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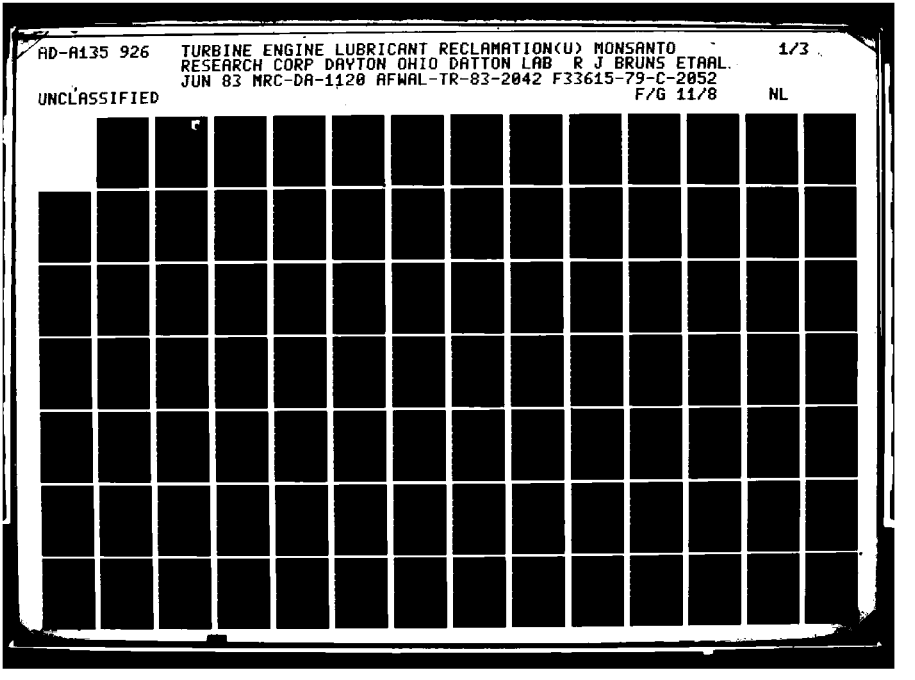

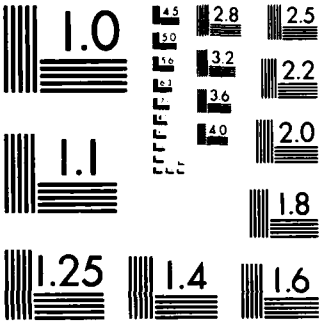

AFWAL-TR-83-2042

TURBINE ENGINE LUBRICANT RECLAMATION

Richard J. Bruns and George L. Beemsterboer
MONSANTO RESEARCH CORPORATION
Dayton Laboratory
Dayton, Ohio 45407

June 1983
Final Report for Period 1 September 1979 - 1 March 1983

AERO PROPULSION LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES
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This technical report has been reviewed and is approved for publication.

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1 REPORT NUMBER AFWAL-TR-83-2042	2. GOVT ACCESSION NO. AD-A135926	3. RECIPIENT'S CATALOG NUMBER
4 TITLE (and Subtitle) TURBINE ENGINE LUBRICANT RECLAMATION		5. TYPE OF REPORT & PERIOD COVERED Final Report 1 September 1979 - 1 March 1983 6. PERFORMING ORG. REPORT NUMBER MRC-DA-1120
7. AUTHOR(s) Richard J. Bruns and George L. Beemsterboer		8. CONTRACT OR GRANT NUMBER(s) F33615-79-C-2052
9 PERFORMING ORGANIZATION NAME AND ADDRESS Monsanto Research Corporation Dayton Laboratory, 1515 Nicholas Road Dayton, OH 45407		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 3048 06 11
11. CONTROLLING OFFICE NAME AND ADDRESS Aero Propulsion Laboratory (AFWAL/POSL) Air Force Wright Aeronautical Laboratories (AFSC) Wright-Patterson AFB, OH 45433		12. REPORT DATE June 1983 13. NUMBER OF PAGES 252
14 MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION DOWNGRADING SCHEDULE

16 DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18 SUPPLEMENTARY NOTES

19 KEY WORDS (Continue on reverse side if necessary and identify by block number)

Reclamation	Distillation	High performance liquid chromatography
Synthetic turbine oils	Adsorbents	
MIL-L-7808	Reclaimed base stock	Pilot plant
Additives	Gas chromatography	Cost analysis
Esters	Synthetic lubricants	

20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

A distillation and adsorption treatment for reclaiming used MIL-L-7808 turbine oils was investigated. A viable additive package was tested on different MIL-L-7808 type virgin base stocks. Fifteen used oils were analyzed by acid number, high performance liquid chromatography, and gas chromatography. A distillation process utilizing caustic (sodium hydroxide) pretreatment was developed on 500-mL and 13-litre scales. Adsorption treatment of distilled oils consisted of barium hydroxide monohydrate. A total of ten 15-25 gallon batches of oil were

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reclaimed/reformulated and MIL-L-7808H tested. Test results were generally good. However, none of the 10 batches completely passed all of the tests.

An extensive screening procedure was found necessary to identify contaminated used oil samples.

An engineering cost study is included.

FOREWORD

This final technical report was prepared by Monsanto Research Corporation (MRC), 1515 Nicholas Road, Dayton, Ohio 45407. The effort was sponsored by the Aero Propulsion Laboratory (APL), Air Force Wright Aeronautical Laboratories (AFWAL), Air Force Systems Command, Wright-Patterson AFB, Ohio, under Contract No. F33615-79-C-2052 during the period 1 September 1979 to 1 March 1983. The work herein was accomplished under Project 3048, Task 304806, Work Unit No. 30480611, "Turbine Engine Lubricant Reclamation," with Mr. G. A. Beane IV, AFWAL/POSL, as Project Engineer.

The authors are indebted to Mr. George Richardson, and to Drs. J. A. Ellard and D. G. Glasgow for their many helpful suggestions.

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SUMMARY

The objective of this program is to improve upon the purification process and additive replenishment package, developed in an earlier program (Technical Report AFAPL-TR-78-50), for reclaiming used synthetic turbine engine oil meeting MIL-L-7808G specifications. The program is also to demonstrate the effectiveness of the total process by successfully reclaiming 12 batches of used synthetic oil representing a variety of MIL-L-7808 compositions.

Base stocks formulated by five different manufacturers were selected for MIL-L-7808H testing using MRC's proposed additive package.

Conditions were established for optimizing a sodium hydroxide distillation followed by barium hydroxide monohydrate treatment. The use of clay and activated charcoal was found to provide no distinct performance advantages over nonuse.

Ten-25 gallons large scale batches were reclaimed and reformulated. These oils were evaluated with MIL-L-7808H tests with good results. However, none of the ten batches completely passed all tests.

An extensive used oil screening method was found necessary to identify samples contaminated beyond reclaimability.

A cost analysis of the process was carried out.

INTRODUCTION

The re-refining and reuse of petroleum base oils has been a successful standard practice in railroad, automotive, and aircraft industries for many years. More recently, the reclamation of synthetic aviation oils has proven successful in those applications involving a specific and known formulation. However, this is not representative of normal U.S. military aircraft operational experience where numerous brands of specification products consisting of widely differing base stocks and additive packages are normally mixed in service. An estimated total of up to one half million quarts of used ester based oils are generated each year and could be collected for reclamation. Reclamation of these oils for reuse in military aircraft turbine engines represents a significant potential source of supply in the event of serious availability problems. Furthermore, reclamation could offer a significant cost savings if the technique developed is also economical and technically effective. Currently available information suggests that the ester base stocks are not significantly degraded during use. Therefore, there is potential for recovery of large portions of the used oils.

An earlier program, described in AFAPL-TR-78-50, demonstrated the potential feasibility of recycling used MIL-L-7808G oils to a satisfactory performance level.

The objective of this program is to improve upon the purification process and additive replenishment package described in AFAPL-TR-78-50, and to demonstrate the effectiveness of the total process by successfully reclaiming 12 batches of used synthetic oil representing a wide variety of MIL-L-7808 compositions.

2. SUMMARY OF THE FIRST OIL RECLAMATION PROGRAM

A process for reclaiming of a used synthetic turbine engine oil meeting MIL-L-7808G specifications was developed. Techniques for characterizing new and used 7808G oils were developed and applied. The components of a reclamation process were defined and the technical feasibility of the process was established.

Two additive packages developed for use in diester/triester base stock mixtures showed considerable promise.

A cost analysis of the process indicated that a continuous batch reclamation process at the 5000-gallon batch size would be cost effective.

Details of the first oil reclamation program can be found in Technical Report AFAPL-TR-78-50, "Reclamation of Synthetic Turbine Engine Oil Mixtures."

3. RESULTS AND DISCUSSION

The object of this program is to further improve the reclamation process developed earlier (summarized in Figure 1), define the variables within the process, verify the additive replenishment package, and demonstrate the effectiveness of the total process by reclaiming 12 batches of used synthetic oil to MIL-L-7808H specifications.

3.1 USED OIL CHARACTERIZATION

To determine how widely properties vary between used oil lots and to decide under what conditions some lots should be rejected for reclamation, Aero Propulsion Laboratory (APL) furnished us with 15 different used oil lots. Each lot was examined and characterized (See Appendix C for analysis procedures) by gas chromatography (GC), high performance liquid chromatography (HPLC), infrared (IR) analysis, and acid number. Table 1 lists the quantities and acid numbers of the 15 used oil lots received.

3.1.1 Infrared Spectrophotometry

Infrared (IR) analysis was performed on filtered and dried samples of the used oils to see if any gross differences were observable which would interfere with reclamation.

Examination of the IR spectra of used oils 0-79-01 through 0-79-15 and a diester base stock (Figures 2 through 7) show that all are quite similar and made from mixtures of both diester and triester type base stocks. All 15 oil samples show characteristic absorption at 1350 cm^{-1} for the diester and at 720 cm^{-1} for the typical triester. Oils 0-79-03, -04, -05, and -09 show absorption at about 1100 cm^{-1}. This absorption is quite strong in 0-79-05 and is present to a lesser degree in samples 0-79-03, -04, and -09. The remaining oils show absorption in this area to a still lesser degree. This absorption is no doubt associated with the C-O-C bond in the ester, and its shape is influenced by the type of acid structure in the ester.

The IR spectra for all these oils suggest they are composed of di and tri esters, the diester portion being quite similar to the di-2-ethylhexyl azelate virgin base stock shown in Figure 7.

3.1.2 Gas Chromatography

Gas chromatography (GC) was also used to characterize the used oils. The chromatograms of the 15 used oils are shown in Figures 8 through 15. Also shown (Figure 16) is the chromatogram for a commercially available diazelate base stock. The major component of this base stock, which elutes at ~29.22 min, and the

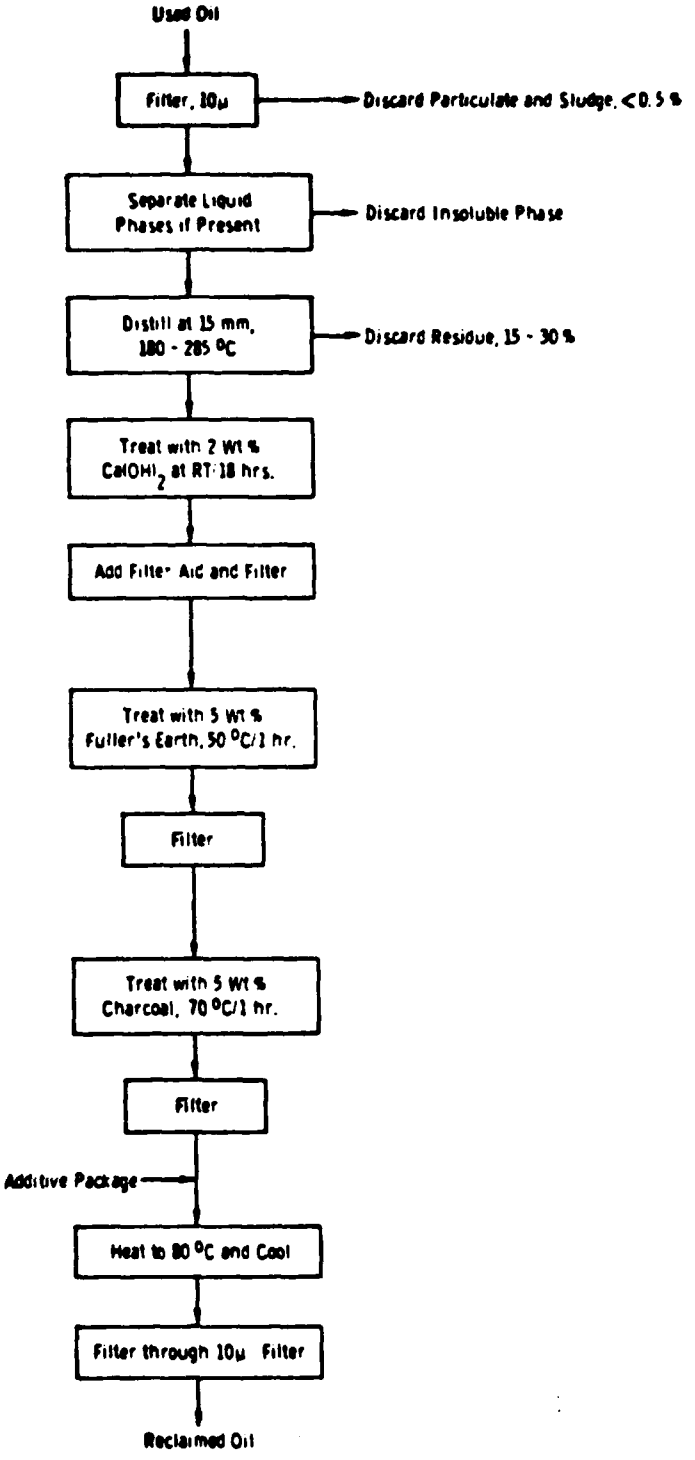
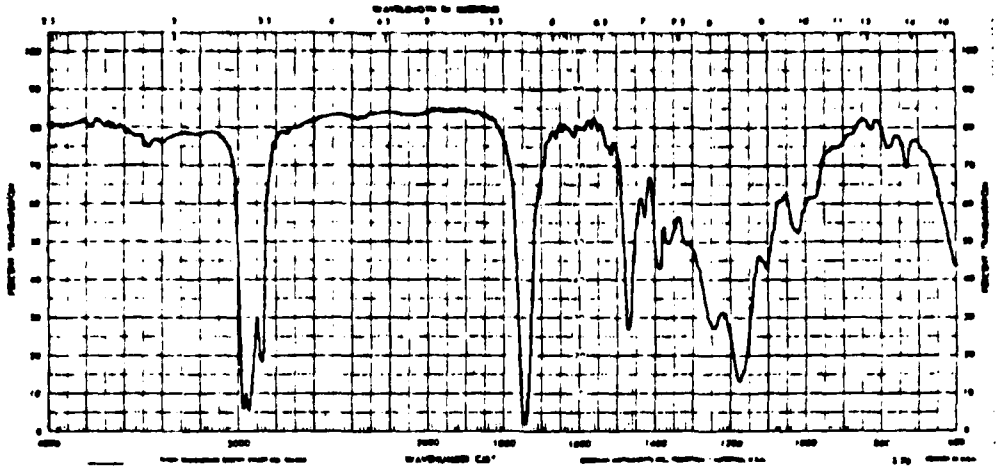


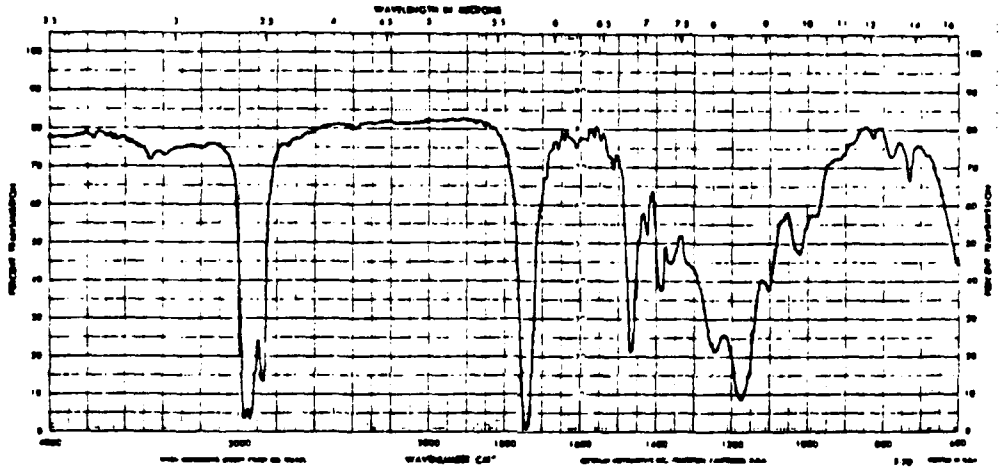
Figure 1. Reclamation process, first program.

TABLE 1. ACID NUMBERS AND QUANTITIES OF OILS RECEIVED FROM APL

Oil number	Quantity, gal	Acid number
0-79-01	35	0.42
0-79-02	55	0.47
0-79-03	~35	0.43
0-79-04	~35	14.45
0-79-05	~35	23.78
0-79-06	55	1.02
0-79-07	~35	1.09
0-79-08	55	1.15
0-79-09	55	0.65
0-79-10	55	0.64
0-79-11	55	0.88
0-79-12	55	0.52
0-79-13	55	0.63
0-79-14	55	1.50
0-79-15	55	1.72



0-79-01



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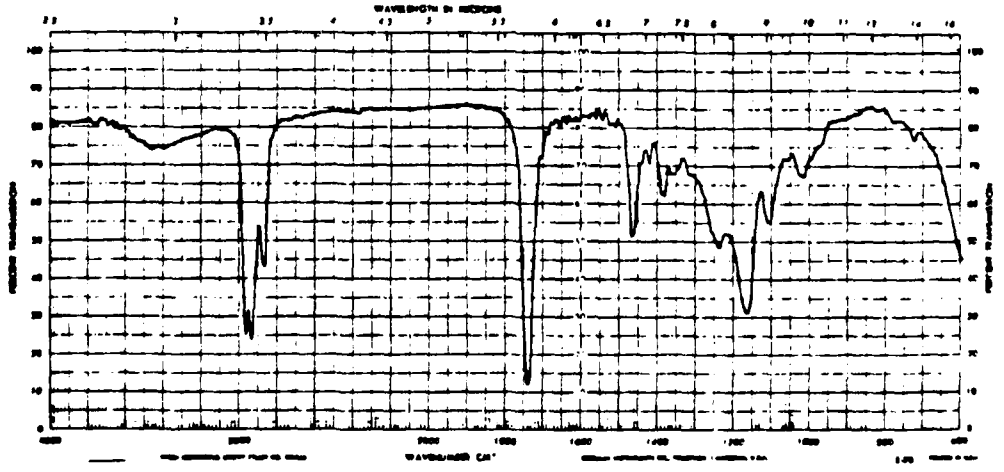
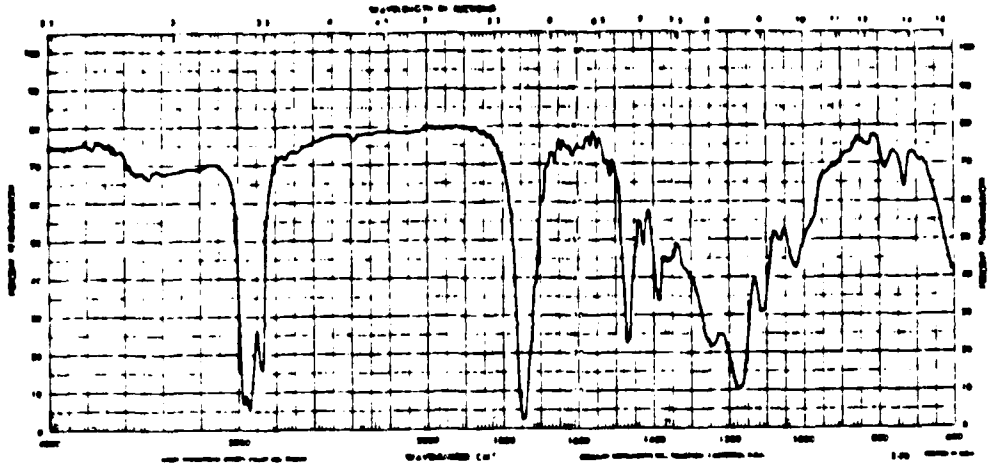


0-79-03

Figure 2. Infrared spectra of used oils.



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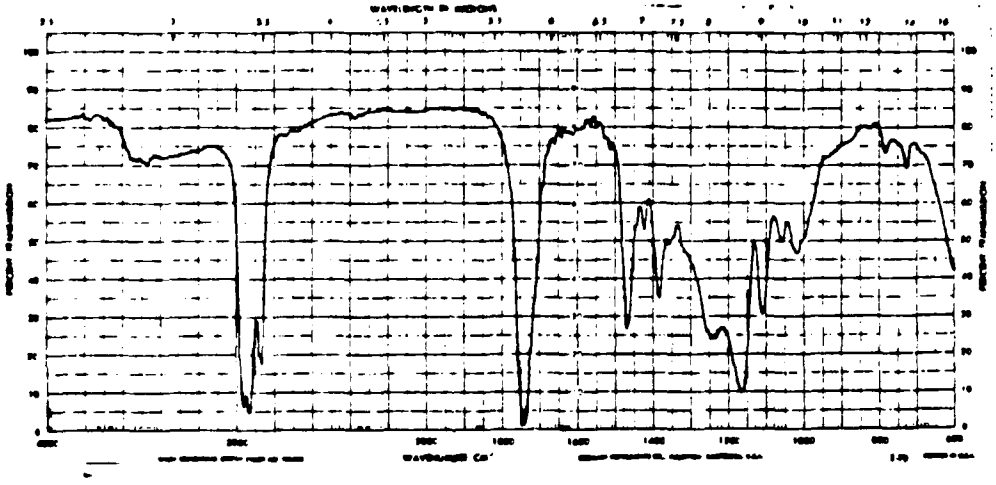

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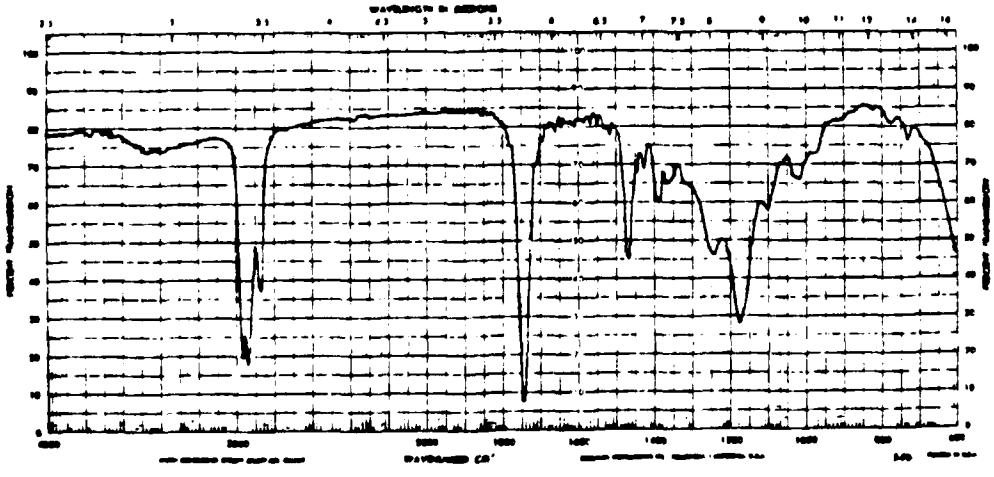
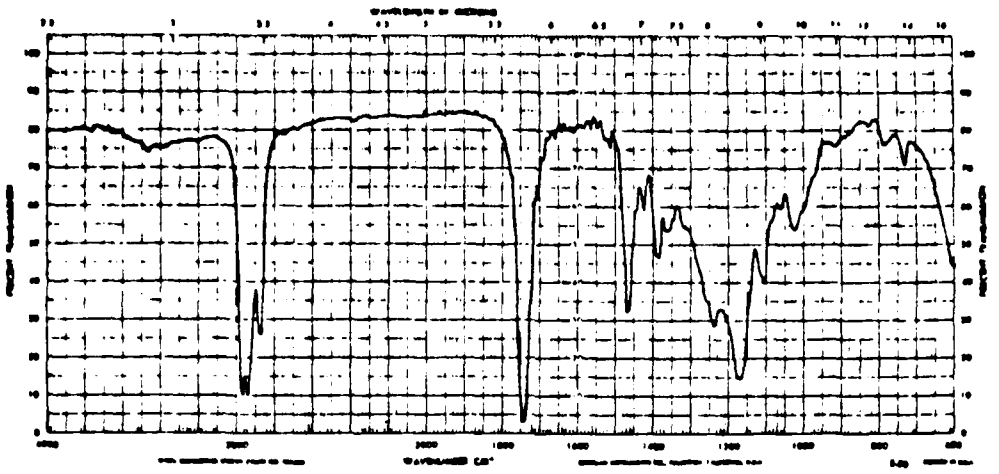


0-79-06

Figure 3. Infrared spectra of used oils.



