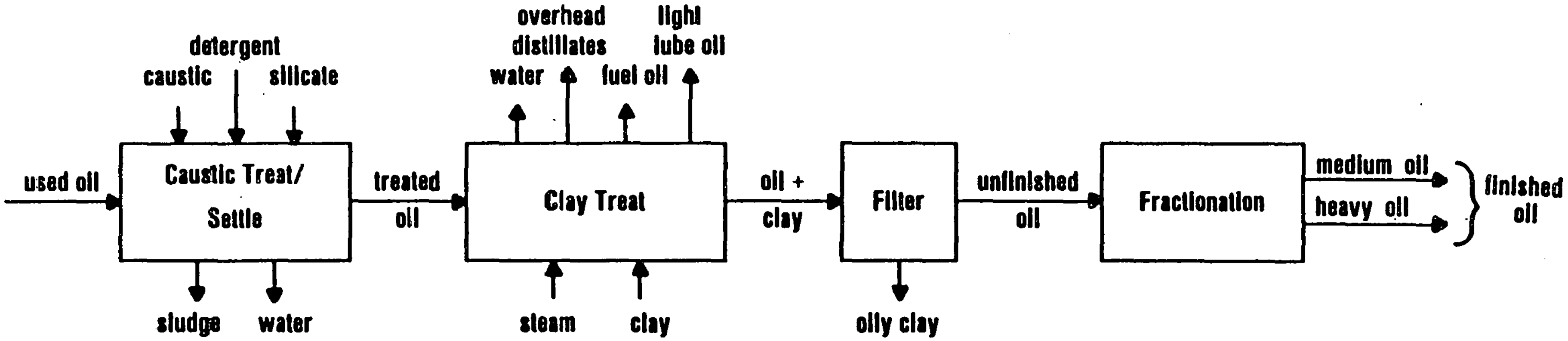


Figure 12.- Schematic of a caustic-clay process

of this development, use of some salvaged equipment as well as standard off-the-shelf apparatus could affect the availability of equipment. Much of the equipment is fairly standard in terms of tanks, pumps, valves, mixers, and heaters. What is less standard is the hydrotreater and the availability of such small capacity hydrotreating equipment as an off-the-shelf item is highly unlikely. Whether or not the chemical treatment step of PROP would be sufficient to convert RP to F-76 is not known although it does not seem likely that such a treatment, alone, would be sufficient.

3.1.4.2 Cost (Capital and Operational)

As a turn-key operation, the cost of a $10 million gallon per year PROP plant including land, permits, tankage, and a fully-assembled and commissioned PROP unit has been estimated at $22 million. This cost did not include distillation equipment except for the flash evaporator to remove water and light hydrocarbons. A unit for chemical treating, only, would cost only a fraction of the estimate, however.

Capital costs for equipment to caustic treat RP would probably be in the range of acid/clay treatment equipment. The type of apparatus and application is very similar to the acid technology. Thus, it is possible that a turn-key 5 million gallon per year caustic treatment facility would require an initial capital investment of $1 million to $2 million assuming that all new equipment was needed.

Operational costs for a 10 million gallon per year PROP plant made by Bigda and Cowan (62) were estimated at $0.13 per gallon which included chemicals, utilities, labor, and maintenance but not feedstock costs. Even adjustment for the increase in the consumer price index does not correspond to current estimates. The operating costs for the PROP technology is currently about $0.35 per gallon.

Operational costs for caustic treatment can only be very roughly estimated. However, costs would be very similar to those of an equivalent acid/clay plant and could range anywhere from $0.20 to $0.40 per gallon.

O'Blasny has estimated the operational cost of his sodium borohydride treatment at about $0.05 per gallon but this does not include utility costs. For lack of a better estimate, this value has been recorded in Table 1.

3.1.4.3 Reliability of Chemical Treatment

The PROP process does not have a good track record for reliability based upon the first two plants that were constructed. Later data from newer plants is not available. Caustic treatment needs considerable operator attention to prevent formation of soaps and therefore is considered of only fair reliability. Sodium borohydride is of unknown reliability but should be considered in Phase II investigations.

Two new PROP plants have been sold and erected recently. One for Shell in Toronto, Canada, and the other for Texaco in Mexico. Both of these plants intend to use distillation as part of the over-all process. Although the construction of both plants is complete and the Canadian plant is in operation, independent reports are not yet available on its success either technically or economically.

3.1.4.4 Flexibility of Chemical Treatment

The PROP process is not extremely flexible in that the removal of gross contaminants depends upon using a sufficient quantity but not an excess of an ammonium salt. Further, the PROP process becomes quite inflexible unless a distillation step is incorporated. Mixtures of products can not be separated. Needless to say, chemical treatment as proposed by PROP does not appear extremely attractive for separating used lubricating oil from RP.

The same limitations on flexibility seem to apply to the caustic chemical treatment and the flexibility of sodium borohydride is unknown.

3.1.4.5 Simplicity of Chemical Treatment

The PROP process probably approaches the BERC solvent process and the IFP propane re-refining schemes in its complexity and therefore is one of the more complex re-refining technologies available today. Taking the chemical step only, from the over-all process, changes the aspects of complexity considerably. Likewise the caustic treatment technology, at first glance, seems quite simple but can become quite complex if soaps or emulsions are formed providing poor yields of finished product. Sodium borohydride probably represents a slightly more complex process.

3.1.4.6 Environmental Impact of Chemical Treatment

The environmental concerns of chemical treatment as related to the PROP process can be enumerated as follows:

metallic phosphate salt-type filter cake
water of dehydration and free water
non-condensable gases
volatile organic compounds

The metallic phosphate filter cake is reportedly a neutral salt and therefore would be amenable to some controlled landfills. The water of dehydration will probably be combined with free water that is off-loaded with the feedstock. This water contains a variety of contaminants including halogens, phenolics, volatile organic compounds, and in some cases traces of pesticides. A secondary water-treatment system is the only way to handle this by-product stream from the process.

Mercaptans and non-condensable gases including hydrogen sulfide can be removed by scrubber technology using caustic and other appropriate chemicals. Flaring of toxic hydrogen sulfide is not permitted in many states although it represents the simplest and less costly procedure.

The environmental impact of caustic treatment is similar to other chemical treatments. Whether a neutralization step of the caustic-precipitated sludge would be required prior to landfilling is not known. It is possible that the use of caustic could solve some environmental problems such as those created by halogenated organics that thermally decompose into acidic materials. It is also probable that spent caustic would present little or no problem in disposition for it has been found that most handlers of hazardous waste must purchase large quantities of caustic to treat their wastes and therefore will often haul away spent caustic without charge.

3.1.5 Hydrotreatment

Hydrogenation combined with one or more of the primary treatment steps such as distillation or chemical treatment or both has tremendous potential in producing a quality product essentially free of color, odor and oxygenates. The attractiveness of such a combination of techniques was sufficient to warrant preliminary pilot-scale tests on 15,000 gallons of RP by Mohawk

Lubricants Ltd. in Vancouver, B.C. during Phase I investigations. Mohawk is a re-refiner of used lubricating oil and uses thin-film evaporator/hydrogenation technology. The details of this preliminary work are in section 4.2 of this report.

In September of 1973, Bethea et al (56) published details of successful research that had been performed for the hydrotreatment of waste lube oil. They found that quite ordinary catalyst such as Nalcomo 471 (cobalt-molybdate) at 600° F and 650 psig, 1 V/V, and 1,600 scf/b of hydrogen was adequate to produce a finished oil with a color of 0.5 ASTM and low neutralization numbers, sulfur, oxygen, and nitrogen contents.

Hydrogenation is considered as one of two or three major options in the over-all re-refining of used oil. Hydrogenation has not been advocated as a "sole" technology for processing of any petroleum product, whether virgin derived or from previously used products. The primary reason that hydrotreatment is not a viable process in itself is the inability of hydrogenation to remove particulates and to separate different boiling species or to cope with contaminants that form coke or poison the catalyst.

Hydrogenation, as applied to re-refining processes, is shown in Figure 13. It is similar to desulfurization in the petroleum industry. It is actually a mild selective treatment; so mild that aromatics are not usually hydrogenated to naphthenes but sulfur is removed as hydrogen sulfide and the remaining hydrocarbon part of the molecule is hydrogenated. The transfer of hydrogen with heteroatoms (sulfur, nitrogen, and oxygen) in the sample is accomplished by passing the liquid hydrocarbon sample at an elevated temperature through a catalyst with a counter-current flow of hydrogen gas at the same temperature. Catalysts commonly used in re-refining include cobalt-molybdate and nickel-molybdate. Temperatures are usually 650° F or less and pressures range from 700 to 1000 psig. Desulfurization of most petroleum fractions requires up to 150 standard cubic feet (scf) of hydrogen per barrel of oil.

To prevent poisoning the catalyst with contaminants a guard-bed is often placed between the incoming feedstock and the catalyst reactor which is packed with a material such as a silicate that will adsorb the poisoning agents before they reach the catalyst.

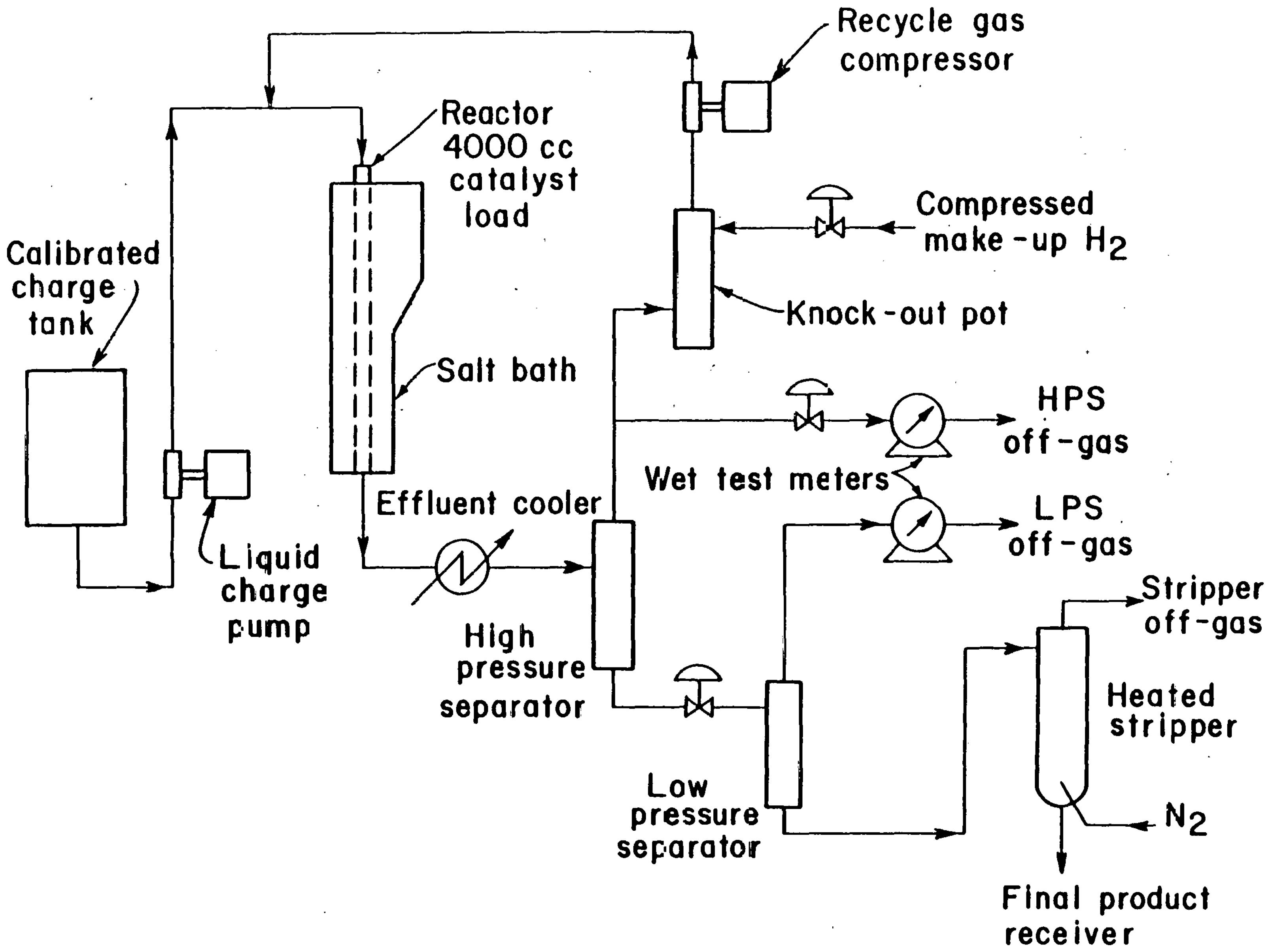


Figure 13.- Simplified flow diagram of Suntech hydrogenation unit

3.1.5.1 Availability of Equipment for Hydrogenation

Small hydrogenation equipment, sized to handle 800 barrels a day or less, is not a common off-the-shelf item and therefore is not readily available. The petroleum industry typically processes much larger quantities than those processed in re-refining. Even so, the hydrogenation equipment used by the petroleum industry is typically custom designed for the process, feedstock, and application of the processor.

Although basic engineering skill needed to acquire a hydrotreater for a particular application is available, the apparatus cannot be obtained in a turn-key mode.

Phillips has provided a hydrotreater as part of their PROP skid-mount plant in the past, but since discontinuing the marketing of such a turn-key re-refinery, it is doubtful they will be providing such in the future. It is possible that Phillips would license or provide the design of their hydrogenator without the rest of the process and this possibility could be explored in Phase II studies.

KTI, a Netherlands based company, developed a process (257) for the re-refining of used lubricating oil that uses hydrogenation and is shown in Figure 14. KTI was approached a few years ago for purposes of obtaining rights and design details for the hydrotreating section of their process. KTI was not willing at that time to provide such services or information.

The IFP process (139,585,586) developed by the French, advocates hydrogenation and at least two plants are in operation in Europe. This equipment, too, was designed and constructed specifically for the two plants that employed the technique.

3.1.5.2 Cost (Capital and Operational) for Hydrogenation

In view of the lack of skid-mount turn-key hydrogenation equipment, it is necessary to look to other sources for estimates of equipment costs. It is reported that a re-refinery in the United States that recently installed a custom designed hydrotreater for the re-refining of used lubricating oil at a rate of about 800 barrels per day (10 million gallons per year) cost in the

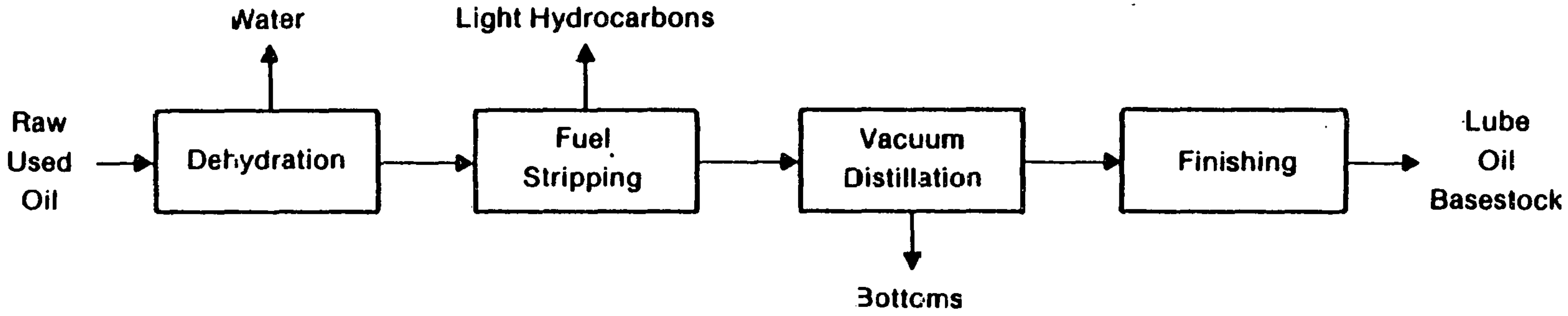


Figure 14.- Schematic of KTI process

vicinity of $1 million completely engineered and constructed. This cost was broken down to $700K for the equipment and another $300K for assembly and construction.

Operational costs which include hydrogen and catalyst are nearly as illusive as are capital costs. Hydrogen costs can vary greatly depending upon the location of the plant. Often a major petroleum refinery will have an excess of hydrogen and a plant close to such a source of hydrogen will significantly lower the cost of that item. Based upon a consumption of 160 cubic feet of hydrogen per barrel of oil (a factor of four higher than Bethea (56) found necessary) and assuming a cost of about $3.50 per thousand cubic feet of hydrogen, the annual cost for hydrogen would be near $130K or about $0.013 per gallon of oil.

Catalyst life is another factor that must be considered in operational costs. Good documented data do not currently exist for the life of a hydrogenation catalyst in a re-refining operation but a rule of thumb used by the petroluem industry is that hydrogenation is marginally economically acceptable if 1 pound of catalyst will process 20 barrels of oil. A more acceptable standard would be 1 pound of catalyst per 100 barrels of oil (120). At a cost of $3.00 to $4.00 per pound of catalyst, and using an intermediate value of 50 barrels of oil per pound of catalyst, the annual cost of catalyst would be near $17K or $0.00167 per gallon of oil.

To protect the catalyst against accidental or incidious deterioration, guard-bed techniques are used. Typically a guard-bed ahead of the catalyst will contain about the same amount of material as catalyst but the cost is less. For estimation purposes, doubling the estimated cost of catalyst would cover the cost of guard-bed material. This would bring the annual cost for catalyst materials to $34K and about $0.003 per gallon of oil.

Although hydrogenation is a more complex technique than clay-contacting, once the plant is commissioned and personnel are trained the process is not labor intensive and it represents a continuous process rather than batch-mode. Utilities would cost about 30 percent more for a hydrogenation unit than for a clay contacting unit but the labor costs would be reversed, somewhat compensating these costs.

In light of the limited data available for capital and operational costs, this information would have to be developed later if this mode of operation was of apparent attractiveness in the conversion of RP to F-76.

3.1.5.3 Reliability of Hydrotreaters

Well-designed and constructed hydrogenators provided with feedstock for which the unit was designed, are reliable. This conclusion is based upon experience of the petroleum companies in their hydrofining operations and upon the limited amount of hydrotreating data that has been forthcoming in the re-refining of used oils by Phillips Petroleum Company and at Mohawk Refining Company in Vancouver, B.C.

3.1.5.4 Flexibility of Hydrotreaters

Generally, hydrogenation equipment is not extremely flexible. The flexibility is limited to the extent that feedstocks of varying viscosities ranging from fuel oil to the heaviest automotive lubricating oil basestocks can be hydrotreated if initial contamination has been reduced to acceptable levels. The same equipment can be, but is not necessarily, flexible over a wide range of temperatures and pressures.

3.1.5.5 Complexity of Hydrotreaters

Hydrotreaters are complex in a relative sense but a well-designed unit will have built-in level and flow controls and safety devices that permit ordinary operation with only a single operator in attendance to monitor instrumentation. As long as the unit continues to operate within the limits of its design capability one could say that hydrotreaters are simple. But given problems that often arise with such operations, one would have to categorize hydrotreaters somewhere between simple and complex.

3.1.5.6 Environmental Impact of Hydrotreating

The by-product streams from hydrotreating are tabulated below and include:

noncondensable gases
volatile organic compounds
contaminated guard-bed material
spent catalyst

The control of by-product streams from hydrotreating operations has been well researched by the petroleum industry and other users of this technology and the environmental control of these by-products is well understood making this an acceptable environmental rating.

The non-condensable gases include hydrogen sulfide which is a toxic substance and cannot be discharged to the atmosphere. Two common techniques of handling the non-condensables is by flares where acceptable and, otherwise, scrubber technology which removes these non-condensables through reaction with chemicals such as caustic (sodium hydroxide).

The volatile organic compounds can be legally flared if they can be separated from the toxic gases. Generally, the temperature of these volatile compounds is reduced through either venturi or heat exchanger techniques and collected after condensation.

In some cases, the guard-bed material can be regenerated through the use of backwashing techniques using steam or other suitable agents. Guard-bed material that cannot be regenerated is land-filled in the type of facility appropriate to the contaminants involved.

Some catalyst manufacturers are prepared to take spent catalyst and in some cases regenerate or otherwise dispose of that material in an environmentally acceptable manner.

3.1.6 Clay Contacting

Perhaps the simplest of all re-refining methods is clay-contacting as shown in Figure 15. Such a technology requires the use of relatively large quantities of activated clay and careful control of feedstock to eliminate mixing of industrial oils, fuels, greases, and other contaminants that require distillation for separation from the primary product. Although Figure 14 shows a distillation step, there has been some evidence that clay contacting, alone, can produce a high quality finished product. Therefore, one cannot discount the possiblity of such technology for the conversion of RP to F-76. The price for activated clay makes this a relatively expensive technical approach despite its simplicity, but not outside the realm of economic feasibility.

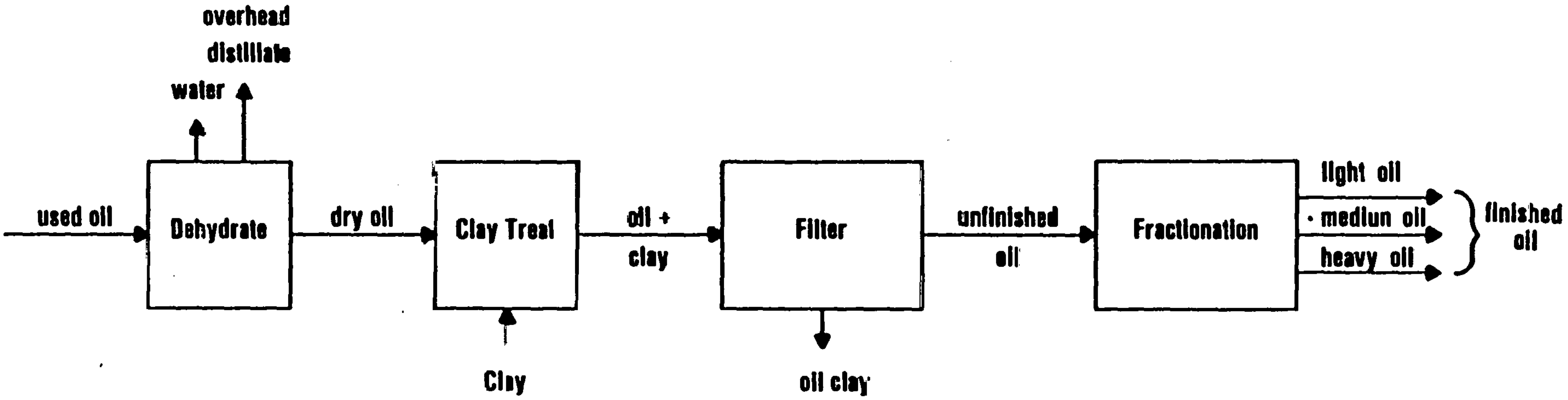


Figure 15.- Schematic of clay treatment

Although clay contacting has been widely used by the petroleum industry, and in particular by the re-refining industry, over the past half century or longer, very little can be found in the literature regarding clay contacting parameters. A study published by the Bartlesville Energy Technology Center in 1979 (598) reported optimum temperature, oil-to-clay ratio, steam-sparge rate, and other parameters. Figure 16 is a simple diagram of equipment used in this study. Otherwise, literature searches have produced little or no information regarding clay contacting parameters.

The types of clay used for re-refining treatment are of two common types. One is manufactured in the U.S. under the tradename of Filtrol which is a sulfuric acid activated montmorillonite (Bentonite) clay. Another type of clay is activated with hydrochloric acid.

3.1.6.1 Availability of Equipment for Clay Contacting

The method used in most clay contacting processes today is so simple that only a few tanks, mixers, valves, and controls are required to put together a batch-oriented clay contacting device. If one were to attempt to automate and build a clay contactor that would work in a continuous mode, engineering design would be required and the result would be a more complex system of controls and apparatus. Even so, the individual items to assemble such a machine are generally available either new or in some instances used equipment can be obtained at a considerable discount in cost.

Since large-scale commerically-available clay contacting equipment (off-the-shelf, turn-key, clay contacting apparatus) does not exist one might assume that the availability of equipment is a serious problem. However, at least one company, Refinoil (597), does manufacture a machine that uses clay and filtration to "re-refine" oil. The unit consists of a mixing tank heated by electric heating elements. It uses a water cooled condenser and vacuum to facilitate removal of light ends during the process. The unit has a filter press in a plate and frame configuration that are faced with both paper and cloth filters. Refinoil claims 60 to 90 percent oil recovery and a finished oil cost between $0.08 and $0.15 per gallon. The time per run, in continuous service is 2 to 3 hours so one can assume the unit can process from 40 to 60 gallons per hour. Refinoil reportedly also makes a larger model which could increase the throughput capacity by a factor of four.

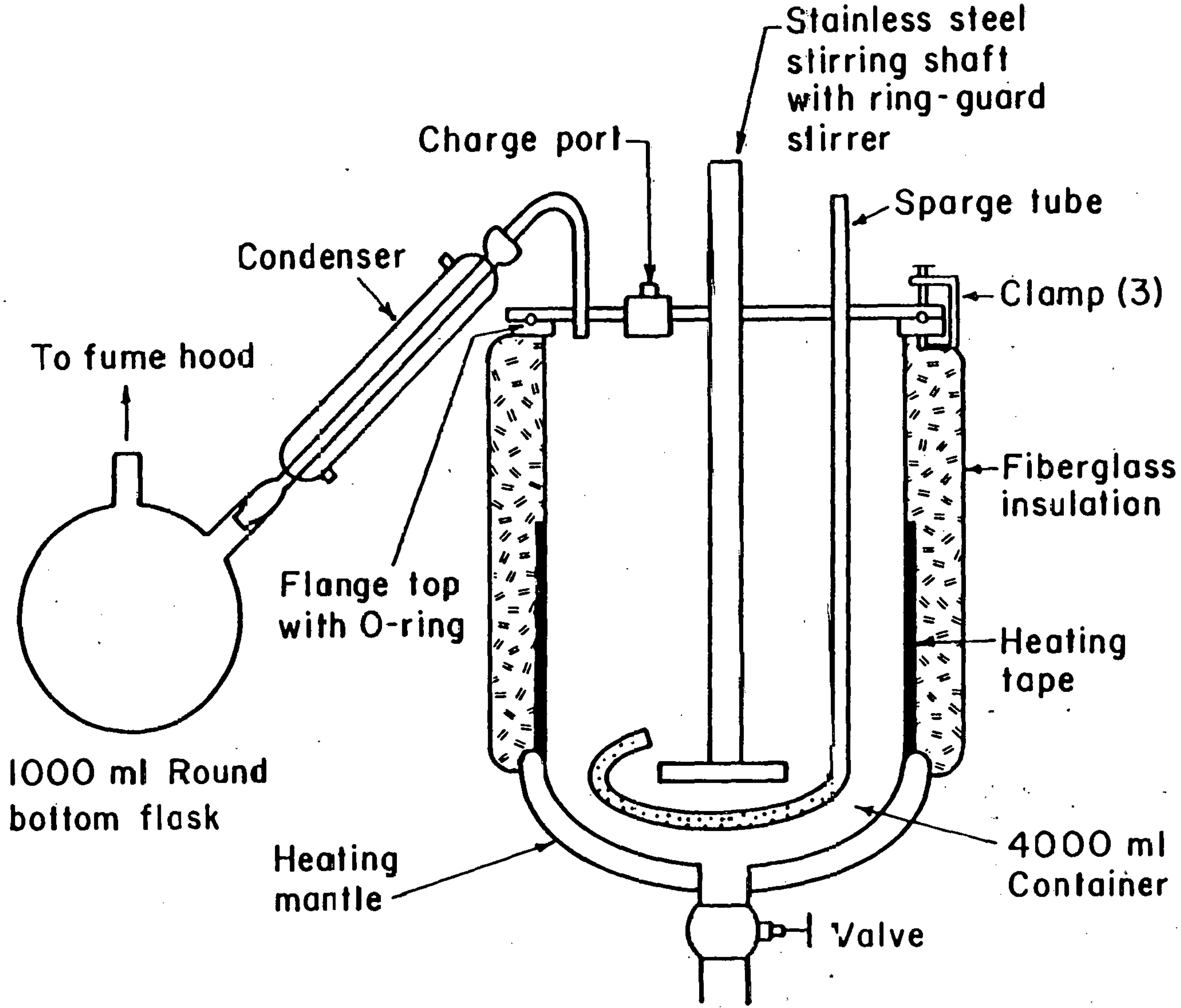


Figure 16.- Clay contacting apparatus

3.1.6.2 Cost (Capital and Operational)

Large scale clay contacting equipment is not available commercially and therefore capital costs are unknown. There are a number of smaller units that combine clay and filtration that could be considered a form of clay contactor but appear to have no application to the conversion of RP to F-76. To assemble a custom designed clay contactor for use with RP or F-76 requires only simple equipment that can be assembled inexpensively. However, clay contacting is a batch process requiring some operator judgments related to temperature, amount of clay, and reaction time. The equipment needed to install clay contacting would probably cost in the vicinity of $100K to $200K if new equipment were used and if some automation were built in to reduce labor requirements.

Operational costs are not well defined. The cost of clay is about $280 per ton (bagged), FOB Jackson, MS, making the cost over $0.14 per pound (not including shipping, which can run $100 per ton) and an annual expenditure of over $100K (based upon 0.75 pounds per gallon). If clay were the only cleansing process used, the amount required might be near 0.3 to 0.75 pounds of clay per gallon or between $0.15 and $0.20 cents per gallon of oil. It is highly probable that much less clay would be required to decontaminate RP--perhaps as little as one-half the amount needed for used lube oil. It has been estimated that the spent clay often contains as much as 20 percent by weight of oil which is not recoverable. This, also, should be a much smaller amount of absorbed RP than for the heavier lubricating oil fractions. However, this loss factor must be taken into consideration in economic evaluations. Other cost factors are the power requirements to heat the oil-clay slurry to an appropriate temperature (550° F for lubricating oil and probably lower for RP) and the disposition of the spent clay.

3.1.6.3 Reliability of Clay Contactors

Clay contactors could generally be considered reliable as long as feedstock to the contactor is fairly consistent. In lieu of automation to make the clay contactor a continuous operation, there is little to cause problems other than operator errors.

3.1.6.4 Flexibility of Clay Contactors

Clay contacting, as a batch process, is flexible but generally each batch of oil or fuel processed requires close monitoring to determine the total amount of clay, the highest temperature to which the slurry is taken and the time the slurry is held at that temperature. One other parameter that is generally monitored carefully is the final temperature at which the oil-clay slurry is filtered. Some operators have reported difficulty in filtering the mixture at ambient temperatures. The viscosity of RP should eliminate any similar problems for filtering.

3.1.6.5 Simplicity of Clay Contactors

Generally speaking, clay contacting is simple reliable and flexible. For example, in a typical re-refining operation using clay contacting, a tank is filled to a predetermined level with oil ready for clay. A measured amount of activated clay is dumped into a tank and the mixer is started while the slurry is circulated through a heat exchanger to bring the temperature up to some desired level. The recirculation is continued, with vapors from the system being condensed in a water-cooled condenser until a "grab" sample from the slurry indicates that the color requirement has been attained. The slurry is then pumped to a filtration device where the clay is removed and the oil is ready for marketing or formulating. In summary, clay contacting equipment is simple and most problems arising can be solved by the addition of more or less clay. A further simplification that might be applicable to RP's is the use of a fixed bed of clay through which the fuel is passed. This could decrease the quantity of caly required dramatically.