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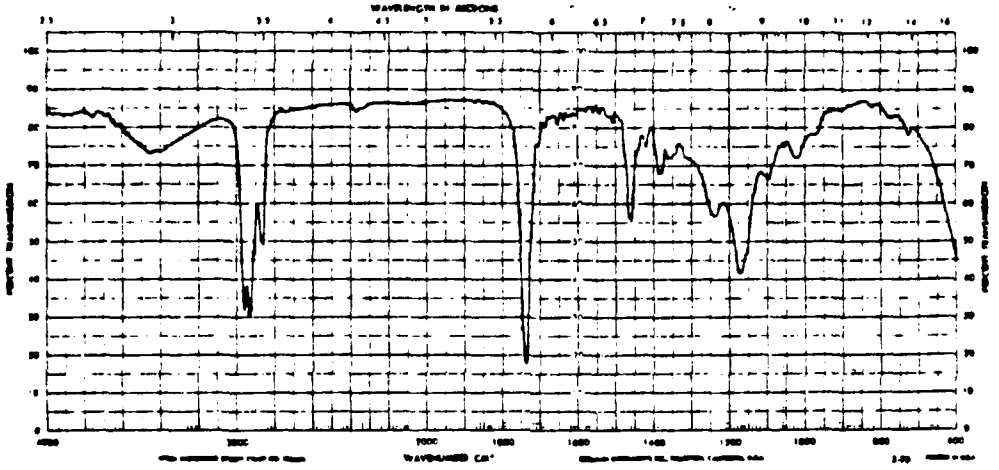

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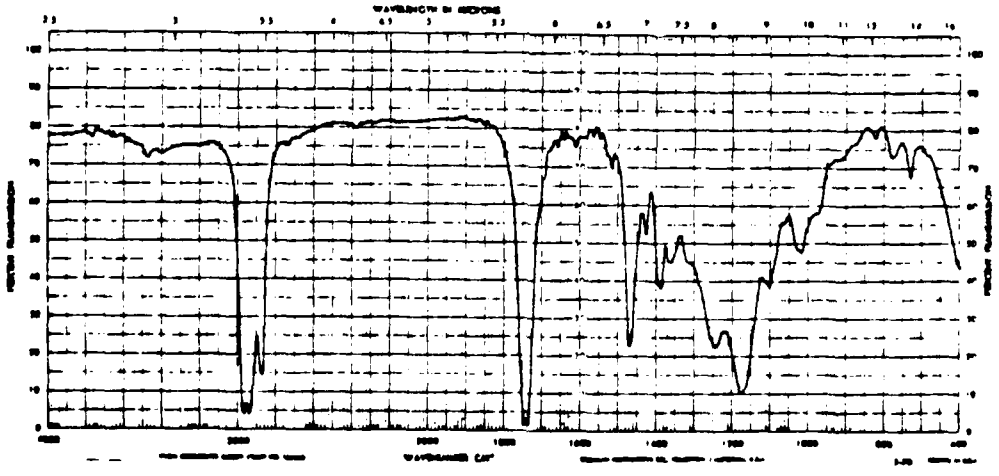
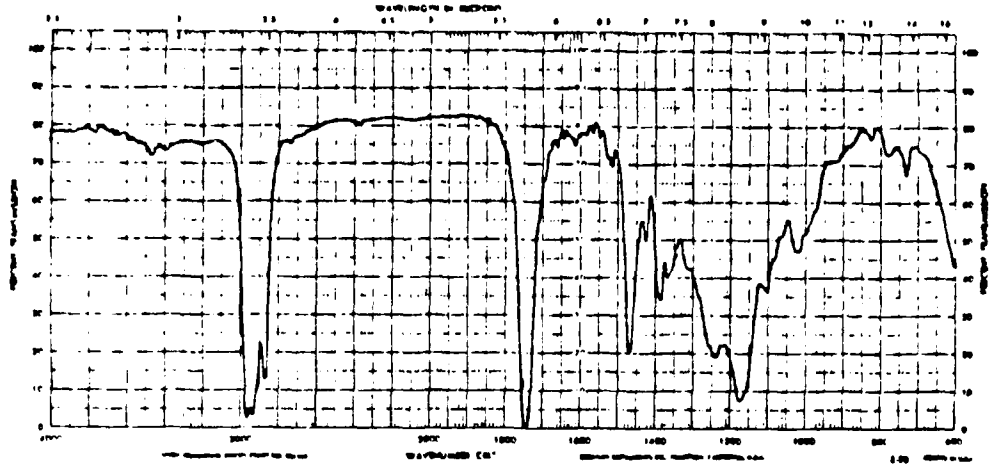


0-79-09

Figure 4. Infrared spectra of used oils.



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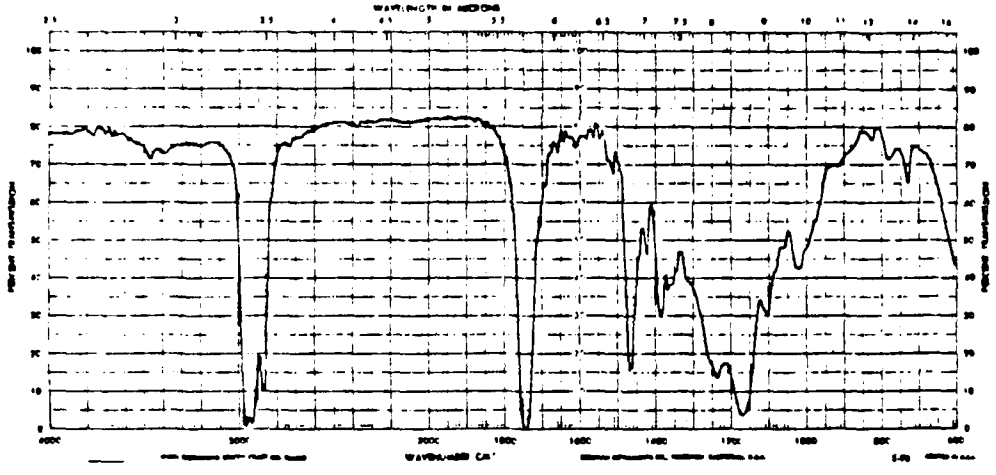

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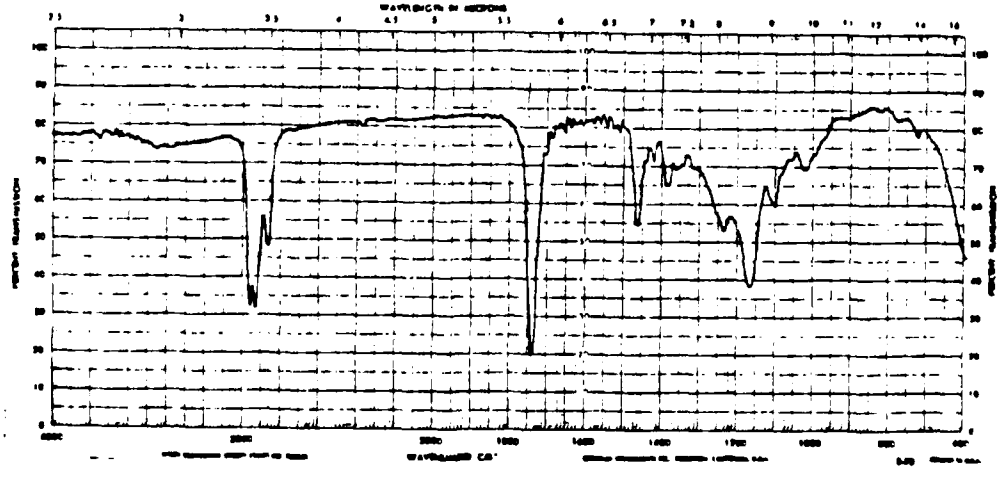
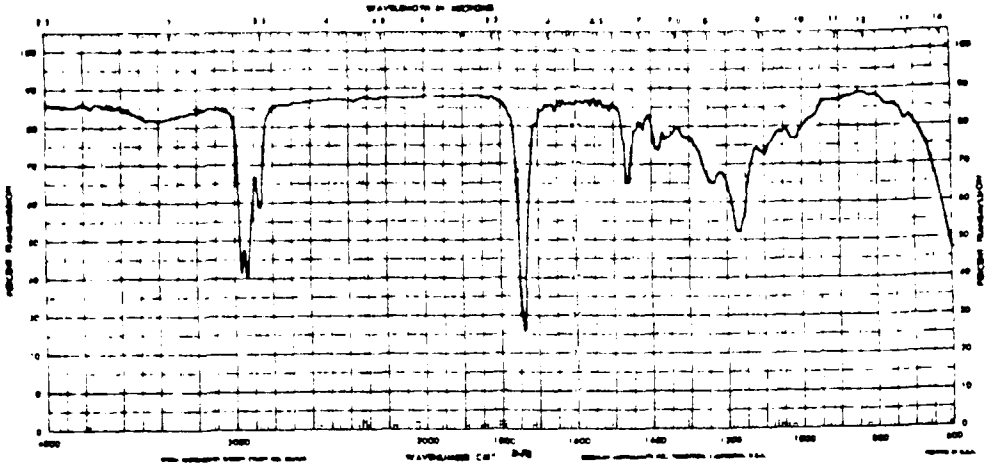
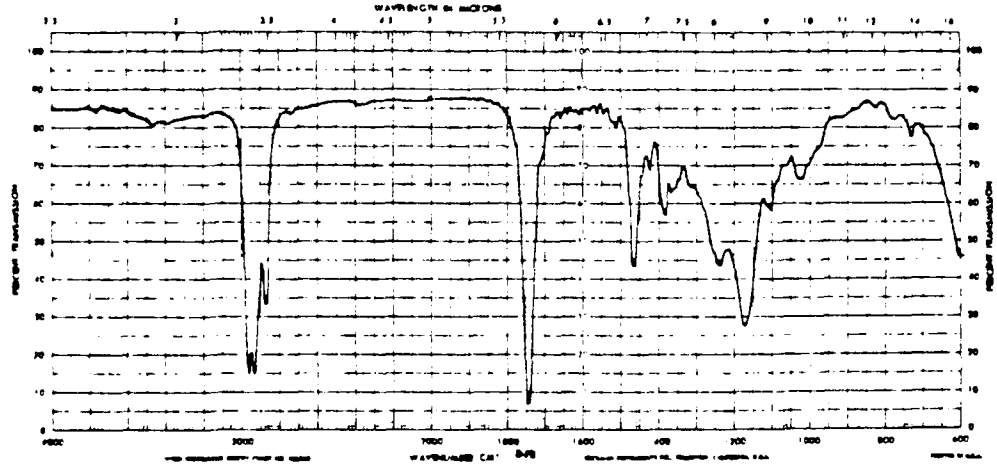


0-79-12

Figure 5. Infrared spectra of used oils.



0-79-13



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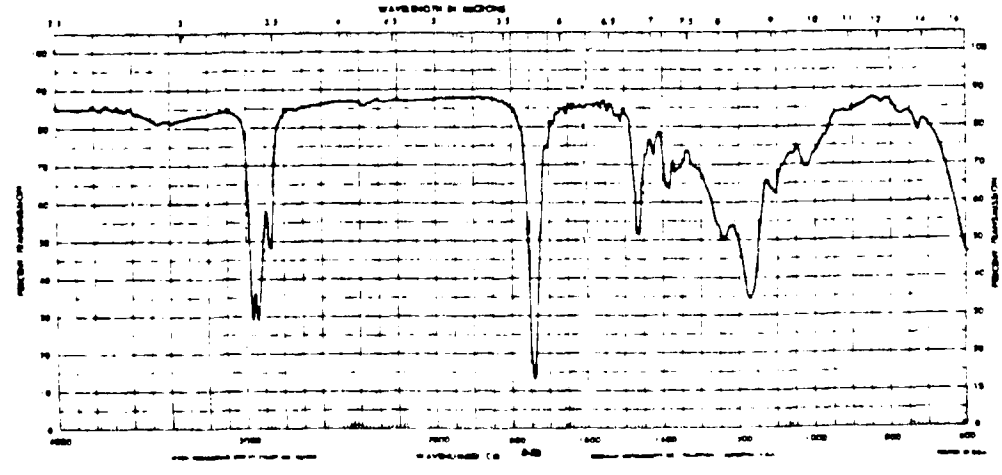


0-79-15

Figure 6. Infrared spectra of used oils.

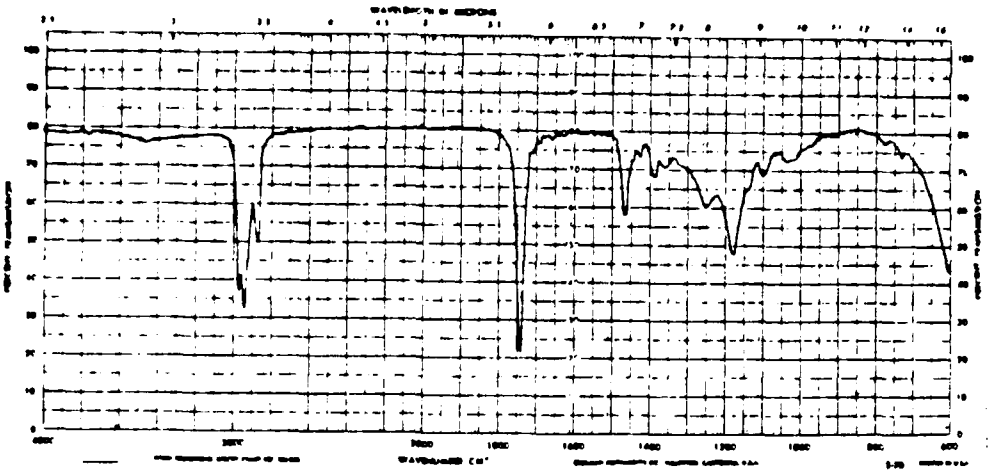


Figure 7. Infrared spectrum of commercially available di-2-ethylhexyl azelate base stock.

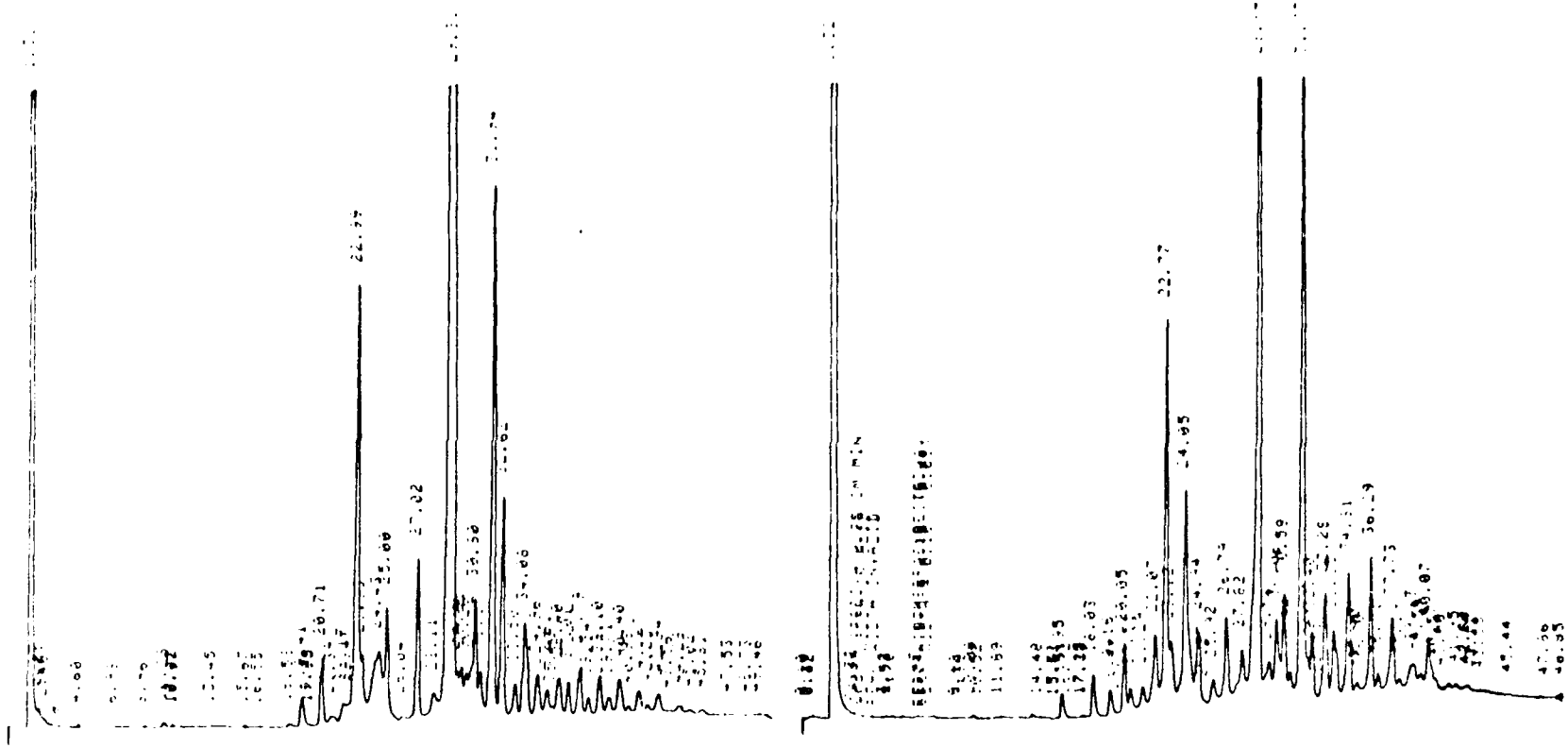
minor peaks at ~20.54, 22.76, 24.83, 26.85, 30.62, 32.4, and 34.07 min are seen in every sample of used oil that has been analyzed. All analyzed oil samples show peaks corresponding to those of the commercial diester but in varying concentrations. These oils are apparently based on mixtures of several components, some of which are quite high boiling in all the samples, but particularly in 0-79-03 ,-05, and -04.

Such close similarities exist between oil samples 0-79-01, 0-79-07, and 0-79-08 that they may be from the same base stock. Oil samples 0-79-09, 0-79-10, 0-79-11, 0-79-12, and 0-79-13, showing only minor differences, are very similar in composition to each other. Peak analysis of possible diester components suggests that in oils 0-79-03 and 0-79-04 approximately 48% of the composition is due to diester; in 0-79-01, 0-79-07, and 0-79-08 diester comprises approximately 70% of the composition. Of the remaining ten oils approximately 45% of the composition is due to diester.

3.1.3 High Performance Liquid Chromatography

Samples which were used for gas chromatographic analysis were examined by high performance liquid chromatography (HPLC) to detect aromatic additives and breakdown products and to establish patterns, if any, for the used oils.

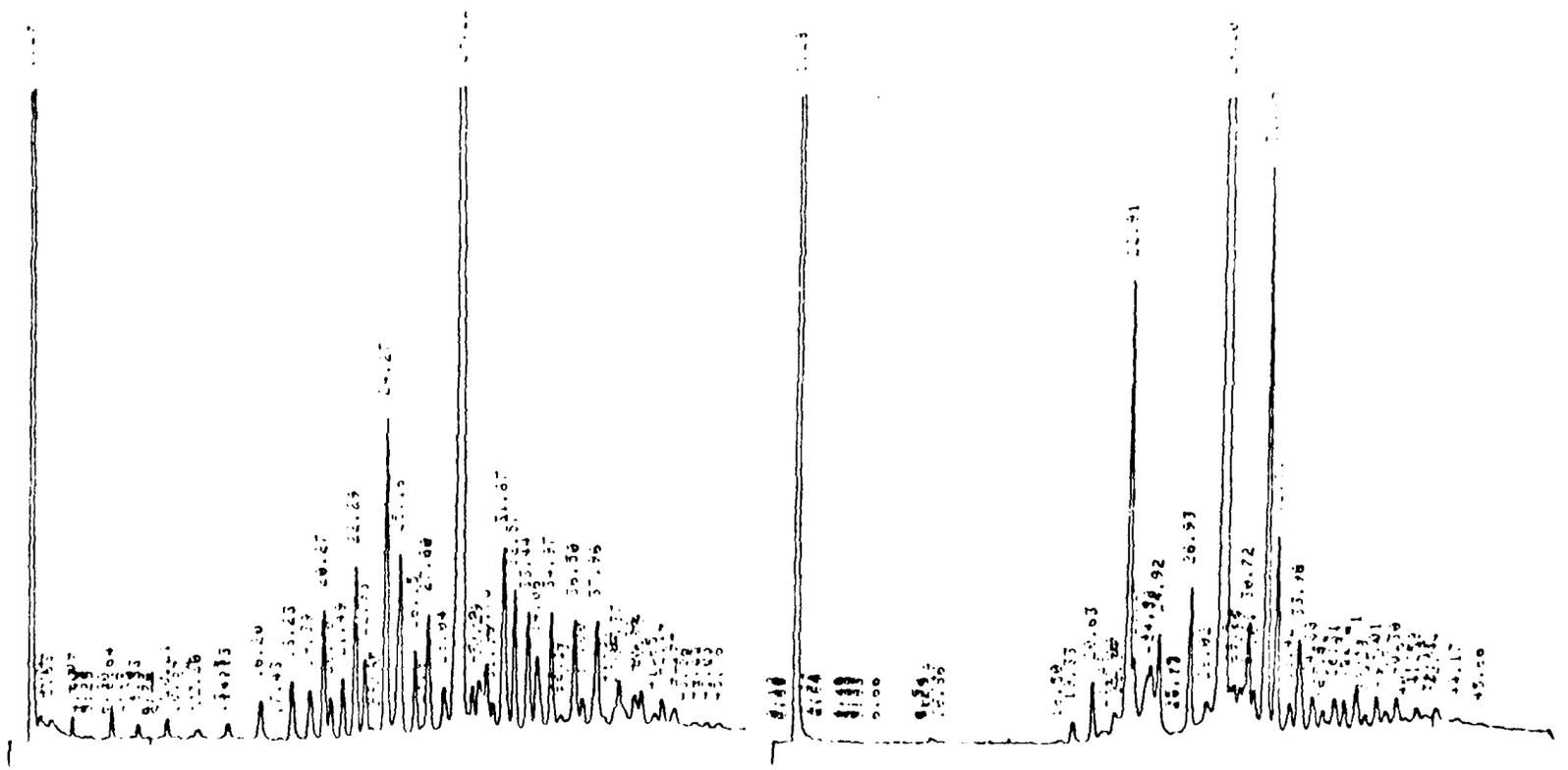
Basic patterns emerged, showing many similarities between all oils. Samples 0-79-01, -02, -06, -07, -08, -10, -11, -12, -13, -14, and -15 are very similar. Sample 0-79-03 is unique in that its gross appearance is that of an almost unused oil. Samples 0-79-04 and 0-79-05 are similar, showing high concentrations of



0-79-01

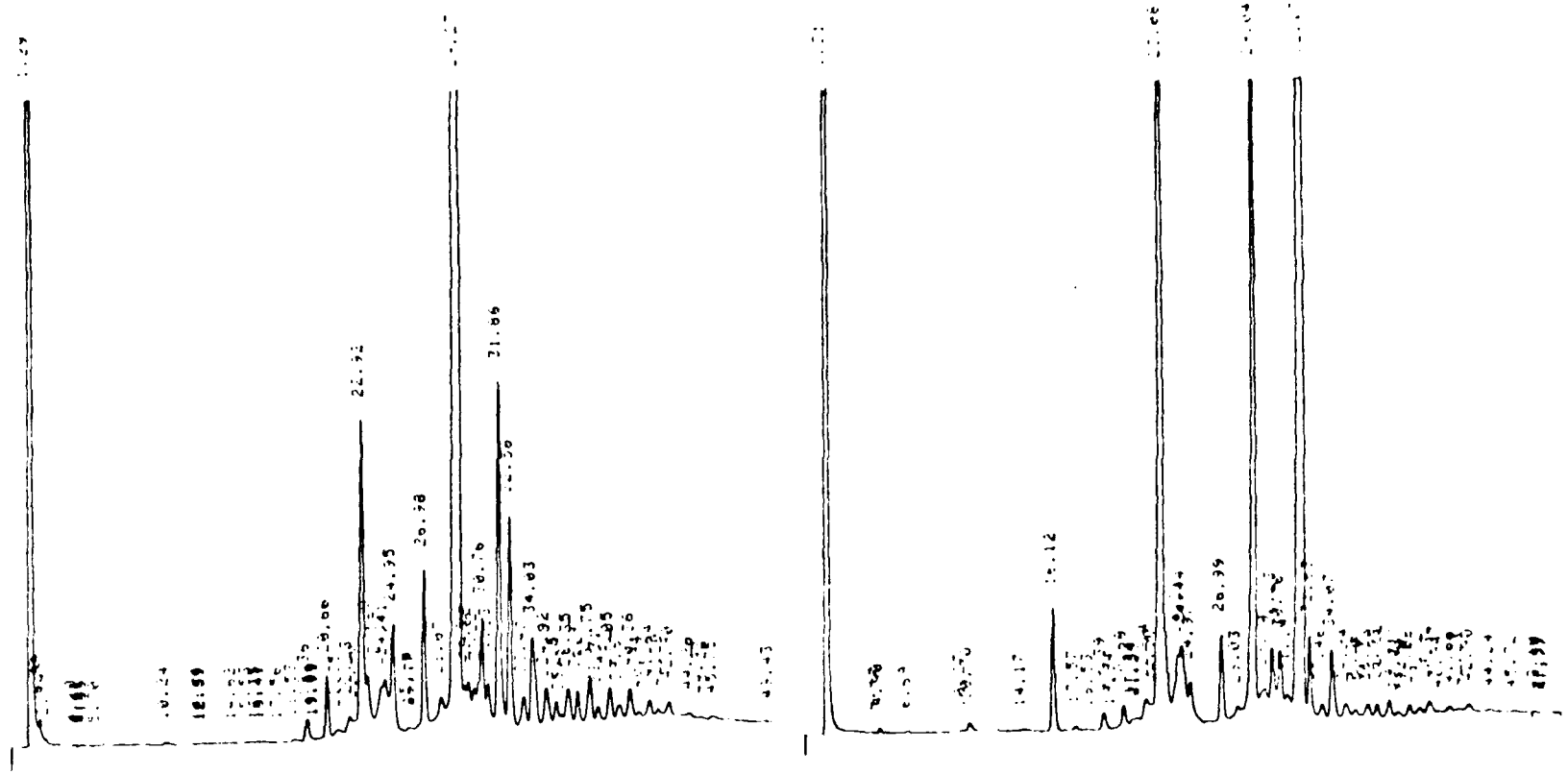
0-79-03

Figure 8. Gas chromatograms of used oils.



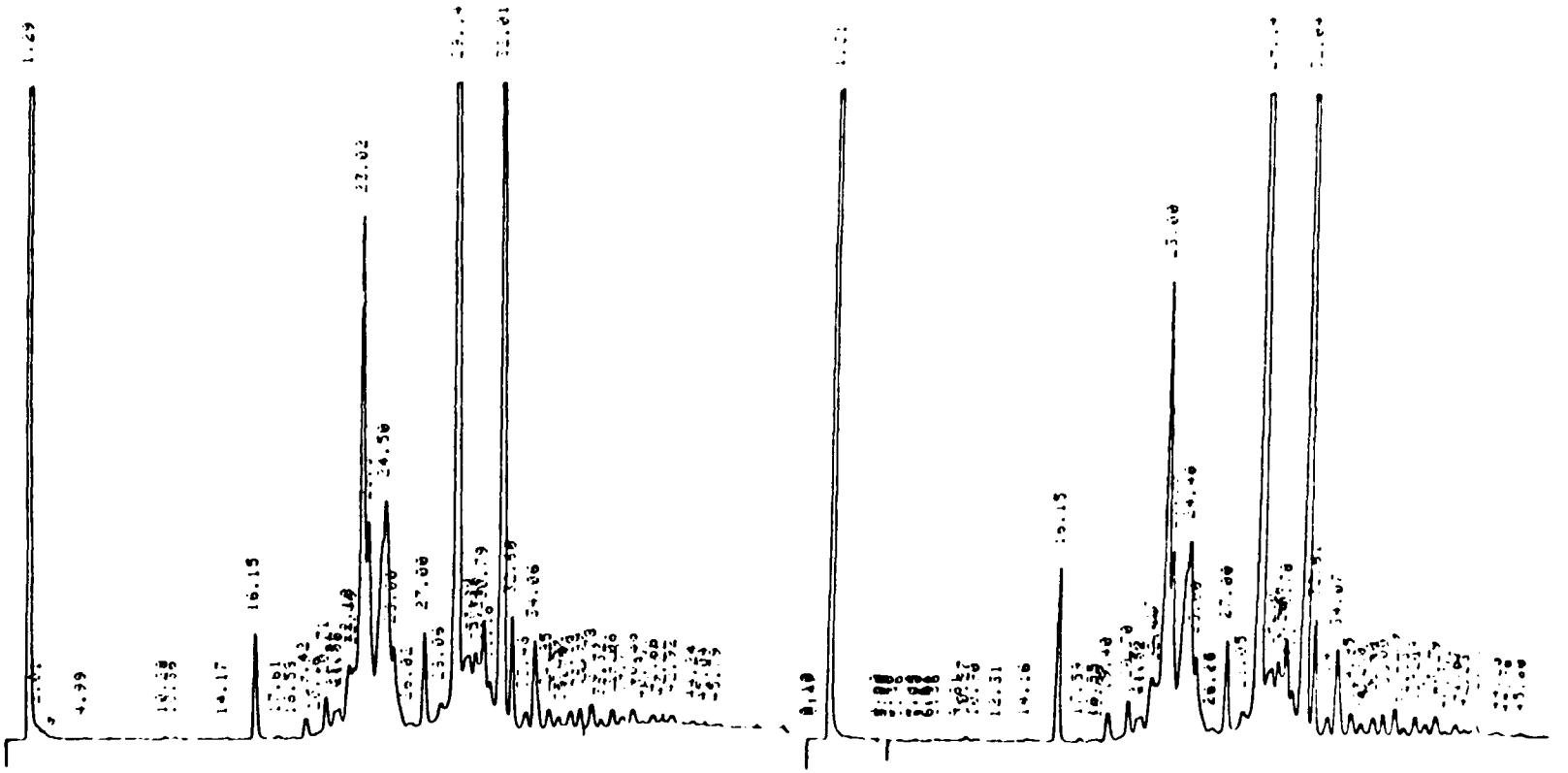
0-79-04 0-79-07

Figure 9. Gas chromatograms of used oils.



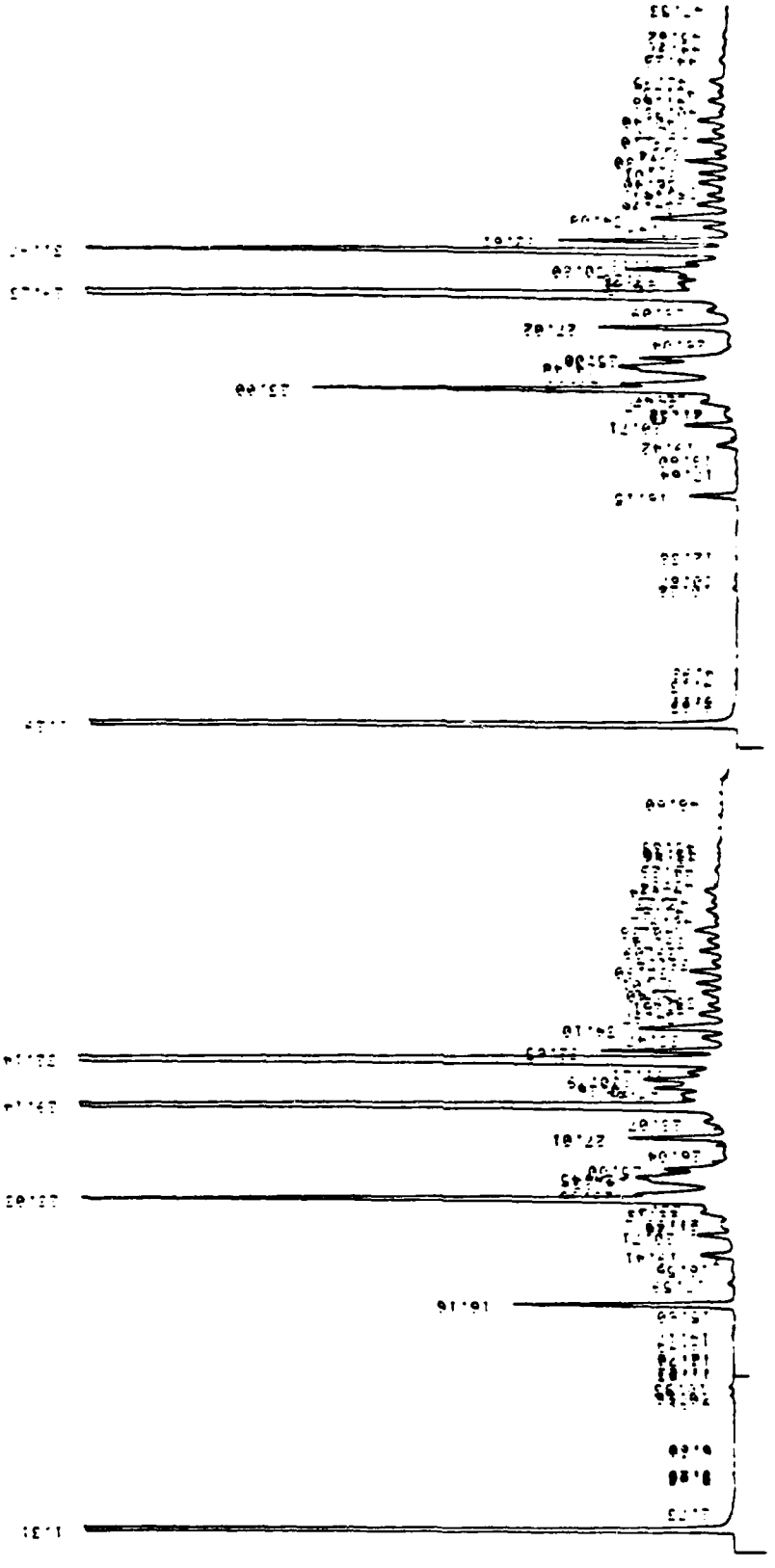
0-79-08 0-79-09

Figure 10. Gas chromatograms of used oils.



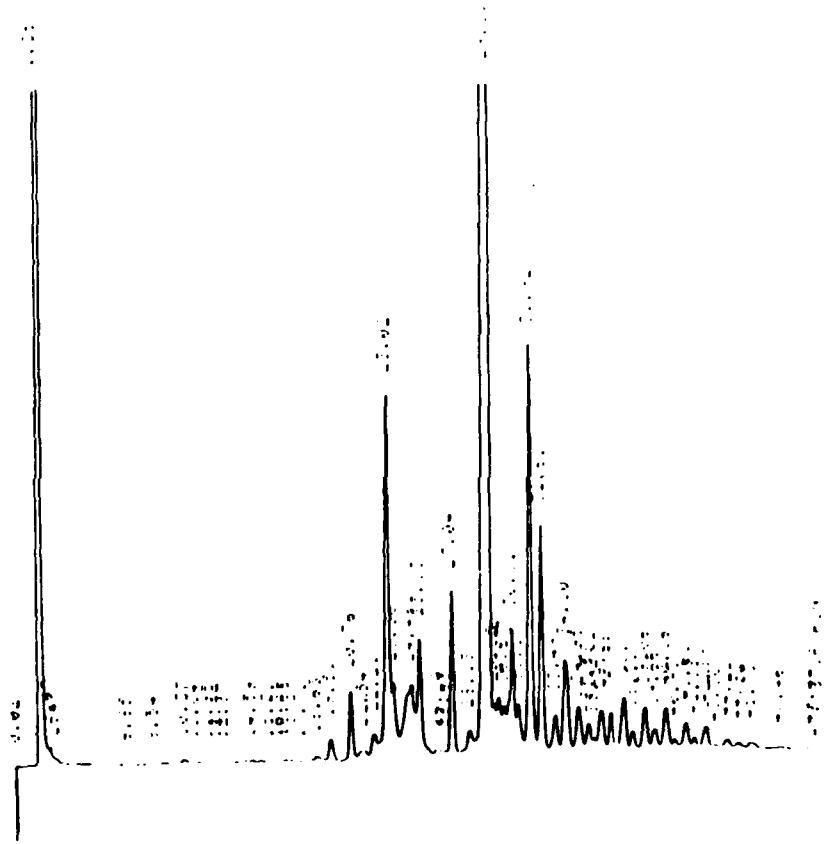
0-79-10 0-79-11

Figure 11. Gas chromatograms of used oils.



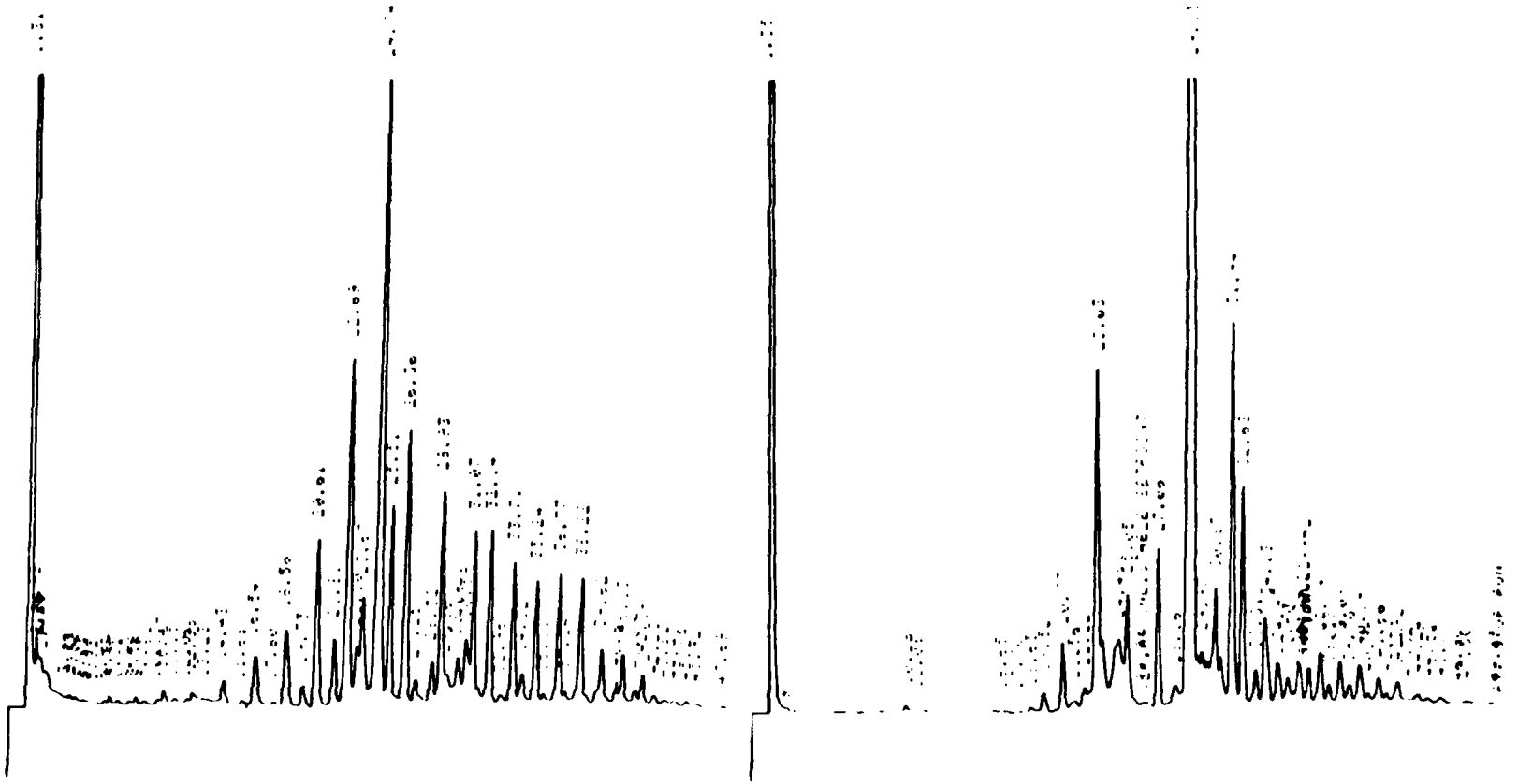
0-79-12 0-79-13

Figure 12. Gas chromatograms of used oils.



0-79-02

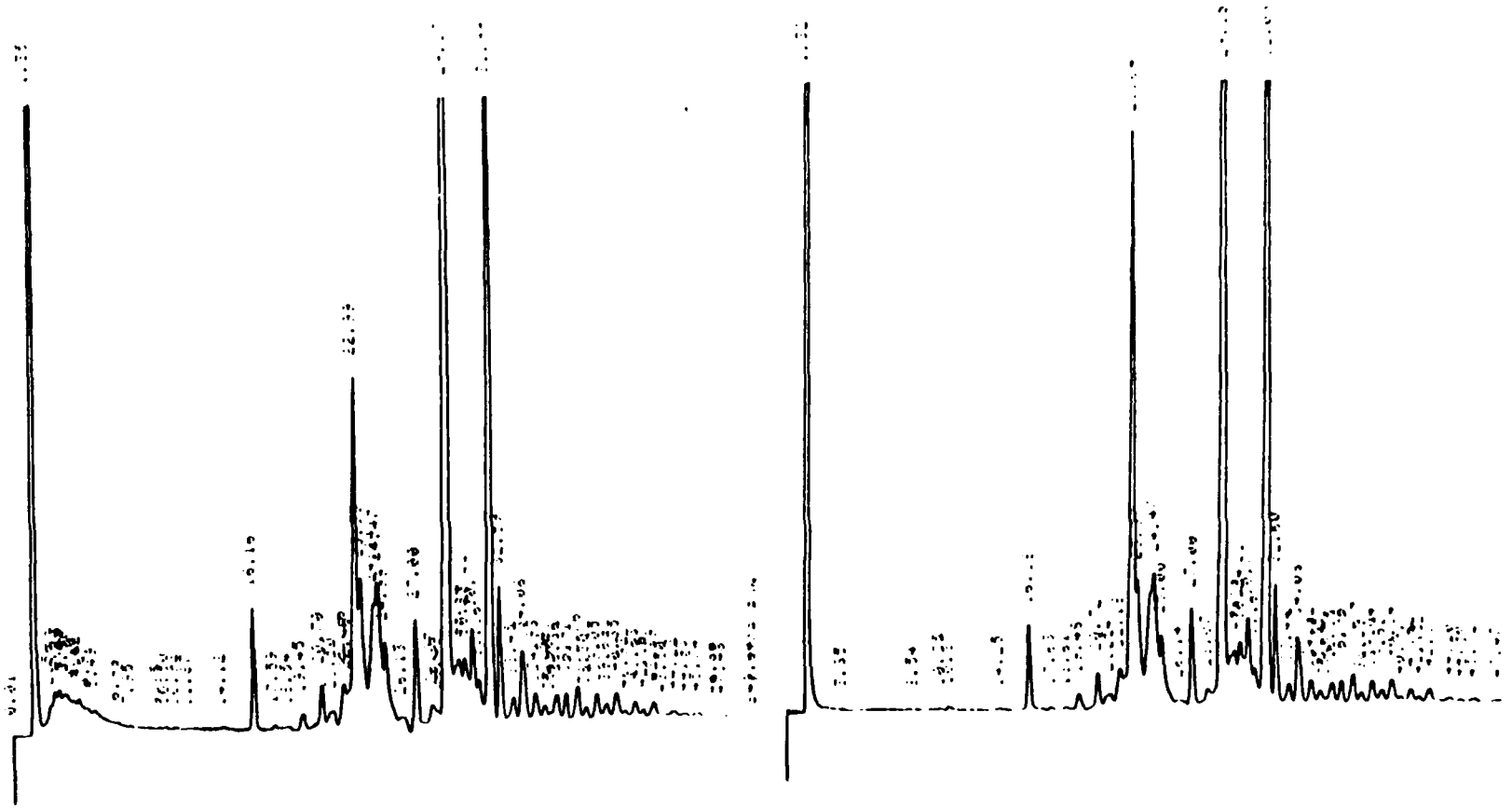
Figure 13. Gas chromatogram of used oils.



0-79-05

0-79-06

Figure 14. Gas chromatograms of used oils.



0-79-14 0-79-15

Figure 15. Gas chromatograms of used oils.

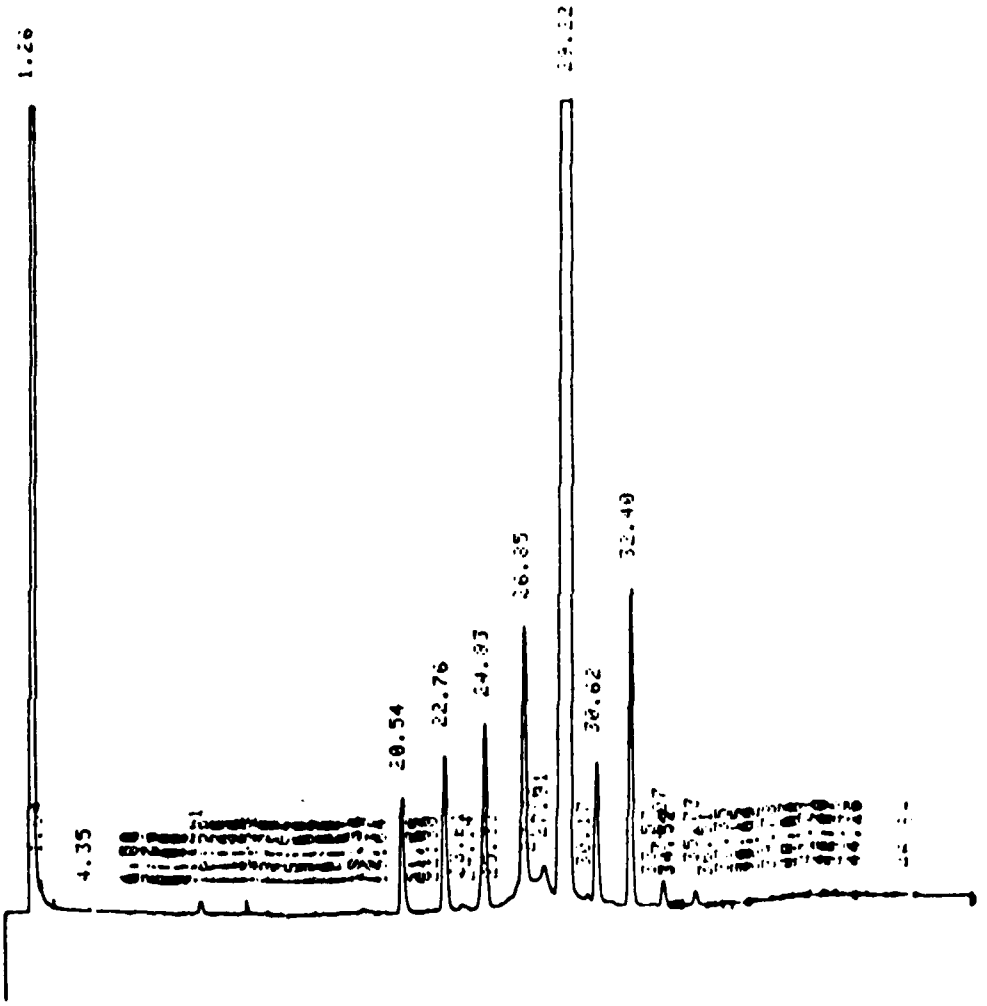


Figure 16. Gas chromatogram of commercially available di-2-ethylhexyl azelate base stock.

cresols. Note that these two oils had very high acid numbers, possibly attributable to the high cresol concentration at approximately 24+ min. Sample 0-79-09 is also unique and not readily comparable with the other 14 oils. Representative chromatograms of each group are shown in Figures 17 and 18. Chromatograms of all the used oils are provided in Appendix D.