3.1.6.6 Environmental Impact of Clay Contacting

The by-products from clay contacting include the following materials that require consideration in an environmental context:

non-condensable vapors
volatile organic compounds
oily clay waste
used filter paper or cartridges

Oily clay waste represents the greatest disposal problem. Weinstein (752) and a DOE/BETC publication by Booz, Allen, and Hamilton (749) addressed some of the problems associated with handling discharges such as vapors and oily clay from waste oil processing. The non-condensable vapors that distill from the clay contactor were not considered of sufficient significance to warrant any special treatment or disposition. Note, however, that in some states and some localities it might be necessary to collect and neutralize these non-condensables because of their offensive odor. Flaring and scrubber technology are two apparent solutions to the disposition of these non-condensables.

Volatile organic vapors represent a problem with most petroleum storage and/or processing systems. When organic vapors reach a level that represents a hazardous condition, as defined by EPA, then measures such as water-cooled condensers must be installed to knock back (condense and collect) these volatile hydrocarbons. They have potential as a fuel and so the cost of collection is partially compensated.

Oily clay wastes represent the most serious environmental problem associated with clay contacting. The clay contains active sites created during acid activation that adsorb contaminants in the fuel containing metals, sulfur, nitrogen, and oxygen. Additionally, a sizable amount of oil is absorbed into the clay. An efficient filtering system will squeeze a portion of the oil from the spent clay leaving what appears to be an almost dry residue. However, tests have shown that as much as 20 percent by weight of the spent clay can be absorbed oil or fuel. This lost material varies with efficiency of the filtering system but at best represents a substantial quantity of oil.

The absorbed oil and contaminants in spent clay are a problem both environmentally and economically. In the latter context, one can assume that the lost oil has been restored to its orginal quality and therefore it's value is near that of the finished product rather than that of the feedstock. So economically, one must assume that the clay treatment loses as much as one-fifth the total finished product. More disturbing, however, is the difficulty of disposition of oily clay. Formerly, one could landfill such material without question or political problems. However, as environmental awareness has increased over the past 50 years, so has the scrutiny to which such disposables as oily clay are subjected. Today, few municipal landfills will accept oily clay wastes because of the leachability of both the oil and the adsorbed contaminants. Therefore, many users of clay contacting have either taken their oily clay to hazardous waste landfills, which is at considerable expense, or discontinued the use of the technique.

There is some indication, however, that there may be alternatives to landfilling of oily clay wastes. Berkau et al, (51) reported that spent clay from the finishing of special lubricating oils containing 25 to 30 percent oil still had enough residual activity to be used in processing used industrial oils. British Petroleum (77) has reported that oily clay can be made acceptable for landfilling by removing oil with a series of solvents. And Thibodeaux (698) has successfully studied landfarming of biodegradable petroleum wastes such as oily clay. Other uses of oily clay include drilling mud emulsification (331). There has also been some undocumented work in Europe to reclaim the caloric content of the oily clay through incineration. The clay residue is then acceptable as landfill but it has also been suggested that the fired clay might have some commerical use in the ceramics industry.

3.1.7 Electromagnetic Radiation and Other Miscellaneous Techniques

In 1983, Klaila, (394) patented a process for controlling the fluidity of hydrocarbons through electromagnetic heating. The principle of electromagnetic heating is similar to that used in microwave appliances. The technique appears to have application where the flow characteristics are adversely affected by viscosity. Heating by electromagnetic radiation reduces the viscosity to the point where desired fluidity can be achieved. Possibly the technique could be used to facilitate the precipitation of particulates or the coagulation of potential sludges from contaminated fuels. The costs would seem to be minimal.

Centrifuging is a popular concept that is the essential technology in many smaller devices that are currently marketed as oil purifiers. Svensson (676) and Schwarz (626) have recommended a shipboard centrifugal separator to pretreat and cleanse F-76. These devices work well on particulate contaminants that can be settled out. A centrifuge will ordinarily not achieve any separation that cannot be accomplished by simple settling. The advantage is in the time required to achieve the desired separation. Centrifuging is a technique that can succeed to a limited extent in removing water and particulates from samples of petroleum that do not contain detergents or dispersants. However, when these latter additives are present, centrifuging generally is unsuccessful in substantially reducing suspended materials.

Still another breed of commercially available devices marketed as oil purifiers are essentially a combination of a heater to vaporize water that accumulates with the fuel or oil and a cartridge-type filter to remove particulates (112,542,577). These devices are available from a size small enough to fit easily beneath the hood of a modern automobile to plantsized apparatus that will handle hundreds of gallons of oil per hour. A study made in 1981 (270) concluded that although such devices do no harm to the engine, they were essentially useless in removing sludge precursors and not cost effective in extending the life of an engine oil. In this light, it appears unlikely that such devices would have significant application in the conversion of RP to F-76.

Ultrafiltration (32,139,233,278,390,453,775), which includes the concept of reverse osmosis, appears to be applicable to the general area of waste oil re-refining and therefore has potential in the conversion of RP to F-76. In the ultrafiltration techniques, a solvent and components of low molecular weight can transfer through a membrane, but the heavy components cannot.

A variety of membranes can be used in ultrafiltration. For example, Defives (139) used acrylo-nitrile copolymer membranes to re-refine used lubricating oil and found that only clay contacting was required in addition to the ultrafiltration to produce a good quality product.

One of the classic ultrafiltration applications is separation of emulsions. An emulsion can be visualized as a homogeneous dispersion of small droplets of oil in water. Ultrafiltration can remove a large portion of the water from the mixture. Claims for ultrafiltration include a compact design, low pressure operation, high membrane output, low maintenance, outstanding cleanability, versatility, low first cost, low membrane replacement cost, and low utility consumption. Ultrafiltration appears to have potential justifying investigation in Phase II studies.

4. INDUSTRIAL CAPABILITY

There are a relatively large number of businesses in the U.S. that combine the collecting of waste hydrocarbons with the marketing of fuel derived from these liquids (352,547,555). These operations probably number in the hundreds but there is no central association through which these collectors/marketers can be identified easily.

The used/waste hydrocarbon collection business in the United States is not one of the better managed and disciplined endeavors. Oil collection is highly competitive in most metropolitan areas, and the opportunities for indiscretions in everyday operations are numerous. Therefore, the oil that is collected is more often than not contaminated with solvents, paint thinners, pesticides, PCB's, fuel, greases, antifreeze, water and halogenated parts-cleaners. It is not too unusual to find vegetable and animal fats and oils included with mineral oil collections.

The above facts makes the production of an acceptable fuel oil from used oil (street drainings) a difficult task and should be kept in mind when considering the application of commercial processing to RP for conversion to F-76. It points to the high risk of contamination at the processing facility if the Navy has a less than outstanding recycler process their RP.

A few of the more active processors of used oil into fuel oil with viable collection, processing, and marketing facilities include:

Dearborn Refining, Dearborn, MI
Petrocon, Philadelphia, PA
Baumgardner Company, Fayetteville, PA
Louisiana Oil and Refining Co., Baton Rouge, LA
Intermountain Oil, UT
International Petroleum Corp., FL
Research Oil Co., Cleveland, OH

4.1 LOCATION OF PRINCIPAL RE-REFINING INDUSTRIES

In addition to the hundreds of similar operations to those described above, there are a few re-refining operations in the United States that take used lubricating oil and produce a re-refined product that is suitable for many, if not all, lubricant applications. The major companies that are known to be operational include:

Booth Refining Co., Buffalo, NY
Cam-Or, Westville, IN
Cam-Or of Texas, Houston, TX
Central Refining Corp., Springfield, IL
Consolidated Recycling, Troy, IN
Gurley Oil Co., Memphis, TN
Midland Refining, Wichita, KS
Motor Oil Refining, Chicago, IL
Lakewood Oil Service, Los Angeles, CA
Warden Oil Co., Minneapolis, MN

The largest of these operations are Motor Oils Refining who have a plant in Chicago, and Cam-Or with plants in Westville, IN, and Houston, TX. Booth Refining Co. is probably the third largest in the United States. Recently a large plant was built in Shippensburg, PA by Energy Resources, but financial problems halted construction just before the plant was completed, and the new plant is currently idle. Another plant that uses the Phillips PROP process and which was installed by the State of North Carolina has recently shut down for financial and political reasons.

Among the manufacturers of small-scale portable equipment for general application to problems related to contaminated fuels and oil, there are a few with laboratory and pilot plant facilities. Those that were contacted and returned affirmative replies included:

Lancy, Zelienople, PA - Oil/water separation equipment. Lancy maintains a complete treatability laboratory facility as well as a certified analytical laboratory under the direction of Lancy Laboratories.

Racor, Modesto, CA - Primarily oil/water separation using filtration systems. Racor offers two types of "Recycle/Blending" units and has pilot-scale testing facilities.

Hilliard, Elmira, NY - Cartridge filtration type of apparatus. Hilliard has laboratory facilities and invites samples sent for testing.

Howe-Baker Engineers, Inc. and Riley-Beaird - Market small skid mounted crude oil distillation units that might be applicable to reclaiming FOR.

Centrico, Inc. Northvale, NJ - Markets centrifuges for reclaiming diesel oils which are contaminated by water and/or heavy fuel oils.

Emulsions Control, Inc., National City, CA - Suggest chemicals and dehydration to remove water and sediment from waste oil. They provide an ECO demulsifier.

Baron & Associates, Inc. Cookville, TN - Design and custom build vacuum distillation process equipment for removing water and volatile contaminants.

4.2 DESCRIPTION OF PROCESSES, CAPACITIES, AND PRODUCTS

The process combination that is common to the largest commercial re-refiners in the U.S. is that of distillation/clay. Water is removed by a flash evaporation, generally at atomspheric conditions. This is followed by fuel stripping using a vacuum flash evaporator. The oil is then sent through one or more thin-film evaporators to produce one or more overhead fractions and a distillation residue. The overhead fractions are subsequently clay-contacted to improve color and odor. The finished products usually have acceptable properties for use as a lubricant basestock. The basestock(s) is sold as a blending stock or in some cases is formulated with additives and sold as a finished lubricating oil. The plants using technology similar to this include Motor Oils Refining, Cam-Or, Inc., Gurley, Central Refining, Consolidated Recycling, and Booth Refining.

At least one manufacturer, Warden Oil, is using dehydration/clay as the sole technology for converting used oil into a re-refined product. By careful control of the feedstock quality and by segregation of industrial oils, fuels, lube oils, greases, solvents, etc. the process works quite successfully in producing a good finished product although the economics of such treatment are considered marginal in the re-refining of used lubricating oil.

The capacities of a few of the major re-refiners in the U.S. are tabulated below. Numbers are assumed to be rough estimates but actual throughput of any given plant is of proprietary concern and so accurate data are not available for this report. Volumes are assumed to reflect input to the plant.

Motor Oils Refining, Chicago - 15 million gallons per year
Cam-Or, Westville - 15 million gallons per year
Cam-Or of Texas - 10 million gallons per year
Booth Oil - 8 million gallons per year
Lakewood, Los Angeles - 7 million gallons per year

The products produced by each of these facilities fall into the categories of fuel oil and lubricating oil. The fuel is used on-site within the process or is sold as a no. 2 fuel oil. In most cases, lube basestocks are marketed to blenders who formulate the oil to the specificaton of their consumers. In a few cases, the re-refiner formulates the basestock and markets finished lubricating oil products.

The types of lubricating oil that can be produced from these re-refined basestocks include:

motor oil
hydraulic oil
automatic transmission fluid
diesel motor oil
gear lubricants
spindle oil

Although this report has not attempted to define the re-refining capacity or capabilities of countries other than the U.S., it should be noted that Mohawk Lubricants, Ltd. of North Vancouver, B.C. has a viable re-refining operation that uses thin-film evaporator/hydrogenation technology and appears to be one of the leaders in the field.

As such, Mohawk was contracted to process 15,000 gallons of Reclaimed Product (RP) (Navy contract N00406-83-M-5816; Requisition No. N00406-83-RQK 1023) through a technology consisting of distillation and hydrotreating. The test method that was followed was distillation at 500° F and 100 mm of absolute pressure, followed by hydrotreatment at 400° F and 450 psig. Tests on the distilled product were used to determine whether the hydrotreatment was required to process the fuel to satisfactory specification.

Mohawk used an appreciable volume of the test sample to flush existing material in the dehydration/distillation system before they subjected the test material (RP) to a controlled test. Of the 15,000 U.S. gallons of RP received, Mohawk actually used 9,000 gallons in flushing the system and finally obtained 6,000 gallons of distilled product.

By the time the hydrotreater had also been flushed they had a 20 percent final yield (approximately 3,000 gallons) of processed fuel, samples of which were analyzed and retained. During hydrotreatment, the steam stripping column was not used in view of the low boiling range of the RP. As a result, the final fuel was saturated with hydrogen sulfide gas.

Conclusions obtained from the short-term test run verified the operating conditions which could be used to recover a diesel fuel from RP, but pointed out the difficulties in attempting to use a lubricating oil re-refinery configuration for processing RP without some modifications. Mohawk recommended the installation of at least one item of equipment, i.e. a larger heater for their vacuum column. Mohawk was convinced that they can satisfactorily re-refine RP to diesel fuel marine (F-76) of specification grade.

In a letter of April 1983, Mohawk said, "So far as we are concerned, we have proved to our satisfaction that we can satisfactorily re-refine FOR (RP) to diesel fuel marine (DFM) specification material. On a large volume, continuous basis we are confident we can reclaim 85 percent of DFM (F-76) from FOR (RP) and our charge for this service would be U.S. 20 cents per U.S. gallon for material delivered to our plant. It would be the responsibility of the U.S. Navy to deliver FOR (RP) to our plant and to subsequently withdraw (F-76) DFM from our storage."

The data obtained from this pilot study are included in Appendix I of this report. The significant findings were that distillation alone produced a fuel that met all specifications for F-76 except for copper strip corrosion. Hydrogenation improved the copper strip corrosion test from an unacceptable 3b to a good 1a. However, hydrogenation often leaves a petroleum product vulnerable to rapid oxidation by removal of the natural oxidation inhibitors and thus the storage stability characteristics of the final product would need to be tested. If necessary oxidation inhibitor could be added after hydrogenation. There may be better ways of dealing with the copper strip corrosion problem than hydrogenation, however, such as a preliminary chemical treatment.

4.3 SUMMARY OF INDUSTRY CAPACITY

The re-refining industry in the United States has deteriorated in number from perhaps as many as 160 re-refining operations following WW II to as few as a half-dozen in the early 70's. With the development of new technology, the re-refining industry is gradually turning itself around, adopting new processing methods, becoming fully aware of the environment, and producing a better quality product than ever before. The total number of processors of used oil to fuel oil probably numbers in the hundreds if each little operation that collects used oil, fuel, solvents, etc., and resells them as a burner fuel supplement is counted. However, the major processors with extensive collection fleets and at least a flash evaporation step to remove water and solvents by distillation probably number less than 25 operations in the United States. Re-refiners in the U.S. are limited to fewer than a dozen viable operations most of which are using some form of thin-film distillation in conjunction with clay or hydrogenation to produce a re-refined basestock. Probably near 100 million gallons of used oil are re-refined by these operations annually.

Few, if any, of these re-refiners attempt to produce a high quality fuel such as F-76. Most produce a fuel cut which is highly contaminated with halogenated organics and thermal decomposition products of lubricating oil additives. This fuel can be cleaned up to meet the specifications of no. 2 fuel oil by a light treatment with clay or by hydrogenation but only one re-refiner has expressed the need for such a fuel and the intention to attempt to produce such by hydrogenation. This re-refiner is Mohawk Oil Co. of Vancouver, B.C. At present, no such product is being produced insofar as the authors of this report know.

5. REVIEW OF NAVY RECLAMATION FACILITIES AND OPERATIONS

In 1982, the NUS Corporation made an extensive study of six Navy reclamation facilities which included some schematic diagrams of processing schemes and an inventory of tankage volumes and operating personnel (533). Some of this information has been extracted from that report and tabulated in Table 4. Note that four of the six installations have some sort of heated tankage for the breakdown of oil/water emulsions and four of the six use a chemical demulsifier to promote separation of oil from water in the generation of RP. The NUS report uses the term FOR extensively in referring to Reclaimed Product although there were indications that an appreciable portion of the product of reclamation was used internally after blending with NSF thus putting it in the nomenclature category of RP rather than FOR.

5.1 NAVY DISTILLATE FUEL (DIESEL FUEL MARINE)

The Navy's preferred fuel for all non-nuclear, surface ships is the middle distillate fuel, formerly called diesel fuel marine (DFM) and now redesignated as Navy Distillate Fuel (F-76). NATO F-76 conforms to the requirements of military specification MIL-F-16884H, while DFM met a similar specification MIL-F-16884G. The Navy selected MIL-F-16884G as a single, multipurpose fuel during a conversion program conducted early in the 1970's that changed the Navy from a heavy blended fuel to a mid-distillate fuel and later to F-76 (402). Specification values for F-76 (MIL-F-16884H) are shown in Table I-37. The DFM was a higher grade and more expensive fuel than were its predecessors.

The recent background for specifications for marine diesel fuels are presented below[1/]:

July 1969	Navy Distillate Fuel	MIL-F-24397
March 1973	Diesel Fuel Marine	MIL-F-16884G
May 1983	Naval Distillate Fuel (F-76)	MIL-F-16884H

[1/] Source, E. W. White and N. F. Lynn. "Recent Trends in the Production and Properties of the Navy's Main Ship Fuel". Symposium on Marine Fuels, ASTM Committee D-2, Miami, FL, December 1983.

Table 4. - Naval treatment/reclamation facilities [1]

Location	Tankage, gallons			Oil chemical treat	Disposition of reclaimed product	Vol oil recovered	Personnel
	Oil recovery						
	Heated	Unheated	Oil storage				
North Island-Naval Air Sta. San Diego, CA	None	75K	150K	No	Trucked to Miramar	1K gal/day	1/shift
Naval Supply Center, Point Loma, San Diego, CA	60K	60K	2/	Yes	FOR and NSFO	1.8K gal/day	1/shift
Naval Supply Center, Pearl Harbor, HA	100K	175K	6.4 million	Yes	FOR and NSFO	5.4K gal/day	1+
Naval Supply Depot, Puget Sound, Manchester, WA	420K	56K	2/	Yes	FOR and NSFO	4K gal/day	2/shift
Naval Station Mayport, FL	None	420K	225K	No	FOR and NSFO	2.4K gal/day	1/shift
Naval Supply Center Craney Isl. Fuel Depot, Norfolk, VA	210K	1.268 million	2/	Yes	FOR and NSFO	13.7K gal/day	2/shift

1/ Source, reference 533

2/ Not defined

The Navy relies, in general, on contracts with small, independent refineries in the continental U.S. to supply military specification fuel (402). Many small refineries hit by the world-wide recession and the high cost of production have limited production or have closed down completely increasing the ultimate likelihood of fuel shortages for the Navy. Problems associated with the use of poor quality fuel by diesel-driven ships have been reported by Wiborg (756). The re-refining of FOR is one important consideration that could, if successfully implemented, provide four to five percent of the Navy's requirement for MIL-F-16884H annually and at a considerable savings in cost.

The primary concerns of the Navy in selecting and specifying fuels for use are tabulated in the following list:

all-ocean operation
multiple prime movers (boilers, diesels, gas
  turbines)/logistics
underway replenishing
minimal fire hazards
extended storage life

The concerns of NAVSEA that any recycled fuel meet necessary specifications and not degrade the virgin F-76 supply are understandable when one considers all the restrictions placed upon the Navy fleets as tabulated above.

The expected trends over the next five years (1983-1988) in marine diesel fuel will be controlled by the necessity of processing heavier, more sour crudes; to increase spot crude purchases; the use of more desulfurizing, coking, and hydrocracking refining procedures; the use of residuum in fluid catalytic cracker (FCC) feed; and, more cracked stocks in the fuels (White, 71,387,510,511).

The end results of these trends may be more problems with future fuels. It may actually be to the Navy's benefit to recycle RP simply because the most reactive compounds will have formed gums and dropped out. In addition, this fuel will have been processed twice. Thus, it should be a clean, reliable product.

5.1.1 Handling Methods

The fuel specification is the first line of defense (71) for obtaining a fuel of suitable quality for the Navy's ships. However, this must be supported by a dependable quality-control program between the contractor's refinery and delivery into the combatant ship. The Defense Fuel Supply Center (DFSC) is responsible for the material management of bulk petroleum products. This responsibility includes procurement, contracting, and initial product distribution from refineries to bulk distribution terminals.

The procurement system is arranged so that separate purchase programs are established for different areas of the world at different times during the calendar year. An invitation for bids against the Navy's estimated F-76 fuel consumption for the particular geographic area is forwarded to potential suppliers for price quotations (71).

According to Boyle (71) a production batch of fuel is checked by an on-site Government inspector to ensure that the fuel meets the requirements of the current MIL-F-16884 specification. If the batch fails to meet a spec requirement, the refiner may request a waiver of the deficiency through the contracting officer, who must then obtain the Navy's technical approval. A waiver may or may not be granted depending upon the nature and degree of the deficiency.

The Government is usually responsible for the transportation of an accepted batch of fuel from the refinery by sea-going tanker, coastal barge, or commercial pipeline. Once the fuel has been transferred to the Government-furnished transport system, the contractor is relieved of responsibility for fuel quality. It is the Government's Quality Assurance Representative who is responsible for inspecting tanker and barge compartments to insure freedom from fuel contamination during loading (71).

Upon arrival at its first destination, which is normally a bulk petroleum, oil, and lubricants terminal, the fuel is again sampled and tested for quality. The contents of each tank receiving part of the cargo are examined. If the fuel has become contaminated, the bulk terminal will reblend product as necessary to meet required property limits.

Boyle (71) further reports that fuel can remain in bulk terminal tanks for periods ranging from six weeks to two years, with the average under a year. The turnover period depends on the location of the terminal. In port, a ship is fueled alongside a pier via pipelines from the terminal, or at anchor from self-propelled yard oilers or barges.

If a task force is to be refueled while underway at sea, a fleet oiler is attached to the task force. This oiler is normally loaded-out at a terminal or replenished at sea by another oiler. The oiler conducts minimum quality-control tests prior to transferring its cargo at sea. However, cargo compartments are normally stripped of water prior to pumping.

5.1.2 Contamination Circumstances

Boyle (71) has reported that Navy shipboard fuels are subjected to conditions not normally experienced at the stationary, shore-based installations in which the most ASTM fuels are consumed. The sea environment combined with design features insures contact between the fuel and saline water. The influx of moisture-laden air into a tank as fuel is drawn out, combined with ambient temperature differences, would alone insure the presence of condensate water in the fuel tank. However, there is also the purposeful addition of seawater to the tank to maintain ship trim and to increase stability in rough seas. In the older ships there is a provision for flooding a tank with water after it has been emptied of fuel. In some newer ships a compensating system is used, so that water enters a tank as fuel is removed. These water-contact situations impose the first fuel requirement, that the fuel have good water-shedding properties. The Navy satisfies those needs through specifications imposing more constraints than similar ASTM specifications.

The Navy has a requirement for high reliability of the ship's propulsion system, especially under combat conditions. Filters plugged by dirty fuel or fuel-related failures of engine parts is undesirable. Such possibilities impose further fuel requirements--that the fuel must be clean on purchase and kept clean throughout the fuel distribution system.

The Navy may store fuel for much longer periods than the commerical consumer, who is supplied at regular intervals by a short, secure distribution system. The Navy maintains some stocks of fuel in what are termed "prepositioned war reserves". That is, stocks drawn on at the beginning of hostilities to carry the fleet over the period before the system gets up to a war footing. Although such stocks are replenished periodically, the retention time is normally longer than that between the refiner's production and the homeowner's consumption of no. 2 fuel oil. Further, ships keep fuel in a number of tanks and, although policy dictates that fuel be consumed from all tanks in a specified rotation, human nature will result in preferential use of fuel from the more conveniently situated tanks. Such practices impose another fuel requirement--a very stable fuel--so that sediment from fuel aging does not cause operating problems such as filter plugging.

As a result of these requirements there have been guidelines and recommendations (762) proposed for shipboard treatment and conditioning of fuel oils that have become contaminated either through residues left in the ships fuel tanks during docking-cleaning operations or through water-compensated fuel tanks in which water ballast is taken onboard as fuel is consumed.

5.1.2.1 Definition of Contaminants in RP

A complete definition of the contaminants in RP was recently completed as described in section 2.2.1.3 and Appendix I of this report. That work was done by the Southwest Research Institute and was both a bulk property survey and a hazardous materials investigation in search of contaminants.

The requirements for FOR (the designation under which RP is sold as utility boiler fuel) have been tabulated in Table I-36 of Appendix I and can best be evaluated by a comparison with F-76 specifications as tabulated in Table I-37 of Appendix I. The sulfur content of FOR is acceptable up to 2.0 wt percent while the maximum for F-76 is 1.0 wt percent. There is a limit of 0.2 wt percent carbon residue on the 10 percent bottoms from F-76 but there is no limit on carbon residue in FOR. The minimum flash point for FOR is 10 degrees below that for F-76. There is no stability requirement for FOR while the maximum allowable accelerated stability rating of F-76 is 1.5 mg/100 ml. There currently is no Navy limit with regard to chlorinated material other than a "no green flame" limit. The water and sediment limit of FOR is 2.0 volume percent. There are no requirements for net heat of combustion for either FOR or F-76.

A concern with handling and recycling RP or FOR is that it may become mixed with hazardous materials (as defined by the U.S. Environmental Protection Agency) or contaminants that effectively render the RP untreatable. An example of the latter would be an excessive amount of vegetable or animal oils in the RP. These latter materials are a problem because they thermally degrade at lower temperatures than mineral oils. They also are generally subject to oxidation at a more rapid rate than mineral oils and therefore have poor stability characteristics.

The inclusion of slop oil and ballast water oils in the feedstream which is ultimately separated and processed into RP makes it highly vulnerable to contamination. Chlorinated solvents are one of the most likely contaminants because of the wide usage of such solvents for cleaning engine parts and greasy equipment and for dry cleaning of clothes. Pesticides as well as some transformer oils can contain halogenated organic compounds that represent a serious contamination problem. While some of these contaminants would not be present on a ship, they must be considered when reviewing the option of sending the RP out to a commerical recycler who handles used oil from a wide spectrum of sources.

Figure 17 shows the gas chromatograms for a typical F-76 (solid line). Overlaying this curve is the detector response under identical conditions for a sample of RP. The differences are seen at retention times between 5 and 9 minutes which is associated with lower boiling species of compounds and between 15 and 27 minutes. This latter portion of the chromatogram represents the higher boiling compounds. The data indicate that RP contains both low-boiling and high boiling contaminants. The lower boiling materials are probably solvent-type materials while the higher boiling components are most likely lubricating oil and greases. The obvious similarities between the two curves strongly underscores the conclusion that recovery of F-76 is a reasonable objective.

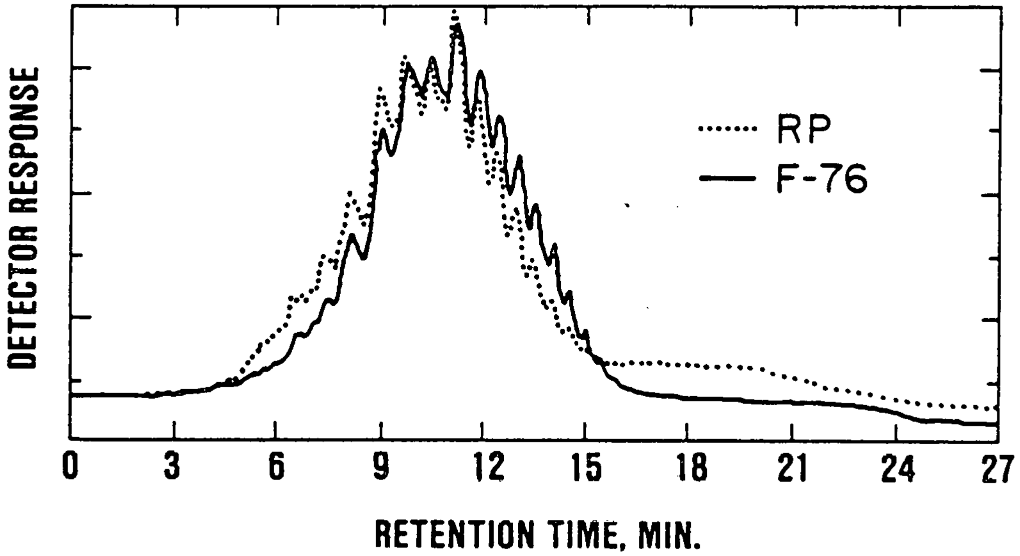


Figure 17.- Differences between RP and F-76 as indicated by GC separation

Most chlorinated organics are rather low in heat content so gross quantities of chlorinated solvents can effectively reduce the net heat of combustion of a fuel. Distillation will remove some chlorinated organics from diesel fuel. But chlorinated organics can decompose at temperatures attained during distillation. The resultant vapors are very corrosive and can destroy ordinary steel equipment in a matter of days. If the chlorinated compounds are not removed by distillation, then some technique such as clay contacting or hydrogenation is required. Clay contacting removes a portion of the chlorinated compounds through adsorption at active sites on the clay. Hydrogenation, on the other hand, converts the chlorine into hydrogen chloride which must be chemically removed from the vapor stream before it is permitted to condense into a liquid. Contact with water converts the gaseous hydrogen chloride to hydrochloric acid which is highly corrosive.

Samples of RP were analyzed by the Southwest Research Institute for hazardous species. The results of gas chromatography/MS analysis showed that neither the F-76 nor the RP contained significant levels of pesticides or

PCB's (polychlorinated biphenyls). However, there were substantial levels of some volatile halogenated organics in the RP indicating contamination with chlorinated solvents in handling.

Although the literature (444,445,448,449) contains considerable material related to gross and detailed chemical characterization of oily waters, there is less to be found that gives detailed information regarding the identification of individual compounds that constitute the contamination in RP (196, 200). Total, suspended, and dissolved organic content and hydrocarbon levels in oily waters have been determined and volatile and water-soluble fractions have been characterized in great detail. Lower aliphatic and aromatic hydrocarbons that are separated from water by nitrogen sparging and collected in an activated carbon adsorption column have been defined. Class characterization of these fractions recovered from oily waters have been performed using High Performance Liquid Chromatographic (HPLC) procedures as well as GC/MS. Little of this information is useful for selecting reclaiming processes of RP to F-76.

5.1.2.2 Stream Segregation

In 1982, Recon Systems (592) made a study designed to provide guidance in recycling Navy used petroleum products in the most cost effective and environmentally sound manner possible. The underlying philosophy of this study was to achieve recyclability of used oil products and solvents for their original use or similar applications as close to the source of generation as possible. But, full consideration was given to all potential recycling alternatives including blending/reprocessing for fuels, re-refining, and sale. Segregation was an integral part of this recommendation.

Segregation, if carefully practiced, would greatly simplify the problem of converting RP into F-76. Contamination with certain materials such as sea water is inadvertent and cannot be significantly deterred by segregation procedures after the ship has reached the depot. However, contamination of offspecification F-76 with solvents, greases, lubricating oil, chlorinated organics, and other petroleum and non-petroleum products could be averted with care, instruction, and appropriate facilities. Segregation in large commercial industrial sites, where a variety of lubricants, fuels, and solvents are used, has shown that segregation pays off eventually in terms of improved and extended usage of products through simple cleaning procedures. The re-refining procedures become increasingly complex as the range of contaminants in any given product is extended. Therefore, segregation of off-specification F-76 from other petroleum products should be given careful consideration.

5.2 RECLAIMED PRODUCT (RP)

The oil that is separated, processed, and ultimately designated as RP or FOR is derived from several sources. Waste oils are delivered often as relatively clean F-76 and downgraded JP-5. Other waste oil with sediment and water contamination must be separated from the water before it can be combined with the cleaner F-76 for further processing.