3.2 OIL CHARACTERIZATION

Six new oils, ATL 9118 through 9123, supplied by AFAPL represent new MIL-L-7808H that could ultimately be found in current used oil mixtures. These new oils were analyzed by HPLC and GC. Among the six oils, only four patterns were seen.

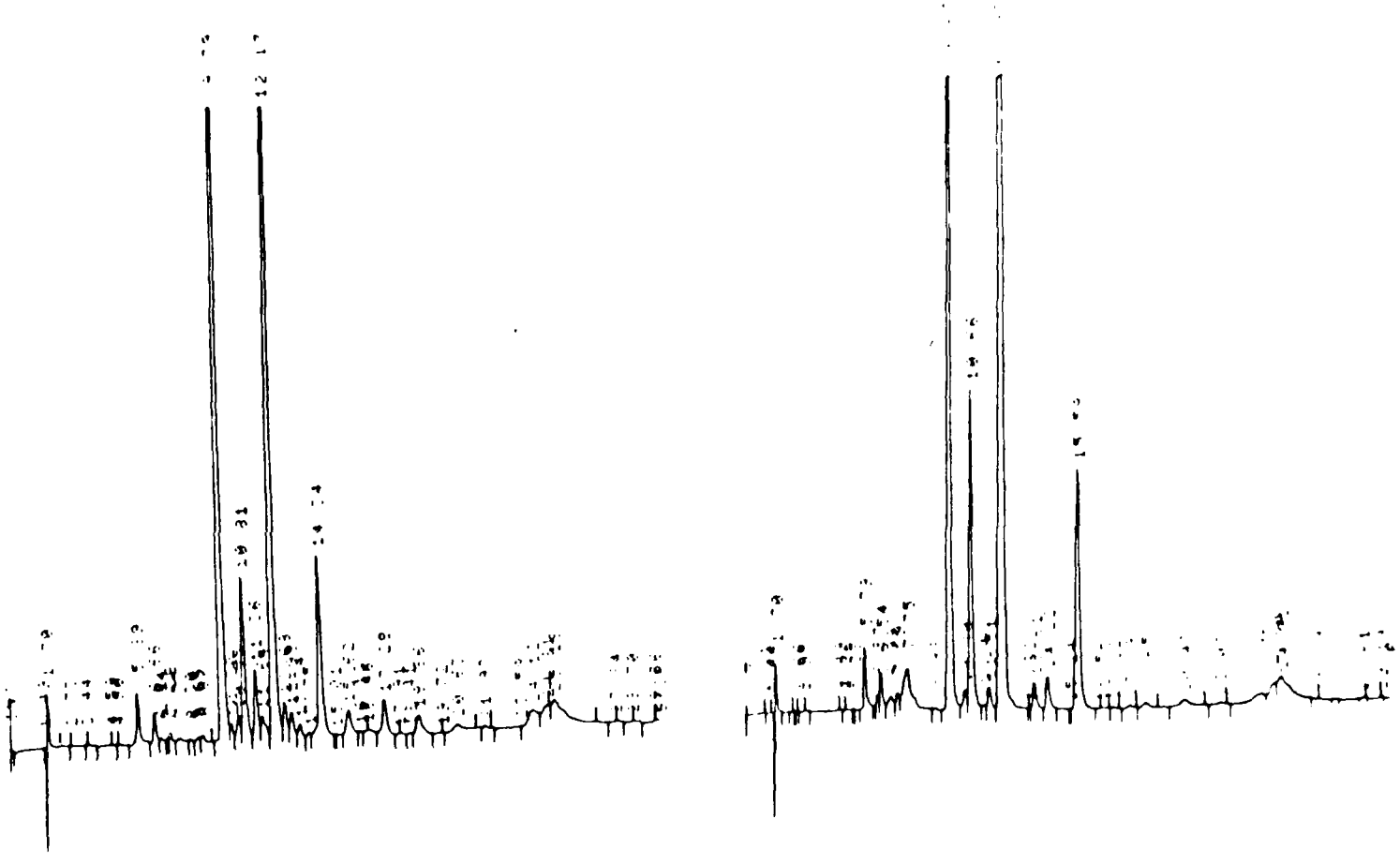
Three oils showed the same HPLC pattern: ATL 9118, 9119, and 9121 (the pattern is represented in Figure 19). They have peaks with the same retention times (rt's) as 4,4'-di-octyldiphenylamine (DODPA) or triphenyl phosphite (TPP), approximately 9.5-10 min; a peak corresponding to the rt's of 3,7-dioctylphenothiazine or N-phenyl-α-naphthylamine approximately 12.0-12.5 min; and a group of peaks corresponding the rt's of tricresyl phosphate and other cresols, 24.0 to 26 min. Sample ATL 9123 (Figure 20) has an additional major peak that corresponds to the rt of phenothiazine, approximately 15.7 min.

Samples ATL 9120 and 9122 (Figure 21) have certain identifiable peaks and, as of yet, major unidentified peaks. Many of these additive peaks are seen in the used oils also, but absolute identification was not completed.

GC's of the new oils are presented in Figures 22, 23, and 24. Molecular weight distribution in ATL 9118 and 9121, also between 9120 and 9123, would suggest possibly the same base stock components.

Throughout this report, the following designations will be used for oils at various stages of reclamation to more readily indicate the state in the reclamation process for any particular sample and eliminate the necessity for lengthy identification of each sample.

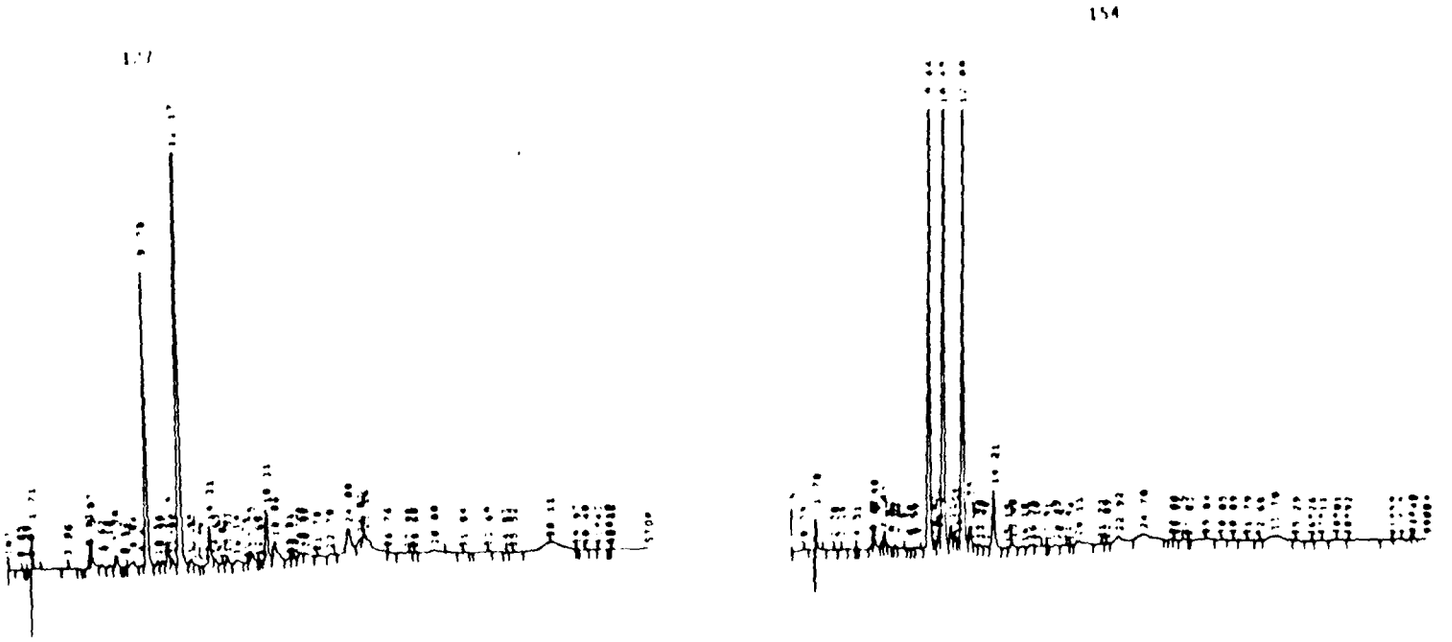
Designation	Sample description
Used oil	Oil as received from AFAPL.
Reclaimed base stock	Oil that has been processed, no replenishment of additives.
Reclaimed oil	Oil that has been processed and additives replenished.



0-79-01

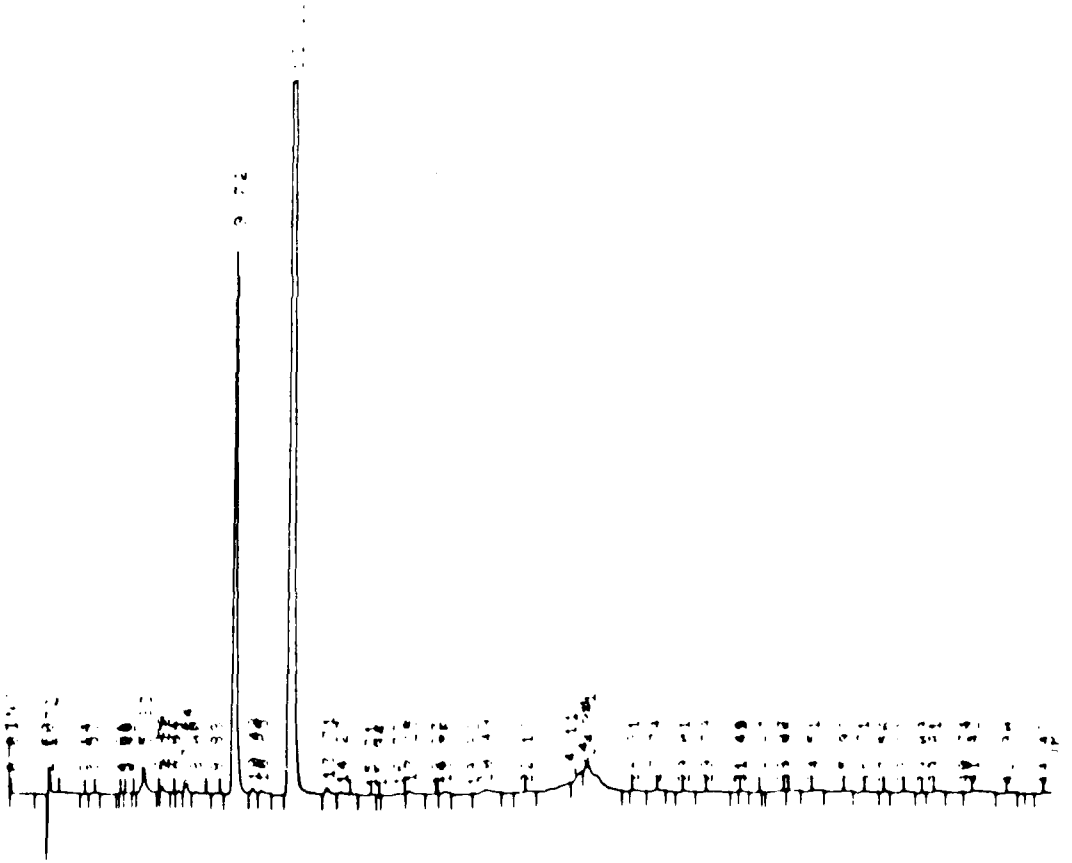
0-79-03

Figure 17. High pressure liquid chromatography of used oils.



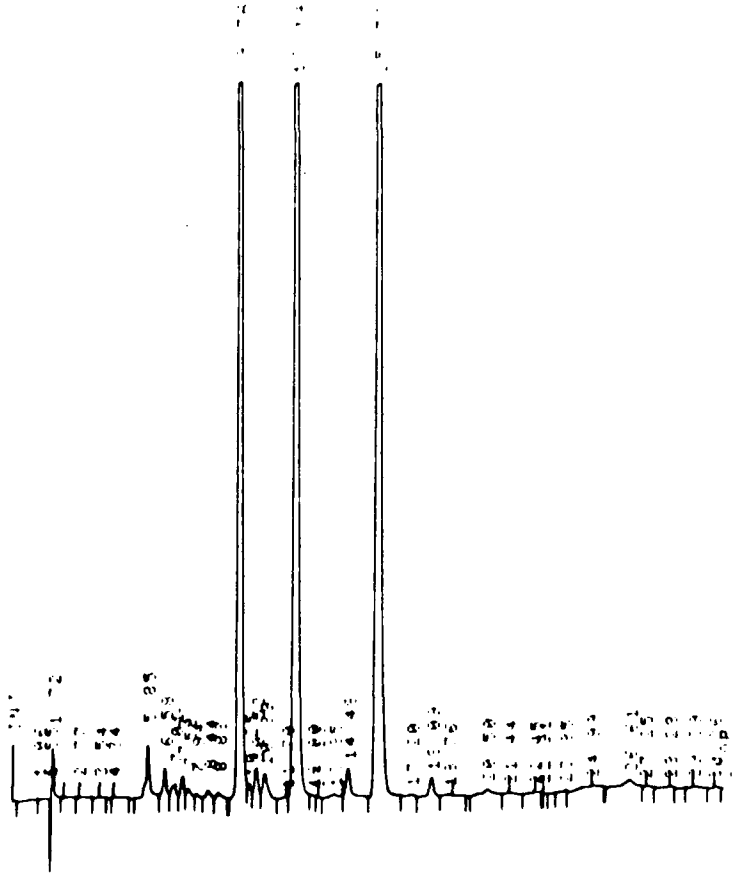
0-79-05 0-79-09

Figure 18. High pressure liquid chromatography of used oils.



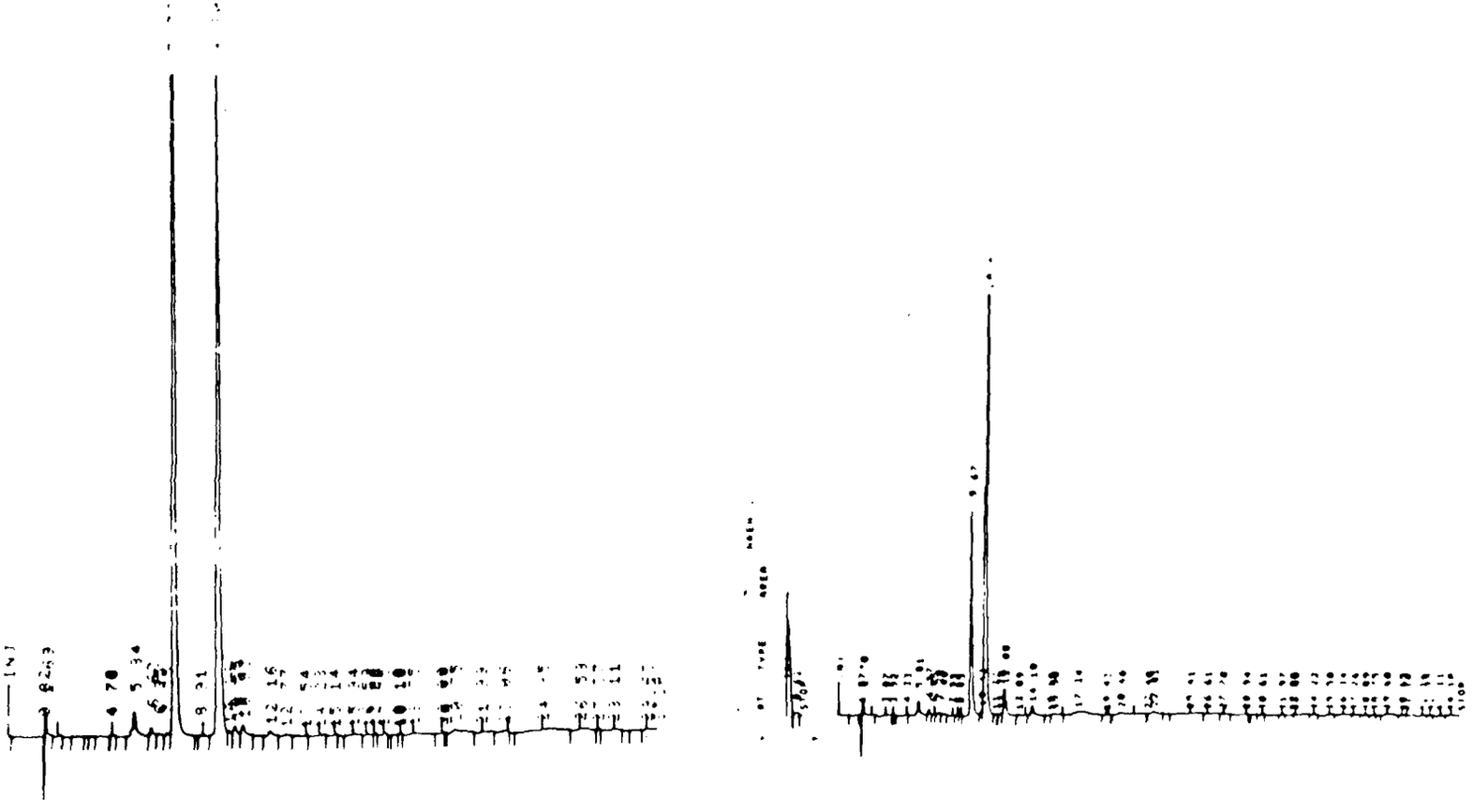
ATL 9121

Figure 19. HPLC representative of new oil ATL 9121.



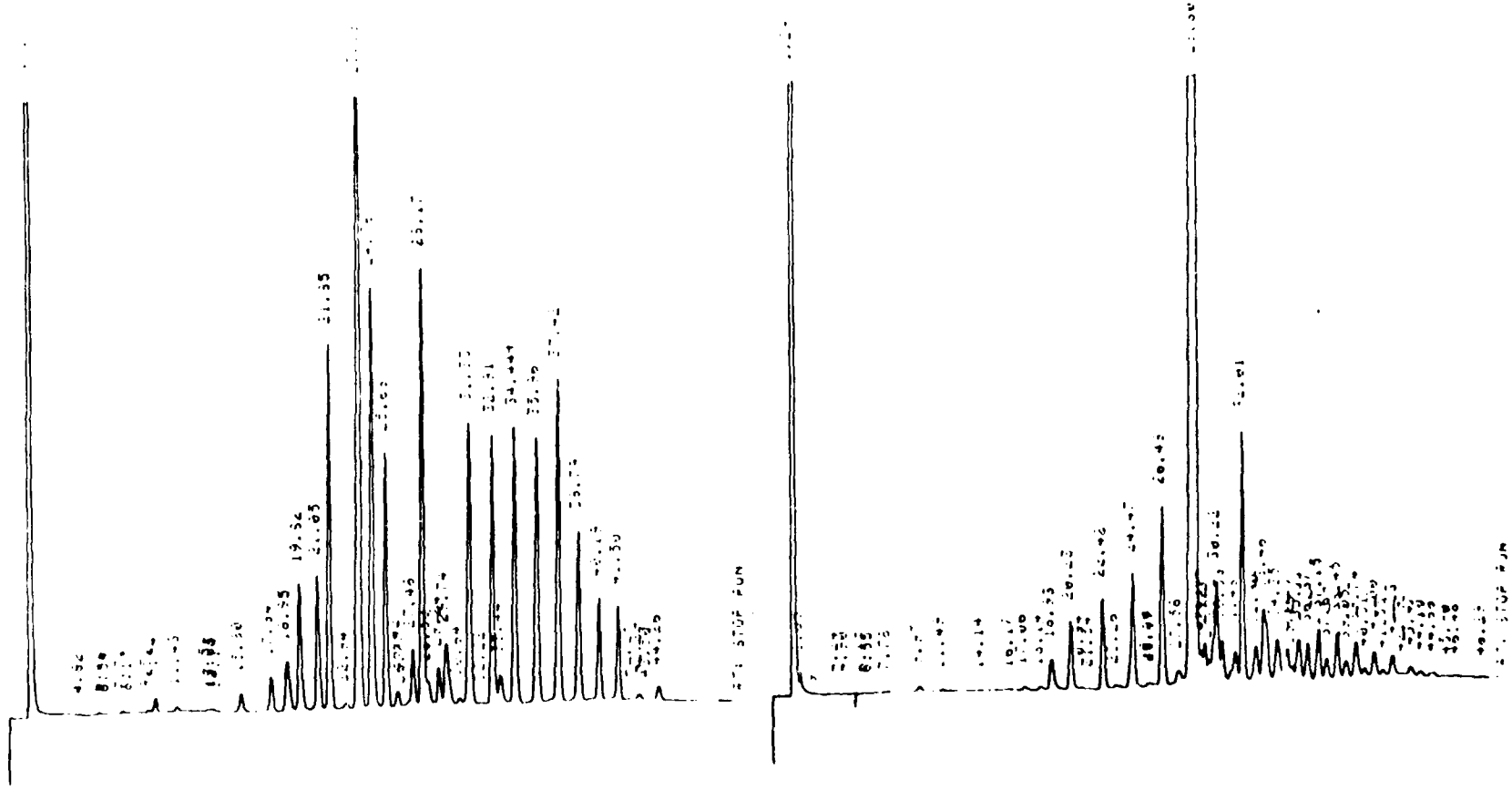
ATL 9123

Figure 20. HPLC representative of new oil ATL 9123.



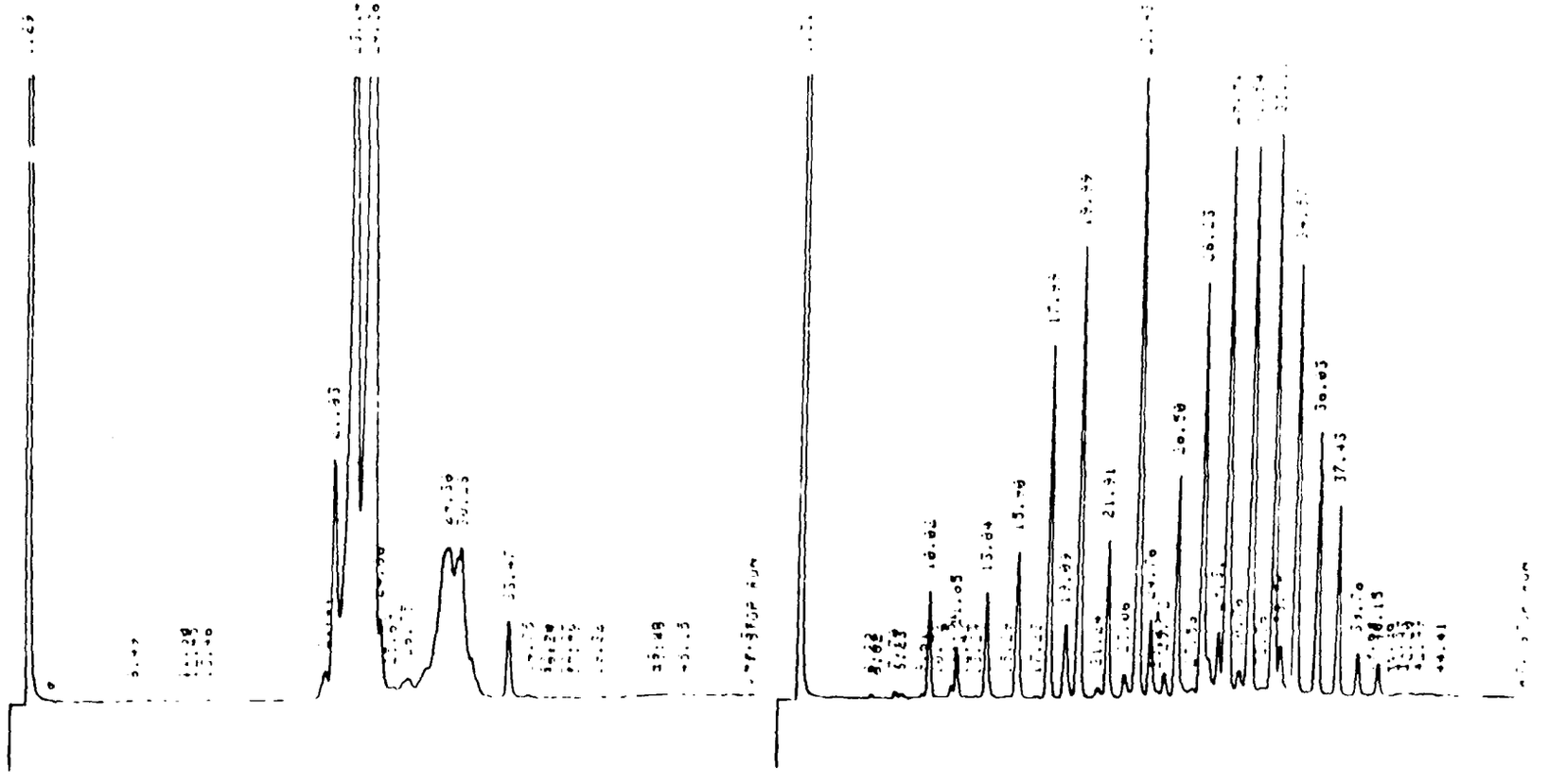
ATL 9120 ATL 9122

Figure 21. HPLC representative of new oils ATL 9120 and 9122.



ATL 9118 ATL 9119

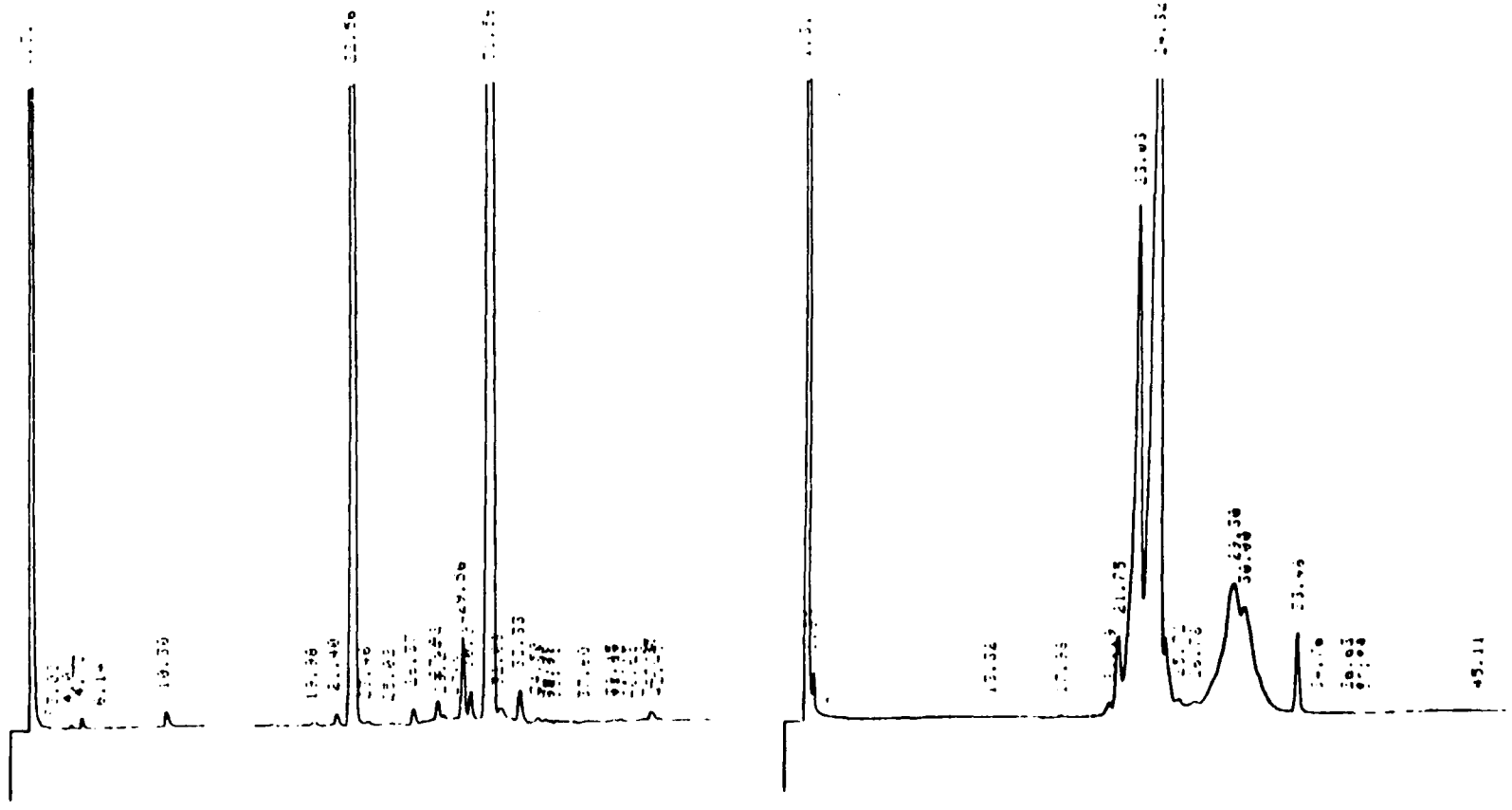
Figure 22. Gas chromatograms of new oils.



ATL 9120

ATL 9121

Figure 23. Gas chromatograms of new oils.



ATL 9122

ATL 9123

Figure 24. Gas chromatograms of new oils.

3.3 ADDITIVE PACKAGE VALIDATION

To validate our proposed additive package, GC's were run on six new oil samples supplied by APL, representing various manufacturers. Gas chromatograms labeled ATL 9118 through 9123 are seen in Figures 22, 23, and 24. From these chromatograms, we were able to select five base stock blends (without additives), supplied by various manufacturers and APL, that closely resembled the new oils with which to validate MRC's additive package.

3.3.1 Selection of Base Stocks for Use in Additive Package Validation

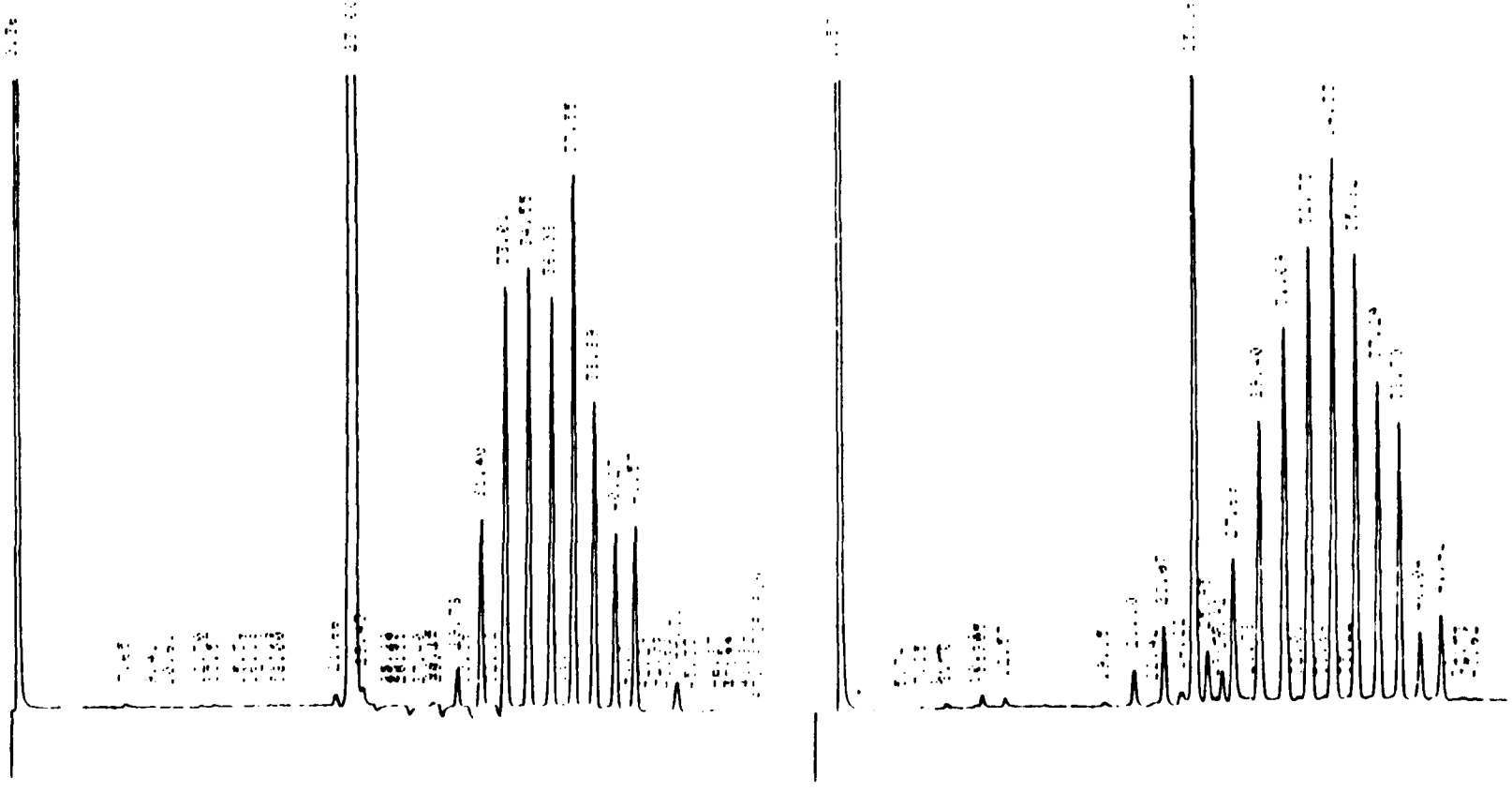
The following MIL-L-7808 type base stock and base stock blends were received from manufacturers:

Stauffer Chemical Company	~10 gal 7808H
Hatco	5 gal 7808H
Royal Lube	10 gal 7808H
ATL 9148 (from APL)	5 gal mix of polyols
ATL 9149 (from APL)	5 gal mix of diesters and polyols
Rohm and Haas	5 gal diisooctyl adipate
	5 gal diisodecyl adipate
Emery Industries	5 gal di-2-ethylhexyl azelate
	5 gal trimethylol propane triester
	10 gal 5-cst polyol ester
Hercules	5 gal Herculube 401

Figures 25, 26, and 27 show gas chromatograms of these base stocks. Figure 28 shows a base stock formulated (1:1:1:1 Royal Lube/Emery 2958/Emery 2932/Herculube 401) and tested with the additive package from the previous program (F33615-76-C-2037), from which a gas chromatogram was not run at the time. Sample 1568834-2A (see Figure 29) represents another, similar sample which was also tested on the prior program.

Our initial tries at blending for viscosity requirements (Figures 29 and 30) show that GC patterns of blended base stocks somewhat match some of the new oils supplied by APL. By using 76.9% Plexol 244 (diisooctyl adipate, Rohm and Haas) and 23.1% Plexol 273 (diisodecyl adipate, Rohm and Haas) a pattern similar to those of oils 9120 and 9123 was produced. A combination of 85% Emery 2958 (di-2-ethylhexyl azelate Emery Lubricants) and 15% Emery 2932 (trimethylol propane triester, Emery Lubricants) gave a pattern somewhat similar to those of oils 9121 and 9118. 92.6% Emery 2958 and 7.4% Emery 2939 (a 5-cSt polyol ester), produced a chromatogram similar to that of new oil 9119.

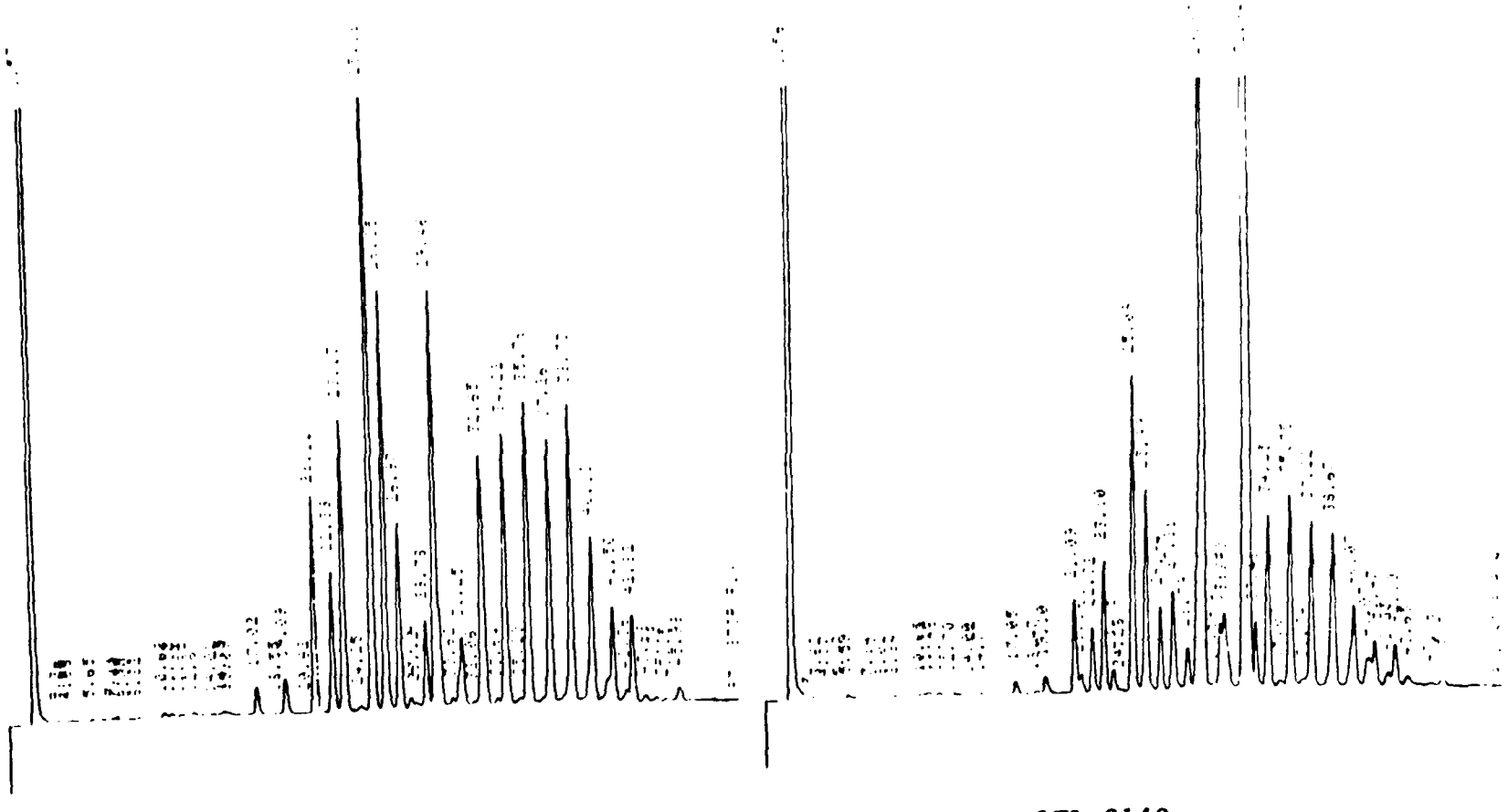
From these data we formulated and tested five additional base stocks with our additive package: ATL 9149, ATL 9148, Stauffer base stock, Hatco base stock, and a base stock of Plexol 244 and Plexol 273 (see Figure 29). They represent a wide variety of base stock formulations and seem to represent most of the used oils we have received and many of the new oil formulations we have examined.



Hatco

Royal Lube

Figure 25. Gas chromatograms of formulated base stocks.



ATL 9148

ATL 9149

Figure 26. Gas chromatograms of formulated base stocks.

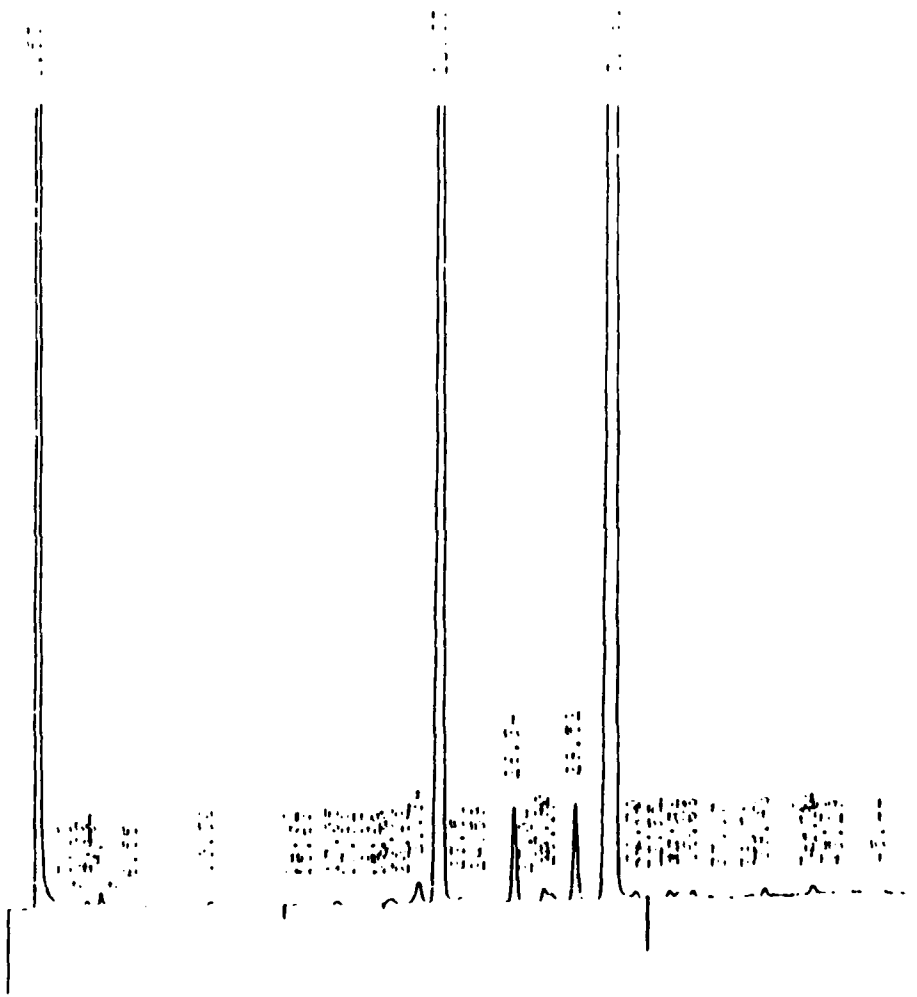
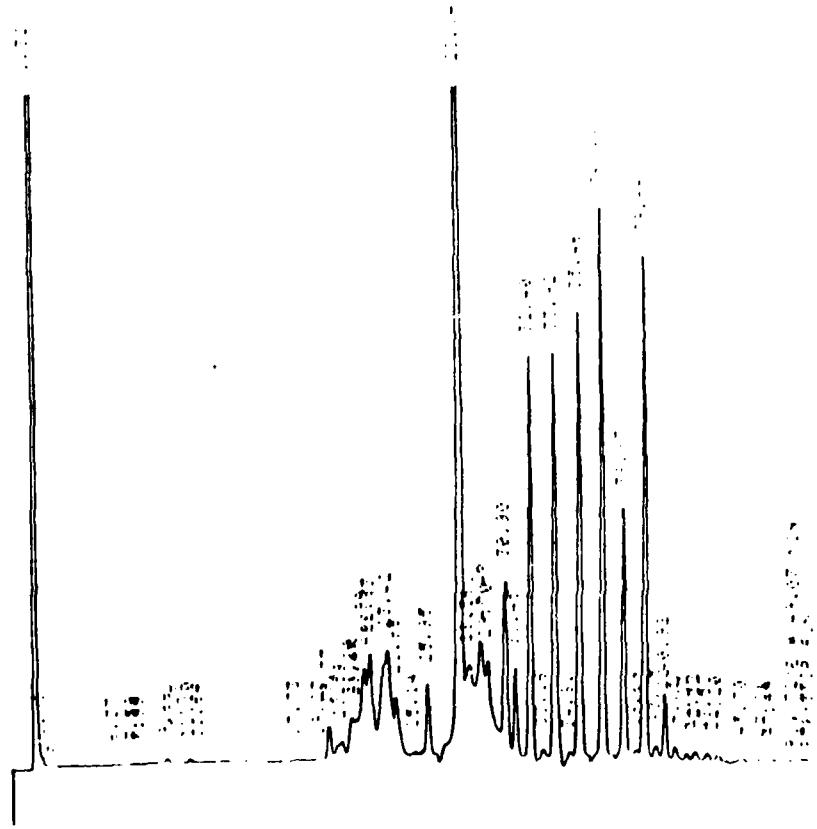


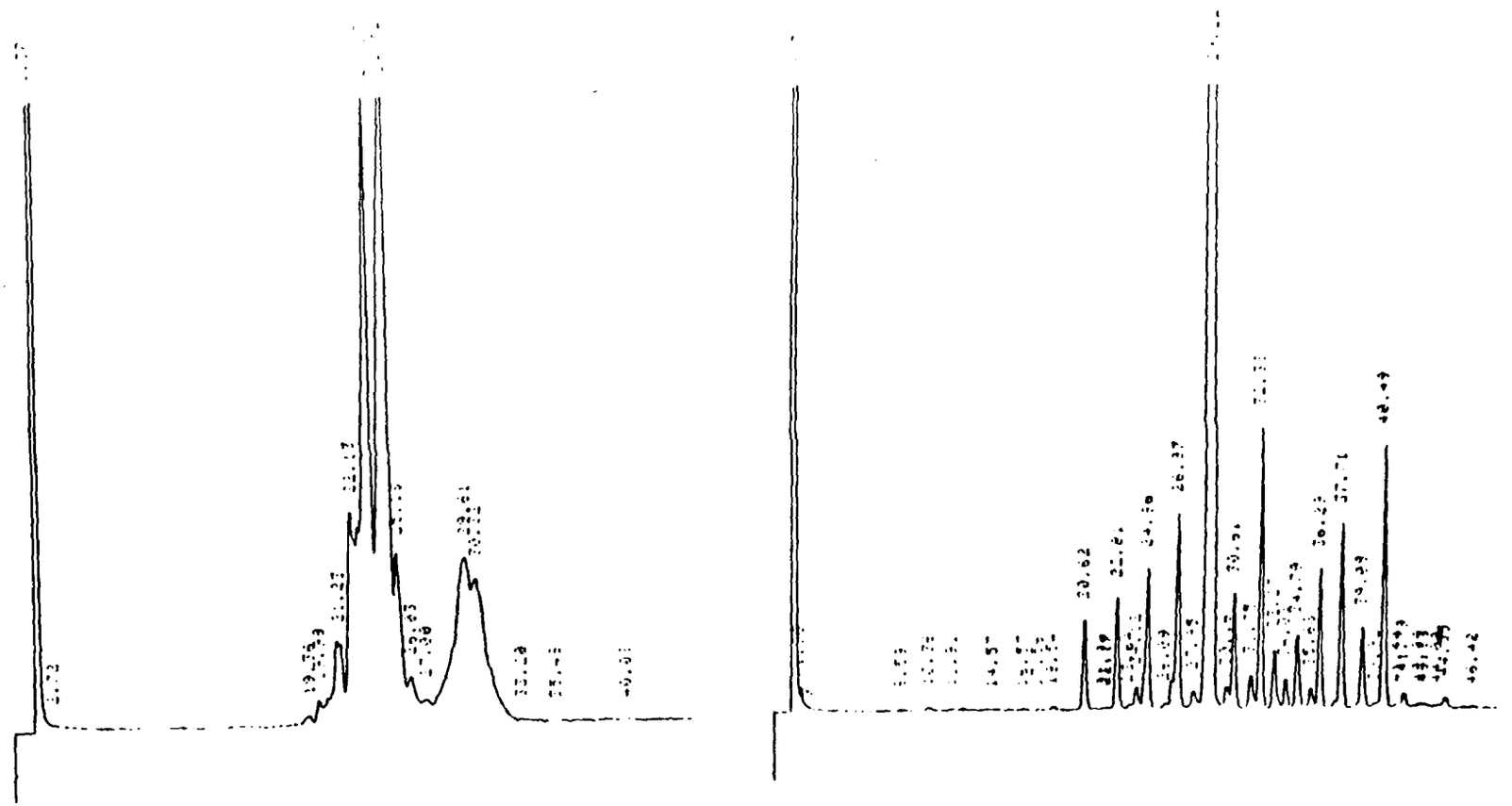
Figure 27. Gas chromatogram of Stauffer formulated base stock.



1:1:1:1

Royal Lube Diester, Emery 2958 and 2932, and Herculube 401

Figure 28. Gas chromatogram of base stock mixture tested earlier with a proposed additive package.