The primary problem in the generation of RP appears to be the separation of small amounts of oil from large quantities of water. Conventional oil/water separation techniques are generally applied which include API separators, skimmers, and dissolved air flotation and/or other separation procedures. The recovered oil is then sent to tanks for further processing. Eventually, all the waste oil is combined and treated in settling (and cooking) tanks in a batch-mode.

Typically (533), facilities with tank heater capability route the waste oil to a settling tank via a piping system. Two tanks are available so that one can be filled while the other is in a quiescient state.

After the settling period, the operator uses a sampling mechanism to draw off samples of the tank to determine the heights of oil and water layers. Water is drawn off first and is stopped at the water/emulsion interface. The oil layer is pumped to a cooking tank which is typically a welded steel tank with a capacity of 30,000 gallons or more. The oily wastes that go to the cooker come from two sources, either bilge oil from ships delivered by their pumps, or from the settling tanks via the transfer pump. The flow from the bilge oil pumps is basically automatic, but considerable operator attention is required to obtain proper levels in the cookers.

In those facilities with "cooker" capabilities, normally, only one tank is used while the other tank is on standby. The cookers are heated by means of a steam tank heater. The heater keeps the tank temperature in the range of 165 to 185° F. The oil is kept in the tank at this temperature for approximately three days. At that time samples are drawn to determine the level of the oil layer and remaining emulsion sludge. If a distinct separation has been achieved, the emulsion sludge is drawn off. The operator must maintain constant watch to insure that when the free oil level is reached the operation is halted. Periodically, some of the recovered oil is pumped to a tank for in-plant use as fuel for the steam generator.

There appear to be some differences in cooking techniques among the several Naval stations that process oily wastes. Some use chemical demulsifiers while others do not. The temperature and period of time of cooking are also somewhat variable among the processing units. Probably the net result of these differences is only in quantity of oil recovered rather than in quality of RP.

5.2.1 Existing NAVY Facilities for Reclamation

The Naval Facilities Engineering Command (NAVFAC) has developed an Oily Waste/Waste Oil Management Program as a result of a series of Federal directives and regulations related to energy usage and environmental pollution. In 1982 NUS Corporation was retained by NAVFAC to complete an Oily Waste/Waste Oil Management Study (533). The study involved two primary tasks. The first was to visit six oily waste/waste oil treatment and reclamation facilities and evaluate the performance and operation of each facility. Task 2 involved development of standardized design criteria and system layouts for future application.

As a result of Task 1, NUS Corp. visited six Navy bases with oily waste collection and treatment systems tabulated in Table 4, Section 5.1:

Naval Air Station, North Island, San Diego, California
Naval Supply Center, Manchester, Washington
Naval Station, Mayport, Florida
Naval Supply Center, Point Loma, San Diego, California
Naval Supply Center, Pearl Harbor, Hawaii
Naval Supply Center, Craney Island, Norfolk, VA

5.2.2 Process(es) Used to Produce RP

After reviewing the six facilities, NUS Corp. (533) reported a lack of standardized procedures for conceptual evaluation and design development of proposed oil waste facilities. The diversity of oily waste collection and storage schemes and combinations of unit operations pointed up the need for some means of central control over projects to assure the consistent application of standard design criteria.

Further, NUS Corp. reported that most of the waste oil received in the collection systems is recovered. In almost all cases reclaimed product (RP) is used as fuel for land-based utility boilers. RP is either blended with Navy Special oil or sold as FOR to be used in utility boilers. Two of the fuel departments had self-imposed standards for RP quality before the RP could be reissued. There is an apparent discrepancy among reclamation plants as to the required treatment for RP. Fuel departments with self-imposed standards indicate polymer addition and cooking are required to meet the RP quality. Other fuel departments indicate that only initial separation is required to provide RP of an adequate quality to burn in utility boilers. In order to optimize RP re-refining to F-76, it will be necessary to develop a single quality standard that is acceptable to all.

Generally, the difference in RP recovery systems is in the cooking procedure. Obviously, all facilities must separate the gross amounts of water from the oil. The final separation of oil/emulsion/water is then achieved in tanks with quiescent settling for a matter of several days. Some of the reclaimers use heat and chemicals to assist in breaking existing emulsions while others depend solely upon settling and take whatever separation is thus achieved.

5.2.3 Quantities of RP Processed

In 1982 the NUS Corp. (533) surveyed six Naval oil reclamation sites and estimated both the quantity of waste water processed as well as the reclaimed product (RP) recovered. These quantities were admittedly estimates and it was interesting to note that as little as 1 percent oil was recovered at some sites from the waste water while as high as 35 percent oil was recovered from one site. The latter was attributed to a large quantity of clean off-spec fuel received at that depot.

The Naval Station and the volumes of oily waste processed and the amount of RP recovered were tabulated in Table 4. The sum of the RP recovered annually according to the NUS Corp. estimates approaches 8.75 million gallons from these six Naval stations.

5.2.4 Properties of Typical RP

The six Naval stations listed above appear to each handle the processing of oily wastes to recover RP in slightly different techniques. The average properties of RP from some of the same stations plus others are tabulated in Table 2 of section 2.2.1.3.

6. TECHNICAL AND ECONOMIC ANALYSES OF PROCESS OPTIONS

At least three reports over the past few years have attempted to assess the economic and technical advantages and disadvantages of selected re-refining processes. The conversion of RP to F-76 may well be essentially a re-refining task requiring one or more of the techniques and types of equipment used to re-refine used lubricating oil. It appears logical to compare some of the results of the past studies as well as to offer a more timely comparison based upon today's economic situation.

6.1 TECHNICAL EVALUATION OF PROCESS OPTIONS

Based upon the extensive information provided in Appendix III and upon the in-depth studies performed by NIPER personnel in the field of reclaiming fuels and oils, viable technologies for converting RP to F-76 have emerged. Some processes such as acid/clay must be discarded initially because of environmental considerations. By-products generated by acid treatment have no presently known disposition other than through hazardous landfills. Technologies such as solvent treatment or chemical treatment are potential candidates for converting RP but may not be completely effective without additional steps to produce a MIL spec F-76 from RP. Other techniques have not been proven in large-scale testing. However, based on limited available knowledge, some of these technologies appear to be candidates for investigation in a second phase of this study.

After identifying, the primary contaminants in RP as water, solvents, lubricating oil, sludges, and trace contaminants including metals, pesticides, and halogenated organic compounds, the preferred process, from a strictly technical standpoint, to effectively remove these contaminants, would be very similar to that used successfully by the majority of viable re-refiners in the U.S. today. That technology would include:

dehydration (distillation at atmospheric pressure),
distillation (thin-film evaporator technology), and
polishing (hydrogenation).

These three essential steps will first remove the water that contaminates RP and also will remove solvents boiling up to the lower boiling point of F-76. Since the initial boiling point (IBP) of F-76 is in the range of 450° F, one would be inclined to dehydrate at this temperature. This distillation could be either a batch or a continuous process depending upon the complexity of controls that are desired.

Assuming that all hydrocarbons boiling below the IBP of F-76 are removed during the dehydration, the oil could then be charged directly to a reduced pressure thin-film evaporator. Here the oil would be separated into an overhead cut which should have an endpoint of about 725° F and a small residue which has potential as feedstock to a lubricating oil re-refining operation.

The final step required to remove last traces of contamination from the distilled F-76 would be a light hydrogenation which would provide excellent color and odor and lower the sulfur, nitrogen, and oxygen content to zero. Metal contamination inherent to the RP would be removed by distillation and adsorption on guard-bed packing. Hydrogenation would also destroy any halogenated organic compounds that have inadvertently contaminated the RP. Clay treatment might produce similar results, but the environmental problems of disposing of oily-clay wastes must be considered. A third possibility for final polishing would be the use of solvents as described by Fletcher and O'Blasny (172) in their patent for the removal of color bodies and odor. This technique has only been demonstrated on a bench-scale basis and therefore would require some pilot-scale studies.

The treatment scheme of dehydration, distillation, and polishing would produce a fuel that meets or exceeds current specifications for MIL-F-16884H.

However, consideration of the limitations imposed by available personnel and initial capital investment and considering flexibility, reliability, and simplicity of equipment and operation, would suggest an alternate approach.

The following techniques are recommended for bench-scale testing to evaluate the potential of each system. Pilot-scale testing would be contingent upon successful initial results.

chemical treatment/distillation
clay only
supercritical extraction
ultrafiltration
electromagnetic radiation.

The chemical treatment/distillation technique would combine the use of either sodium borohydride or sodium hydroxide with a wiped-film evaporator technology. Preliminary data from Mohawk's re-refinery in Vancouver indicated that distillation using a thin-film evaporator produced a fuel that met F-76 specs except for corrosion. The proposed pretreatment with chemicals or more efficient stripping of H_2S might rectify that deficiency.

Clay alone has been shown to produce a good quality lubricating oil from used crankcase drainings (357). It is possible that this technology, which is simple and flexible could do a good job of returning RP to F-76 specs if a satisfactory solution to the problem of by-product disposal could be achieved. A partial solution may be to use the clay in a fixed bed with the fuel flowing through it. This could significantly reduce the quantity of oily clay generated.

Supercritical extraction, ultrafiltration and electromagnetic radiation have potential enough to warrant further evaluation through Phase II testing.

6.2 ECONOMIC ANALYSIS OF PROCESS OPTIONS

6.2.1 Complete Re-refinery

Mascetti and White (461), in a report prepared for the Department of Energy in 1978 estimated the capital cost for dehydration equipment for a 10 million gallon per year operation at $59K. It should be noted that in this range and size of equipment, costs are not very sensitive to size. That is, halving the size of a plant will not reduce the capital costs by a factor of 2. Weinstein, writing for the Department of Energy in 1980 (750) estimated the costs of dehydration equipment for a 2 million gallon per year plant at

$139K. Obviously there is considerable discrepancy between these numbers even after compensating for the increase in the consumer price index over the period.

A major manufacturer of thin-film evaporators has estimated the complete cost of a thin-film evaporator installation including all peripherals to process 5 million gallons of oil annually, at $600K. Since there are intermediate sizes of thin-film evaporators ranging from 1.5 square foot of heat exchange area to 344 square feet, it is likely that the distillation equipment can be closely sized to the anticipated needs of the particular installation. However, the Norfolk Naval station appears to process about 5 million gallons of contaminated oil to RP annually, so it appears reasonable to size a plant based upon this estimated throughput.

The capital costs for the hydrotreater can only be estimated at this stage of this study, but based on recent costs to build a hydrotreater in Pennsylvania to process 10 million gallons of oil annually, the estimated capital cost of a completed turn-key hydrotreater for 5 million gallons/yr. would probably be in the range of $600K.

Including tankage, piping, design engineering, laboratory and other expenditures, the capital costs to build a re-refinery that would process 5 million gallons of RP annually into specification grade F-76 is currently estimated in the range of $1.3 million to $4 million.

Teknekron (697), in 1978, estimated the production costs of a distillation/hydrogenation plant for processing 6.6 million gallons of used oil per year to produce 5 million gallons of saleable basestocks. They estimated an average production cost of $0.29 per gallon less a feedstock cost of $0.12 making the average production cost near $0.17 per gallon. Based upon an increase of 51.3 percent in the consumer price index since the Teknekron cost estimate would raise the production cost to about $0.26 per gallon.

The Aerospace report (461), also published in 1978, estimated the process energy requirements was 410K Btu per barrel of feedstock for a distillation/hydrotreating plant. Based upon 1978 prices this resulted in a total base oil production cost by distillation/hydrotreating of $0.291 per gallon less $0.15 feedstock cost giving a net production cost of $0.14 per gallon.

The production costs estimated by Teknekron and Aerospace take into consideration the yield of product, indirect and direct processing costs, and labor. The direct process costs include chemicals, energy, waste disposal, and credit for overheads and is based upon a process yield of about 76 percent. The process yield of F-76 from RP is not known but should greatly exceed this 76 percent yield of lubricating oil basestocks from used lubricating oil.

6.2.2 Economic Analysis of Other Options

The other choices that require an economic analysis include a chemical/distillation treatment of RP. In this scenario, it is assumed that a small amount of chemical such as sodium borohydride or a caustic (sodium hydroxide) combined with the RP as it is fed to a distillation apparatus will neutralize acidic components. The distillation, then, will remove residual water and solvents and leave the heavier contaminants as distillation bottoms.

The cost of such a system as just described is primarily in distillation equipment, of which thin-film techniques appear to have an advantage over a fractionation tower from a practical standpoint. Currently, the largest wiped-film evaporator that would be required to process the maximum annual production of RP (about 5 million gallons at Norfolk, VA) could be completely installed for about $700K. The operation of such a system could be handled by one man after sufficient training, and the operational cost to convert a gallon of RP to specification grade F-76 is roughly estimated at less than $0.20 per gallon. Actual costs of this choice could be closely estimated in a Phase II study, if the option were considered acceptable in terms of initial capital investment, flexibility, simplicity, and reliability.

The next option would be a clay-only treatment which has the potential of producing a dry, specification grade F-76 from RP with the penalty of clay disposal. The initial capital cost for a clay contactor is only an estimate, but a device such as that manufactured by Refinoil probably would cost less than $100K initially. Such a device could also be constructed from existing equipment augmented with appropriate pumps, valves, heaters, and tanks at a savings in initial capital investment over a commercial device.

Clay contacting could be conducted as a one-man operation in a batch-wise mode and would undoubtedly represent the simplest of the options and perhaps the most reliable and flexible. Operational costs would depend a great deal upon the amount of clay required but under the worst scenario that can be visualized at this stage, less than 2.0 pounds of clay per gallon of RP would be required. The cost would then probably range between $0.15 and $0.20 per gallon. If a flow-through system proves viable, the costs would be much less, possibly as low as $0.03 to $0.05 per gallon.

The capital and operational costs for supercritical extraction, ultrafiltration, and electromagnetic energy are not well defined at this stage of investigations and this information would have to be developed during Phase II of this study. However very rough estimates are included in Table 1 in section 1.1.

7. PRIORITIZING TREATMENT OPTIONS

To prioritize the treatment options one must consider the specific conditions that exist at the sites where RP is produced and keep the process as simple, reliable, and economical as possible. In this context, lesser known options that would represent a breakthrough in processing technology should be investigated while keeping in mind that the surest technology in terms of successful conversion of RP to F-76 would be a modified re-refinery. The complexity and cost of this latter option removes it from the head of the prioritized listing.

As a result of this study, the priorities are as follows (descending order):

chemical/distillation (thin-film)
clay
supercritical extraction
ultrafiltration
electromagnetic radiation
re-refinery (modified)

The chemical/distillation option should be checked using several chemical treatments followed by thin-film distillation to see if specification grade F-76 can be obtained. Some of the possible chemicals and those most likely to succeed have been listed previously.

The clay-only technique is another that can be performed in bench-scale quantities and should be evaluated from both the technical and environmental aspects. There is at least one commercial clay contactor, and perhaps others that should be investigated as part of this option.

Supercritical extraction will require more sophisticated equipment and is a more complex technology than either of the first two. Essential equipment includes an autoclave and compressors. A number of solvents and gases can be assessed with regard to their effectiveness in removing contaminants from RP.

It may be possible to use an equipment manufacturer's pilot plant in order to evaluate the effectiveness of ultrafiltration. These techniques, while very effective in certain applications, can be very slow. Therefore it will be necessary to evaluate the maximum flowrates applicable to ultrafiltration in addition to the effectiveness in producing specification F-76.

Although a complete re-refinery may not be economically acceptable, it is appropriate to evaluate treatment options within this technology. The investigation should be limited to re-refining techniques that have some basis for evaluation by representing a viable alternative for the recycling of RP to F-76. The dehydration step for removing water from RP probably does not warrant further investigation for this project.

It must be noted that if a technology consisting primarily of thin-film distillation were elected, water might be removed in a single pass through the apparatus by using partial condensation techniques that will produce two or three cuts from a single pass through the evaporator. One of the innovations that have been developed in connection with thin-film evaporator technology which conventionally produces only two fractions--an overhead cut and the distillation bottoms--is a partial condensation unit that works in series after the evaporator. By cooling sections of the condenser to different temperatures, it is possible to take more than one cut from the unit based upon condensation temperature. Since there is no reflux there is no fractionation and the boiling point overlap of these fractions is considerable. However, the technique does represent a method of obtaining more than a single overhead cut from a thin-film evaporator.

The choices for distillation of the dehydrated RP which has been stripped of components boiling below the acceptable range for F-76 are as follows:

thin-film evaporator
skid-mount crude oil still

Without the benefit of pilot-scale tests, it is difficult to completely evaluate the merits of the two systems listed above for distillation. The thin-film evaporator is much more costly than a similar capacity crude oil distillation unit. The complexity of operation is difficult to evaluate.

Efficiency in terms of sample throughput is generally in favor of the thin-film technology. Good definition of boiling point cut-offs can be achieved with the thin-film and probably the crude oil still. It is therefore suggested that further pilot-scale tests be conducted before a final decision is made in this regard.

A vacuum tower can not be justified unless the RP to be converted has a substantial lubricating oil contamination. Therefore this option was not included above and discussions were on the level of a simpler distillation unit.

The polishing step for the distilled F-76 may not be required. A great deal depends upon the nature of contamination. If a final polishing step is required to remove color bodies, odor, metallic components, or halogenated materials, then hydrogenation represents the cleanest and most effective technology and has the capacity to destroy toxic and carcinogenic halogens without creating unmanageable waste streams. However, hydrogenation is initially costly and relatively complex. Clay is less complex but represents a potential problem of spent clay disposal. The third alternative is solvent treatment as a polishing step. The latter is an unproven technique that would require bench- and, perhaps, pilot-scale tests. Solvent polishing represents the simplest technique for removing color and odor bodies and therefore must be considered for Phase II study. The priority rating for polishing would be as follows:

None required (Distillation produces an on-spec product)
Solvent polishing
Clay contacting
Hydrogenation

8. RECOMMENDATION FOR FURTHER RESEARCH

Techniques that have potential for successful conversion of RP to F-76 include an abbreviated re-refining process, treatment with clay, use of supercritical extraction techniques, ultrafiltration, and electromagnetic radiation. These technologies represent the areas that are recommended for further research.

Much of the bench-scale work for Phase II will be performed at NIPER. The Navy will be informed of projected schedules so that visits can be arranged to observe tests of special interest.

The following technologies will be studied:

(a) Distillation - Using both simulated procedures (gas chromatography) and actual bench-scale units, the extent of processing necessary to generate an acceptable product will be evaluated. It may be necessary to combine a pretreatment with distillation if analytical data indicates such a need.

(b) Solvent Treatment - Using both atmospheric and supercritical conditions, several solvents will be evaluated for their potential as a pretreatment step prior to further processing and as a self-sufficient step.

(c) Chemical Treatment - Using commercial demulsifiers and other promising chemicals, their effects on contaminants in RP will be evaluated and in some cases the ability of chemicals to assist distillation or other steps to achieve a specification product will be investigated.

(d) Clay Contacting - Using commercially activated clays, several conditions will be used to evaluate the extent of upgrading. Emphasis will be on minimum levels of clay usage to control the production of waste by-products.

(e) Hydrotreating - Using the bench-scale flow-through reactor, several catalysts operated at a matrix of temperature, pressure and flowrate conditions will be evaluated.

(f) Supercritical Extraction - One or more solvents will be tested in the supercritical region for their ability to separate contaminants preferentially from diesel fuel. Suggested solvents could include propane, other hydrocarbons, and carbon dioxide.

(g) Filtration - While filtration alone does not seem to show much promise, several varieties of filters in conjunction with the preceding technologies will be evaluated as will certain ultrafiltration and reverse osmosis configurations. Criteria will include efficiency, ease of operation, manpower requirements and reliability.

(h) Electromagnetic Radiation - A commercial supplier of scientific microwave equipment has offered to run samples of RP at a nominal charge to evaluate the separation of BS&W. It is planned to take advantage of this service to evaluate the potential of this technology.

8.1 RESOURCES REQUIRED FOR BENCH- AND PILOT-SCALE TESTING

Some equipment is required to test the various options that have been discussed above. Some of the equipment is listed and briefly described below.

To test the combination of chemical/distillation scheme would require only a pilot-scale thin-film evaporator and NIPER has one of these. If larger equipment of similar nature is required there are at least three pilot-scale plants that can be obtained for limited use within the U.S.

Determination of the efficiency of clay-contacting requires simple pilot-scale equipment, which NIPER also has. It may also be possible to work with a clay manufacturer to develop the best combination of parameters to perform the treatment required. Manufacturers of commercial clay contactors will be contacted for potential cooperation in a joint study of converting RP to F-76.

Both bench-scale and pilot-scale hydrotreaters are available at NIPER if the need for polishing becomes apparent as tests progress. If these facilities are not adequate, similar or larger-scale hydrogenation equipment and services can be obtained at Oklahoma State University, Stillwater, OK, Continental Oil Company in Ponca City, OK, and Suntech in Marcus Hook, PA. If the solvent polishing step appears worth investigation, Delta Refining in Nachitoches, LA, has a small laboratory for bench-scale testing and can provide pilot-scale tests upon requests.

Supercritical extraction will require an autoclave and compressors and some essential plumbing that are currently available at NIPER. However, the testing of this technology can probably be obtained either in-house or through the facilities of equipment manufacturers.

Ultrafiltration represents a technique that could best be tested in cooperation with an equipment manufacturer. Equipment to test this technique is not available at NIPER.

Electromagnetic radiation is an area where bench- and pilot-scale facilities are available from a manufacturer and samples can be tested at a very reasonable cost.

Evaluation of various commercially-available re-refining technologies can be performed in-house. Dehydration is a technique that is used by numerous handlers of petroleum products and does not warrant a major test program. Knowing the level of water acceptable in the finished F-76 and the IBP will enable construction of a flash evaporator that will produce good results. However, to provide dry samples with which to work will require that some bench- and pilot-scale dehydrations be performed. NIPER has within its facility a variety of bench-scale distillation apparatus that will produce small (milliliter) quantities of water- and solvent-free RP. Also at NIPER are two thin-film evaporators that can be used to provide larger quantities of water-free RP and to evaluate the possibility of using such equipment instead of more conventional flash evaporators. There are also many waste oil processing facilities with conventional dehydration equipment that could dehydrate large quantitites of RP on a fee basis.

8.1.1 Samples To Be Tested

A representative sample of RP will be required to initiate bench- and pilot-scale studies. The quantity of RP required for the Phase II program will probably be around a thousand gallons. Although bench-scale tests will require only small quantities, the size of pilot-scale processing equipment will dictate sample size. It is important this be a homogeneous sample.

Samples of F-76 for reference testing will also be required. If compatibility studies are to be conducted, several drums of specification F-76 will be needed.

8.1.2 Test Procedures

The test procedures used in the Phase II study will include physical and chemical testing by the analytical services group at NIPER, augmented by those of commercial labs and the Southwest Research Institute, to obtain a measure of the quality of the samples. NIPER has an excellent characterization group that uses unique separation procedures coupled with chromatographic and spectroscopic techniques for the identification and characterization of contaminants, if such information becomes vital to the successful evaluation of technology.

Dehydration will be performed in one of several available configurations of distillation equipment under conditions to obtain the best results for this step of the process.

Likewise, the actual distillation of the dehydrated RP can be performed in two or more distillation configurations at a variety of conditions of feedrate, temperature, and pressure.

Hydrogenation conditions, if required, will undoubtedly be quite mild and probably only one or two of the more common catalysts will be evaluated while varying the available hydrogen, temperature, pressure and feedrate.

8.2 TESTING PROGRAM

It is proposed that Phase II be initiated upon the completion of Phase I and that the test program generally follow the directions outlined above. The objective of Phase II testing would be to evaluate the steps that appear best suited for the re-refining of RP to F-76 and to compare the options that have been suggested for each of these steps. The product of Phase II testing would be another report describing test results and making recommendations for further studies.

8.2.1 Performance/Technical Criteria

There appears to be no real need to perform a complete slate of analyses on each product generated from testing in Phase II. However, certain product streams that seem most attractive will be obtained in sufficient volume to permit complete screening for acceptability under F-76 specifications. The other streams will be analyzed for key properties expected to be affected by the technology being evaluated.

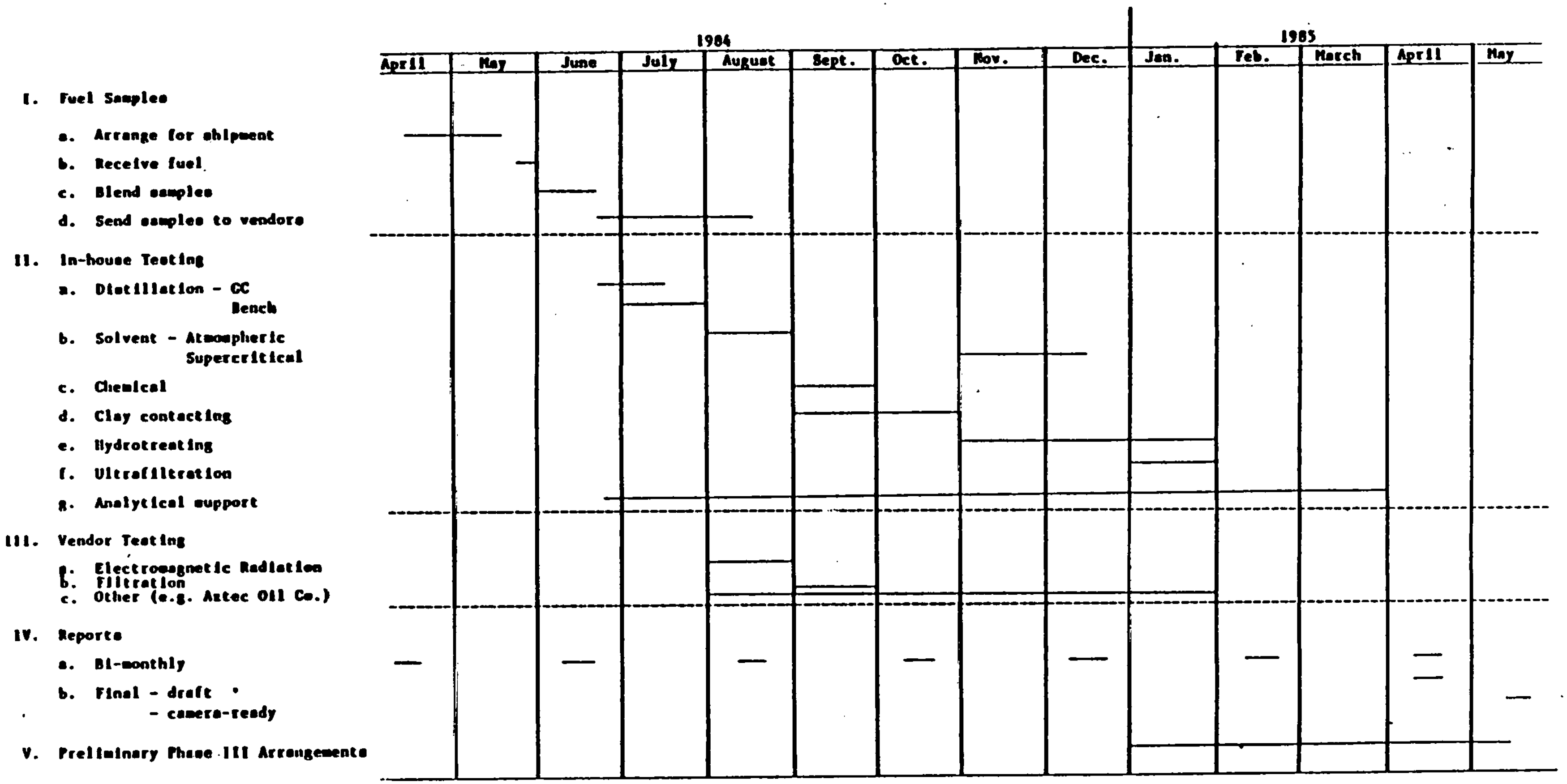
It may not be crucial that the final product meet all F-76 specifications, but only that it be apparent that specifications can be met with a reasonable level of blending. However, a serious attempt will be made to attain a specification product in all cases merely to demonstrate the extent of processing required.

8.2.2 Deliverables

The deliverables from the Phase II research to the Navy will be bimonthly progress reports, oral briefings as appropriate, and a formal final report detailing all experimental work, significance of findings, and recommendations for additional testing, demonstration projects, commercial liaisons, and/or abandonment of this idea.

Figure 18 is a milestone or progress schedule that is proposed for the second phase of this study. The chart shows both the schedule for deliverables and the proposed timing for the various investigations that will be required.

Figure 18.- Proposal Phase II milestone chart



APPENDIX I

CHARACTERIZATION DATA

APPENDIX I

In conjunction with this study for the Navy, the Southwest Research Institute (SwRI) in San Antonio has collected F-76 (DFM) and RP (FOR) samples from six naval refueling depots. Two F-76 (DFM) samples and three monthly RP (FOR) samples were requested from each depot in order to develop good data on starting material for Phase II investigations. This was essential to have sufficient data to permit selection of appropriate starting materials for bench- or pilot-scale studies.

Note that SwRI used two designations for some of the samples requested from the Navy for analysis. The SwRI work was reported before they were aware of the change in nomenclature from Diesel Fuel Marine (DFM) to Naval Distillate Fuel. Consequently, some sample analyses were labeled as DFM and NDF rather than the currently preferred designation of F-76. Likewise, all reclaimed samples obtained from the Navy and analyzed by SwRI were designated as FOR (Fuel Oil Reclaimed) although it is not certain that these fuels were destined for use as a utility fuel.

Table 2 of the text preceeding this appendix contained a summary of physical property data that was obtained on 12 F-76 and 18 RP samples. A copy of the SwRI report is attached. Tables I-1 through I-5 of this report contain the detailed analyses from which those summaries were derived plus the requirements for NDF and FOR.

Table 3 of the text contained similar summarized data for the contaminants found in the same 12 F-76 and 18 RP samples described in the preceeding paragraph. Table I-6 through I-13 of the SwRI report contains the detailed analyses for each of these samples. It will be noted that volatile or purgeable priority pollutants, semivolatile organic contaminants and pesticides plus PCB's were included in the SwRI analyses for pollutants and contaminants. Because none of the pesticides or PCB's were detected in the analyses, these data were not included in the text summaries.

Tables I-14 through I-35 contain gas chromatographic data generated for the NDF and FOR samples that were analyzed by the Southwest Research Institute. Copies of the chromatograms are included.

A copy of the military specification MIL-F-24951 Fuel Oil Reclaimed (FOR) issued February 15, 1983, is included in this appendix as Table I-36. The chemical and physical requirements for FOR are given in this document. Also included is a listing of ASTM test methods, quality assurance provisions, preparation for packaging, packing and marking, and notes on intended use, Navy responsibilities and user responsibilities.

Specifications for the military designation MIL-F-16884H for Fuel, Navy Distillate (NDF) or NATO F-76 which supersedes MIL-F-16884G Diesel Fuel Marine (DFM) were issued May 3, 1983. A copy of this document (FSC 9140) is included in this appendix. The chemical and physical requirements for F-76 are given in Table I-37. Other information included pertains to applicable ASTM tests, permissable additives, quality assurance provisions such as inspections, sampling and test methods, packaging, and notes on intended use, acquisition requirements, and data requirements.

Table I-38 is a tabulation of the laboratory analyses submitted by Mohawk Lubricant, Ltd. which includes their analyses of samples of FOR and DFM furnished by the Naval Supply Center Fuel Department at Manchester, WA. Elemental analyses were performed on a Perkin-Elmer 5500 I.C.P. spectrometer. Only phosphorus was greater than 1 ppm and it was reported to be 4 ppm in the distillate fuel. The results of the accelerated stability test on both the distillate and the hydrotreated sample are tabulated in Table I-39.