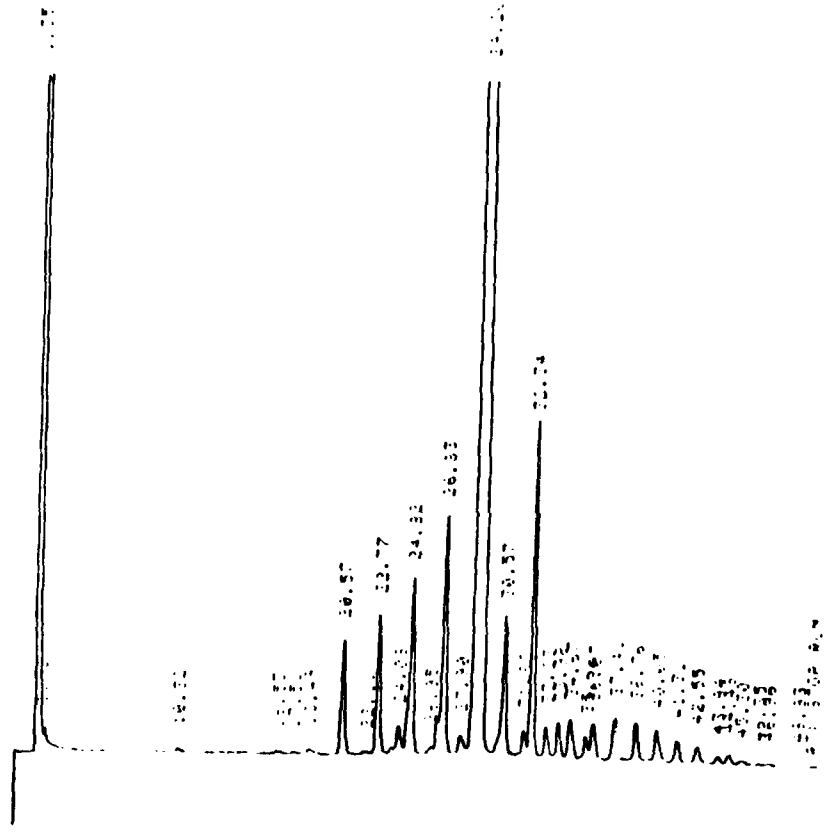
1568834-1

76.9% Plexol 244/23.1% Plexol 273

1568834-2A

85% Emery 2958/15% Emery 2932

Figure 29. Gas chromatograms of blends of commercially available base stocks.



1568834-3

92.6% Emery 2958/7.4% Emery 2939

Figure 30. Gas chromatogram of a blend of commercially available base stocks.

3.3.2 MRC's Additive Package Added to Selected Base Stocks

The 3,7-dioctylphenothiazine (DOPTA) used in the earlier program was replaced with phenyl-α-naphthylamine (PANA) since DOPTA is no longer produced in the United States. We also wanted to determine if the additive had a singular effect on the foam testing of a formulated base stock. Royal Lube base stock was formlated with all of the additives minus PANA and exhibited a foam volume of 5 mL. The formulated base stock with PANA had a foam volume of 45 mL. Additional testing of this base stock with PANA gave similar results (40-45 mL foam volume).

When informed of this foaming problem, the manufacturer provided a new supply of PANA, which produced a foam volume of 15 mL. This newer, cleaner appearing PANA was used to formulate the virgin base stocks.

The five selected virgin base stocks (Hatco, Stauffer, ATL 9148, ATL 9149, and Plexol) were formulated with:

1.0% DODPA (dioctyldiphenylamine) - Van Lube 81
1.0% PANA (phenyl-α-naphthylamine) - Uniroyal
2.0% TCP (tricresyl phosphate) - Kronitrex AA FMC
0.1% Ethyl antioxidant 703 - Ethyl Corp.
0.1% TPP (triphenyl phosphite) - Eastman
0.1% Benzotriazole - photo grade - Sherwin Williams
0.05% Quinizarin (GAF - purified)

These oils were sent to Alcor for specific testing. They were evaluated in the following sequence to allow detection of the most common modes of failure.

1. Total acid number
2. Static foaming characteristics, test method 3213
3. Viscosity at -65°F, 100°F, and 210°F
4. Lead corrosion
5. Corrosion and oxidation stability, 96 hr at 392°F
6. Dynamic foaming characteristics
7. FA elastomer compatibility at 347°F
8. Gear load carrying rating (one gear, two determinations)
9. Silver and bronze corrosion
10. Deposition number

The results of these tests are included in Appendix A of this report. MRC's proposed additive package in the five selected base stocks had met all test specifications except the static foam test in one of the base stocks furnished by APL. A comparison of the MRC versus Alcor static and dynamic foam test results on the five formulated base stocks (see Table 2) suggests that the static foam volume of 115 mL for oil 1732509 (ATL 9149),

TABLE 2. SELECTED DATA FROM MIL-L-7808H TESTING

Oil	MRC static foam volume,[a] mL	Alcor static foam volume,[a] mL	Acid number[b]	Alcor dynamic foam volume,[a,c] mL	Viscosity at 210°F, cSt	Viscosity at -65°F, cSt 35 min
Hatco 1732506	15	15	0.20	10	3.0	12,136
Stauffer 1732510	25	30	0.25	10	3.1	13,225
Rohm and Haas 1732511	35	35	0.14	10	3.0	11,341
ATL 9148 1732508	60	100	0.22	10	3.4	14,462
ATL 9149 1732509	90	115	0.17	10	3.5	16,642
ATL 9148[d]	5					
ATL 9149[d]	0					

[a]Maximum 100 mL.

[b]Maximum allowable acid number is 0.30.

[c]Foam volumes same at both 176°F and 230°F, 1000 cc air.

[d]Base stock with no additives; only static foam volume tested.

is only a marginal failure. Another oil (1732511, Rohm and Haas), though not out of specifications as far as corrosion and oxidation stability (96 hr at 392°F), did exhibit high magnesium corrosion and oxidation values.

Effects on acid numbers, viscosity, and foam volume of the same additive package in various MIL-L-7808 fluids, produced by unique manufacturing processes, are seen in Table 2. The base stocks received from AFAPL seem to be much more affected by the formulation process than the commercially available virgin base stocks. The cause of this effect on the APL base stocks is unknown.

The interaction on PANA with the APL-supplied base stocks may contribute to the higher than normal foam volumes. After the virgin base stocks had already been formulated, PANA from another supplier arrived for testing. Table 3 compares test results for PANA from Union Carbide and from Uniroyal; both produced equal response. The data suggest base stock interaction with the additive package, specifically with PANA.

TABLE 3. COMPARING FOAM TEST RESULTS PANA FROM TWO SUPPLIERS

MRC's additive package with	Foam test volume, mL	
	Royal lube base stock	ATL 9149 base stock
Uniroyal PANA	15	115
Union Carbide PANA	25	120

3.3.3 Effect of Additive Package on Viscosity

From the previous contract, an increase in viscosity was expected from the addition of our proposed additive package to base stock oils. Results were much more sporadic than expected. These random viscosity increases (see Table 4) fall into distinct patterns, making future viscosity increase predictions much more difficult.

TABLE 4. VISCOSITY INCREASES DUE TO ADDITIVE ADDITION

	Percent increase when viscosity measured in cSt at			
	-20°F		100°F	
ATL 9149	16.9	Pattern 1	3.0	Pattern 1
ATL 9148	17.0		3.6	
Rohm and Haas	27.6	Pattern 2	10.0	Pattern 2
Stauffer	22.9		8.0	
Hatco	7.6	Pattern 3	0.4	Pattern 3

3.3.4 Chromatograms of Additives in Base Stock

Chromatograms of MRC's additive package in Stauffers base stock are shown in Figure 31. All the high performance liquid chromatograms of the formulated fluids tested by Alcor are the same. The gas chromatograms of the formulated fluids are duplications of those for the base stocks without additives, although PANA does appear at 18.24 min in each chromatogram.

3.3.5 Estimated Additive Shelf Life

According to their manufacturers, the additives contained in MRC's additive package have different shelf lives. DODPA and PANA should be tested yearly; quinizarin and ethyl antioxidant 703 should not be used if stored over 2 years; and TCP, benzotriazole (BT), and TPP are unaffected by storage.

3.4 DISTILLATION STUDY

Distillation ideally should be included in the early part of any reclamation process to remove any low boiling material (water, toluene, etc.) and major contamination such as degraded additives and polymeric esters. Removal of the major contaminants insures lower effective absorbent treatment levels in subsequent process steps.

3.4.1 Distillation of Neutralized Used Oils

In the earlier program, a straight takeover distillation was performed. In the current program, a literature search revealed a paper on commercial oil reclamation which suggests that treatment of oils with metallic sodium prior to distillation reduces the formation of nondistillable impurities [1]. The metallic sodium also reacts to form nondistillable compounds with some degradation products. Since any water of low boilers would normally be removed during the topping step on distillation, it was felt we could add aqueous or alcoholic caustic solutions to the oil prior to distillation and possibly achieve the same effects. During this study, sodium hydroxide (NaOH) at 5% volume dissolved in water/isopropyl alcohol (1/1) was added to the oil just prior to distillation. The amount of NaOH added to the oil was based on the stoichiometric amount (or proportions thereof) necessary to neutralize the acid number. Figure 32 shows a graph comparing the effects of varying the percentage of NaOH on the acid number of the 2nd (main) and 3rd (secondary) fractions. The samples were distilled under the same conditions and cut-off points. Oil 0-79-14 was used for this experiment. Acid numbers of the same oil distilled without NaOH treatment are presented with the graph.

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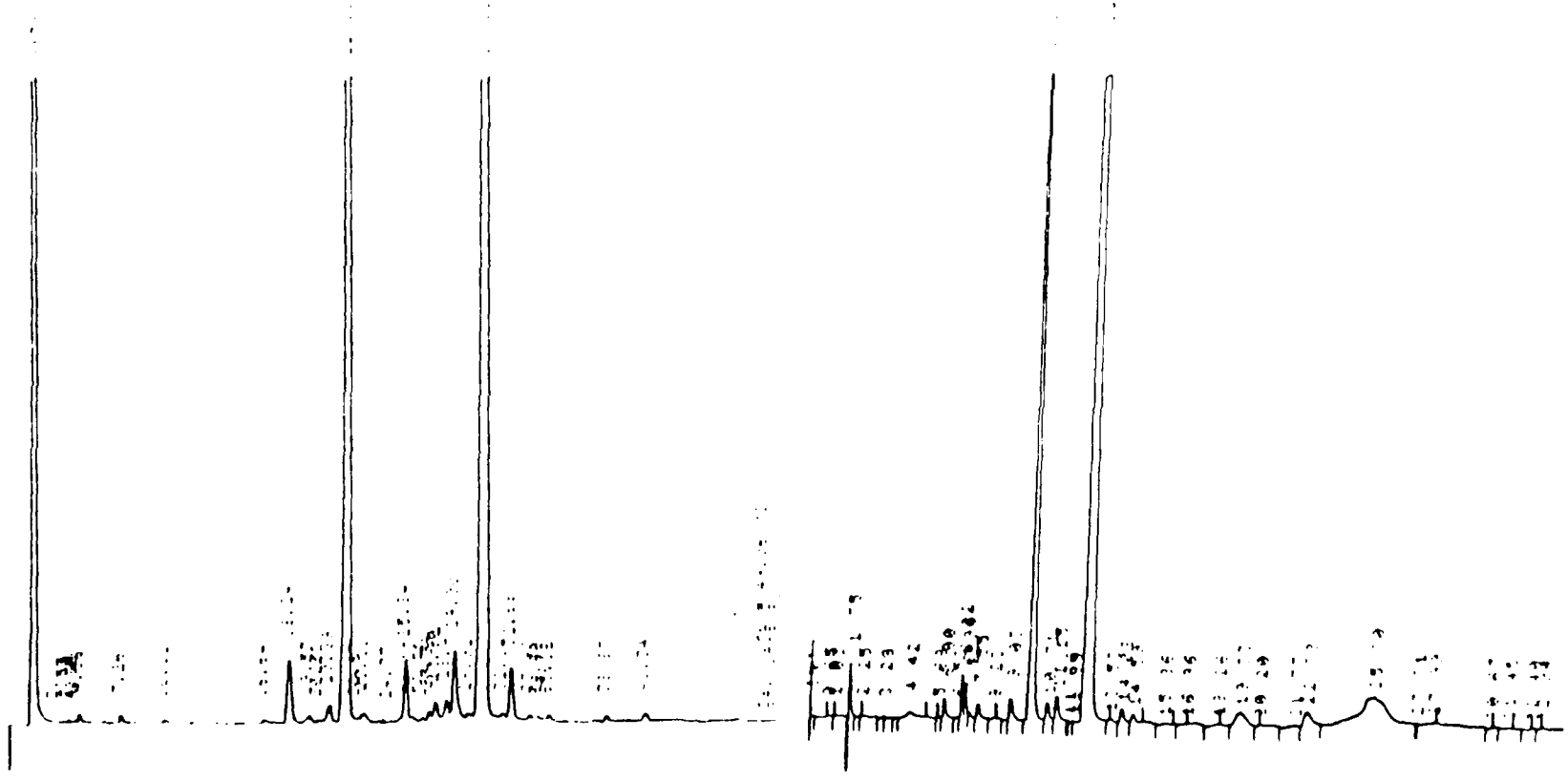
[1] Reynolds, J. W.; Whisman, M. L.; Brinkman, D. W.; Goetzinger, J. W.; Cotton, F. O. "From Oil:Oil," Chemtech, October 1979.



Gas Chromatogram

High Performance Liquid Chromatogram

Figure 31. Chromatograms of Stauffer base stock with MRC additive package.

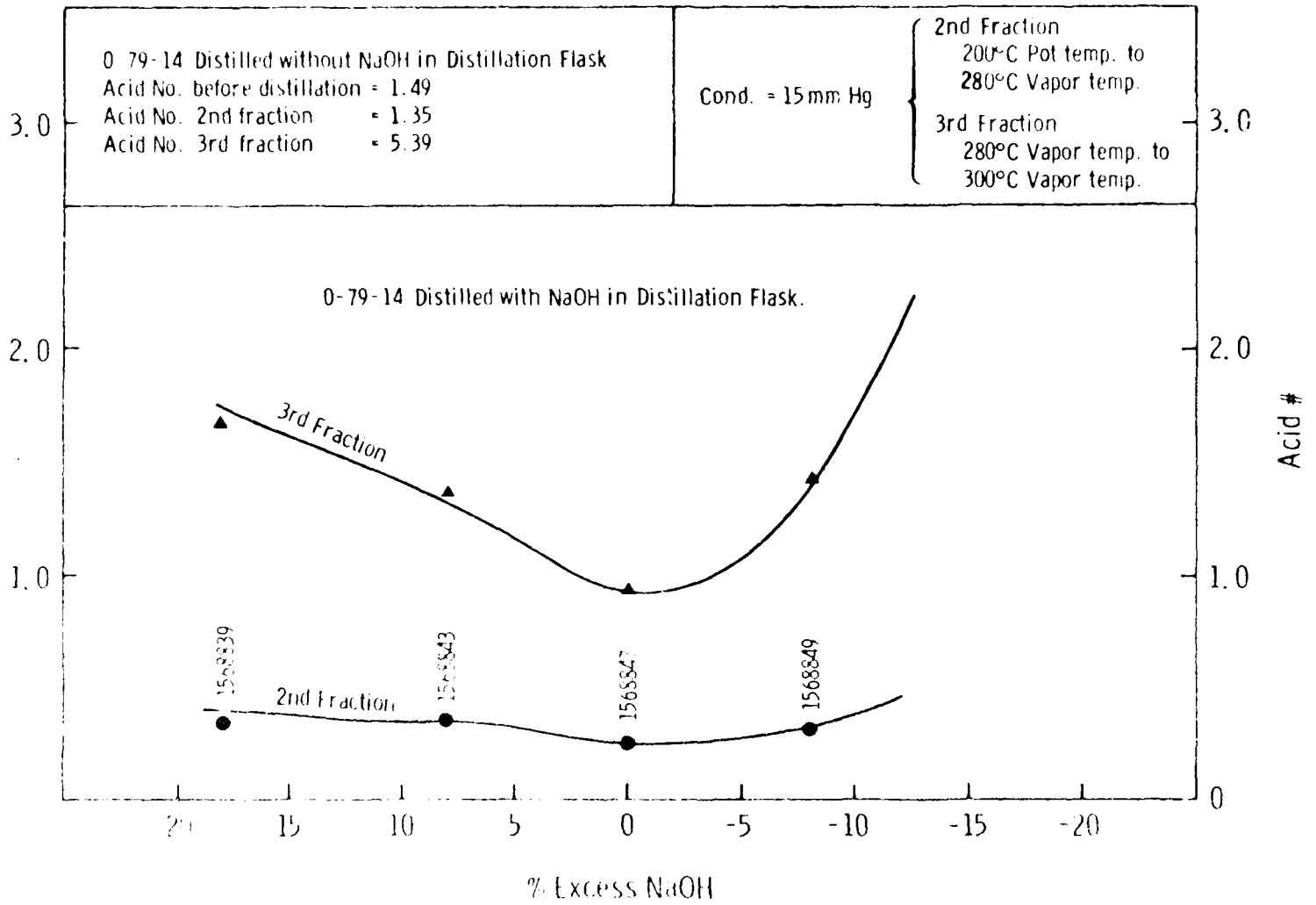


Figure 32. Acid numbers of distillate fractions, after varying NaOH level in used oil 0-79-14.

Because of improved acid numbers and obvious visual improvements in color, additional studies were warranted.

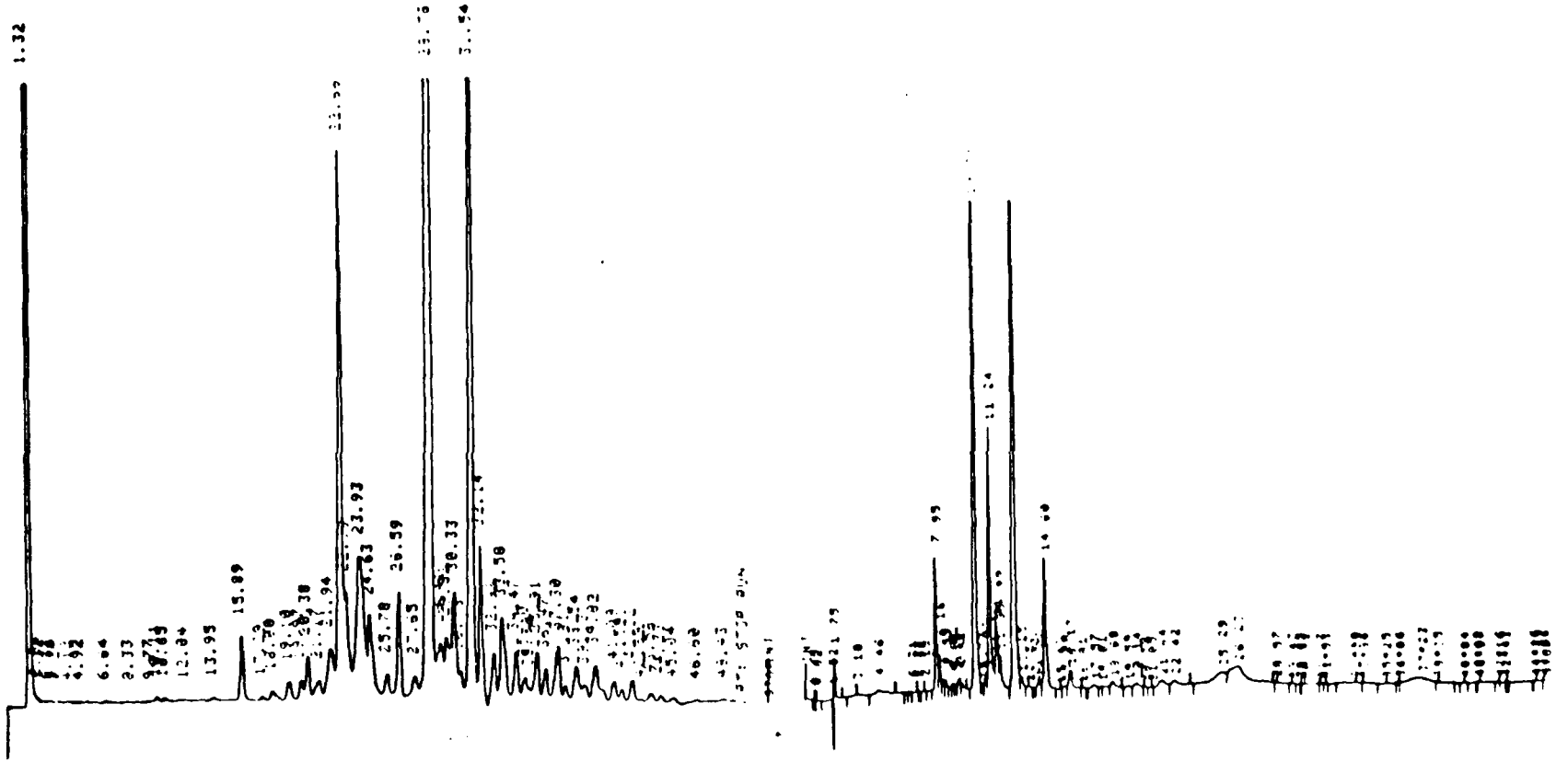
Two samples of a mixture of all 15 oils in the volume proportions received (see Figure 33 for HPLC and gas chromatograms) were distilled, one treated with sufficient NaOH to effect total neutralization and the other without treatment. Samples were taken throughout distillation for analysis. Figure 34 shows results for the mixture distilled without caustic treatment and Figure 35 gives the corresponding results for the mixture treated with NaOH. The circles represent vapor temperature vs. acid number and the squares represent cumulative percent recovery vs. vapor temperature.

There is virtually no difference between the plots of cumulative recovery vs. vapor temperature, suggesting there is no deleterious effect on recovery by NaOH treatment. The acid number data show significant differences. The total amount of distilled material having low acid numbers is much greater in the NaOH treated sample. These lower acid numbers may mean that less postdistillation treatment will be required. This plot might also be used as an accurate means of determining a proper distillation cut-off point. HPLC analyses show no differences between the treated vs. nontreated samples. The treated samples were lighter colored, having fewer visible degradation products present. Thin layer chromatography (TLC) analysis did show removal of additional material from the NaOH-treated samples.

3.4.2 Analysis of Distilled Neutralized Used Oils

Various analyses (GC, HPLC, acid number, and foam testing) were performed on oil samples distilled with and without NaOH treatment. A GC comparison of three equal (vapor temperatures) points on the recovery curves (Figures 36-38) showed no differences between the treated and nontreated samples. HPLC comparisons of combined distillates in the same vapor temperature ranges are shown in Figure 39. No differences are seen. These combined samples were foam tested and acid numbers were run with the following results:

	Foam volume, mL	Acid number
1568887-A (156882 mixed fractions 3 through 11, distilled with no NaOH)	15	1.40
1568887-B (1568865 mixed fractions 4 through 11, distilled with NaOH)	10	0.35



Gas Chromatogram

High Performance Liquid Chromatogram

Figure 33. Chromatograms of the mixture of 15 used oil samples.

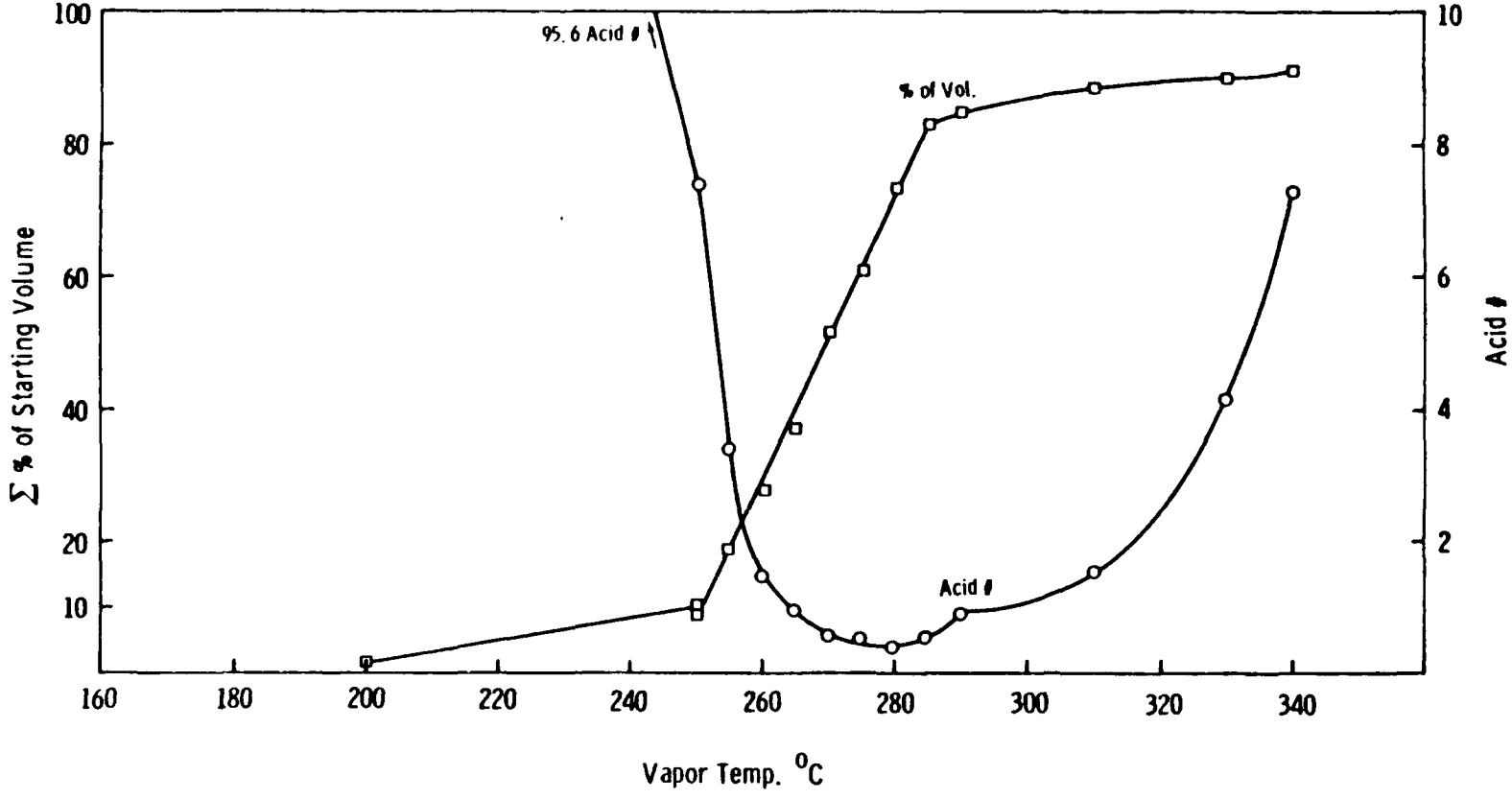


Figure 34. Sample 1568862, mixture of 15 used oils distilled with no NaOH (used oil starting acid number: 2.88).

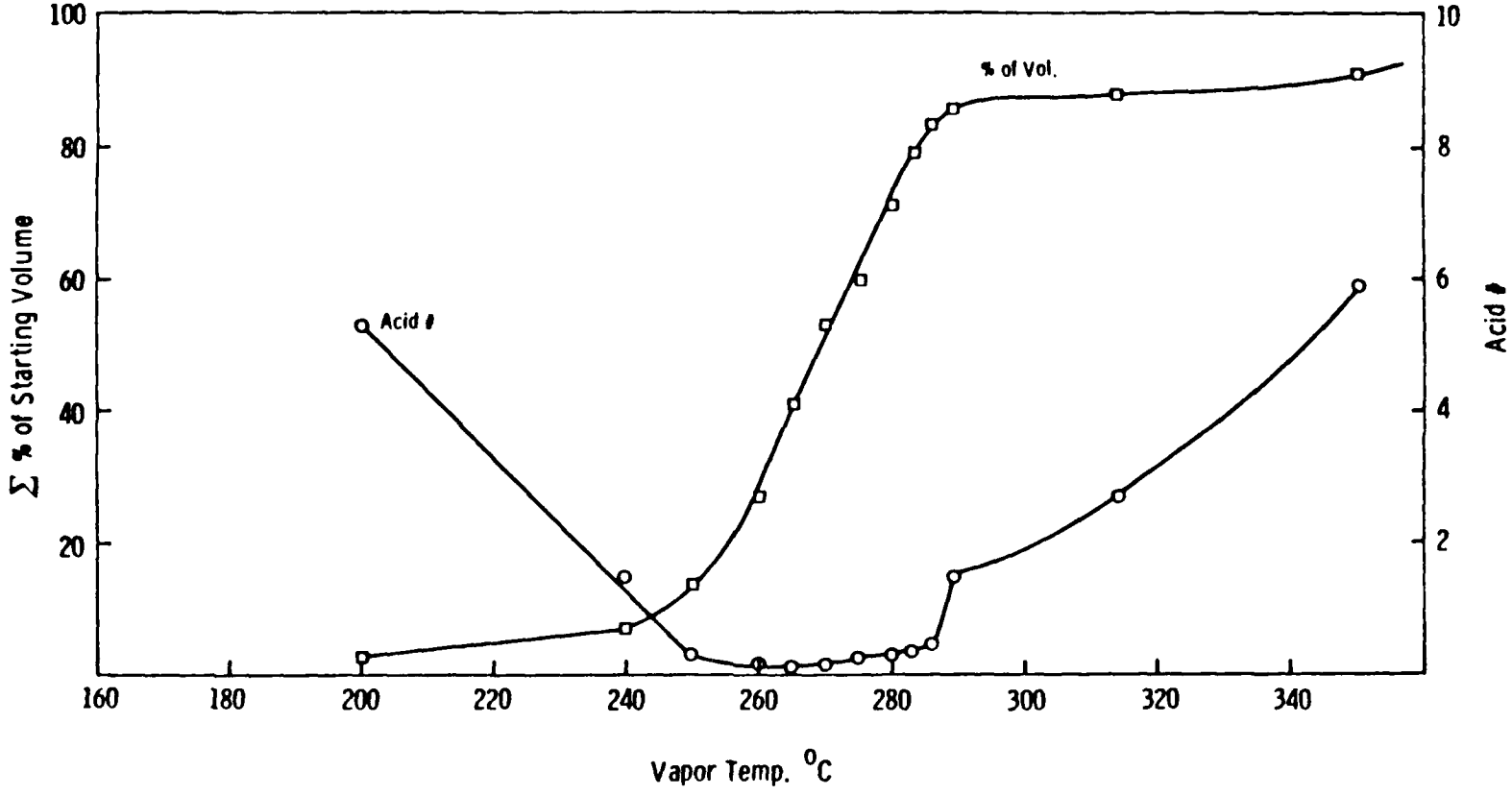
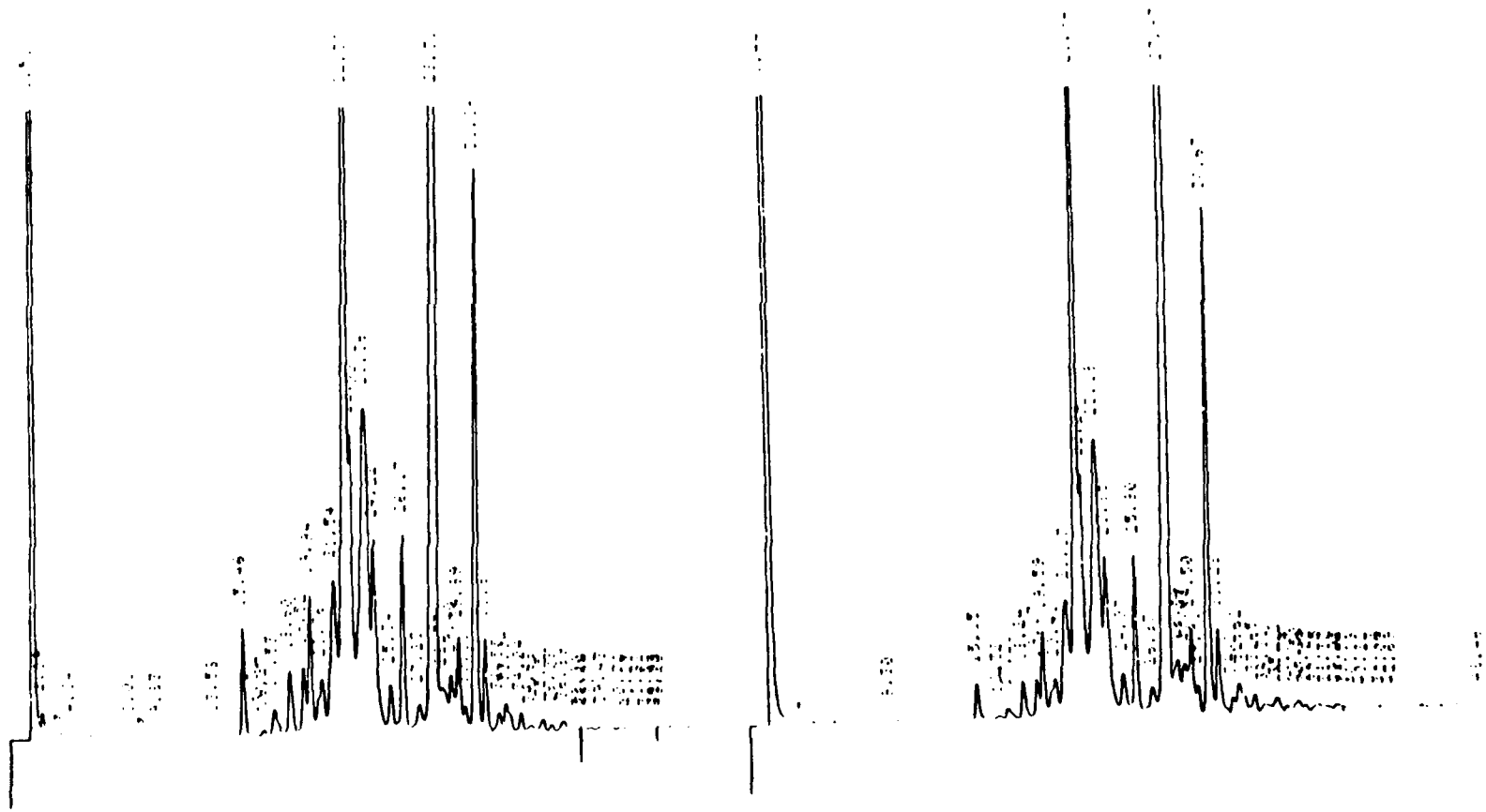


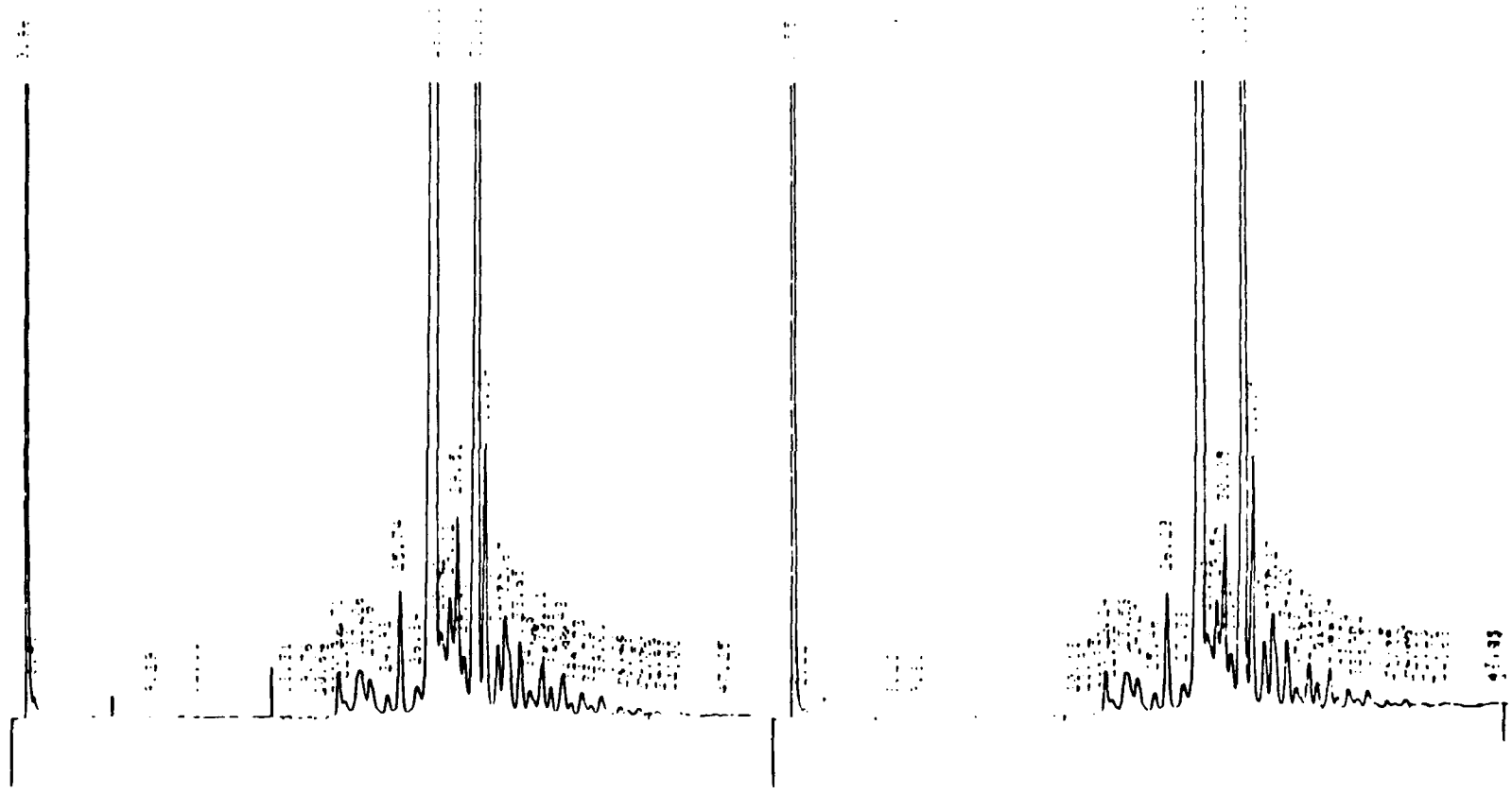
Figure 35. Sample 1568864, mixture of 15 used oils distilled with sufficient NaOH (added with water/isopropyl alcohol) for neutralization (used oil starting acid number: 2.88).



1568865-5 with NaOH

1568862-5 without NaOH

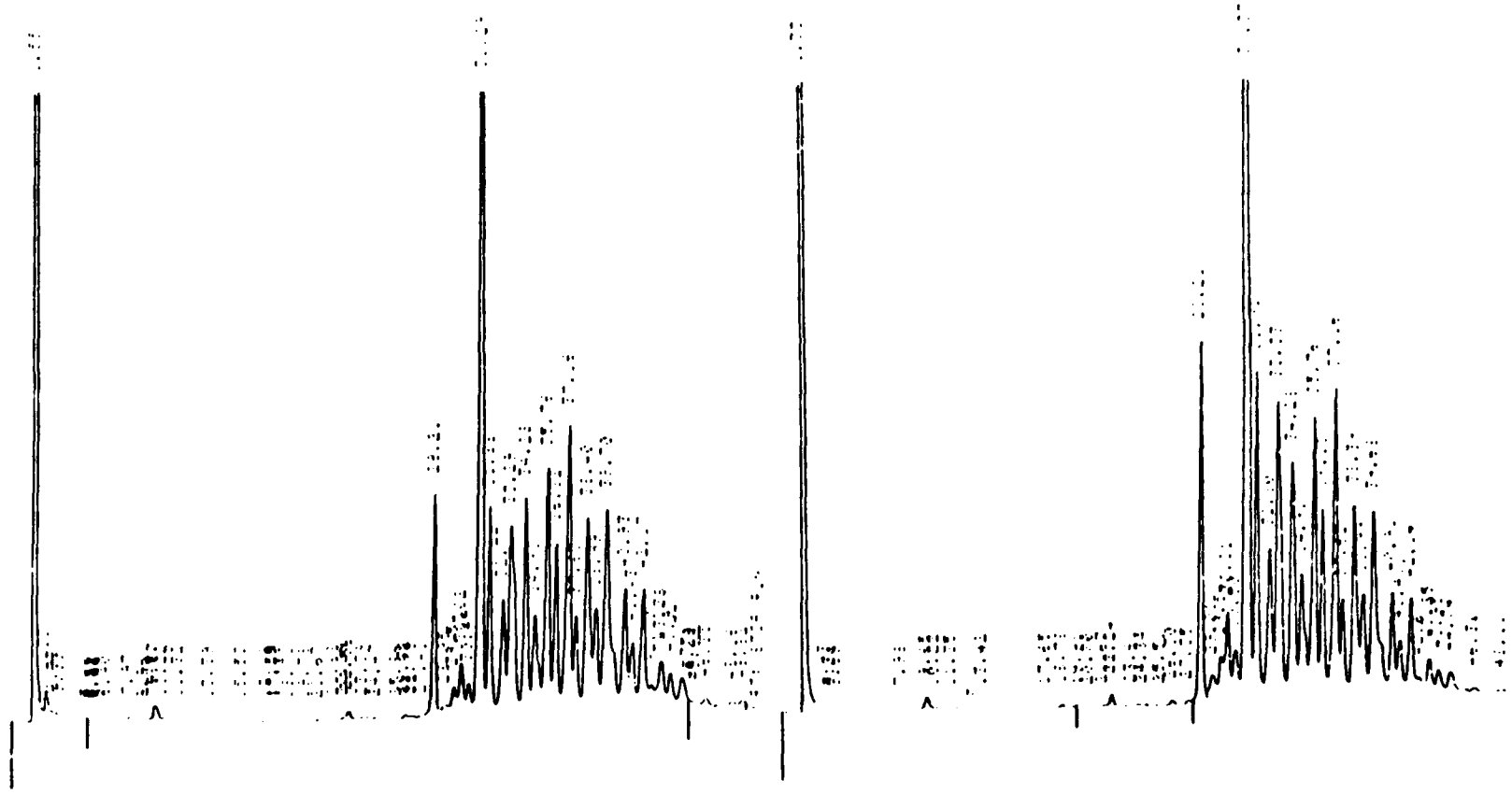
Figure 36. Gas chromatograms of distillation samples at 260°C vapor temperature.



1568865-9 with NaOH

1568862-9 without NaOH

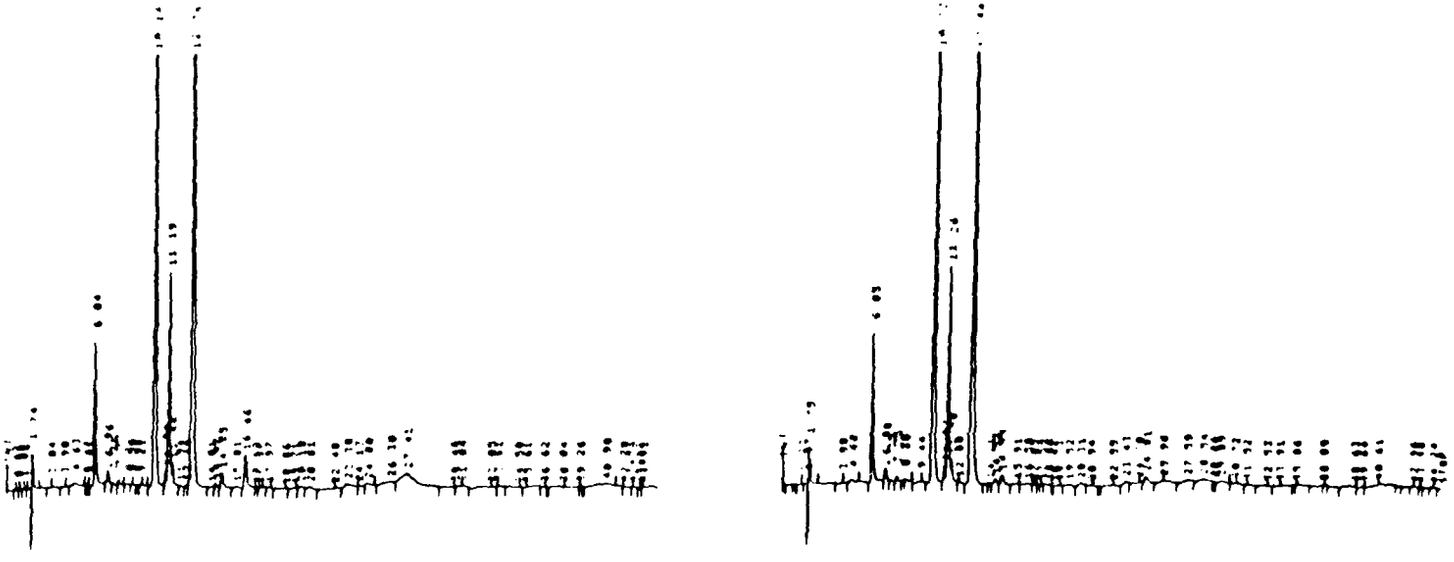
Figure 37. Gas chromatograms of distillation samples at 280°C vapor temperature.



1568865-13 with NaOH

1568862-12 without NaOH

Figure 38. Gas chromatograms of distillation samples at 310°C vapor temperature.



1568887-A without NaOH

1568887-B with NaOH

Figure 39. High performance liquid chromatogram of combined distillation samples.

Acid number was significantly improved by this treatment. Results show that NaOH (caustic) treatment does not adversely affect the combined distillates. In fact, the data suggest that such treatment improves acid number, color, and odor, and may eliminate some portions of posttreatment.

3.4.3 Substitution of Other Basic Material for Sodium Hydroxide

Because results of distillation with NaOH were so promising, we sought a more practical substitute. Since calcium hydroxide [$Ca(OH)_2$] can be added as a powder, its use would save total process time and energy. When $Ca(OH)_2$ was substituted, however, a solidified rubbery gel materialized in the still at 320°C, with a residue level of 13% of the original oil volume. These disappointing results terminated the study.

Other quantities of the mixture of the 15 used oils were distilled with NaOH/methanol, sodium carbonate, magnesium oxide, and a combination of 75:25 NaOH/methanol and magnesium oxide. Comparisons were made with previous distillations of NaOH/water:isopropyl alcohol. Figures 40 through 43 contain graphs showing acid number vs. vapor temperature vs. cumulative volume percent of these distillations. HPLC's of distillates in the same vapor temperature range are shown in Figures 44 and 45. Examination of all data indicates the following decreasing order of effectiveness:

1. NaOH/methanol (Figure 40)
2. NaOH/methanol with magnesium oxide (Figure 41)
3. Magnesium oxide (Figure 42)
4. NaOH/water:isopropyl alcohol (Figure 35)
5. Sodium carbonate (Figure 43)

3.4.4 Comparison of NaOH Distillation of Used Oil and New Oil

Virgin oil containing MRC's proposed additive package was distilled with and without NaOH treatment to examine the distillation process with completely known additives. High performance liquid chromatograms (HPLC) of two selected distillate fractions, at the same vapor temperature range, are shown in Figure 46. It can be readily observed from the chromatograms that NaOH had removed material from the distillate. Thin layer chromatography studies of the same distillates confirmed the HPLC results. A typical chromatogram (HPLC) of MRC's additive package in a virgin base stock is shown in Figure 31.

Plots of acid number vs. vapor temperature vs. cumulative percent recovery of the virgin oil discussed above are shown in Figures 47 and 48. These two figures reemphasize the decrease in acid number with no deleterious effects on recovery by NaOH distillation.