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LIST OF FIGURES

U.S. ARMY FUELS AND LUBRICANTS RESEARCH LABORATORY
6220 CULEBRA ROAD—P.O. DRAWER 28510 PH:512-684-5111 SAN ANTONIO, TEXAS 78284

USAFLRL

File: 02-6800-841
07 February 1984

Dr. Dennis W. Brinkman
National Institute for Petroleum
and Energy Research
P.O. Box 2128
Bartlesville, Oklahoma 74005

SUBJECT: Analyses of "NDF" and "FOR" Samples

Dear Sir:

We are forwarding to you analytical data on additional samples of "NDF" and "FOR" received from six Naval Supply Centers identified as NSC San Diego, NSC Puget Sound, NSC Oakland, NSC Charleston, NSC Norfolk, and NSC Jacksonville. The analytical data consist of the following:

- Specification inspection data for the second and third round of "FOR" samples and for the second round of "NDF" samples;
- GC boiling point distribution data in the form of "distillation reports" and Chromatograms for the second and third round of "FOR" and for the second round of "NDF" samples;
- Analyses of purgeable volatile and semivolatile pollutants and of hazardous compounds for all of the "NDF" and "FOR" samples evaluated in this program.

The second round samples of "NDF", for the most part, met the requirements of MIL-F-16884H, Naval Distillate Fuel. The samples from NSC San Diego and NSC Oakland had color ratings slightly above the specification maximum. The samples from NSC Charleston and NSC Norfolk had total insolubles on the accelerated stability test that were above the maximum limit.

Most of the "FOR" samples met the requirements of MIL-F-24951, Fuel Oil, Reclaimed. The sample from NSC Charleston had an excessive amount of water which appeared to be emulsified. This water interfered with the flash point, distillation, and carbon residue tests. This sample also had a high value for sulfated ash. The presence of water in the sample from NSC Puget Sound apparently interfered with the measurement of flash point in this sample also.

The GC boiling point distribution data show that the "NDF" samples all completely eluted from the chromatographic column after about 20 minutes, while many of the "FOR" samples continued to elute well beyond that time frame. This is an indication that the "FOR" samples contained heavier hydrocarbon fractions such as lubricating oils.

The analyses for purgeable pollutants, both volatile and semivolatile compounds, showed that the "NDF" samples contained numerous aromatic compounds, as would be expected, but no halogenated materials with one exception. The first sample received from NSC Norfolk appears to contain 0.5 mg/kg trichlorofluoromethane. All of the "FOR" samples contained varying amounts of halogenated compounds.

The analyses for pesticides and PCB's indicated that all these samples were free of detectable quantities of these hazardous materials except for one sample of FOR from NSC Jacksonville in which 25 ppm of AR 1248 were apparently detected. This value is still in question and will be further evaluated.

Please feel free to contact us if you have any questions on this material.

Very truly yours,

S.J. Lestz
Director

John N. Bowden

John N. Bowden
Staff Scientist

SJL/JNB/jag (WD05.S)
Enclosures
cf: U.S. Army Belvoir R&D Center, STRBE-VF, M.E. Lepera
Navy Petroleum Office, N. Schmokel
SwRI, BRW

TABLE I-1 ANALYSES OF MIL-F-16884G, "DFM" SAMPLES

	DFM Requirements						
Sample From	----	NSC San Diego Point Loma San Diego CA	NSC Puget Sound Manchester WA	NSC Oakland Point Molate Site Richmond CA	NSC Charleston SC	NSC Norfolk Craney Island VA	NSC Jacksonville FL
AL - code	----	AL-12365-F	AL-12412-F	AL-12423-F	AL-12425-F	AL-12432-F	AL-12466-F
Date received	----	14 Sept 1983	5 Oct 1983	12 Oct 1983	13 Oct 1983	21 Oct 1983	9 Nov 1983
Method of Sampling	----	Thief	Thief	Thief	Thief	Thief	Thief
Tank No	----	42	D-25	7	3900-F	122	31
Initials	----	II	WLM	VBR	LEN	PBJ	CB
PROPERTIES							
Cetane No	45 min	48	50	48	46	53	46
Appearance	Clear, bright and free of particles (1)	C,B, water droplets	C & B	C & B	C & B	C & B	C & B
Distillation, °C(°F)							
IBP	----	214(418)	214(418)	227(441)	209(408)	212(413)	193(380)
10% point	----	242(468)	242(468)	253(488)	251(484)	238(460)	231(448)
50% point	Record	285(545)	277(530)	289(552)	290(554)	280(536)	282(539)
90% point	357(675) max	335(635)	320(608)	332(630)	327(620)	332(629)	331(627)
EP	385(725) max	362(683)	349(661)	367(693)	359(678)	362(683)	362(683)
Residue and loss, vol%	3.0 max	1	1	1	1.5	1	1
Flash point, °C(°F)	60(140) min	90(194)	88(191)	102(216)	72(162)	89(193)	76(169)
Pour point, °C(°F)	-6(20) max	-9(16)	-12(10)	-9(16)	-18(0)	-9(16)	-15(5)
Cloud point, °C(°F)	-1(30) max	-9(16)	-10(14)	-8(18)	-14(7)	-5(23)	-13(9)
Viscosity @ 40°C(104°F), cSt	1.7-4.3	3.76	3.28	4.04	3.93	3.30	3.48
Carbon residue, 10% bottoms, wt%	0.20 max	0.15	0.12	0.12	0.15	0.15	0.14
Sulfur, wt%	1.00 max	0.47	0.45	0.34	0.75	0.52	0.48
Cu corrosion @ 100°C(212°F)	1 max	1A	1A	1A	1A	1A	1A
Color	3 max	2	3	3	2.5	1.5	3.5
Ash wt%	0.005 max	0	0	0	0	0	0
Gravity, °API	Record	31.9	33.3	31.7	32.6	34.8	33.1
Demulsification @ 25°C(77°F) min	10 max	3	3	4	4	3	3
Acid No, mgKOH/g	0.30	0.16	0.07	0.24	0.03	0.15	0.10
Neutrality	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral
Aniline point, °C(°F)	Record	63.0(145.4)	64.0(147.2)	64.4(147.9)	65.6(150.1)	68.6(155.5)	64.4(147.9)
Accelerated stability, mg/100 ml	1.5 max	0.1	0.4	0.2	0.3	0.8	1.8
Trace elements			Fe-13 ppm	Fe-17 ppm P-0.04%	Fe-21 ppm Cr-17 ppm P-0.06% Cl-0.01%	Fe-18 ppm	none
Aromatics, vol%	NR (2)	41	37	38	41	32	38
Net heat of combustion, Btu/lb	NR	18,145	18,196	18,215	18,156	18,253	18,237
MJ/kg		42.205	42.31	42.368	42.230	42.456	42.419
Water and sediment, vol%	0.01 (1)	0.02	-	-	-	-	-

---

(1) A slight haze is acceptable providing a maximum water and sediment of 0.01 vol% is obtained.
(2) No requirement.

TABLE 1-2. ANALYSES OF MIL-F-16884H, "NDF" SAMPLES

Sample From	NDF Requirements ----	NSC San Diego Point Loma San Diego CA	NSC Puget Sound Manchester WA	NSC Oakland Point Molate Site Richmond CA	NSC Charleston SC	NSC Norfolk Craney Island VA	NSC Jacksonville FL
AL - code	----	AL-12572-F	AL-12575-F	AL-12614-F	AL-12618-F	AL-12626-F	AL-12622-F
Date received	----	12-22-83	12-28-83	12-29-83	1-4-84	1-11-84	1-5-84
Method of Sampling	----	Thief	Thief	Thief	Thief	Thief	Thief
Tank No	----	43	D-25	11	3900-F	118	20
Initials	----	II	WLM	RLC	LEN	PBJ	CDB
PROPERTIES							
Cetane No	45 min	50	50	50	49	50	48
Appearance	Clear, bright and free of particles (1)	C & B	C & B	C & B	Cloudy Hazy	C & B	C & B
Distillation, °C(°F)							
IBP	----	216(420)	214(418)	204(400)	194(382)	206(402)	194(381)
10% point	----	246(474)	242(468)	233(452)	229(444)	240(464)	227(440)
50% point	Record	283(541)	277(531)	277(531)	278(533)	285(545)	273(525)
90% point	357(675) max	317(603)	323(613)	326(618)	326(619)	332(630)	326(619)
EP	385(725) max	338(641)	346(655)	354(669)	357(657)	362(683)	356(672)
Residue and loss, vol%	3.0 max	1	1	1	1	1	1
Flash point, °C(°F)	60(140) min	85(185)	86(187)	79(174)	75(167)	82(179)	74(166)
Pour point, °C(°F)	-6(20) max	-11(12)	-13(9)	-12(10)	-13(9)	-9(16)	-15(5)
Cloud point, °C(°F)	-1(30) max	-10(14)	-10(14)	-9(16)	-11(12)	-9(16)	-11(12)
Viscosity @ 40°C(104°F), cSt	1.7-4.3	3.53	3.26	3.26	3.21	3.54	3.04
Carbon residue, 10% bottoms, wt%	0.20 max	0.17	0.12	0.14	0.15	0.14	0.15
Sulfur, wt%	1.00 max	0.28	0.43	0.33	0.57	0.43	0.38
Cu corrosion @ 100°C(212°F)	1 max	1A	1A	1A	1A	1A	1A
Color	3 max	4.0	3.0	3.5	3.0	3.0	3.0
Ash wt%	0.005 max	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Gravity, °API	Record	33.3	33.4	33.6	34.4	33.3	34.8
Demulsification @ 25°C(77°F) min	10 max	5	3	2	4	3	2
Acid No, mg KOH/g	0.30	0.19	0.08	0.19	0.09	0.10	0.15
Neutrality	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral
Aniline point, °C(°F)	Record	65.5(149.9)	63.7(146.7)	64.3(147.7)	65.7(150.3)	65.4(149.7)	64.4(147.9)
Accelerated stability, mg/100 ml	1.5 max	0.7	0.6	0.5	3.4	3.6	0.28
Trace elements		Fe-<15 ppm Cu-14 ppm	Fe-17 ppm Cu-17 ppm	none	Fe-18 ppm Cu-12 ppm	none	none
Aromatics, vol%	NR (2)	35	37	34	34	35	33
Net heat of combustion, Btu/lb	NR	18,323	18,226	18,272	18,263	18,267	18,423
MJ/kg		42.619	42.395	42.500	42.479	42.490	42.852
Water and sediment, vol%	0.01 (1)	-	-	-	0.005	-	-

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(1) A slight haze is acceptable providing a maximum water and sediment of 0.01 vol% is obtained.
(2) No requirement.

(MPR83E.L)

TABLE 1-3. ANALYSES FOR MIL-F-24951, "FOR", SAMPLES

	FOR Requirements						
Sample From	----	NSC San Diego Point Loma San Diego CA	NSC Puget Sound Manchester WA	NSC Oakland Point Molate Site Richmond CA	NSC Charleston SC	NSC Norfolk Craney Island VA	NSC Jacksonville FL
AL - code	----	AL-12360-F	AL-12413-F	AL-12424-F	AL-12426-F	AL-12433-F	AL-12467-F
Date received	----	14 Sept 1983	6 Oct 1983	12 Oct 1983	13 Oct 1983	21 Oct 1983	9 Nov 1983
Method of Sampling	----	Top	Thief	Top Midlevel	Thief Composite	Top	Thief
Tank No	----	44	F-32	E	39A,39D,39060	61	USN 97-31082
Initials	----	14	WLM	BR	LEH	PBJ	CB
PROPERTIES							
Gravity, °API	25-40	32.9	34.4	33.4	30.4	35.6	33.8
Viscosity at 40°C(104°F), cSt	2.0-15.0	4.17	3.94	3.50	7.55	3.33	3.47
Viscosity at 50°C(122°F), SUS	30-90	37.1	36.5	35.6	44.2	35.2	35.5
Flash point, °C(°F)	55(130) min	water vapor	77(171)	80(176)	92(198)	78(173)	75(167)
Pour point, °C(°F)	-6.7(20) max	0(-18)	-42(-44)	<-42(-44)	-27(-17)	-21(-6)	<-42(-44)
Sulfated ash, wt%	0.15 max	0.03	0.04	0.004	0.14	0.02	0.04
Water and sediment, vol%	2.0 max	0.4 sed.	1.0	0.07	0.3 water 5 sed. (1)	0.4	0.02 water 1.8 sed.
Neutrality	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral
Sediment, wt%	0.5 max	0.01	<0.01	<0.01	0.06	0	0.04
Chlorinated material	no green flame	none	none	none	none	none	none
Sulfur, wt%	2.0 max	0.38	0.35	0.51	0.58	0.41	0.24
Explosiveness %	50 max	10	10	5	15	25	12
Cetane number	NR (2)	47	50	48	47	51	48
Total acid, no. mg/KOH/g	NR	0.34	0.26	0.23	0.42	0.24	0.13
Cu corrosion @ 100°C(212°F)	NR	4A	4A	3B	4B	4B	3B
Accelerated stability, mg/100mL	NR	1.4	1.6	2.2	Filter plugged	0.5	5.8
Aniline point, °C(°F)	NR	72.2(162)	75(167)	63.8(147)	82.5(180)	71.9(161)	70.5(158.9)
Net heat of combustion, Btu/lb	NR	18,305	18,169	18,205	18,028	18,301	18,223
Bromine number	NR	1.37	1.26	1.72	1.59	1.09	0.85
Carbon residue, 10% bottoms, wt%	NR	0.81	1.34	6.49	10.8	1.3	1.0
Distillation, °C(°F)							
IBP		206(402)	189(372)	202(395)	183(361)	181(357)	189(372)
10%		227(440)	216(421)	221(429)	223(433)	213(416)	216(420)
50%		280(536)	272(522)	263(506)	297(567)	264(508)	266(510)
90%		347(657)	378(713)	361(682)	383(721)	364(687)	362(683)
EP		382(719)	388(731)	366(690)	383(721)	395(743)	395(743)
Residue		1	0.7	3	4	1	1
Water, Karl Fischer, wt%	NR	0.57	0.96	0.012	1.32	0.17	0.37
Trace metals	NR	Fe-37 ppm Cu-26 ppm Cl-0.04 wt%	Fe-44 ppm Cu-11 ppm Cl-0.10 wt%	Fe-14 ppm P-0.05%	Fe-45 ppm Cr-6 ppm Zn-30 ppm P-0.07% Cl-0.09%	Fe-21 ppm Cu-14 ppm Ca-36 ppm	Fe-45 ppm Cu-<10 ppm Cr-<15 ppm Pb-<40 ppm Ca-0.01 wt% Ba-0.05 wt% Zn-0.02 wt% P-0.06 wt%

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(1) Sludge-like in appearance.

(2) NR - No requirement.

TABLE I-4. ANALYSES OF MIL-F-24951, "FOR", SAMPLES

Sample From	FOR Requirements	NSC San Diego Point Loma San Diego CA	NSC Puget Sound Manchester WA	NSC Oakland Point Molate Site Richmond CA	NSC Charleston SC	NSC Norfolk Craney Island VA	NSC Jacksonville FL
AL - code	----	AL-12521-F	AL-12500-F	AL-12530-F	AL-12515-F	AL-12516-F	AL-12531-F
Date received	----	23 Nov 1983	15 Nov 1983	8 Dec 1983	21 Nov 1983	21 Nov 1983	8 Dec 1983
Method of Sampling	----	Thief	Thief	Tap Midlevel	Thief Composite	Tap	Middle
Tank No	----	44	F-32	E	390,39060	60	USN 83682
Initials	----	II	WLM	BR	LEN	PBJ	CB
PROPERTIES							
Gravity, °API	25-40	33.1	34.4	33.6	31.2	36.1	34.7
Viscosity at 40°C(104°F), cSt	2.0-15.0	4.09	3.95	3.52	6.64	2.95	3.35
Viscosity at 50°C(122°F), SUS	30-90	37.1	36.5	35.6	43.3	34.1	35.2
Flash point, °C(°F)	55(130) min	78(173)	No Flash (1)	81(177)	82(179)	74(165)	75(167)
Pour point, °C(°F)	-6.7(20) max	-22(-7.6)	-42(-44)	-40(-40)	-27(-17)	-45(-49)	-40(-40)
Sulfated ash, wt%	0.15 max	0.02	0.04	0.002	0.06	0.01	0.03
Water and sediment, vol%	2.0 max	0.58	3.6	0.02	1.4	0.06	0.62
Neutrality	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral
Sediment, wt%	0.5 max	0.01	0.02	0.01	0.04	0.02	0.02
Chlorinated material	no green flame	None	None	None	None	None	None
Sulfur, wt%	2.0 max	0.34	0.37	0.49	0.53	0.37	0.27
Explosiveness %	50 max	13	15	5	14	13	7
Cetane number	NR (2)	48	48	46	47	50	47
Total acid, no. mg/KOH/g	NR	0.25	0.26	0.31	1.36	0.24	0.34
Cu corrosion @ 100°C(212°F)	NR	3B	4A	3B	4A	4C	4B
Accelerated stability, mg/100mL	NR	1.64	1.86	3.51	(3)	12.33	3.32
Aniline point, °C(°F)	NR	71.0(159.8)	75.5(167.9)	64.6(148.3)	76.7(170.1)	69.4(156.9)	69.6(157.3)
Net heat of combustion, Btu/lb	NR	18,265	18,185	18,230	18,089	18,343	18,249
Bromine number	NR	1.09	0.73	1.45	1.45	1.5	1.0
Carbon residue, 10% bottoms, wt%	NR	0.7	1.1	7.89	7.59	3.05	1.38
Distillation, °C(°F)							
IBP		184(364)	183(362)	201(394)	182(360)	183(362)	186(367)
10%		229(444)	213(416)	233(452)	223(434)	208(407)	211(412)
50%		282(541)	270(519)	261(502)	292(559)	250(483)	265(509)
90%		356(672)	370(699)	354(670)	376(709)	357(675)	357(675)
EP		378(713)	387(728)	370(698)	377(710)	364(688)	382(720)
Residue		2.0	2.0	0	1.0	4.8	1.0
Water, Karl Fischer, wt%	NR	0.25	0.80	0.014	0.97	0.53	0.34
Trace elements	NR	Fe-24 ppm Cl-0.02 wt%	Fe-42 ppm Cu-0.002 wt% Cr-0.002 wt% Pb-<0.006 wt% P-0.04 wt% Cl-0.12 wt%	ND (4)	Fe-215 ppm Cu-0.003 wt% P-0.03 wt% Cl-0.1 wt% Ca-0.03 wt% Zn-0.02 wt%	Fe-23 ppm Cl-0.02 wt%	Fe-46 ppm Cu-0.001 wt% Cl-0.03 wt% Zn-0.02 wt% Ca-0.01 wt%

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(1) Due to water vapor
(2) NR - No requirement
(3) Filter plugged
(4) ND - None detected

TABLE I-5. ANALYSES FOR MIL-F-24951, "FOR" SAMPLES

Sample From	FOR Requirements	NSC San Diego Point Loma San Diego CA	NSC Puget Sound Manchester WA	NSC Oakland Point Molate Site Richmond CA	NSC Charleston SC	NSC Norfolk Craney Island VA	NSC Jacksonville FL
AL - code	----	AL-12573-F	AL-12576-F	AL-12615-F	AL-12619-F	AL-12627-F	AL-12623-F
Date received	----	12-22-83	12-28-83	12-29-83	1-4-84	1-11-84	1-5-84
Method of Sampling	----	Thief	Thief	Tap	Thief	Tap 8 ft	Thief
Tank No	----	63	F-32	E	3906-0	61	97-31062
Initials	----	II	WLM	RLC	LEN	PBJ	CDB
PROPERTIES							
Gravity, °API	25-40	32.9	34.4	35.5	33.6	35.4	34.9
Viscosity at 40°C(104°F), cSt	2.0-15.0	4.08	3.85	3.49	7.43	3.31	3.33
Viscosity at 50°C(122°F), SUS	30-90	36.9	36.4	35.6	45.9	35.2	35.0
Flash point, °C(°F)	55(130) min	85(185)	No flash(1)	81(177)	No flash(1)	72(161)	74(166)
Pour point, °C(°F)	-6.7(20) max	-20(-4)	-45(-49)	-39(-38)	-26(-15)	-45(-49)	-45(-49)
Sulfated ash, wt%	0.15 max	0.015	0.031	0.002	0.18	0.016	0.027
Water and sediment, vol%	2.0 max	0.1	1.0	0.08	8.0	0.10	0.12
Neutrality	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral	Neutral
Sediment, wt%	0.5 max	0.01	0.01	0.01	0.14	0.01	0.02
Chlorinated material	no green flame	none	none	none	none	none	none
Sulfur, wt%	2.0 max	0.39	0.36	0.52	0.41	0.39	0.27
Explosiveness, %	50 max	13	15	7	15	14	14
Cetane number	NR (2)	51	50	50	50	50	49
Total acid No, mg KOH/g	NR	0.26	0.38	0.51	0.64	0.38	0.38
Cu corrosion @ 100°C(212°F)	NR	3B	4A	3B	4A	4B	4A
Accelerated stability, mg/100mL	NR	4.8	1.4	2.7	(3)	4.97	5.84
Aniline point, °C(°F)	NR	69.7(157.5)	72.5(162.5)	64.5(148.1)	77.0(170.6)	69.9(157.8)	69.9(157.8)
Net heat of combustion, Btu/lb	NR	18,248	17,933	18,250	17,177	18,302	18,288
Bromine number	NR	1.18	1.00	1.63	1.54	1.27	0.93
Carbon residue, 10% bottoms, wt%	NR	0.78	1.32	7.2	(5)	3.73	1.51
Distillation, °C(°F)							
IBP		200(392)	192(378)	196(385)	(5)	179(354)	174(346)
10%		236(456)	220(428)	218(425)		211(411)	217(422)
50%		284(543)	272(521)	259(499)		258(496)	266(510)
90%		357(675)	384(723)	348(658)		372(702)	361(682)
EP		399(751)	432(809)	382(720)		379(714)	387(728)
Residue		1	1	1		1.5	1
Water, Karl Fischer, wt%	NR	0.07	0.74	0.02	4.17	0.10	0.19
Trace metals	NR	Fe-37 ppm Cu-15 ppm	Fe-43 ppm Cu-12 ppm	Fe-20 ppm	Fe-759 ppm Cr-33 ppm Cu-44 ppm Cn-0.03 wt% Zn-0.02 wt% Cl-0.12 wt%	Fe-16 ppm P-0.03 wt% Cl-0.04 wt%	Fe-29 ppm P-0.02 wt% Cl-0.03 wt%

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(1) Due to water vapor

(2) NR - No requirement

(3) Filter plugged

(4) ND - None detected

(5) Due to excessive emulsified water, tests could not be conducted

TABLE I-6. PURGEABLE PRIORITY POLLUTANTS, mg/kg (ppm)
(First Round Samples)

	NSC San Diego		NSC Puget Sound		NSC Oakland		NSC Charleston		NSC Norfolk		NSC Jacksonville	
AL-F Code	12364	12367	12412	12413	12423	12424	12425	12426	12432	12433	12466	12467
	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR
Volatile Compounds												
Methylene chloride	ND	5.6	ND	30	ND	ND	ND	5.3	ND	ND	ND	ND
Trichlorofluoromethane	ND	26	ND	45	ND	ND	ND	1.9	0.5	100	ND	ND
Trichlorotrifluoroethane	ND	40	ND	140	ND	ND	ND	600	ND	35	ND	52
1,1-Dichloroethane	ND	ND	ND	3.6	ND	ND	ND	1.6	ND	0.1	ND	0.3
trans-1,2-Dichloroethylene	ND	ND	ND	14	ND	ND	ND	0.4	ND	0.1	ND	1.9
1,1,1-Trichloroethane	ND	18	ND	400	ND	ND	ND	61	ND	29	ND	18
1,2-Dichloropropane	ND	ND	ND	ND	ND	ND	ND	0.2	ND	ND	ND	ND
Trichloroethylene	ND	0.2	ND	180	ND	0.3	ND	0.3	ND	0.8	ND	1.4
1,1,2-Trichloroethane	ND	ND	ND	ND	ND	0.3	ND	ND	ND	ND	ND	ND
Benzene	10	23	18	21	4.4	5.3	38	11	8.5	4.9	29	18
Tetrachloroethylene	ND	28	ND	63	ND	ND	ND	38	ND	20	ND	170
Toluene	110	180	160	190	47	93	150	170	60	100	190	290
Ethylbenzene	130	110	120	160	38	81	120	80	42	130	100	210

ND = None detected, detection limit 0.1 mg/kg

(MPR83E.M)

TABLE I-7. SEMIVOLATILE ORGANIC COMPOUNDS, mg/kg (ppm)
(First Round Samples)

AL-F Code	NSC San Diego		NSC Puget Sound		NSC Oakland		NSC Charleston		NSC Norfolk		NSC Jacksonville	
	12364	12367	12412	12413	12423	12424	12425	12426	12432	12433	12466	12467
	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR
Compounds												
Naphthalene	760	520	270	880	780	1200	160	1200	410	1100	710	2600
Fluorene	1100	670	480	230	410	320	360	330	350	280	270	290
Phenanthrene	960	540	800	360	710	580	680	400	610	390	480	510
Anthracene	ND	ND	41	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-butyl phthalate	ND	ND	ND	ND	20	18	23	ND	25	ND	ND	ND
Pyrene	ND	ND	ND	16	32	41	30	34	20	44	30	100
Benzo(a)anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chrysene	ND	ND	ND	ND	ND	20	ND	16	ND	ND	ND	ND
bis-2-Ethylhexyl-phthalate	ND	ND	690	ND	ND	ND	ND	ND	ND	39	ND	ND
1,2,4-Trimethyl benzene	500	600	ND	38	ND	230	ND	310	ND	230	680	1500
Tetramethyl benzene	140	160	ND	160	55	280	ND	210	27	160	250	570
Tetralin	ND	ND	ND	310	190	730	ND	160	22	190	200	670
2-Methyl naphthalene	1400	840	660	1000	850	1100	380	1300	63	1100	1300	1500
Biphenyl	360	210	500	220	ND	390	210	330	290	260	910	310
2-Ethyl naphthalene	470	160	190	350	390	550	120	480	200	400	920	590
2,3-Dimethyl naphthalene	1900	1100	680	650	560	750	410	820	530	790	600	750
2,6-Dibutyl methyl phenol	ND	ND	ND	66	ND	160	ND	130	12	64	ND	165
Dibenzofuran	ND	ND	190	72	94	96	60	73	97	ND	58	78
Triphenyl phosphate	ND	93	ND	ND	ND	ND	Tr<1.2	26	Tr<1.2	12	ND	120
Diethyl hexyl sebacate	ND	ND	ND	ND	ND	ND	ND	21	ND	Tr<2	ND	93

ND - None detected, less than 1 mg/kg

(HPR83E.M)

TABLE I-8. PESTICIDES AND PCB'S, mg/kg (ppm)
(First Round Samples)

Source	NSC San Diego		NSC Puget Sound		NSC Oakland		NSC Charleston		NSC Norfolk		NSC Jacksonville	
Sample No.	AL-12364-F	AL-12367-F	AL-12412-F	AL-12413-F	AL-12423-F	AL-12424-F	AL-12425-F	AL-12426-F	AL-12432-F	AL-12433-F	AL-12466-F	AL-12467-F
	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR
<u>Compounds</u>												
Aldrin	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
Alpha-BHC	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
Beta-BHC	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
Delta-BHC	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
Gamma-BHC	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
4,4'-DDD	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
4,4'-DDE	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
4,4'-DDT	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
Dieldrin	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
Endosulfan I	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
Endosulfan II	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
Endrin	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5
Heptachlor	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
Hepatchlor epoxide	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <0.5	ND <1	ND <1	ND <0.5	ND <0.5
Toxaphene	ND <2	ND <2	ND <2	ND <10	ND <2	ND <10	ND <10	ND <15	ND <5	ND <15	ND <2	ND <2
Chlordane	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1254	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1016	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1260	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1221	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1232	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1242	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1
AR 1248	ND <1	ND <1	ND <2.5	ND <10	ND <2	ND <10	ND <10	ND <10	ND <5	ND <15	ND <1	ND <1

ND = Not detected; value shows limit of detection.

(MPR83E:H)

TABLE I-9. PURGEABLE PRIORITY POLLUTANTS, mg/kg (ppm)
(Second Round "FOR" Samples)

	NSC San Diego AL-1252[illegible]-F	NSC Puget Sound AL-12500-F	NSC Oakland AL-12530-F	NSC Charleston AL-12515-F	NSC Norfolk AL-12514-F	NSC Jacksonville AL-12531-F
Volatile Compounds						
Methylene chloride	6.5	33	ND	7.0	0.6	ND
Trichlorofluoromethane	18	73	ND	2.3	29	ND
Trichlorotrifluoroethane	52	590	ND	940	31	46
1,1-Dichloroethane	0.1	4.2	ND	2.1	0.1	ND
trans-1,2-Dichloroethylene	ND	14	ND	0.7	ND	3.2
1,1,1-Trichloroethane	16	490	ND	120	98	11
1,2-Dichloropropane	ND	ND	ND	0.3	ND	ND
Trichloroethylene	0.2	190	ND	0.5	0.6	0.9
1,1,2-Trichloroethane	ND	ND	ND	ND	ND	ND
Benzene	26	24	5.4	14	9.3	18
Tetrachloroethylene	28	63	ND	62	12	120
Toluene	270	220	95	240	160	200
Ethylbenzene	160	170	100	150	170	210

ND = None detected, detection limit 0.1 mg/kg

	NSC San Diego AL-1252[illegible]-F	NSC Puget Sound AL-12500-F	NSC Oakland AL-12530-F	NSC Charleston AL-12515-F	NSC Norfolk AL-12514-F	NSC Jacksonville AL-12531-F
Semivolatile Organic Compounds						
Naphthalene	1400	1700	1200	2000	2200	2500
Fluorene	320	200	220	260	160	330
Phenanthrene	330	290	450	400	340	ND
Anthracene	ND	ND	ND	ND	ND	ND
Di-n-butyl phthalate	ND	ND	ND	ND	ND	ND
Pyrene	20	30	60	60	60	110
Benzo(a)anthracene	ND	ND	ND	ND	ND	ND
Chrysene	ND	ND	ND	ND	ND	ND
bis-2-Ethylhexyl-phthalate	35	61	8	21	15	19
1,2,4-Trimethyl benzene	860	1400	760	990	1100	1400
Tetramethyl benzene	250	420	470	420	500	640
Tetralin	610	800	1600	350	1800	1400
2-Methyl naphthalene	960	1000	890	1200	1200	1400
Biphenyl	220	180	260	270	250	300
2-Ethyl naphthalene	620	510	600	550	470	540
2,3-Dimethyl naphthalene	540	430	470	550	530	630
2,6-Dibutyl methyl phenol	100	280	ND	340	110	168
Dibenzofuran	79	57	58	61	51	67
Triphenyl phosphate	170	240	31	100	43	87
Diethyl hexyl sebacate	4	21	ND	39	13	74

ND = None detected, less than 2 mg/kg

(MPR83E.M)

TABLE I-10. PESTICIDES AND PCB'S, mg/kg (ppm)
(Second Round "FOR" Samples)

Source Sample No.	NSC San Diego AL-12521-F FOR	NSC Puget Sound AL-12500-F FOR	NSC Oakland AL-12530-F FOR	NSC Charleston AL-12515-F FOR	NSC Norfolk AL-12514-F FOR	NSC Jacksonville AL-12531-F FOR
Compounds						
Aldrin	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Alpha-BHC	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Beta-BHC	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Delta-BHC	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Gamma-BHC	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
4,4'-DDD	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
4,4'-DDE	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
4,4'-DDT	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Dieldrin	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Endosulfan I	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Endosulfan II	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Endrin	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Heptachlor	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Hepatchlor epoxide	ND <3	ND <5	ND <1	ND <5	ND <3	ND <7.5
Toxaphene	ND <15	ND <20	ND <4	ND <20	ND <15	ND <25
Chlordane	ND <7.5	ND <10	ND <2	ND <10	ND <7.5	ND <15
AR 1254	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30
AR 1016	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30
AR 1260	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30
AR 1221	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30
AR 1232	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30
AR 1242	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30
AR 1248	ND <10	ND <20	ND <10	ND <20	ND <10	ND <30

ND = Not detected; value shows limit of detection.

(MPR83E.M)

TABLE I-11. PURGEABLE PRIORITY POLLUTANTS, mg/kg (ppm)
(Second Round "NDF" and Third Round "FOR" Samples)

AL-F Code	NSC San Diego		NSC Puget Sound		NSC Oakland		NSC Charleston		NSC Norfolk		NSC Jacksonville	
	12572	12573	12575	12576	12614	12615	12618	12619	12626	12627	12622	12623
	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR
Volatile Compounds												
Methylene chloride	ND	1.1	ND	7.8	ND	ND	ND	ND	ND	8.1	ND	ND
Trichlorofluoromethane	ND	9.1	ND	71	ND	ND	ND	8.0	ND	20	ND	ND
Trichlorotrifluoroethane	ND	59	ND	380	ND	ND	ND	680	ND	250	ND	53
1,1-Dichloroethane	ND	0.3	ND	3.8	ND	ND	ND	3.1	ND	0.3	ND	0.4
trans-1,2-Dichloroethylene	ND	ND	ND	15	ND	ND	ND	0.5	ND	ND	ND	3.6
1,1,1-Trichloroethane	ND	24	ND	520	ND	ND	ND	83	ND	68	ND	11
1,2-Dichloropropane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethylene	ND	0.8	ND	130	ND	ND	ND	0.4	ND	1.6	ND	1.1
1,1,2-Trichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	53	20	19	19	41	5.8	45	16	48	9.2	38	18
Tetrachloroethylene	ND	18	ND	65	ND	ND	ND	38	ND	180	ND	110
Toluene	170	180	190	180	180	89	170	230	180	140	190	210
Ethylbenzene	120	140	160	160	140	89	150	150	120	180	180	170

ND = None detected, detection limit 0.1 mg/kg

(MPR83E.M)

TABLE I-12. SEMIVOLATILE ORGANIC COMPOUNDS, mg/kg (ppm)
(Second Round "NDF" and Third Round "FOR" Samples)

AL-F Code	NSC San Diego		NSC Puget Sound		NSC Oakland		NSC Charleston		NSC Norfolk		NSC Jacksonville	
	12572	12573	12575	12576	12614	12615	12618	12619	12626	12627	12622	12623
	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR
<u>Compounds</u>												
Naphthalene	330	640	1000	580	760	640	790	830	730	680	770	890
Fluorene	240	260	350	150	300	210	340	190	400	160	260	220
Phenanthrene	460	430	680	230	430	360	530	310	640	180	420	370
Anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Di-n-butyl phthalate	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Pyrene	20	20	Tr<20	Tr<20	20	20	30	25	50	20	30	30
Benzo(a)anthracene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chrysene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
bis-2-Ethylhexyl-phthalate	ND	36	ND	43	ND	16	ND	11	21	14	ND	24
1,2,4-Trimethyl benzene	260	620	630	710	550	550	1000	510	770	1200	870	610
Tetramethyl benzene	110	230	220	360	220	440	280	350	230	400	350	470
Tetralin	160	410	270	560	360	1100	320	270	210	480	260	360
2-Methyl naphthalene	630	1200	2200	1200	1300	1200	1500	1300	1700	1000	1300	1400
Biphenyl	190	310	520	210	270	420	570	340	710	250	380	390
2-Ethyl naphthalene	400	760	930	610	540	790	670	680	930	550	810	760
2,3-Dimethyl naphthalene	580	890	1700	720	1000	730	1100	740	980	570	910	980
2,6-Dibutyl methyl phenol	ND	58	ND	150	ND	ND	ND	240	ND	96	ND	94
Dibenzofuran	60	96	180	53	100	70	70	62	80	42	70	63
Triphenyl phosphate	ND	63	ND	76	ND	ND	ND	39	ND	12	ND	21
Diethyl hexyl sebacate	ND	8	ND	11	ND	ND	ND	24	ND	ND	ND	41

ND = None detected, less than 2 mg/kg

(HPR83E.M)

TABLE I-13. PESTICIDES AND PCB'S, mg/kg (ppm)
(Second Round "NDF" and Third Round "FOR" Samples)

Source	NSC San Diego		NSC Puget Sound		NSC Oakland		NSC Charleston		NSC Norfolk		NSC Jacksonville	
Sample No.	AL-12572-F	AL-12573-F	AL-12575-F	AL-12576-F	AL-12614-F	AL-12615-F	AL-12618-F	AL-12619-F	AL-12626-F	AL-12627-F	AL-12622-F	AL-12623-F
	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR	NDF	FOR
Compounds												
Aldrin	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Alpha-BHC	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Beta-BHC	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Delta-BHC	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Gamma-BHC	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
4,4'-DDD	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
4,4'-DDE	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
4,4'-DDT	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Dieldrin	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Endosulfan I	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Endosulfan II	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Endrin	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Heptachlor	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Hepatchlor epoxide	ND <2	ND <3	ND <0.5	ND <2	ND <0.5	ND <2	ND <1	ND <1	ND <3	ND <5	ND <1	ND <5
Toxaphene	ND <10	ND <15	ND <5	ND <10	ND <10	ND <10	ND <5	ND <10	ND <15	ND <20	ND <10	ND <20
Chlordane	ND <5	ND <7.5	ND <5	ND <5	ND <5	ND <5	ND <2	ND <5	ND <7.5	ND <10	ND <5	ND <10
AR 1254	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25
AR 1016	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25
AR 1260	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25
AR 1221	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25
AR 1232	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25
AR 1242	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25
AR 1248	ND <10	ND <10	ND <5	ND <25	ND <5	ND <25	ND <10	ND <5	ND <10	ND <20	ND <5	ND <25

ND - Not detected; value shows limit of detection.

(MPR83E.M)

Tables I-14 through I-31 contain GC distillation data of quality unsuitable for reproduction. Copies can be provided upon request.

TABLE I-32. GC Distillation Report AL-12467F+C_6,
"FOR" Sample From NSC Jacksonville

WT% OFF	DEG C	WT% OFF	DEG C	WT% OFF	DEG C
.1	116.5	26	236.1	60	302
.2	124.7	27	237.6	61	303.4
.3	133.2	28	239.8	62	304.8
.4	138.9	29	242	63	306.2
.5	142.4	30	244.2	64	309
.6	144.5	31	247.1	65	311.1
.7	148	32	249.3	66	313.9
.8	150.9	33	251.5	67	316.8
.9	152.2	34	253	68	318.3
1	154.3	35	254.4	69	321.2
2	167.4	36	255.9	70	324.2
3	174.3	37	256.6	71	327.2
4	180.1	38	258.1	72	330.2
5	185.2	39	260.2	73	332.4
6	189.5	40	262.4	74	336.1
7	193.8	41	264.5	75	339.8
8	196.7	42	266	76	343.6
9	198	43	268.1	77	347.1
10	200.7	44	269.6	78	350.7
11	204.1	45	271.7	79	355.7
12	206.8	46	272.4	80	359.9
13	209.5	47	274.5	81	366.3
14	212.2	48	275.9	82	372.7
15	214.2	49	278	83	381.2
16	216.2	50	280.7	84	393.3
17	217.7	51	282.1	85	408.8
18	219.2	52	284.2	86	426.5
19	222.1	53	286.3	87	442.9
20	225.1	54	287.7	88	459.3
21	227.3	55	289.8	89	474.7
22	229.5	56	292	90	490
23	231.7	57	294.9	91	504.4
24	233.9	58	297	92	518.5
25	235.3	59	299.2	93	526.6

RESIDUE= 7.4

TABLE I-33. GC Distillation Report AL-12467F+C$_6$, "FOR" Sample From NSC Jacksonville

RT	AREA		AREA %	NAME
2.70	259109	VV	14.141	#NC6:69
4.22	597	BB	.033	
4.92	1267	BB	.069	
5.22	864	BV	.047	%NC8:126
5.62	1483	VV	.081	
5.96	3587	VV	.196	
6.24	1838	VV	.100	
6.40	4152	VV	.227	#NC9:151
6.62	1236	VV	.067	
6.78	3811	VV	.208	
6.89	1768	VV	.096	
7.09	13321	VV	.727	
7.35	7246	VV	.395	
7.50	21373	VV	1.166	#NC10:174
7.76	8485	VV	.463	
8.21	52746	VV	2.879	
8.53	62971	VV	3.437	#NC11:196
8.90	28067	VV	1.532	
9.12	47753	VV	2.606	
9.49	102723	VV	5.606	#NC12:216
9.87	15711	VV	.857	
10.16	55481	VV	3.028	
10.40	128625	VV	7.020	
11.08	66052	VV	3.605	
11.25	125441	VV	6.846	#NC14:254
11.77	81357	VV	4.440	
12.05	101686	VV	5.550	#NC15:271
12.52	63111	VV	3.444	
12.80	82717	VV	4.514	#NC16:287
13.20	52886	VV	2.886	
13.57	95217	VV	5.196	#NC17:302
13.90	33532	VV	1.830	
14.21	79988	VV	4.365	#NC18:316
14.59	16408	VV	.895	
14.84	74845	VV	4.085	
15.45	55166	VV	3.011	#NC20:344
16.02	35297	VV	1.926	
16.58	21094	VV	1.151	
17.12	10506	VV	.573	
17.63	4347	VV	.237	#NC24:391
18.14	1390	VB	.076	
24.53	7076	BB	.386	#NC40:522

TOTAL AREA = 1832333 TOTAL AREA % = 100.000

TABLE I-34 GC Distillation Report AL-12467F, "FOR" Sample From NSC Jacksonville

WT% OFF	DEG C
.1	107.6
.2	120.2
.3	127.6
.4	134.6
.5	139.6
.6	142.4
.7	145.2
.8	148
.9	150.1
1	151.5
2	165.4
3	172.3
4	177.2
5	182.3
6	186.6
7	191
8	194.6
9	196
10	198
11	200.7
12	203.4
13	206.1
14	208.8
15	210.8
16	213.5
17	214.9
18	216.2
19	217.7
20	219.9
21	222.9
22	225.1
23	227.3
24	229.5
25	231.7
26	233.1
27	234.6
28	235.3
29	236.8
30	238.3
31	240.5
32	242.7
33	245.6
34	247.8
35	249.3
36	251.5
37	253
38	253.7
39	255.2
40	256.6
41	258.1
42	259.5
43	261.7
44	263.1
45	265.3
46	266.7
47	268.9
48	270.3
49	271
50	272.4
51	274.5
52	275.9
53	278
54	280
55	282.1
56	284.2
57	285.6
58	287
59	288.4
60	290.6
61	293.4
62	295.6
63	297.7
64	299.9
65	302
66	302.7
67	304.1
68	306.2
69	308.3
70	311.1
71	313.9
72	316
73	317.5
74	319.8
75	322.7
76	325.7
77	328.7
78	330.9
79	333.2
80	336.9
81	340.6
82	343.6
83	347.1
84	350.7
85	355.7
86	359.2
87	365.6
88	371.3
89	379.1
90	389
91	401.8
92	416.6
93	432.2
94	446.4
95	461.4
96	475.4
97	488.6
98	501.7
99	514.5
99.5	520.6
100	526.6

RESIDUE= 0

TABLE I-34. GC Distillation Report (continued)

```
 RT          AREA        AREA %        NAME

  4.20        606 VB      .032
  4.90       1407 BB      .074
  5.20        968 BV      .051   %NC8:126
  5.62       1596 VV      .084
  5.94       4179 VV      .221
  6.22       2198 VV      .116
  6.38       4929 VV      .261   #NC9:151
  6.60       1458 VV      .077
  6.77       4207 VV      .223
  6.86       2254 VV      .119
  7.07      15697 VV      .831
  7.32       8557 VV      .453
  7.48      25495 VV     1.349   #NC10:174
  7.74       9898 VV      .524
  8.19      63534 VV     3.362
  8.51      75655 VV     4.003   #NC11:196
  8.87      33421 VV     1.769
  9.10      58001 VV     3.069
  9.47     124745 VV     6.601   #NC12:216
  9.84      18772 VV      .993
 10.15      63633 VV     3.367
 10.38     159260 VV     8.428
 11.24     232903 VV    12.325   #NC14:254
 11.62      24236 VV     1.282
 11.75      71439 VV     3.780
 12.03     123019 VV     6.510   #NC15:271
 12.50      76580 VV     4.052
 12.79     100671 VV     5.327   #NC16:287
 13.19      63285 VV     3.349
 13.55     119536 VV     6.326   #NC17:302
 13.88      35657 VV     1.887
 14.21     116565 VV     6.168   #NC18:316
 14.82      90727 VV     4.801
 15.43      67071 VV     3.549   #NC20:344
 16.00      42334 VV     2.240
 16.56      25438 VV     1.346
 17.10      12852 VV      .680
 17.61       5282 VV      .280   #NC24:391
 18.12       1678 VV      .089

TOTAL AREA =        1889742        TOTAL AREA % =     100.000
```

TABLE I-35. GC Distillation Report AL-12466-F,
"DFM" Sample From NSC Jacksonville

WT% OFF	DEG C	WT% OFF	DEG C	WT% OFF	DEG C
.1	100.9	28	261.7	64	311.8
.2	116.5	29	263.8	65	313.2
.3	124	30	265.3	66	315.3
.4	132.5	31	267.4	67	316
.5	137.5	32	268.9	68	317.5
.6	141.7	33	270.3	69	319
.7	144.5	34	271.7	70	320.5
.8	148	35	273.1	71	321.2
.9	150.9	36	274.5	72	322.7
1	153.6	37	275.9	73	325
2	170.9	38	277.3	74	326.5
3	180.8	39	279.3	75	327.9
4	188.8	40	280.7	76	329.4
5	195.3	41	282.1	77	330.9
6	199.4	42	283.5	78	332.4
7	204.1	43	284.9	79	333.9
8	208.8	44	286.3	80	335.4
9	212.8	45	287.7	81	337.6
10	216.2	46	288.4	82	339.8
11	219.2	47	289.8	83	341.3
12	223.6	48	291.3	84	343.6
13	227.3	49	292.7	85	345.7
14	230.2	50	294.1	86	347.1
15	233.1	51	295.6	87	349.3
16	235.3	52	297	88	351.4
17	237.6	53	298.4	89	354.2
18	239.8	54	299.9	90	356.4
19	242.7	55	301.3	91	359.2
20	246.4	56	302.7	92	362
21	248.6	57	303.4	93	366.3
22	251.5	58	304.8	94	369.1
23	253	59	305.5	95	374.1
24	255.2	60	306.9	96	379.1
25	256.6	61	308.3	97	385.5
26	258.1	62	309	98	394
27	259.5	63	310.4	99	407.4
				99.5	420.9
				100	473.3

RESIDUE= 0

TABLE I-35. GC Distillation Report (continued)

RT	AREA		AREA %	NAME
3.79	647	VV	.026	
4.23	961	VB	.038	
4.93	2541	BV	.101	
5.23	1985	VV	.079	%NC8:126
5.66	2526	VV	.101	
5.97	4836	VV	.193	
6.24	2571	VV	.102	
6.41	4305	VV	.172	#NC9:151
6.62	1420	VV	.057	
6.79	3721	VV	.148	
7.09	13778	VV	.549	
7.36	6465	VV	.258	
7.51	11136	VV	.444	#NC10:174
7.64	4427	VV	.177	
7.77	7110	VV	.283	
8.23	38877	VV	1.550	
8.54	42606	VV	1.699	#NC11:196
8.92	22341	VV	.891	
9.12	21671	VV	.864	
9.24	13637	VV	.544	
9.49	81553	VV	3.252	#NC12:216
9.88	13194	VV	.526	
10.40	168794	VV	6.730	
11.27	133961	VV	5.341	#NC14:254
11.35	81733	VV	3.259	
11.79	104033	VV	4.148	
12.07	163128	VV	6.504	#NC15:271
12.57	114590	VV	4.569	
12.84	168522	VV	6.719	#NC16:287
13.26	117928	VV	4.702	
13.61	213555	VV	8.514	#NC17:302
13.92	84980	VV	3.388	
14.29	246591	VV	9.832	#NC18:316
14.89	202405	VV	8.070	
15.49	98300	VV	3.919	#NC20:344
15.72	51771	VV	2.064	
16.05	100426	VV	4.004	
16.60	39040	VV	1.557	
16.81	24358	VV	.971	
17.12	40042	VV	1.596	
17.63	24186	VV	.964	#NC24:391
18.12	13780	VV	.549	
18.59	6922	VV	.276	
19.04	2821	VV	.112	
19.47	585	VB	.023	#NC28:431
24.65	3416	BB	.136	

TOTAL AREA = 2508175 TOTAL AREA % = 100.000

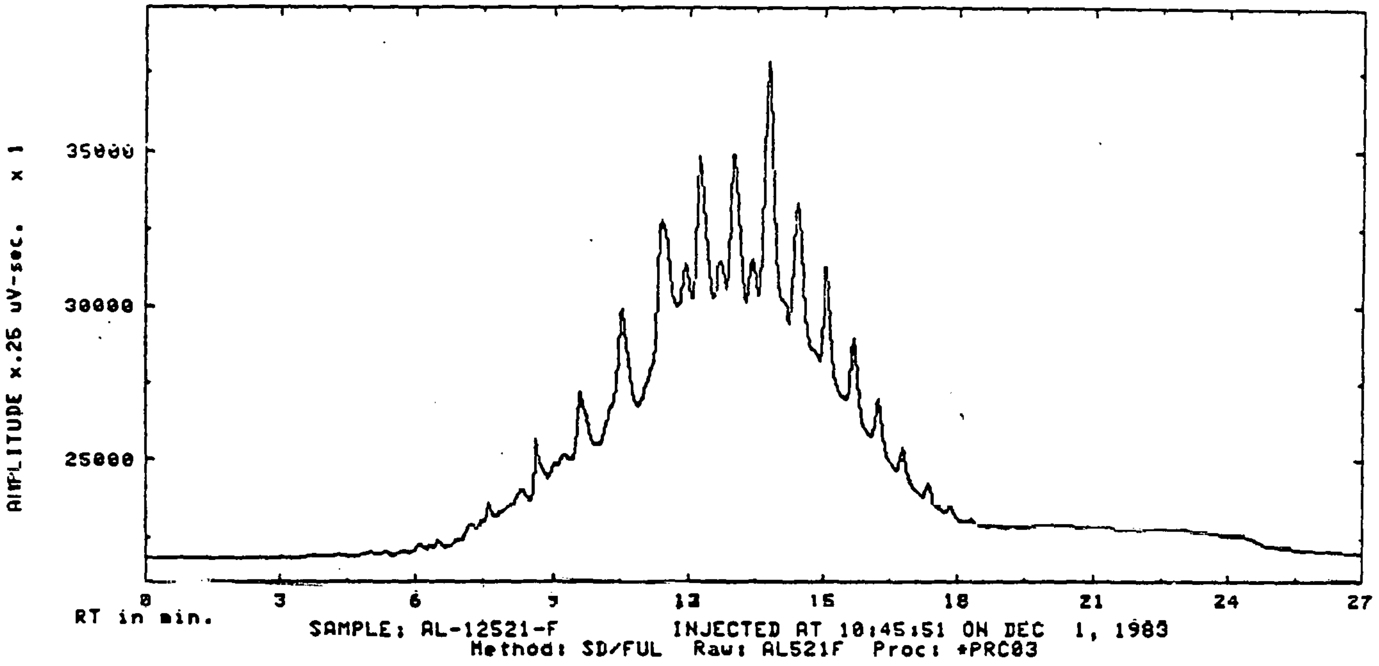

FIGURE 1. CHROMATOGRAM--"FOR" SAMPLE 2
FROM NSC SAN DIEGO

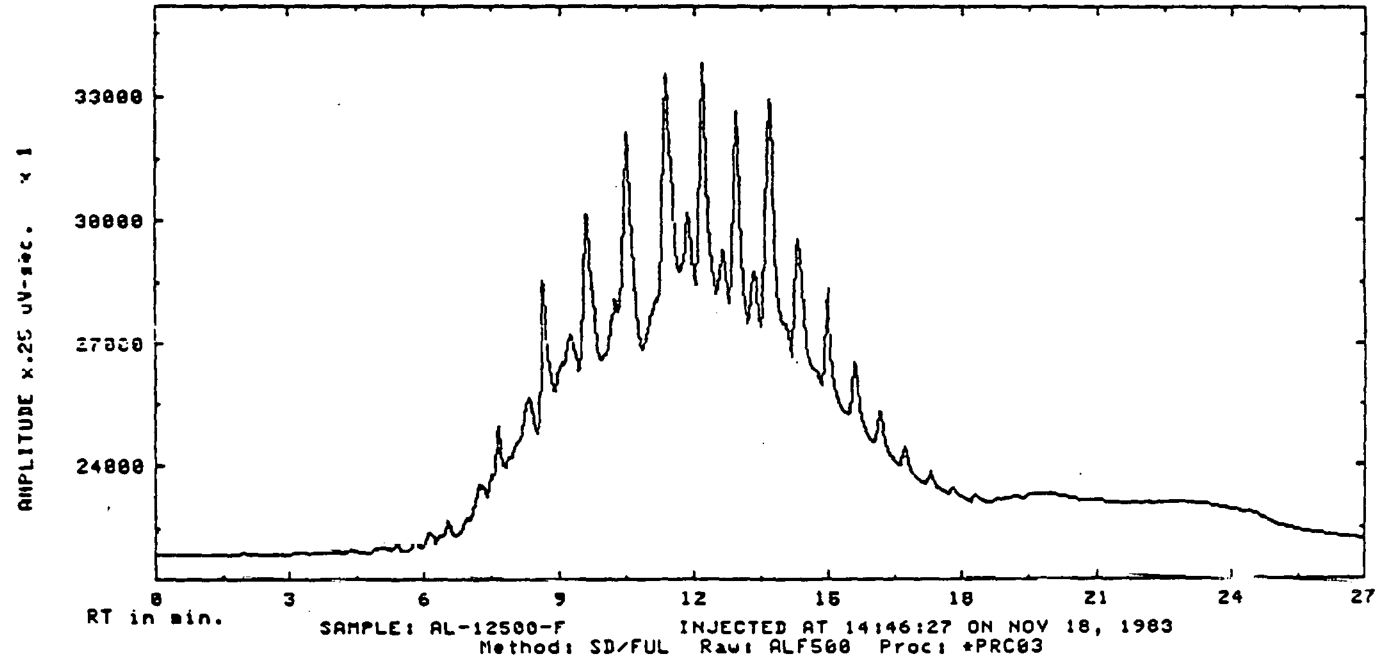

FIGURE 2. CHROMATOGRAM--"FOR" SAMPLE 2
FROM NSC PUGET SOUND

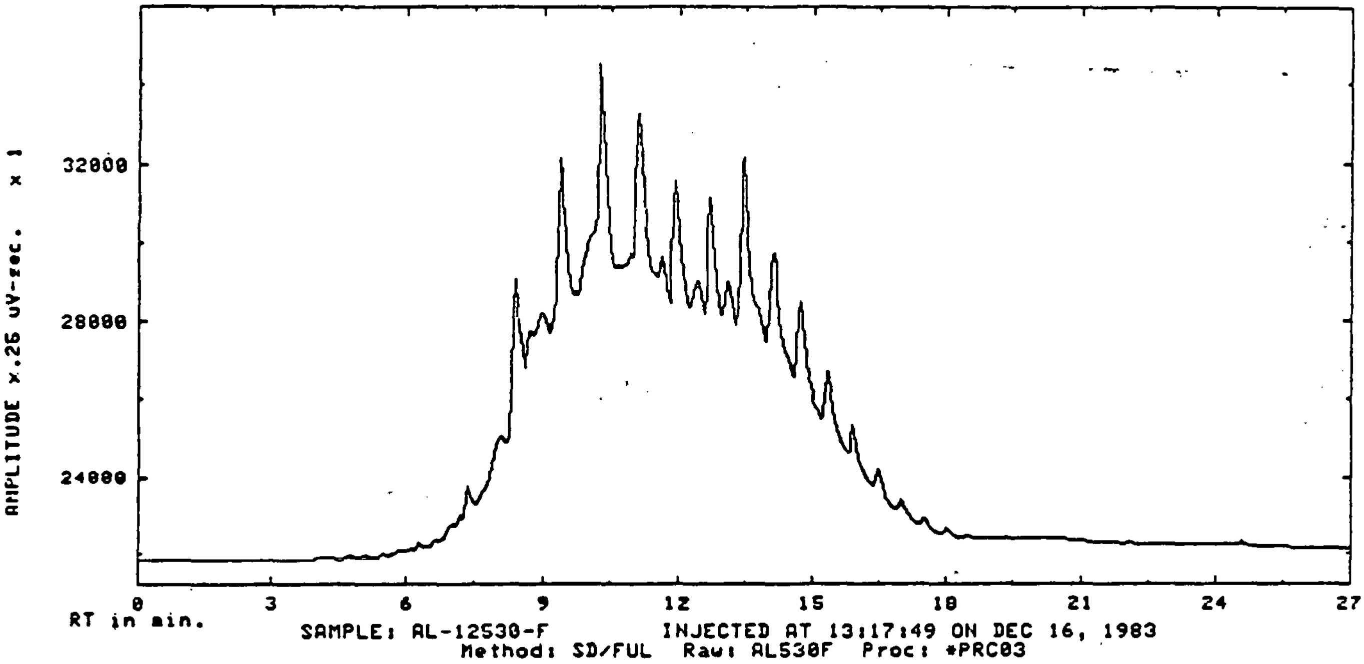

FIGURE 3. CHROMATOGRAM--"FOR" SAMPLE 2
FROM NSC OAKLAND

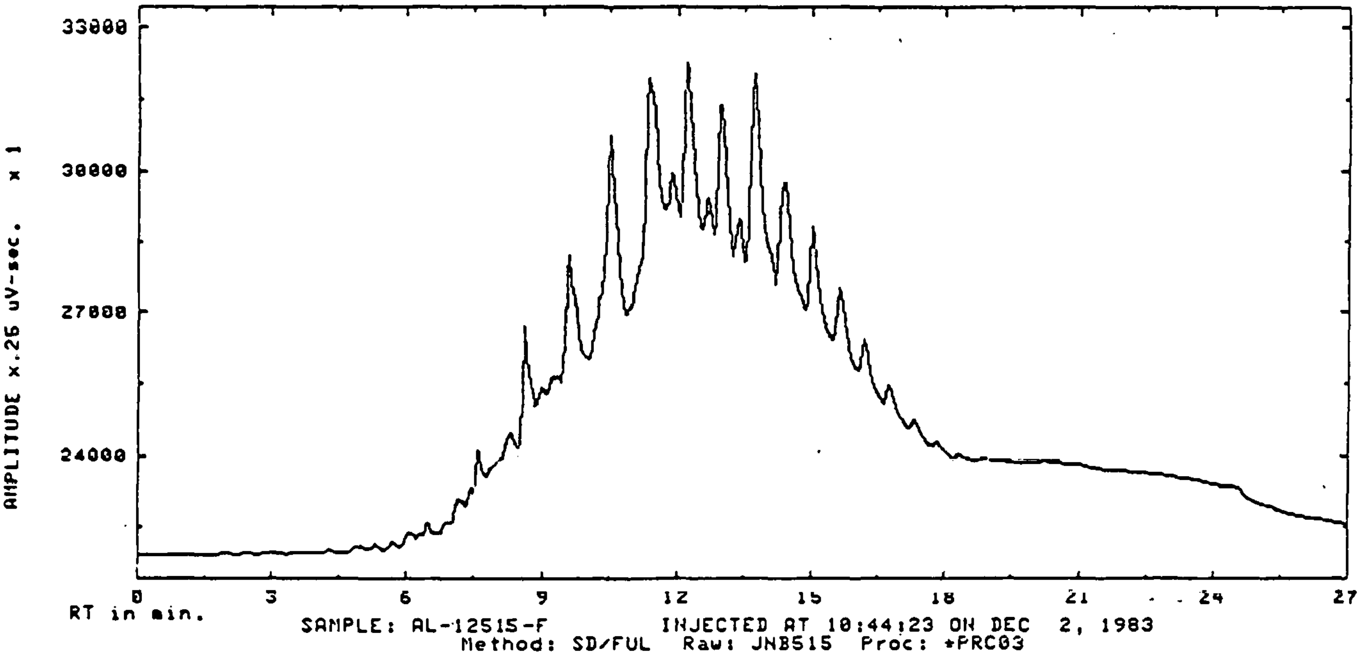

FIGURE 4. CHROMATOGRAM--"FOR SAMPLE 2
FROM NSC CHARLESTON

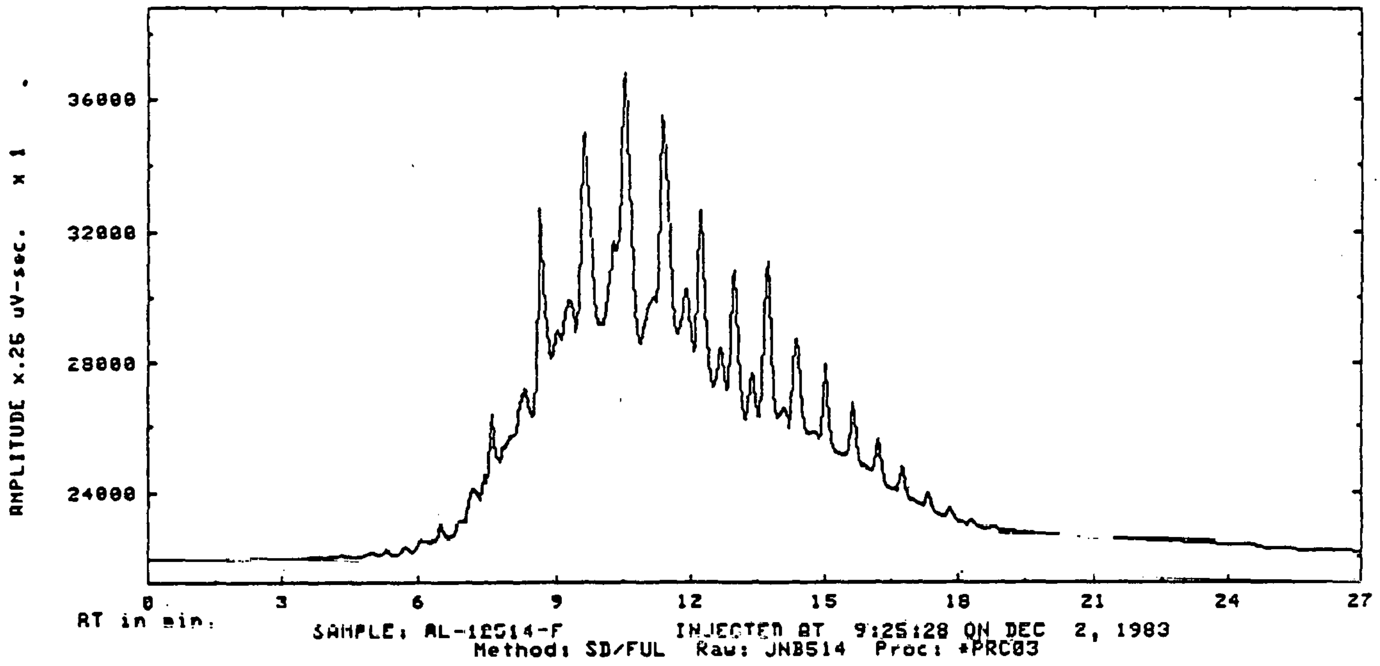

FIGURE 5. CHROMATOGRAM--"FOR" SAMPLE 2
FROM NSC NORFOLK

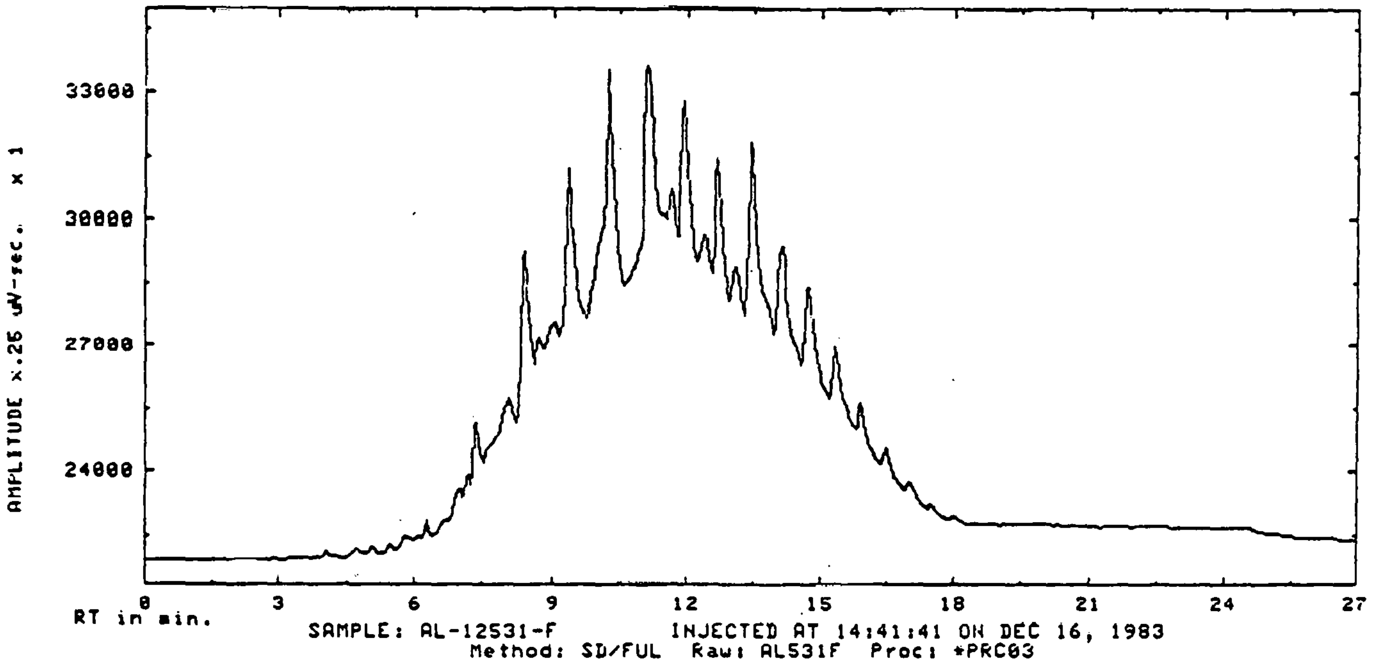

FIGURE 6. CHROMATOGRAM--"FOR" SAMPLE 2
FROM NSC JACKSONVILLE

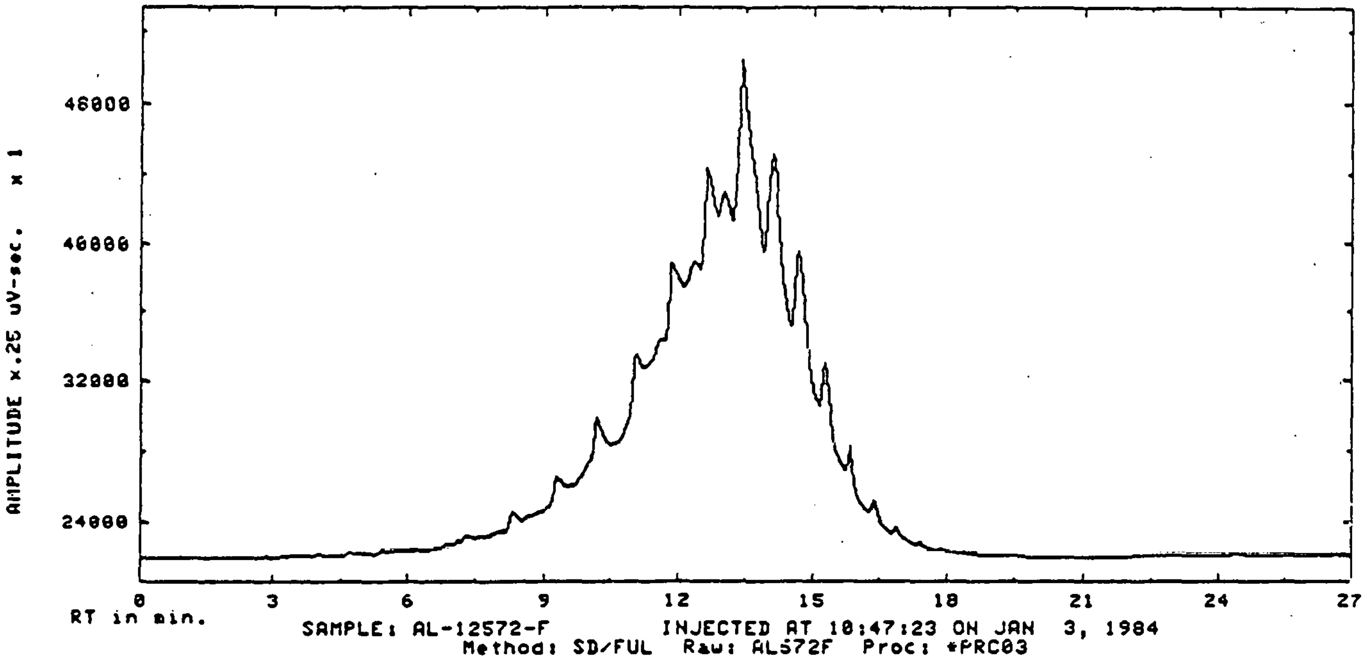

FIGURE 7. CHROMATOGRAM--"NDF" SAMPLE 2
FROM NSC SAN DIEGO

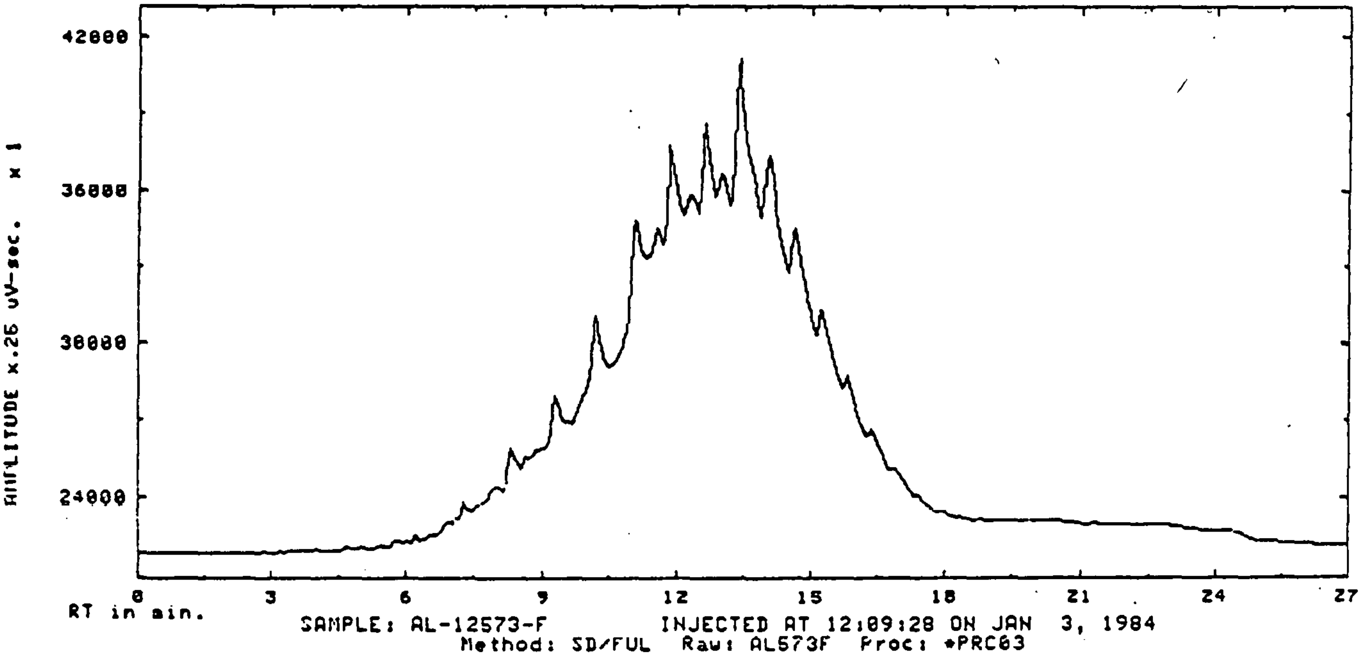

FIGURE 8. CHROMATOGRAM--"FOR" SAMPLE 3
FROM NSC SAN DIEGO

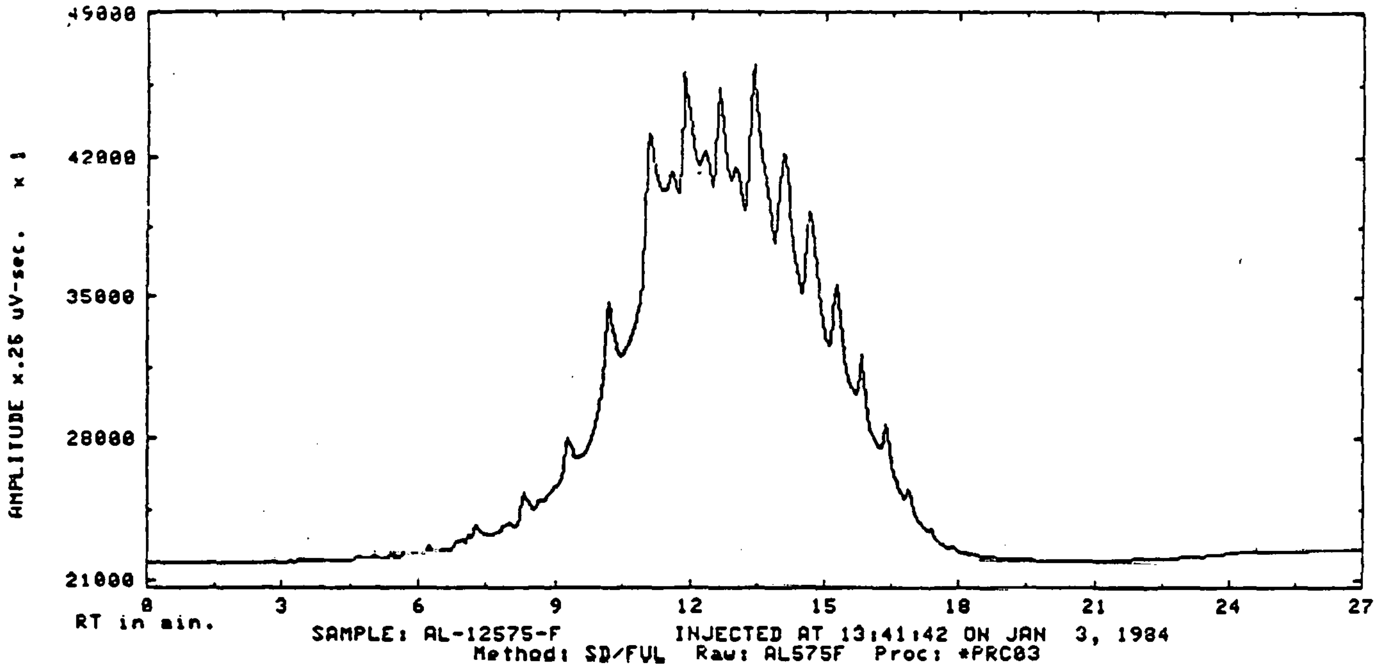

FIGURE 9. CHROMATOGRAM--"NDF" SAMPLE 2
FROM NSC PUGET SOUND

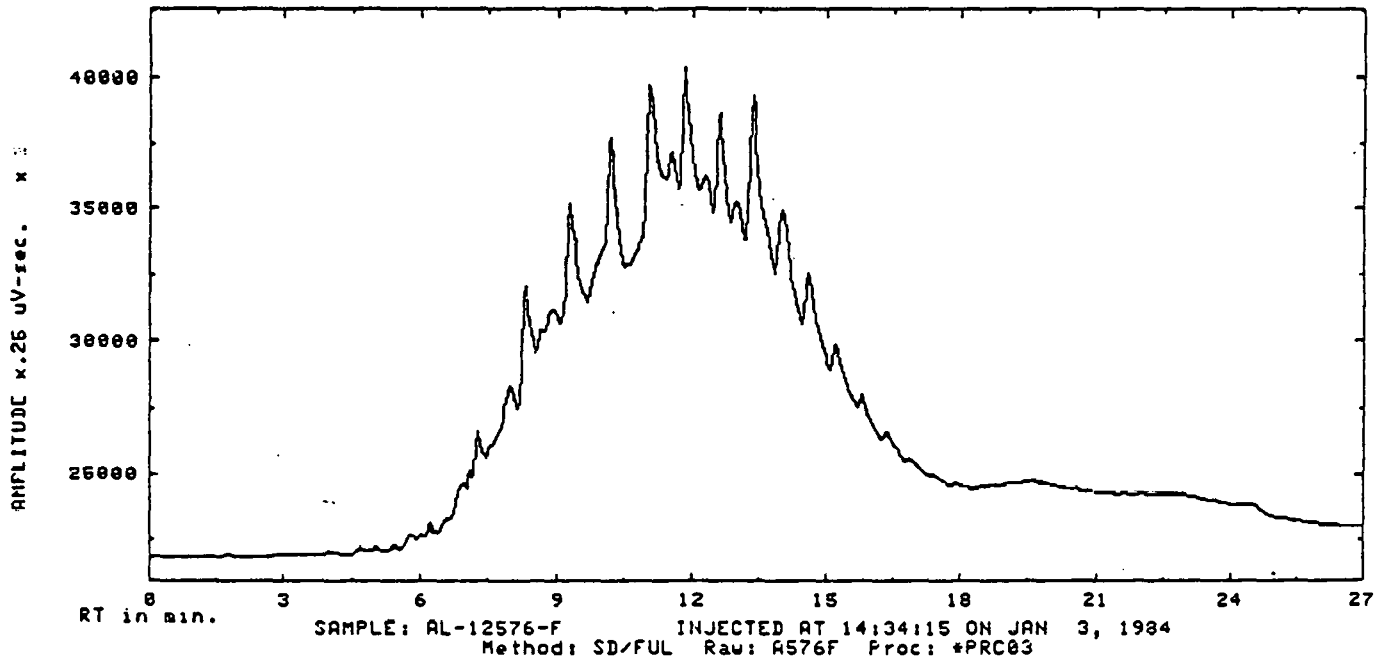

FIGURE 10. CHROMATOGRAM--"FOR" SAMPLE 3
FROM NSC PUGET SOUND

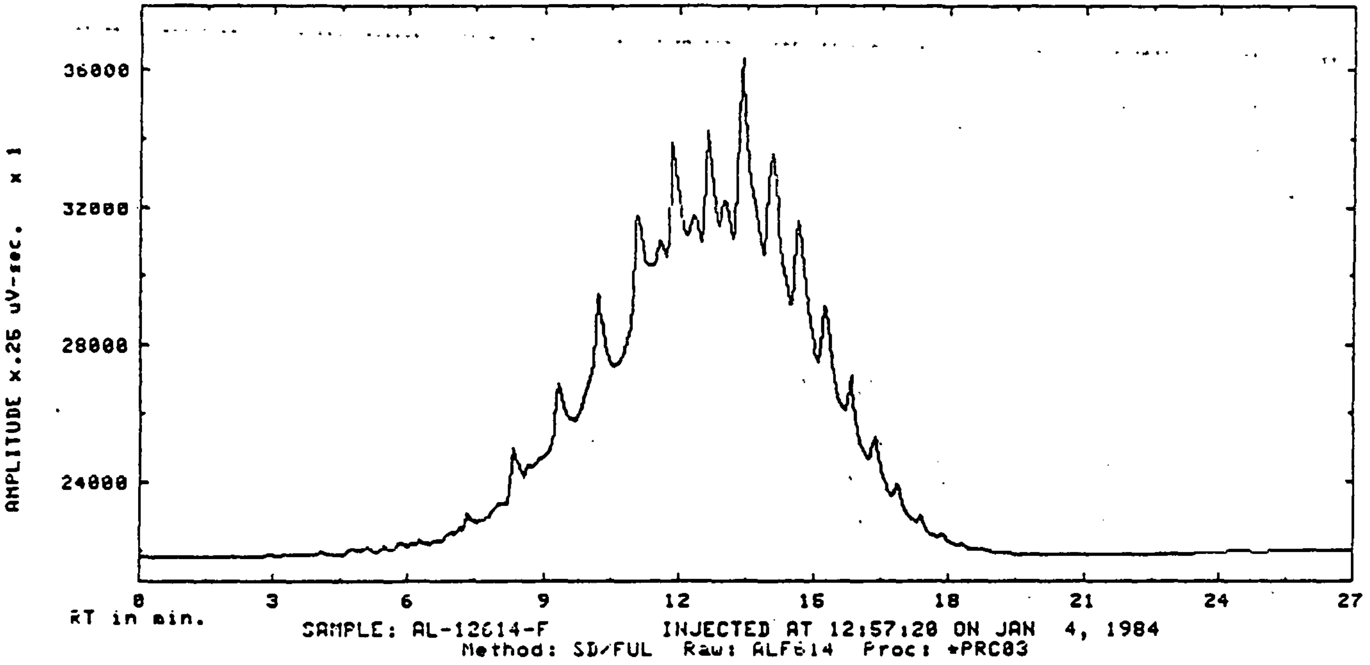

FIGURE 11. CHROMATOGRAM--"NDF" SAMPLE 2
FROM NSC OAKLAND

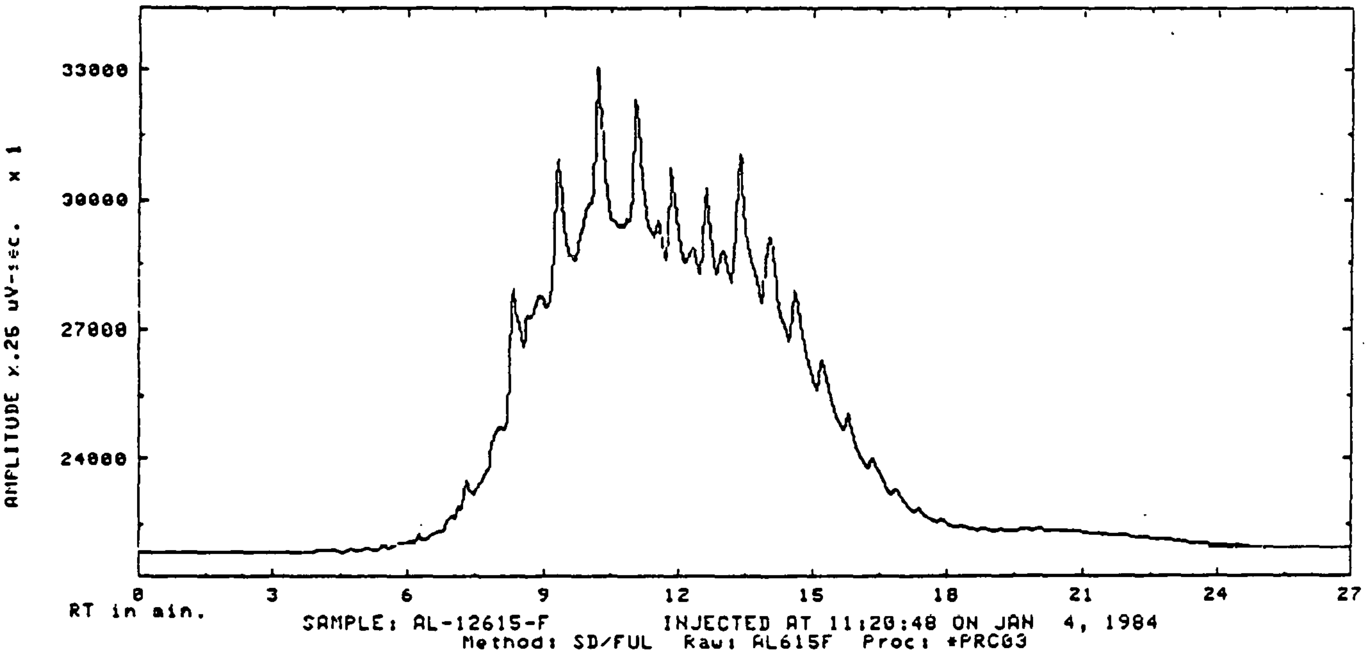

FIGURE 12. CHROMATOGRAM--"FOR" SAMPLE 3
FROM NSC OAKLAND

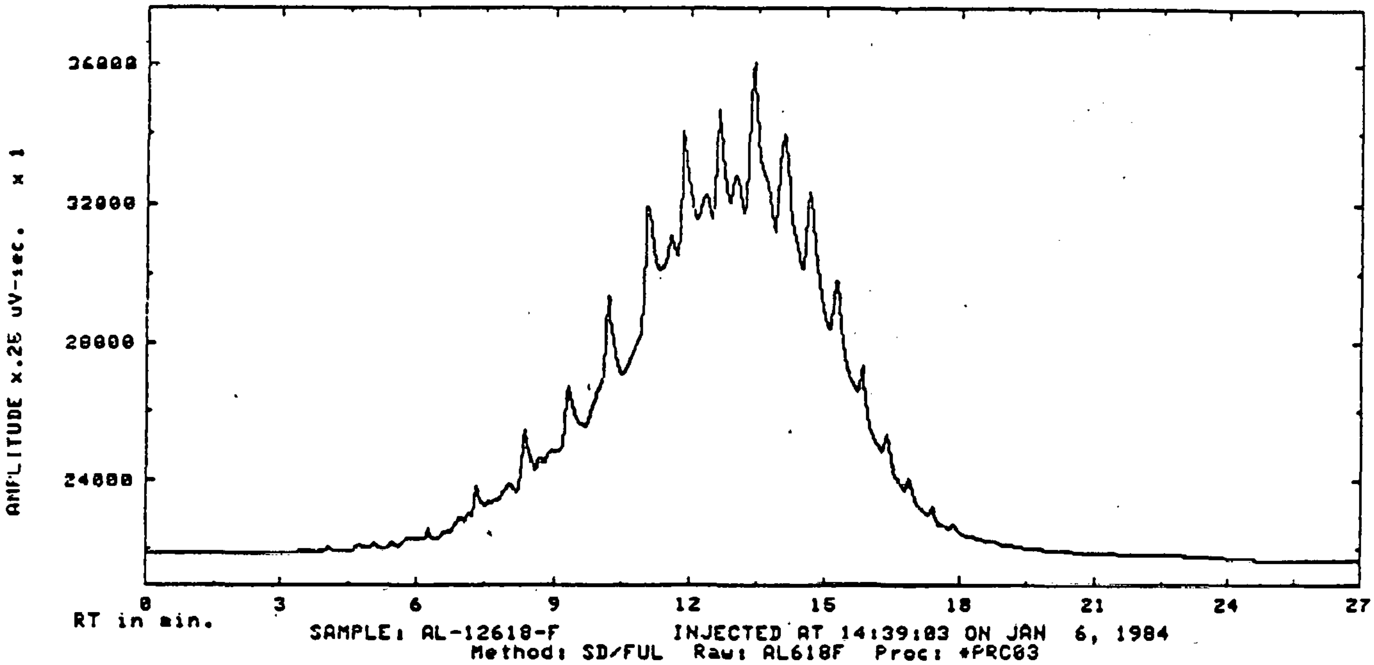

FIGURE 13. CHROMATOGRAM--"NDF" SAMPLE 2
FROM NSC CHARLESTON

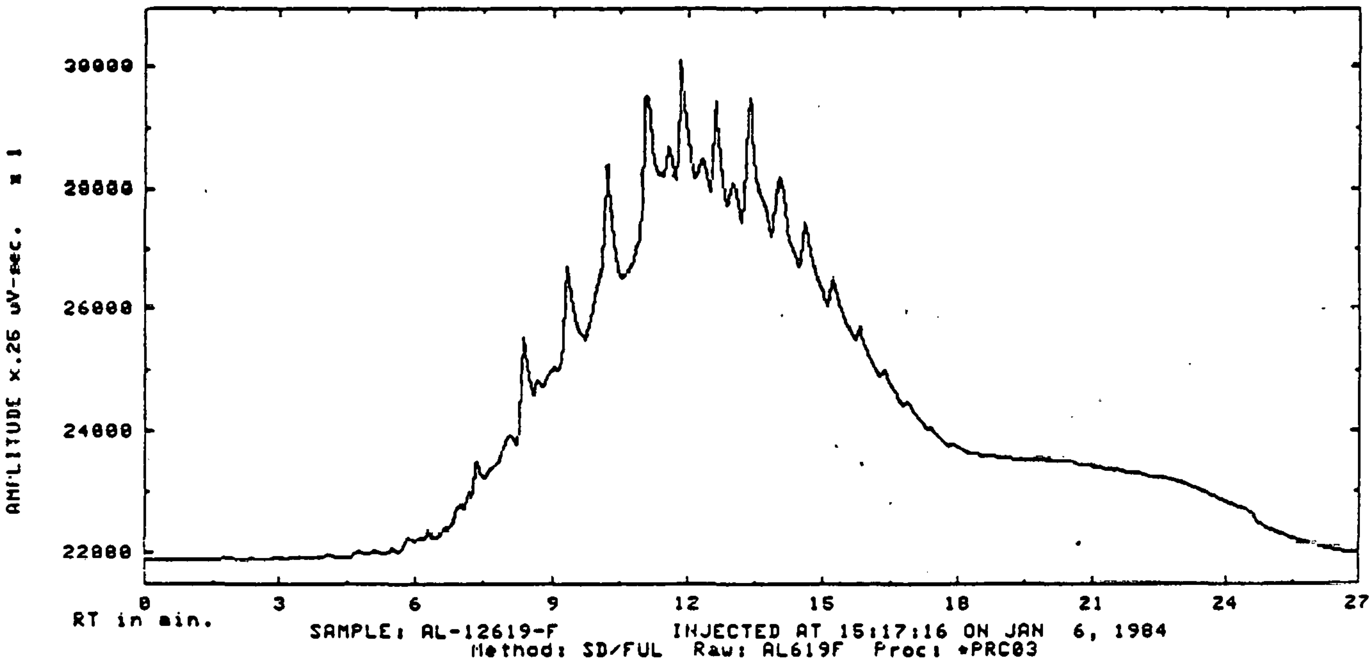

FIGURE 14. CHROMATOGRAM--"FOR" SAMPLE 3
FROM NSC CHARLESTON

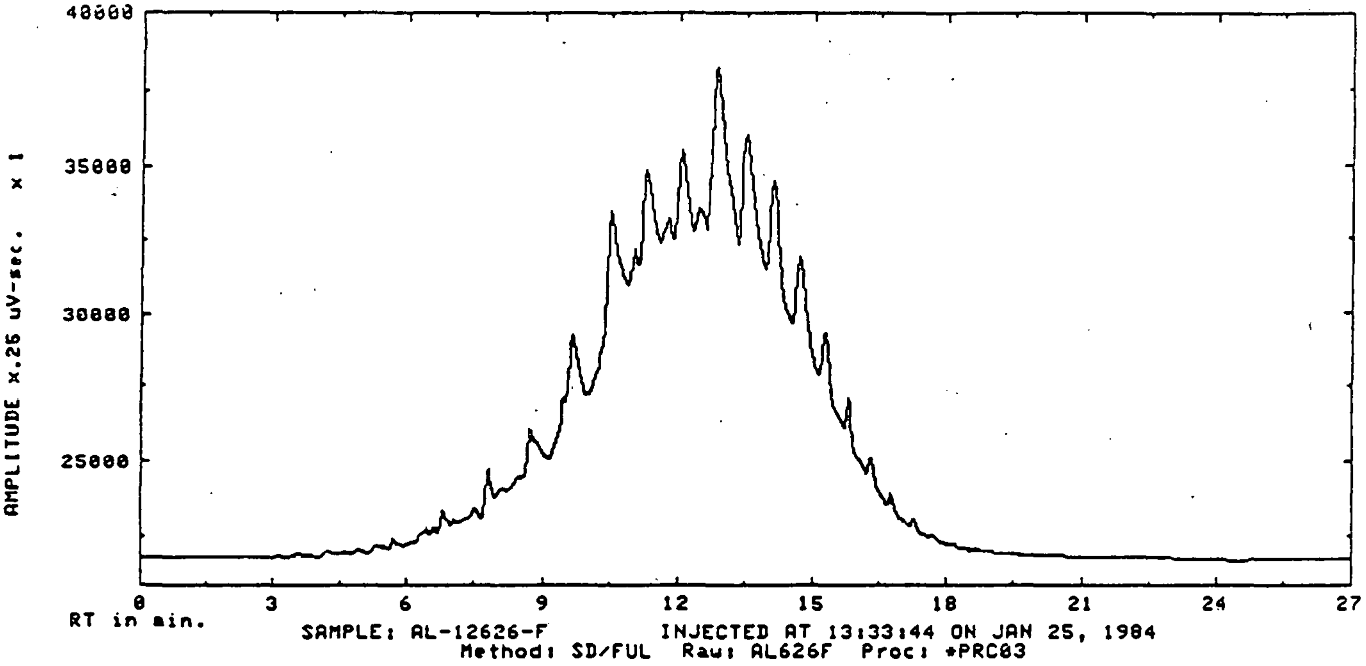

FIGURE 15. CHROMATOGRAM--"NDF" SAMPLE 2
FROM NSC NORFOLK

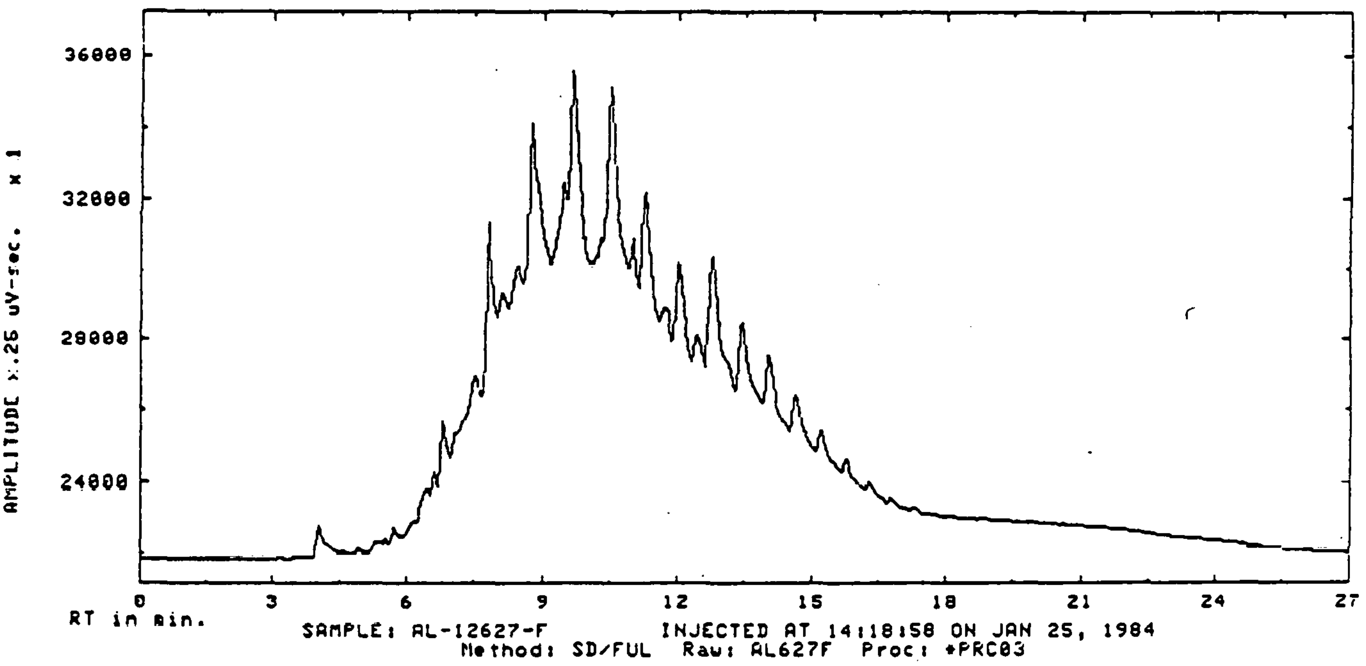

FIGURE 16. CHROMATOGRAM--"FOR" SAMPLE 3
FROM NSC NORFOLK

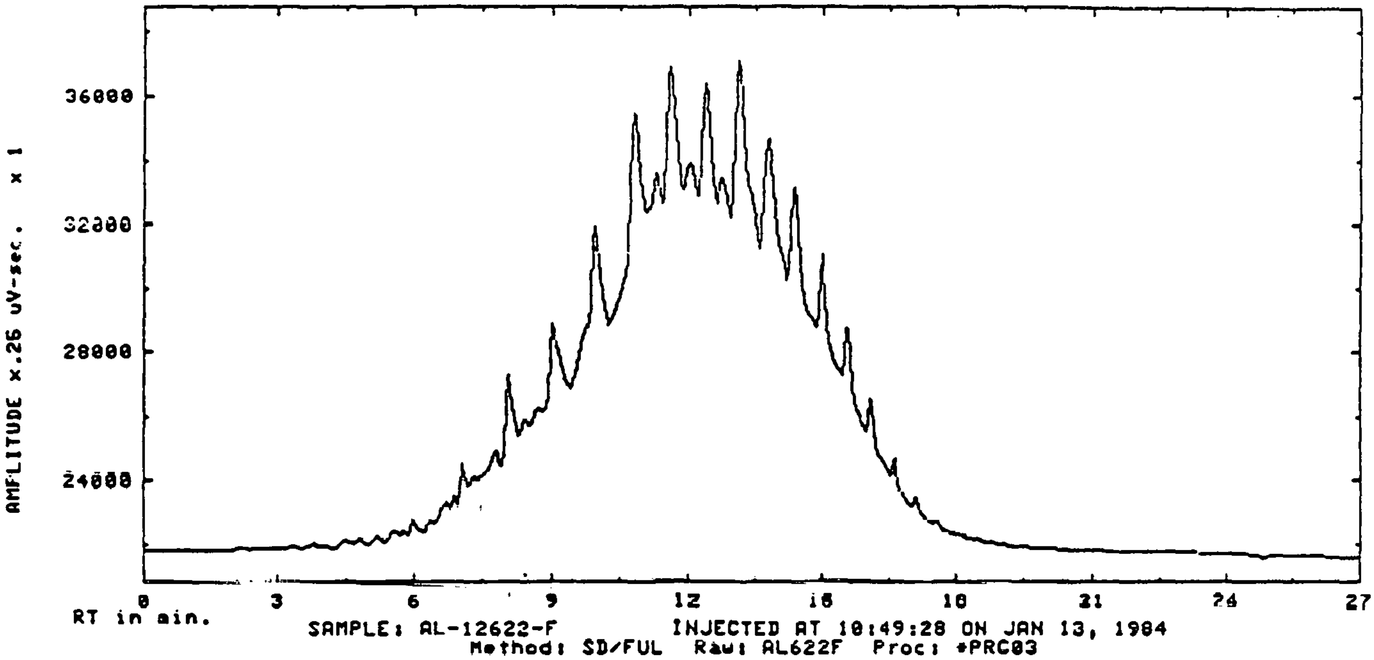

FIGURE 17. CHROMATOGRAM--"NDF" SAMPLE 2
FROM NSC JACKSONVILLE

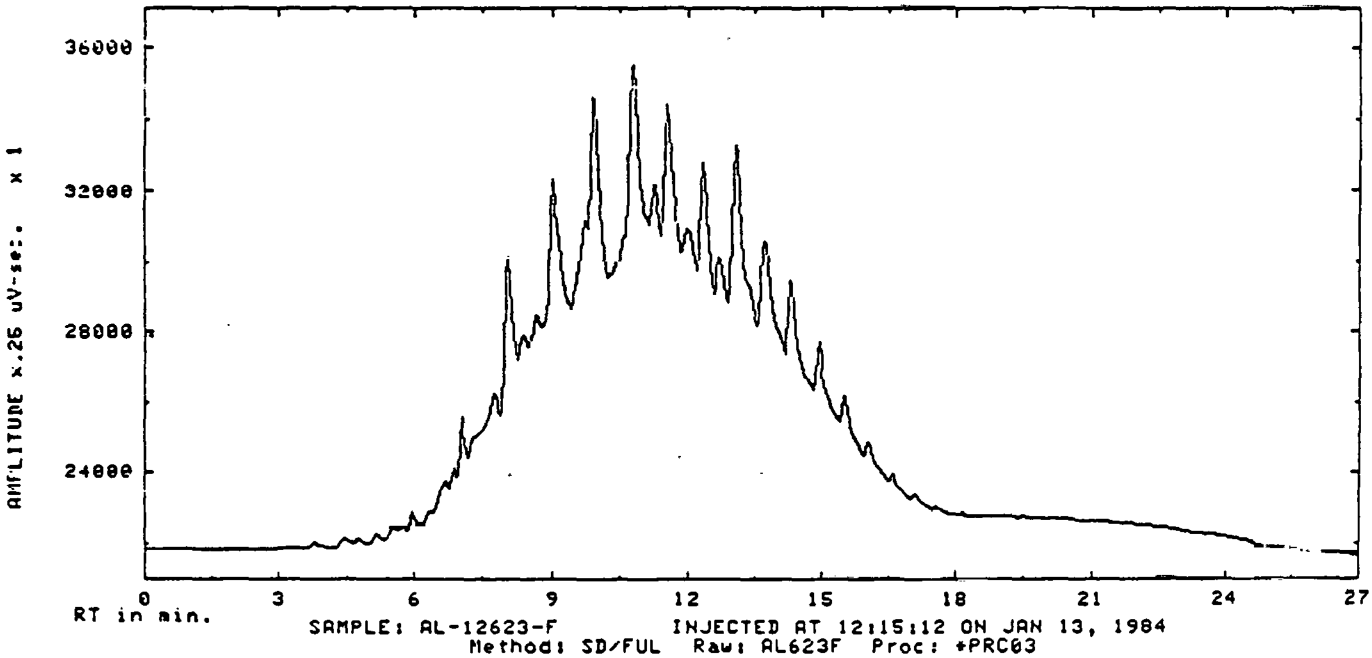

FIGURE 18. CHROMATOGRAM--"FOR" SAMPLE 3
FROM NSC JACKSONVILLE

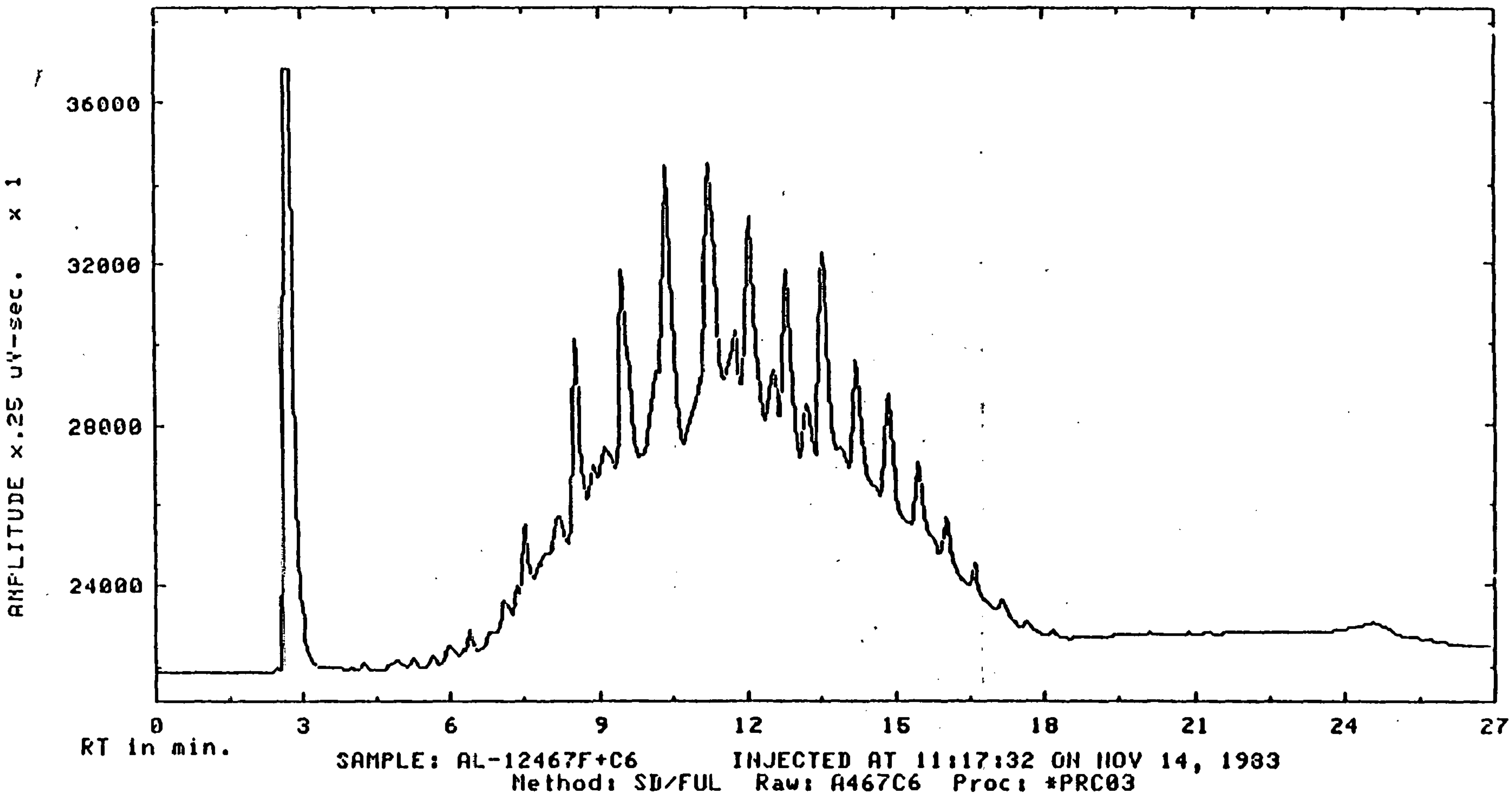

FIGURE 19. - Chromatogram - "FOR" Sample AL-12467F+C_6 From NSC Jacksonville

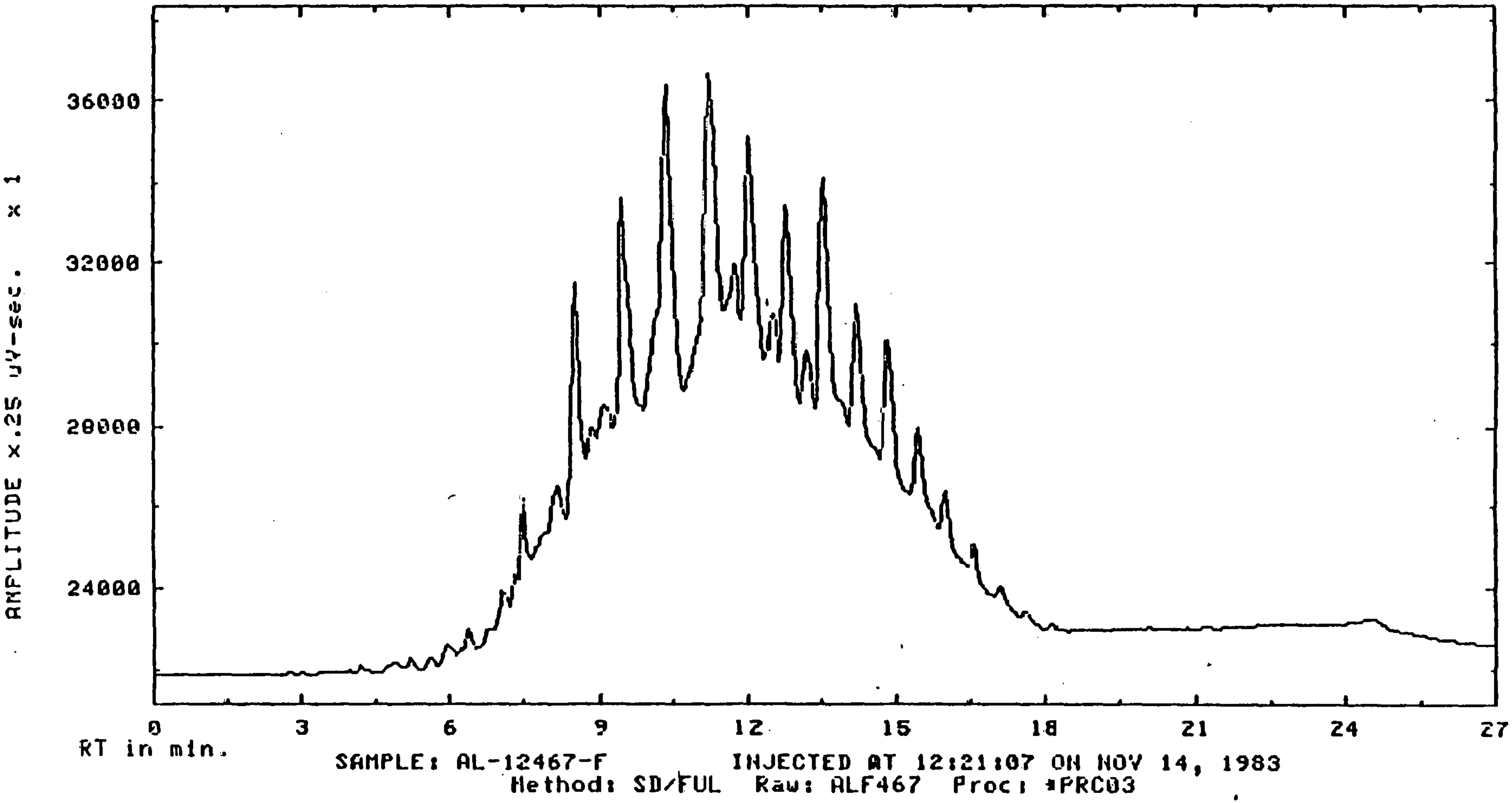

FIGURE 20. - Chromatogram - "FOR" Sample AL-12467 From NSC Jacksonville

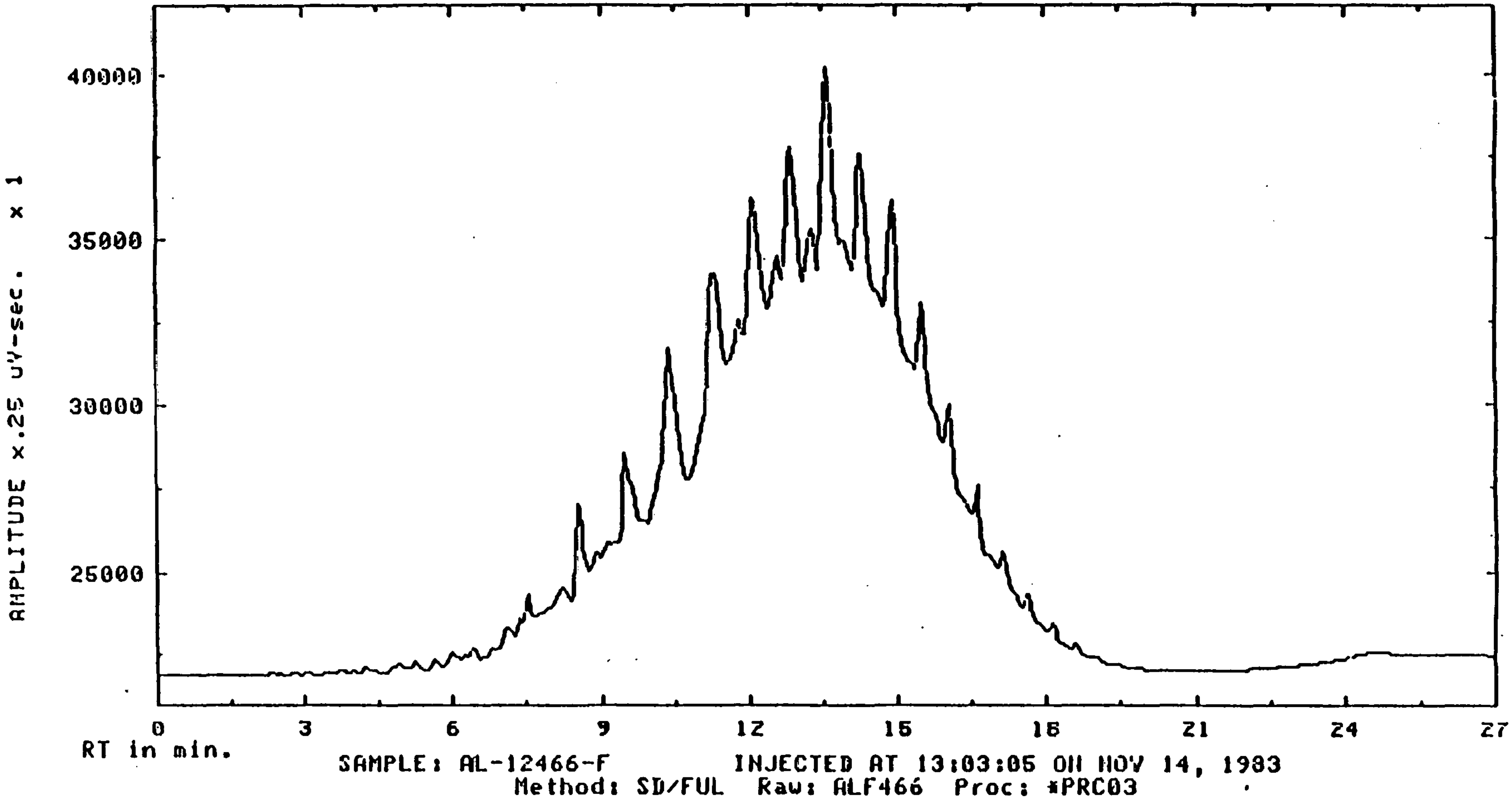

FIGURE 21. - Chromatogram = "DFM" Sample AL-12466-F From NSC Jacksonville

MIL-F-24951 (SA)
1 February 1983

TABLE I-36.

MILITARY SPECIFICATION

FUEL OIL RECLAIMED

This specification is approved for use by the Department of the Navy.

1. SCOPE

1.1 Scope. This specification covers Fuel Oil Reclaimed (Stock Number NSN 9140-01-068-6903) which is produced as a product of Navy reclamation operations (product use is described in 6.1).

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of sale by the Navy, form a part of the specification to the extent specified herein.

STANDARDS

FEDERAL
FED-STD-791 - Lubricants, Liquid Fuels, and Related Products: Methods of Testings
MILITARY
MIL-STD-290 - Packaging, Packing and Marking Petroleum and Related Products

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the date of sale by the Navy shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)
D 88 - Saybolt Viscosity
D 93 - Flash Point by Pensky-Martens Closed Tester.
D 97 - Pour Point.
D 129 - Sulfur in Petroleum Products by the Bomb Method.
D 270 - Sampling Petroleum and Petroleum Products.
D 287 - API Gravity of Crude Petroleum and Petroleum Products. (Hydrometer Method).
D 445 - Viscosity of Transparent and Opaque Liquids (Kinematic and Dynamic Viscosities).
D 473 - Sediment in Crude and Fuel Oils by Extraction
D 874 - Sulfated Ash from Lubricating Oils and Additives
D 1796 - Water and Sediment.

(The ASTM methods listed above are included in parts 23 or 24 of the Annual Book of ASTM Standards and are available individually. Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

3. REQUIREMENTS

3.1 Material. Fuel Oil Reclaimed shall consist of a mixture of distillates and residual fuel and may contain waste lubricants or other recycled products.

3.2 Physical and chemical requirements. Fuel Oil Reclaimed shall conform to the physical and chemical requirements given in Table 1. Any requests for waivers from these requirements will be addressed to Navy Petroleum Office for approval.

TABLE-1- Chemical and Physical Requirements

Characteristics	Requirements	FED-STD 791 test method	ASTM Test method
API Gravity @ 60°F (hydrometer)(range)	25-40		D 287
Viscosity at 104°F (40°C) Kinematic, centistokes (range)	2.0 - 15.0		D 445
Viscosity @ 122°F (50°) Saybolt Universal (range)	30 - 90		D 88
Flash point °F (min)	130°F (55°C)		D 93
Pour point °F (max)	20°F(-6.7°C)		D 97
Sulfated Ash, percent (max)	0.15		D 874
Water & sediment, percent (max)	2.0		D 1796
Neutrality	Neutral	5101	
Sediment percent (max)	0.5		D 473
Chlorinated Material 1/	No Green Flame		
Sulfur content, percent(max)	2.0		D 129 2/ or other approved ASTM ~~[illegible]~~ methods
Explosiveness, percent (max)	50	1151.1	

1/ FOR shall be essentially free of chlorinated material. To determine the presence of chlorinated material a clean copper wire is heated in a clear blue gas flame, to red heat, until no green shows in the flame. The wire is dipped while still hot, into a sample of FOR and then put back into the flame. No green shall show in the flame. (For practice, a blend of 1% of Trichloroethane in DFM or other distillate fuel may be used as an example of an oil which fails this test also. The oil should be purged of any sodium chloride by washing with fresh water.)

2/ In the U.S.A. sulfur limits shall be as specified by the Environmental Protection Agency, state or community where the fuel is to be used, whichever is more restrictive. In foreign countries, the sulfur limit shall conform to the limit established in the Status of Forces Agreement.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the purchase order, the Navy is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the purchase order, the Navy may use its own or any other facilities suitable for the performance of the inspection requirements specified herein.

4.2 Bulk Lot. An indefinite quantity of a homogeneous mixture of Fuel Oil Reclaimed offered for acceptance in a single isolated container. Upper, middle and lower samples will be taken as described in ASTM D270, Section 14. Samples may be composited to a single sample if the lot is homogeneous. If the lot is not homogeneous, specification tests will be performed separately on the upper, middle and lower samples.

4.3 Homogeneity. The homogeneity of product will be determined by measuring API gravity using ASTM test method D287. Lots will be considered homogeneous if the determinations for the upper, middle and lower samples do not vary from the average by more than 0.5° for API gravity.

4.4 Sampling. Take samples for tests in accordance with ASTM method D270.

4.5 Inspection. Perform inspection in accordance with 9601 of FED-STD-791.

4.6 Classification of tests. All tests are quality conformance tests.

4.7 Test methods. Perform tests in accordance with Table 1.

5. PREPARATION FOR DELIVERY

5.1 Packaging, packing and marking. Unless otherwise specified in the purchase order packaging, packing and marketing shall be performed in accordance with MIL-STD-290.

6. NOTES

6.1 Intended use. Fuel Oil Reclaimed can be used as a substitute for Burner Fuel Oil (FED-SPEC VV-F-815D), either directly or as a blend in stationary fuel-burning furnaces for heating buildings, for the generation of steam or for other purposes.

6.2 Navy Responsibilities. The Navy is responsible for assuring that the Fuel Oil Reclaimed meets all the requirements listed in Table 1.

6.3 User responsibilities. The user is responsible for any onsite blending and all operational or equipment modifications necessary to assure that the Fuel Oil Reclaimed is burned in a safe and efficient manner.

Preparing activity:
Navy - SA

Project No. 9140-N105

Custodian:
Navy - SA
Review activities:
To be determined
User activities:
To be determined

F-76
MIL-F-16884H
3 May 1983
SUPERSEDING
MIL-F-16884G
7 March 1973
(See 6.4)

TABLE I-37. MILITARY SPECIFICATION

FUEL, NAVAL DISTILLATE

This specification is approved for use by all Departments and Agencies of the Department of the Defense.

1. SCOPE

1.1 Scope. This specification covers one grade of Naval distillate fuel (NATO symbol F-76).

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Standards. Unless otherwise specified, the following standards of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

STANDARDS

FEDERAL
FED-STD-791 - Lubricants, Liquid Fuels, and Related Products; Method of Testing.

MILITARY
MIL-STD-105 - Sampling Procedures and Tables For Inspection by Attributes.
MIL-STD-290 - Packaging of Petroleum and Related Products.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Sea Systems Command, SEA 5523, Department of the Navy, Washington, DC 20362 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 9140

(Copies of standards required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

D 86 - Distillation of Petroleum Products, Method For. (DoD adopted)
D 93 - Flash Point by Pensky-Martens Closed Tester, Test Method For. (DoD adopted)
D 97 - Pour Point of Petroleum Oils, Test Method For. (DoD adopted)
D 129 - Sulfur in Petroleum Products (General Bomb Method), Test Method For. (DoD adopted)
D 130 - Detection of Copper Corrosion from Petroleum Products by the Copper Strip Tarnish Test, Method For. (DoD adopted)
D 270 - Standard Method of Sampling Petroleum and Petroleum Products. (DoD adopted)
D 287 - API Gravity of Crude Petroleum and Petroleum Products (Hydrometer Method), Test Method For. (DoD adopted)
D 445 - Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity), Test Method For. (DoD adopted)
D 482 - Ash from Petroleum Products, Test Method For. (DoD adopted)
D 524 - Ramsbottom Carbon Residue of Petroleum Products, Test Method For. (DoD adopted)
D 611 - Aniline Point and Mixed Aniline Point of Petroleum Products and Hydrocarbon Solvents, Test Method For. (DoD adopted)
D 613 - Ignition Quality of Diesel Fuels by the Cetane Method, Test Method For.
D 665 - Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water, Test Method For. (DoD adopted)
D 974 - Neutralization Number by Color-Indicator Titration, Test Method For. (DoD adopted)
D 976 - Calculated Cetane Index of Distillate Fuels, Test Method For.
D 1298 - Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method, Test Method For. (DoD adopted)
D 1401 - Emulsion Characteristics of Petroleum Oils and Synthetic Fluids, Test Method For.
D 1500 - ASTM Color of Petroleum Products (ASTM Color Scale), Test Method For. (DoD adopted)
D 1552 - Sulfur in Petroleum Products (High-Temperature Method), Test Method For. (DoD adopted)
D 2274 - Oxidation Stability of Distillate Fuel Oil (Accelerated Method), Test Method For.
D 2500 - Cloud Point of Petroleum Oils, Test Method For. (DoD adopted)
D 2622 - Sulfur in Petroleum Products (X-Ray Spectrographic Method), Test Method For. (DoD adopted)

D 2709 - Water and Sediment in Distillate Fuels by Centrifuge, Test Method For. (DoD adopted)
E 29 - Recommended Practice for Indicating Which Places of Figures Are To Be Considered Significant in Specified Limiting Values. (DoD adopted)

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups using Federal agencies.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 General. Requirements contained herein are not subject to corrections for tolerance of test methods. If multiple determinations are made by the inspecting laboratory, average results will be used except for those test methods where repeatability data are given. In those cases, the average value derived from the individual results that agree within the repeatability limits given may be used at the discretion of the inspection authority, provided an indication is given of the total number or results obtained and the number falling outside the repeatability limits. For purposes of determining conformance with each requirement, an observed value or calculated value shall be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off procedure given in ASTM E 29.

3.2 Material. The fuel supplied under this specification shall be distillate fuel and may contain only those additives specified in 3.2.1 through 3.2.4.

3.2.1 Additives. The additives listed herein may be used singly or in combination in amounts not to exceed those specified.

3.2.2 Antioxidants. The following active inhibitors may be blended separately or in combination into the fuel in total concentration not in excess of 8.4 pounds of inhibitor (not including weight of solvent) per 1,000 barrels of fuel (9.1 grams/100 gallons (U.S.), 24 milligrams (mg)/liter or 109 mg/gallons (U.K.)) in order to prevent the formation of gum:

(a) N,N' - diisopropyl-para-phenylenediamine
(b) N,N' - disecondary butyl-para-phenylenediamine
(c) 2,6 - ditertiary butyl-4-methylphenol
(d) 2,4 - dimethyl-6-tertiary butylphenol
(e) 2,6 - ditertiary butylphenol
(f) 75 percent minimum 2,6-ditertiary butylphenol
25 percent maximum tertiary and tritertiary butylphenols

TABLE I-37. - MIL-F-16884H

3.2.3 Metal deactivator. A metal deactivator, N, N' - disalicyclidene-1, 2 propanediamine may be blended into the fuel in an amount not to exceed 2 pounds of active ingredient per 1,000 barrels of fuel (2.2 grams/100 gallons (U.S.), 5.8 mg/liter or 25 mg/gallons (U.K.)).

3.2.4 Ignition improver. The following additives, to raise the ignition quality of the fuel, may be used as required to conform to this specification:

Amyl nitrate (mixed primary nitrates).
Hexyl nitrate (N-hexyl nitrate).
Cyclohexyl nitrate.
Octyl nitrate.

3.3 Chemical and physical requirements. The diesel fuel shall conform to the physical and chemical requirements specified in table I.

TABLE I. Chemical and physical requirements.

Characteristics	Requirements	FED-STD-791 test method	ASTM test method
Ignition quality, cetane number (min) (see 4.5.1)	45		D 613
Appearance at 21°C (70°F) or ambient temperature whichever is higher	Clear, bright, and free from visible particulate matter1/		
Distillation:			
50 percent point, °C (°F)	Record		
90 percent point, °C (°F) (max)	357°C (675°F)		D 86
End point, °C (°F) (max)2/	385°C (725°F)		
Residue plus loss, percent (max)	3.0		
Flash point, °C (°F) (min)	60°C (140°F)		D 93
Pour point, °C (°F) (max)	-6°C (20°F)5/		D 97
Cloud point, °C (°F) (max)	-1°C (30°F)5/		D 2500
Viscosity at 40°C (104°F) Kinematic, centistokes	1.7 - 4.3		D 445
Carbon residue, on 10 percent bottoms, percent (max) (see 4.5.2)	0.20		D 524
Sulfur, percent (max)	1.00		3/D 129
Corrosion (max) at 100°C (212°F)	No. 1 ASTM		D 130
Color (max)	3		D 1500
Ash, percent (max)	0.005		D 482

See footnotes at end of table.

TABLE I. Chemical and physical requirements. - Continued

Characteristics	Requirements	FED-STD-791 test method	ASTM test method
Gravity (hydrometer)	Record		4/D 1298
Demulsification at 25°C (77°F), minutes (max) (see 4.5.3)	10		D 1401
Acid number (max)	0.30		D 974
Neutrality	Neutral	5101	-------
Aniline point, °C (°F)	Record		D 611
Accelerated stability, total insolubles mg/100 mL (max)	6/1.5		D 2274

1/ A slight haze is acceptable providing a maximum (max) water and sediment of 0.01 percent is obtained using procedure ASTM D 2709.

2/ As the end point of the distillation is approached, if either a thermometer reading 385°C (725°F) or a decomposition point is observed, discontinue the heating and resume the procedure as directed in ASTM D 86.

3/ ASTM D 1552 and ASTM D 2622 may be used as alternative methods.

4/ ASTM D 287 may be used as an alternative method.

5/ The ASTM methods for pour and cloud points permit optional use of either Celsius or Fahrenheit procedures; therefore requirements are specified for either option.

6/ Average of three determinations is acceptable.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specfied in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to the prescribed requirements.

4.2 Lot.

4.2.1 Bulk lot. Bulk lot shall be considered an indefinite quantity of a homogenous mixture of material offered for acceptance in a single isolated container.

4.2.2 Packaged lot. Packaged lot shall be considered an indefinite number of 55-gallon drums or smaller unit containers of identical size and type, offered for acceptance, and filled with a homogenous mixture of material from one isolated container; or filled with a homogenous mixture of material manufactured in a single plant run (not exceeding 24 hours) through the same processing equipment, with no change in ingredient material.

4.3 Sampling.

4.3.1 Sampling for examination of the preparation for delivery. A random sample of packed containers shall be taken from each lot in accordance with MIL-STD-105, at inspection level II, and acceptable quality level (AQL) equals 2.5 percent defective. The sample shall be examined in accordance with 4.4.1.

4.3.2 Sampling for tests. Samples for tests shall be taken in accordance with ASTM D 270. Samples shall be tested in accordance with table I and 4.5.

4.4 Inspection. Inspection shall be performed in accordance with method 9601 of FED-STD-791.

4.4.1 Examination of the preparation for delivery. Samples taken in accordance with 4.3.1 shall be examined for compliance with MIL-STD-290 with regard to fill, closure, sealing, leakage, packaging, packing, and marking requirements. Any container having one or more defects, or under the required fill shall be rejected. If the number of defective or underfilled containers exceeds the acceptance number for the appropriate plan of MIL-STD-105, the lot represented by the sample shall be rejected.

4.5 Test methods.

4.5.1 Ignition quality. When the apparatus specified in ASTM D 613 is not available for product inspection purposes, the cetane index may be authorized in lieu of the cetane number, provided that sufficient data are available to establish the cetane index number correlation for a finished product or a blend of products from the same manufacturing process or processes and the same specific crude source. In all instances, the product submitted shall be of sufficiently high cetane index to assure a cetane number at least as high as that shown in table I. In no case shall the cetane index be less than 45. The calculated cetane index shall not be used in determining the ignition quality of fuel containing ignition improvers. The cetane index shall be determined by ASTM D 976 and the corresponding cetane number from the manufacturers correlation data shall be reported.

4.5.2 Carbon residue. When the finished fuel contains a cetane improver the carbon residue requirement specified in table I shall apply to the base fuel without the cetane improver.

4.5.3 Demulsification. The demulsification test shall be conducted in accordance with ASTM D 1401 with the following exceptions:

(a) Synthetic sea water prepared in accordance with ASTM D 665 shall be used as the emulsifying fluid.
(b) The test temperature shall be 25° ± 1.1°C (77° ± 2°F).
(c) The demulsification time shall be that required for separation into two layers with no cuff at the interface. A lacy emulsion which does not form a band or cuff on the wall of the cylinder shall be disregarded. The fuel, water, and emulsion layer volumes shall be recorded at 1 minute intervals and the demulsification time reported shall be to the nearest minute.

4.6 Test reports. The contractor shall prepare test reports in accordance with the data ordering document (see 6.2.2).

4.7 Inspection of preparation for delivery. The packaging, packing, and marking shall be inspected for compliance with section 5 of this specification.

5. PACKAGING

(The preparation for delivery requirements specified herein apply only for direct Government acquisition.)

5.1 Packaging, packing, and marking. Packaging, packing, and marking shall be in accordance with MIL-STD-290. The level of packaging, level of packing, type, and size shall be as specified (see 6.2.1).

6. NOTES

6.1 Intended use. Naval distillate fuel is intended for use in all shipboard boilers, gas turbines, and diesel engines at ambient temperatures above -1.1°C (30°F). Other uses may be specified according to the needs of the Department of Defense. When gas turbines and diesel engines are exposed to ambient temperatures that consistently fall below -1.1°C (30°F), JP-5 per MIL-T-5624 should be used instead of naval distillate.

6.2 Ordering data.

6.2.1 Acquisition requirements. Acquisition documents should specify the following:

(a) Title, number, and date of this specification.
(b) Applicable level of packaging and packing required (see 5.1).
(c) Unit container quantity (see 5.1).

6.2.2 Data requirements. When this specification is used in an acquisition which incorporates a DD Form 1423, Contract Data Requirements List (CDRL), the data requirements identified below shall be developed as specified by an approved Data Item Description (DD Form 1664) and delivered in accordance with the approved CDRL incorporated into the contract. When the provisions of DAR 7-104.9 (n)(2) are invoked and the DD Form 1423 is not used, the data specified below shall be delivered by the contractor in accordance with the contract or purchase order requirements. Deliverable data required by this specification is cited in the following paragraphs.

Paragraph no.	Data requirement title	Applicable DID no.	Option
4.6	Reports, test	DI-T-2072	10.1.b

(Data item descriptions related to this specification, and identified in section 6 will be approved and listed as such in DoD 5000.19L., Vol. II, AMSDL. Copies of data item descriptions required by the contractors in connection with specific acquisition functions should be obtained from the Naval Publications and Forms Center or as directed by the contracting officer.)

6.2.2.1 The data requirements of 6.2.2 and any task in sections 3, 4, or 5 of this specification required to be performed to meet a data requirement may be waived by the contracting/acquisition activity upon certification by the offeror that identical data were submitted by the offeror and accepted by the Government under a previous contract for identical item acquired to this specification. This does not apply to specific data which may be required for each contract regardless of whether an identical item has been supplied previously (for example, test reports).

6.3 International interest. Certain provisions of this specification are the subject of international standardization agreement NATO STANAG-1135. When amendment, revision, or cancellation of this specification is proposed which will modify the international agreement concerned, the preparing activity will take appropriate reconciliation action through international standardization channels including departmental standardization offices to change the agreement or make other appropriate accommodations.

6.4. Changes from previous issue. Asterisks are not used in this revision to identify changes with respect to the previous issue, due to the extensiveness of the changes.

Custodians:
  Army - ME
  Navy - SH
  Air Force - 68
Review activities:
  Army - ME
  Navy - YD, SA
  DLA - PS, GS
User activities:
  Navy - MC, CG

Preparing activity:
  Navy - SH
  (Project 9140-0103)

☆U.S. GOVERNMENT PRINTING OFFICE: 1983-605-034/2588

LABORATORY ANALYSIS REPORT
NSC 4020/1 (Rev. 2-82)

TABLE I-38

DEPARTMENT OF THE NAVY
NAVAL SUPPLY CENTER, PUGET SOUND
Bremerton, Washington 98314

DATE 11 FEB 1983

FROM COMMANDING OFFICER (Code 700)	REPORT NO. 83-2-18
TO	

The following laboratory analysis of the sample(s) identified below is submitted.

SOURCE OF SAMPLE Naval Supply Center, Puget Sound, Manchester Fuel Department, Manchester, WA 98353		DATE SAMPLED 11 Feb 1983
VESSEL OR TANK SAMPLED SHORE TANK #F-14	NO. OF SAMPLES COMPOSITE	DATE SAMPLE RECEIVED 11 FEB 1983
CONTRACTOR/MANUFACTURE	CONTRACT NO.	CONTRACT DATE
TYPE OF FUEL FUEL OIL, RECLAIMED	GRADE OR CLASS FOR	SPECIFICATION NO. MIL-SPEC-FOR

TESTS	REQUIREMENTS	RESULTS	
		LAB #	
		SAMPLE #	
Appearance			
Color			
Gravity A.P.I. @ 60°F	25 - 40	34.7	
Viscosity @ 122 °F SSU	30 - 90	37.1	
Flashpoint °F MIN	130	172	
POUR POINT °F MAX	20	-5	
Freeze Point °F			
Copper strip corrosion ASTM max			
Existent Gum Mg/100Ml max			
Reid Vapor Pressure 100 °F P.S.I.			
Distillation initial boiling point °F			
Fuel evaporated, 10% min @ °F			
Fuel evaporated, 20% min @ °F			
Fuel evaporated, 40% min @ °F			
Fuel evaporated, 50% min @ °F			
Fuel evaporated, 90% min @ °F			
End point, max, °F			
Sum of 10% and 50% temp. min @			
% evaporated at 400 °F			
Neutrality	NEUTRAL	NEUTRAL	
Ash % max	REPORT	0.28	
B.S. & W. % max	2.0	0.8	
Water by distillation max			
Sediment by extraction max	0.5	0.02	
Carbon residue conradson max			
Ignition quality, cetane index No.			
Tetraethyllead content, max Ml/gal			
Chlorinated Material	No Green Flame	PASS	
Demulsification minutes	REPORT	30+	

IN THE ABOVE RESPECTS SAMPLE (COMPLIES/DOES NOT COMPLY) WITH APPLICABLE SPECIFICATION.

ANALYSIS ACCOMPLISHED BY
WILSON L. MEBANE Wilson L Mebane 3/30/83
INSPECTOR

BY DIRECTION

Enclosure (2)

LABORATORY ANALYSIS REPORT
NSC 4020/1 (Rev. 2-82)

TABLE I-38. Continued

DEPARTMENT OF THE NAVY
NAVAL SUPPLY CENTER, PUGET SOUND
Bremerton, Washington 98314

DATE 27 APR 1983

FROM COMMANDING OFFICER (Code 700)	REPORT NO. 83-4-66
TO Typical/Average	

The following laboratory analysis of the sample(s) identified below is submitted.

SOURCE OF SAMPLE Naval Supply Center, Puget Sound Manchester Fuel Dept., Manchester, Washington 98353		DATE SAMPLED 26 Apr 1983
VESSEL OR TANK SAMPLED Shore Tank #D-39	NO. OF SAMPLES COMPOSITE	DATE SAMPLE RECEIVED 26 Apr 1983
CONTRACTOR/MANUFACTURE	CONTRACT NO.	CONTRACT DATE
TYPE OF FUEL FUEL OIL, DIESEL, MARINE	GRADE OR CLASS MARINE	SPECIFICATION NO. MIL-F-16884G

TESTS	REQUIREMENTS	RESULTS LAB # SAMPLE #	
Appearance	CLEAR & BRIGHT	C&B	
Color	3.0 MAX (6)*	L2.5	
Gravity A.P.I. @ 60°F	RECORD	33.6	
Viscosity Kinematic, cs@100°F	1.8 - 4.5	3.50	
Flashpoint °F PMCC	140°F MIN	180	
Cloud Point °F	30°F MAX	14	
Pour Point °F	20°F MAX	5	
Freeze Point °F			
Copper strip corrosion ASTM max 212°F, 3 Hrs.	No. 1b MAX	1a	
Demulsification, minutes max	10 MAX	2.0	
Acid Number, max	0.30 MAX	0.15	
Distillation initial boiling point °F		400	
Fuel evaporated, 10% min @ °F			
Fuel evaporated, 20% min @ °F			
Fuel evaporated, 40% min @ °F			
Fuel evaporated, 50% min @ °F	RECORD	520	
Fuel evaporated, 90% min @ °F	675°F MAX	620	
End point, max, °F	725°F MAX (730)*	668	
Sum of 10% and 50% temp. min @			
% evaporated at 400 °F			
Residue + Loss, % max	3.0 MAX	2.0	
Neutrality	NEUTRAL	NEUTRAL	
Ash % max	0.005 MAX (0.01)*	0.0002	
B.S. & W. % max	0.01 MAX	TRACE	
Water by distillation max			
Sediment by extraction max			
Carbon residue conradson max, 10% Bottoms	0.20 MAX	0.06	
Ignition quality, cetane index No., calculated	45 MIN	47	
Tetraethyllead content, max Ml/gal			
Gravity, Specific, 60/60°F		0.8571	
	* USE LIMIT		

IN THE ABOVE RESPECTS SAMPLE (COMPLIES/DOES NOT COMPLY) WITH APPLICABLE SPECIFICATION.

ANALYSIS ACCOMPLISHED BY
WILSON L. MEBANE Wilson L. Mebane 5/4/83 INSPECTOR

BY DIRECTION

Enclosure (2)

TABLE I-38. Continued

LABORATORY ANALYSIS REPORT
NSC 4020/1 (Rev. 2-82)

DEPARTMENT OF THE NAVY
NAVAL SUPPLY CENTER, PUGET SOUND
Bremerton, Washington 98314

DATE 27 APR 1983

FROM COMMANDING OFFICER (Code 700)
REPORT NO. 83-4-69
TO

The following laboratory analysis of the sample(s) identified below is submitted.

SOURCE OF SAMPLE Naval Supply Center, Puget Sound
Manchester Fuel Dept., Manchester, WA 98353
DATE SAMPLED UNK
VESSEL OR TANK SAMPLED DISTILLATE
NO. OF SAMPLES COMPOSITE
DATE SAMPLE RECEIVED 26 APR 1983
CONTRACTOR/MANUFACTURE
CONTRACT NO.
CONTRACT DATE
TYPE OF FUEL FUEL OIL, DIESEL, MARINE
GRADE OR CLASS MARINE
SPECIFICATION NO. MIL-F-16884G

TESTS	REQUIREMENTS	RESULTS	
		LAB #	
		SAMPLE #	
Appearance	CLEAR & BRIGHT	C&B	
Color	3.0 MAX (6)*	L1.0	
Gravity A.P.I. @ 60°F	RECORD	37.5	
Viscosity Kinematic, cs@100°F	1.8 - 4.5	2.32	
Flashpoint °F PMCC	140°F MIN	178	
Cloud Point °F	30°F MAX	-12	
Pour Point °F	20°F MAX	-15	
Sulfur, % max	1.0 MAX		
Copper strip corrosion ASTM max 212°F, 3 Hrs	No. 1b MAX	3b	
Demulsification, minutes max	10 MAX	0.50	
Acid Number, max	0.30 MAX	0.16	
Distillation initial boiling point °F		400	
Fuel evaporated, 10% min @ °F			
Fuel evaporated, 20% min @ °F			
Fuel evaporated, 40% min @ °F			
Fuel evaporated, 50% min @ °F	RECORD	480	
Fuel evaporated, 90% min @ °F	675°F MAX (680)*	640	
End point, max, °F	725°F MAX (730)*	675	
Sum of 10% and 50% temp. min @			
% evaporated at 400 °F			
Residue + Loss, % max	3.0 MAX	1.5	
Neutrality	NEUTRAL	NEUTRAL	
Ash % max	0.005 MAX (0.01)*	NIL	
B.S. & W. % max	0.01 MAX	NONE	
Water by distillation max			
Sediment by extraction max			
Carbon residue conradson max, 10% Bottoms	0.20 MAX	0.18	
Ignition quality, cetane index No., calculated	45 MIN	48	
Gravity, Specific, 60/60°F		0.8373	
	* USE LIMIT		

IN THE ABOVE RESPECTS SAMPLE (XXXXXXX/DOES NOT COMPLY) WITH APPLICABLE SPECIFICATION.

ANALYSIS ACCOMPLISHED BY WILSON L. MEBANE 5/4/83 INSPECTOR

BY DIRECTION

Enclosure (2)

LABORATORY ANALYSIS REPORT
NSC 4020/1 (Rev. 2-82)

TABLE I-38. Continued

DEPARTMENT OF THE NAVY
NAVAL SUPPLY CENTER, PUGET SOUND
Bremerton, Washington 98314

FROM COMMANDING OFFICER (Code 700)	DATE REPORT NO. 83-4-67
TO	

The following laboratory analysis of the sample(s) identified below is submitted.

SOURCE OF SAMPLE Naval Supply Center, Puget Sound Manchester Fuel Dept., Manchester, WA 98353		DATE SAMPLED UNK
VESSEL OR TANK SAMPLED HYDROTREATED FUEL	NO. OF SAMPLES 2 (gal)	DATE SAMPLE RECEIVED 26 Apr 1983
CONTRACTOR/MANUFACTURE MOHAWK OIL CO., VANCOUVER B.C.	CONTRACT NO.	CONTRACT DATE
TYPE OF FUEL FUEL OIL, DIESEL, MARINE	GRADE OR CLASS MARINE	SPECIFICATION NO. MIL-F-16884G

TESTS	REQUIREMENTS	RESULTS LAB # SAMPLE #	
Appearance	Clear & Bright	C&B	
Color	3.0 MAX (6)*	L0.5	
Gravity A.P.I. @ 60°F	RECORD	37.6	
Viscosity Kinematic, cs@100°F	1.8 - 4.5	2.33	
Flashpoint °F PMCC	140°F MIN	176	
Cloud Point °F	30°F MAX	-16°	
Pour Point °F	20°F MAX	-20	
Copper strip corrosion ASTM max 212°F, 3 Hrs.	No. 1b MAX	1a	
Demulsification, minutes max	10 MAX	0.25 Minutes	
Acid number, max	0.30 MAX	0.09	
Distillation initial boiling point °F	REPORT	386	
Fuel evaporated, 10% min @ °F	REPORT	420	
Fuel evaporated, 20% min @ °F	REPORT	436	
Fuel evaporated, 40% min @ °F			
Fuel evaporated, 50% min @ °F	RECORD	479	
Fuel evaporated, 90% min @ °F	675°F MAX (680)*	554	
End point, max, °F	725°F MAX (730)*	645	
Sum of 10% and 50% temp. min @			
% evaporated at 400 °F			
Residue + Loss, % max	3.0 MAX	2.0	
NEUTRALITY	NEUTRAL	NEUTRAL	
Ash % max	0.005 MAX (0.01)*	NIL	
B.S. & W. % max	0.01 MAX	NONE	
Water by distillation max			
Sediment by extraction max			
Carbon residue conradson max, 10% Bottoms	0.20 MAX	0.02	
Ignition quality, cetane index No., calculated	45 MIN	48	
Gravity, Specific, 60/60°F		0.8368	
	* USE LIMIT		

IN THE ABOVE RESPECTS SAMPLE (COMPLIES/DOES NOT COMPLY) WITH APPLICABLE SPECIFICATION.

ANALYSIS ACCOMPLISHED BY 5/4/83 WILSON L. MEBANE INSPECTOR	BY DIRECTION

Enclosure (2)

TABLE I-39

AEROSPACE FUELS LABORATORY TEST REPORT

Fuel oil, marine diesel

TEST LABORATORY AND LOCATION: Energy Management Laboratory, Mukilteo, WA

SPECIFICATION NUMBER: MIL-F-16884

SAMPLE NUMBER: See below

DATE SAMPLE RECEIVED: 5 MAY 83

DATE SAMPLE TESTED: Sampled 21 APR 83

CONTRACTOR:

CONTRACT NUMBER:

MANUFACTURER: Mohawk Lubricants Ltd.

PURCHASE ORDER NO AND/OR NATIONAL STOCK NUMBER:

SUBMITTED BY: Naval Supply Center, Puget Sound
Manchester, WA

SAMPLE MARKED:

~~TEST RESULTS~~

Test No.	Sample marked	Accelerated Stability Insolubles/100 ml	Total
83-F-915	83-4-69; 6000 gals. Distillate	0.2	0.2
83-F-916	83-4-67; 3000 gals. Hydrotreated reclaimed fuel	0.2	0.2

REMARKS:

REVIEWED BY (Signature, date and organization symbol):

APPROVED BY (Signature, date and organization symbol):
Lionel V. McIntosh
LIONEL V. McINTOSH, Chief, SFTLD 10 MAY 83

SA-ALC FORM JAN 76 216

Enclosure (3)

APPENDIX II

VENDOR INQUIRIES

APPENDIX II

In July of 1983, a letter of inquiry was sent to a number of vendors selected for their potential in providing processes and/or related equipment applicable to the problem of converting reclaimed fuel oil (FOR) into a fuel of quality that meets the specifications for the current Naval Distillate Fuel (F-76). The military specification for this fuel is MIL-F-16884H while the predecessor to F-76 was Diesel Fuel Marine (DFM) with a similar set of specifications (MIL-F-16884G).

A copy of the letter of inquiry and its attachments which included chemical and physical requirements for both FOR and DFM (now F-76) is included in this appendix as Tables II-1, II-2, and II-3.

The vendors to whom this letter was sent were selected from a list prepared by reviewing appropriate categories in the Thomas Register and from contacts provided by both NIPER and Navy personnel. A list of these vendors is also included in this appendix as Table II-4.

The responses to these inquiries are discussed in section 2.2.1.4 of this report.

LIST OF TABLES
APPENDIX II

TABLE II-1

Department of Energy
Bartlesville Energy Technology Center
P.O. Box 1398
Bartlesville, Oklahoma 74005

July 8, 1983

Lubrication Company of America
Oak Street & East Pacific Way
Los Angeles, CA

Dear Sir:

The Department of Navy (DON) has been concerned with management of its oily wastes for many years. All of DON's major fuel terminals have facilities to reclaim oil from the products which are generated in ships' bilges and waste oil tanks, and from tank bottoms, line cleaning and other contaminated oil. After a primary oil/water separation, some of the resulting oil is given secondary treatment consisting of heating and/or chemical addition to remove much of the remaining water and sludge. The product from these processes is suitable for blending with Naval Special Fuel Oil (NSF) or for sale as Fuel Oil Reclaimed (FOR) for use in DON shore boilers.

DON has asked the Department of Energy (MIPR No. N0003783MP39034) to assist it in determining if there is a feasible and practical process(es) by which reclaimed oil (FOR) can be treated to produce a fuel of quality suitable for blending into DFM. Tables summarizing basic properties for both FOR and DFM are enclosed for your information.

Information about existing and developing processes that may be applicable to this problem would be very useful to us in making this determination. We would appreciate it very much if you could supply us with two copies, if possible, (one for DON and one for DOE) of information, brochures, etc. that you have describing your process(es) and/or related equipment, and any pertinent economic data. We would also like to know if you have a laboratory where samples could be tested using your process and, further, if you do processing on a commercial scale. If you do not do processing on a commercial scale, could you provide us with the names of any companies using your process or equipment that do.

If you have any questions, or would like to discuss this further before responding, please feel free to call us at (918) 336-2400.

Sincerely,

Dennis W. Brinkman
Project Manager

Marian L. Olson
Marian Olson, Chief
Planning and Environmental
Compliance Branch

2 Enclosures

TABLE II-2

3. REQUIREMENTS

3.1 Material. Fuel Oil Reclaimed shall consist of a mixture of distillates and residual fuel and may contain waste lubricants or other recycled products.

3.2 Physical and chemical requirements. Fuel Oil Reclaimed shall conform to the physical and chemical requirements given in Table 1. Any requests for waivers from these requirements will be addressed to Navy Petroleum Office for approval.

TABLE-1- Chemical and Physical Requirements

Characteristics	Requirements	FED-STD 791 test method	ASTM Test method
API Gravity @ 60°F (hydrometer)(range)	25-40		D 287
Viscosity at 104°F (40°C) Kinematic, centistokes (range)	2.0 - 15.0		D 445
Viscosity @ 122°F (50°) Saybolt Universal (range)	30 - 90		D 88
Flash point °F (min)	130°F (55°C)		D 93
Pour point °F (max)	20°F(-6.7°C)		D 97
Sulfated Ash, percent (max)	0.15		D 874
Water & sediment, percent (max)	2.0		D 1796
Neutrality	Neutral	5101	
Sediment percent (max)	0.5		D 473
Chlorinated Material 1/	No Green Flame		
Sulfur content, percent (max)	2.0		D 129 2/ or other approved ASTM methods
Explosiveness, percent (max)	50	1151.1	

1/ FOR shall be essentially free of chlorinated material. To determine the presence of chlorinated material a clean copper wire is heated in a clear blue gas flame, to red heat, until no green shows in the flame. The wire is dipped while still hot, into a sample of FOR and then put back into the flame. No green shall show in the flame. (For practice, a blend of 1% of Trichloroethane in DFM or other distillate fuel may be used as an example of an oil which fails this test also. The oil should be purged of any sodium chloride by washing with fresh water.)

2/ In the U.S.A. sulfur limits shall be as specified by the Environmental Protection Agency, state or community where the fuel is to be used, whichever is more restrictive. In foreign countries, the sulfur limit shall conform to the limit established in the Status of Forces Agreement.

TABLE II-3

3.2 Material. Diesel Fuel Marine supplied under this specification shall be distillate fuel and may contain only those additives specified in 3.4.

3.3 Chemical and physical requirements. The diesel fuel shall conform to the physical and chemical requirements specified in table 1.

Table 1 - Chemical and physical requirements

Characteristics	Requirements	FED-STD-791 test method	ASTM test method
Ignition quality, cetane number (min) (see 4.3.1)	45		D 613
Appearance 1/	Clear, bright, and free from visible particulate matter		
Distallation:			
50 percent point, °F	Record		
90 percent point, °F (max)	675°F (357.2°C)		D 86
End point, °F (max) 2/	725°F (385°C)		
Residue plus loss, percent (max)	3.0		
Flash point °F (min)	140°F (60°C)		D 93
Pour point °F (max)	20°F (-6.7°C)		D 97
Cloud point °F (max)	30°F (-1.1°C)		D 2500
Viscosity at 100°F (37.8°C) Kinematic, centistokes	1.8 - 4.5		D 445
Carbon residue, on 10 percent bottoms, percent (max) (see 4.6.2)	0.20		D 524
Sulfur, percent (max)	1.00		D 129 3/
Corrosion (max) at 212°F (100°C)	No. 1 ASTM		D 130
Color (max)	5		D 1500
Ash, percent (max)	0.005		D 482
Gravity (hydrometer)	Record		D 287
Demulsification, minutes (max) (see 4.6.3)	10	3201	-----
Acid number (max)	0.30		D 974
Neutrality	Neutral	5101	-----
Aniline point, °F	Record		D 611
Accelerated stability, total insolubles mg/100 ml (max)	2.5 4/		D 2274

1/ A slight haze is acceptable providing a maximum water and sediment of 0.01 percent is obtained using procedure ASTM D 2709.

2/ As the end point of the distillation is approached, if either a thermometer reading 725°F (385°C) or a decomposition point is observed, discontinue the heating and resume the procedure as directed in ASTM D 86.

3/ ASTM D 1552 and ASTM D 2622 may be used as alternate methods.

4/ Average of three determinations is acceptable.

TABLE II-4

Absolute Oil Separator Corp.
57-15 32nd Avenue
Woodside, NY 11377
(212)721-1138

Alfa-Laval, Inc.
2115 Linwood Avenue
Fort Lee, NJ 07024
(201)592-7800

Allen Filters, Inc.
533 N. Fremont Street
Springfield, MO 65801
(417)865-2844

AMF Incorporated
AMF Cuno Division
402 Research Pkwy.
Meriden, Conn 06450
(203)237-5541

Aquanetics, Inc.
111 Milbar Blvd.
Farmingdale, NY 11735
(516)454-7600

ARO Corporation, The
One ARO Center
Bryan, OH 43506
(419)636-4242

Bardahl Mfr. Corp.
1400 NW 52nd Street
Seattle, WA 98107
(206)783-4861

Baron & Associates, Inc.
PO Box 1140-T
Cookeville, TN

Barret Centrifugals
PO Box 551
Worcester, MA 01613
(617)755-4306

BASF Wyandotte Corp.
Industrial Chemicals Group
100-T Cherry Hill Road
Parsippany, NJ

Bendix Corp., Fram Corp.
105 Pawtucket Avenue
East Providence, RI

Betz Laboratories, Inc.
Somerton Road
Trevose, PA 19047
(215)355-3300

BS & B Engineering Co.,
8303-T Southwest Frwy.
Houston, TX

Bulk Systems Division
Webb Drive
Farmington Hills, MI 48018
(313)553-1220

Cecor Inc.
102 Lincoln Street
Verona, WI 53593

Centrico, Inc.
100 Fairway Court
Northdale, NJ 07647
(201)767-3900

Cincinnati-Milicron
Products Division
PO Box 9013
Cincinnati, OH 45209

Electro Impulse, Inc.
116-T Chestnut Street
Red Bank, NJ 07701
(201)741-0404

Electro Lube Devices Inc.
16 N. Georgia Street
Jacksonville, FL 32202
(904)353-3147

Engineered Lubricants
11525 Rock Island Court
Maryland Heights, MO 63043
(314)873-9540

ERC/Lancy A Dart & Kraft, Co.
525 W. New Castle Street
Zelienople, PA 16063
(412)452-9360

Ferguson Perforating & Wire Co.
138-T Ernest Street
Providence, RI

Filterite/Brunswick
2033 Greenspring Drive
Timonium, MD 21093
(301)252-0800

Gulf Oil Co.
PO Box 1563
Houston, TX 77001
(713)754-2870

TABLE II-4

Hilliard Corp.
The Hilco Division
100 W. 4th Street
Elmira, NY 14902
(607)733-7121

Hyde Products Inc.
810 Sharon Drive
Cleveland, OH 44145
(216)871-4885

Keene Corp,
Bohannon Avenue
Greenville, TN 37743
(615)638-8156

Kennecott Corp.
Commercial Filters Division
State Route 32 West
Lebanon, IN 46052
(317)482-3900

Lubrication Company of America
Oak Street & East Pacific Way
Los Angeles, CA

Luscon Industries Corp.
361 Silver Sands Road
East Haven, CT 06512
(203)469-2336

Metalworking Lubricants Co.
6785 Telegraph-Suite 200
Birmingham, MI 48010
(313)642-0410

Millipore Corp.
80-T Ashby Road
Bedford, Mass

Montgomery Co.

The 50 Canal Street
Windsor Locks, CN 06096
(203)623-3336

Nalco Chemical Co.
2901 Butterfield Road
Oak Brook, IL 60521
(312)887- 500

National Chemsearch
2727 Chemsearch Blvd.
Irving, TX 75060
(214)438-0511

Niagara Lubricant Co.
PO Box 76
Buffalo, NY 14240
(716)822-2300

Pall Industrial Hydraulics Corp.
2200 Northern Blvd.
East Hills, NY 11548
(516)484-4000

Pennwalt Corp.
Sharples, Stokes Division
955 Mearns Road
Warminster, PA 18974
(215)443-4000

Petreco Division
PO Box 2546
Houston, TX

Sanborn Associates, Inc.
25-T Commercial Drive at Route One
Wrentham, Mass 02093
(617)384-3181

SCA Chemical Waste Service Inc.
60 State Street
Boston, Mass

S.D. Myers Inc.
PO Box 3628
Akron, OH 44310
(216)929-6800

Serfilco, LTD
1234 Depot Street
Glenview, IL 60025
(800)323-5431

Southwest Filter Co.
1534-T N. 75th East Ave.
Tulsa, OK

Tretolite Division
369 Marshall Avenue
St. Louis, MO

United States Filter Systems Corp.
Subsidiary of U.S. Filter Corp.
12442-T E. Putnum Street
Whittier, CA 90602
(213)69 -9414

Universal Silencer Process Products Group
Division of Nelson Industries, Inc.
PO Box 411
Stoughton, WI 53589
(608)873-4272

Velcon Filters Inc.
1750 Rogers Avenue
San Jose, CA 95112
(408)298-6525

Westmont Industrial Products
3116 N. Kedzie Avenue
Chicago, IL 60618
(312)478-2930

Wind, Ken, Co.
PO Box 19056
Houston, TX

Witco Chemical Corp.
PO Box 4239
Grand Central Station
New York, NY 10164
(212)775-1395

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