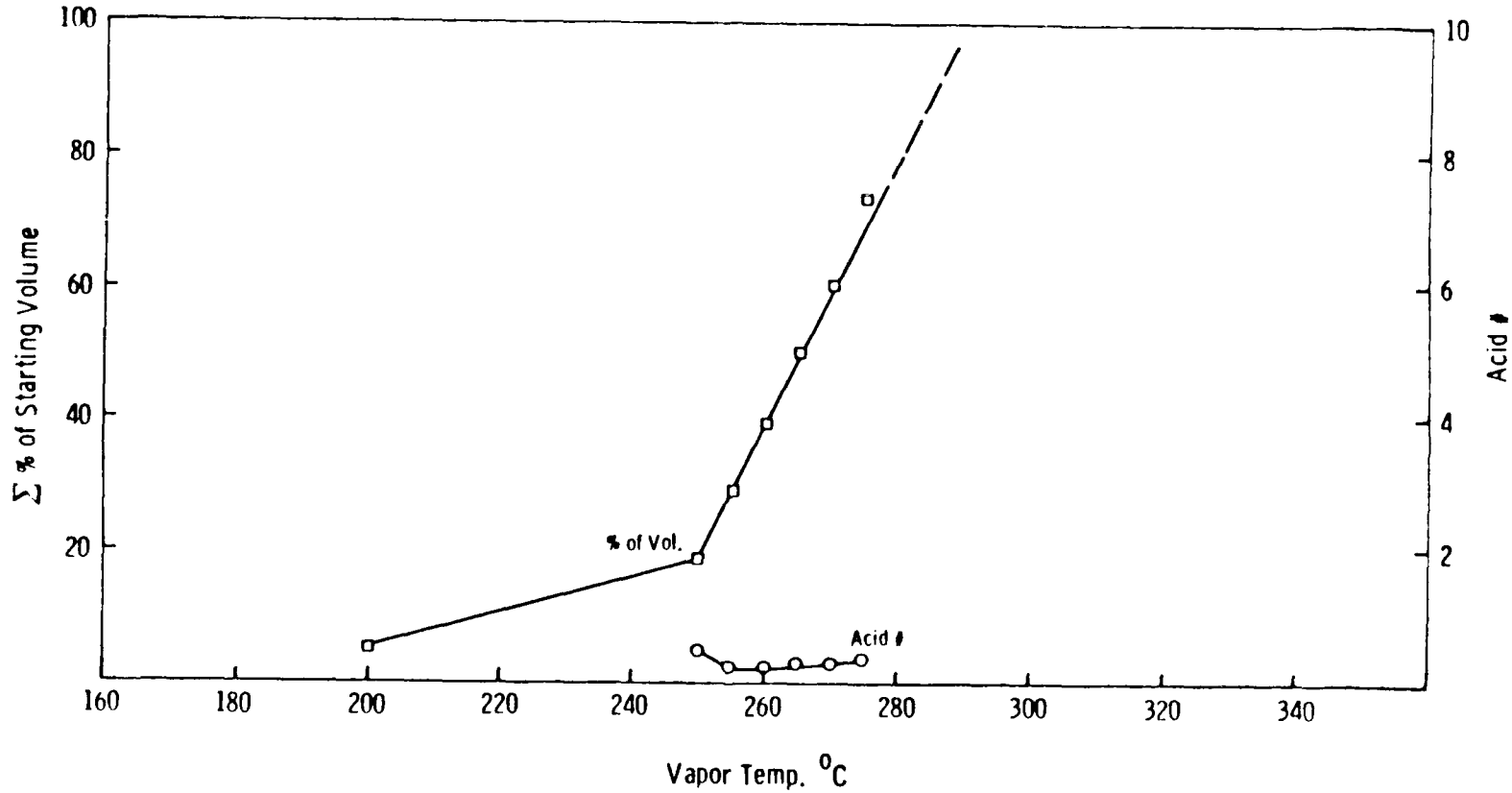


Figure 40. Sample 1732568, mixture of 15 used oils distilled with NaOH/methanol.

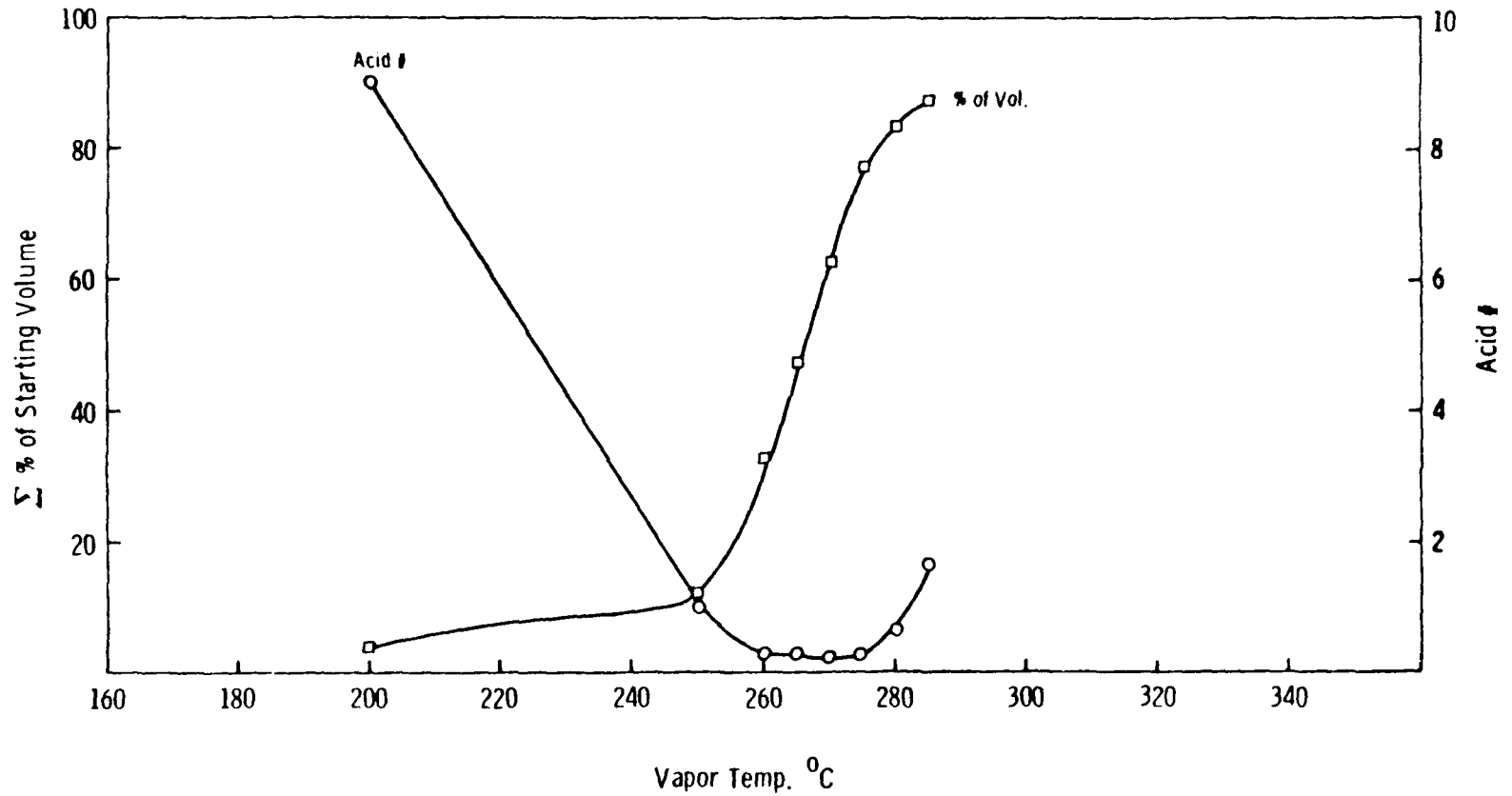


Figure 41. Sample 1732583, mixture of 15 used oils distilled with 75:25 NaOH/methanol:magnesium oxide.

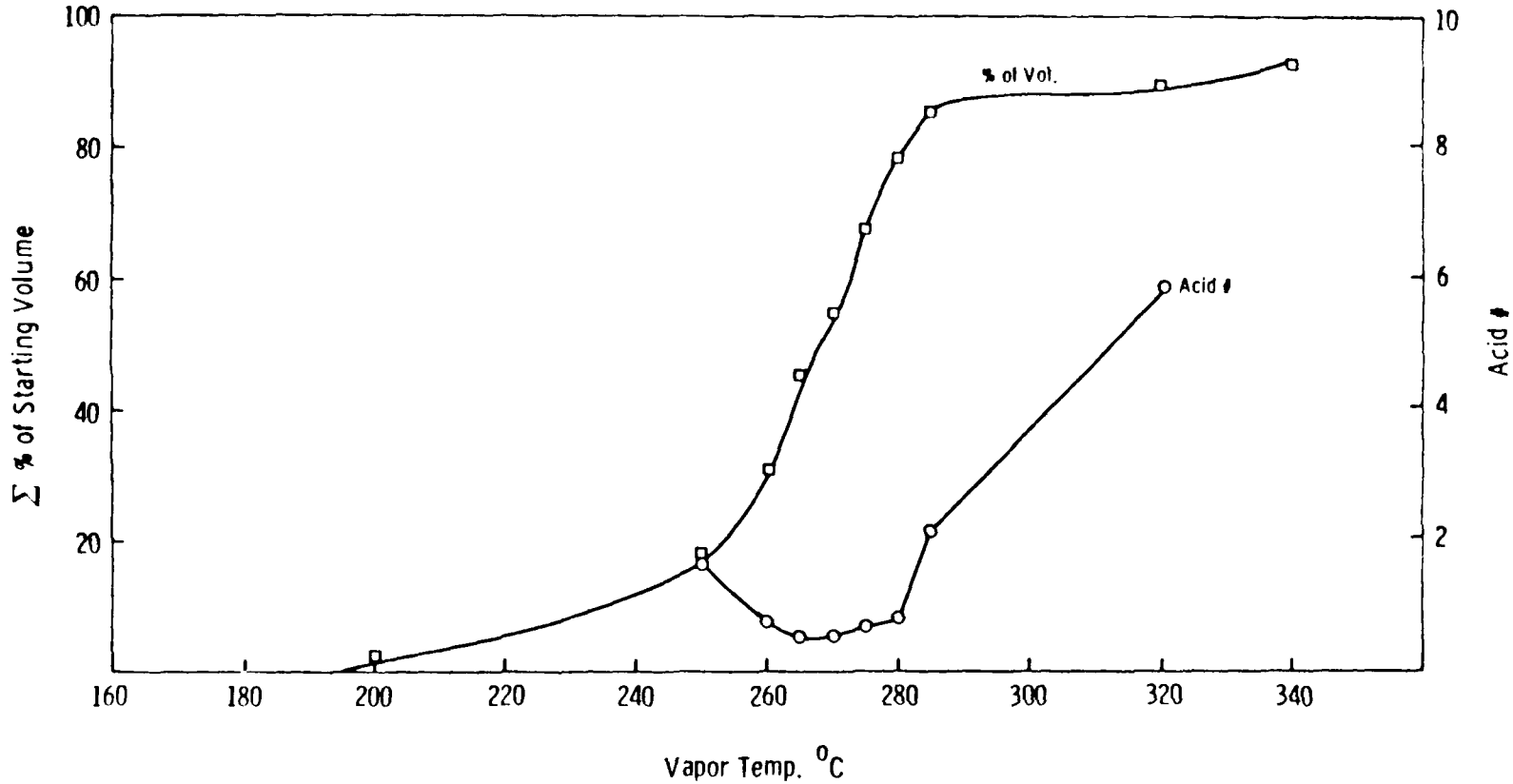


Figure 42. Sample 1732570, mixture of 15 used oils distilled with magnesium oxide.

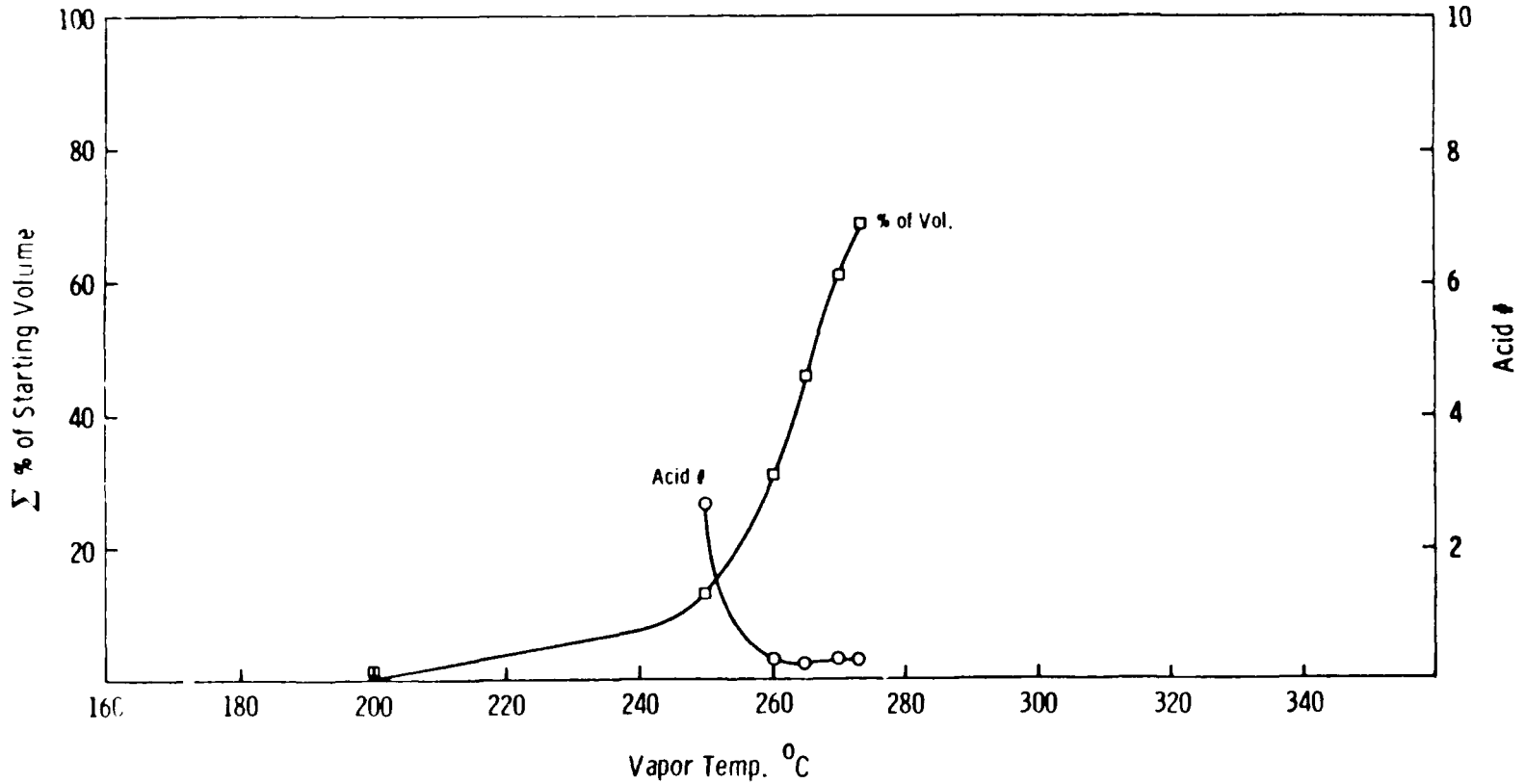
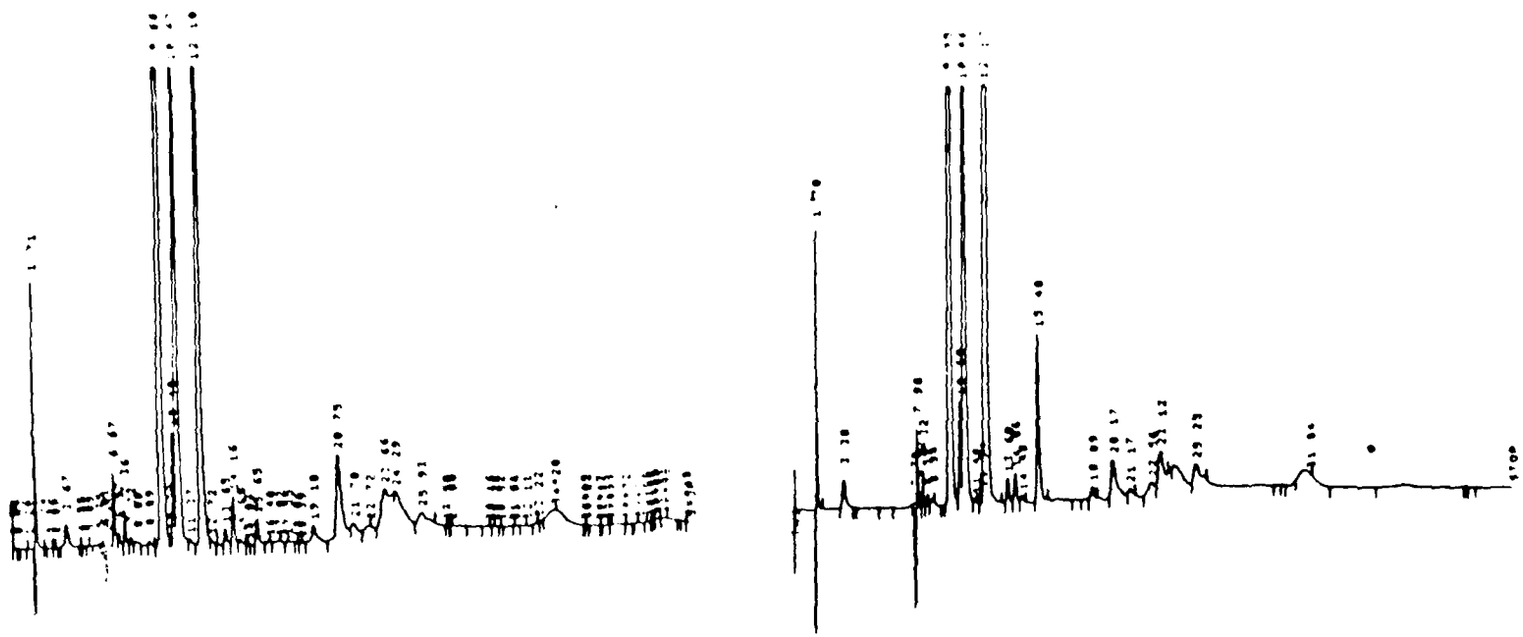


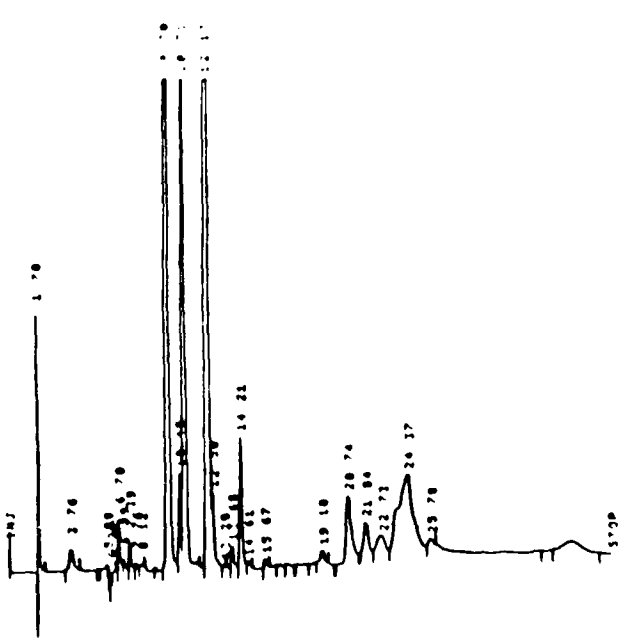
Figure 43. Sample 1732572, mixture of 15 used oils distilled with sodium carbonate.



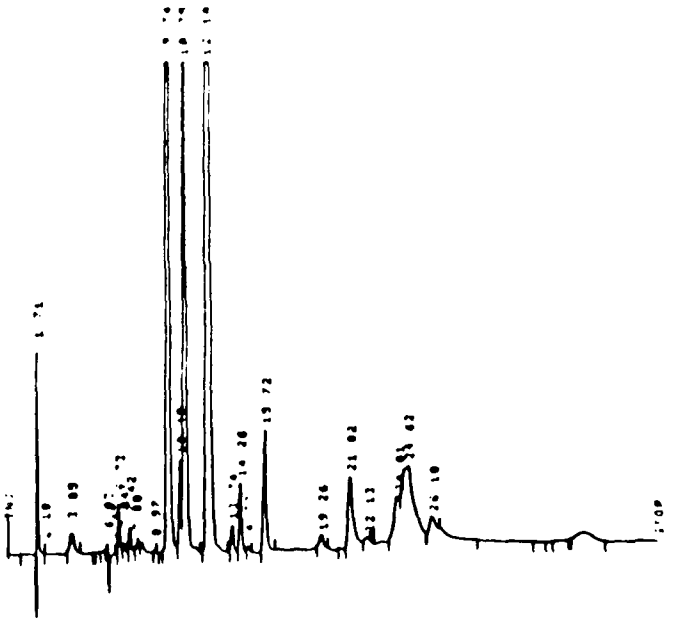
Sample 1732573(68),
fractions 4 through 7
distilled with NaOH/methanol

Sample 1732583,
fractions 3 through 6
distilled with 75:25 combination of
NaOH/methanol with magnesium oxide

Figure 44. High performance liquid chromatograms of oil distillates in 260-275°C vapor temperature range.

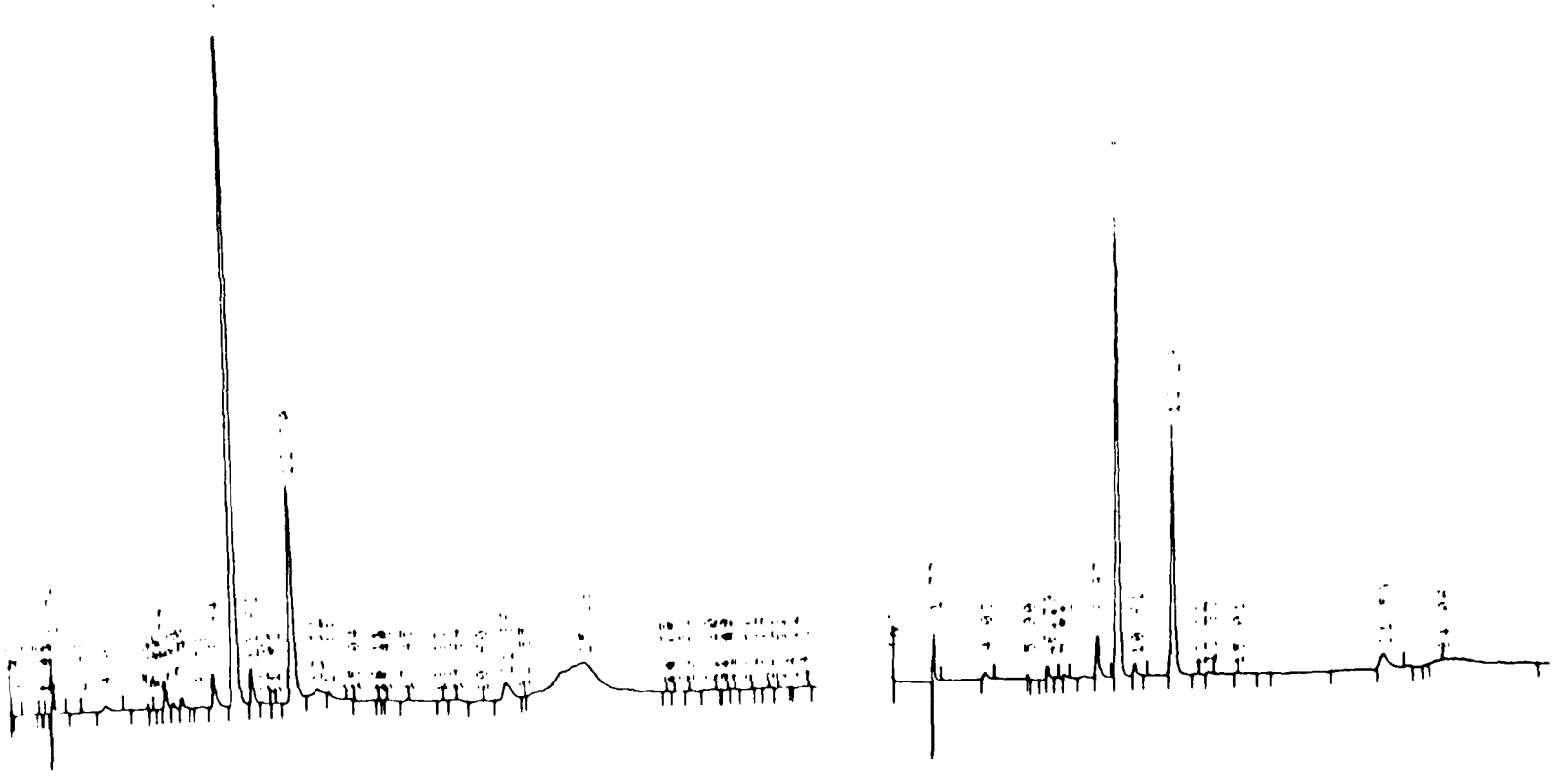


Sample 1732573(70),
fractions 3 through 6
distilled with magnesium oxide



Sample 1732573(72),
fractions 3 through 6
distilled with sodium carbonate

Figure 45. High performance liquid chromatograms of oil distillates in 260-275°C vapor temperature range.



Sample 1732518-8,
regular distillation

Sample 1732526-9,
NaOH distillation

Figure 46. HPLC's of two selected fractions of virgin oil containing MRC proposed additive package.

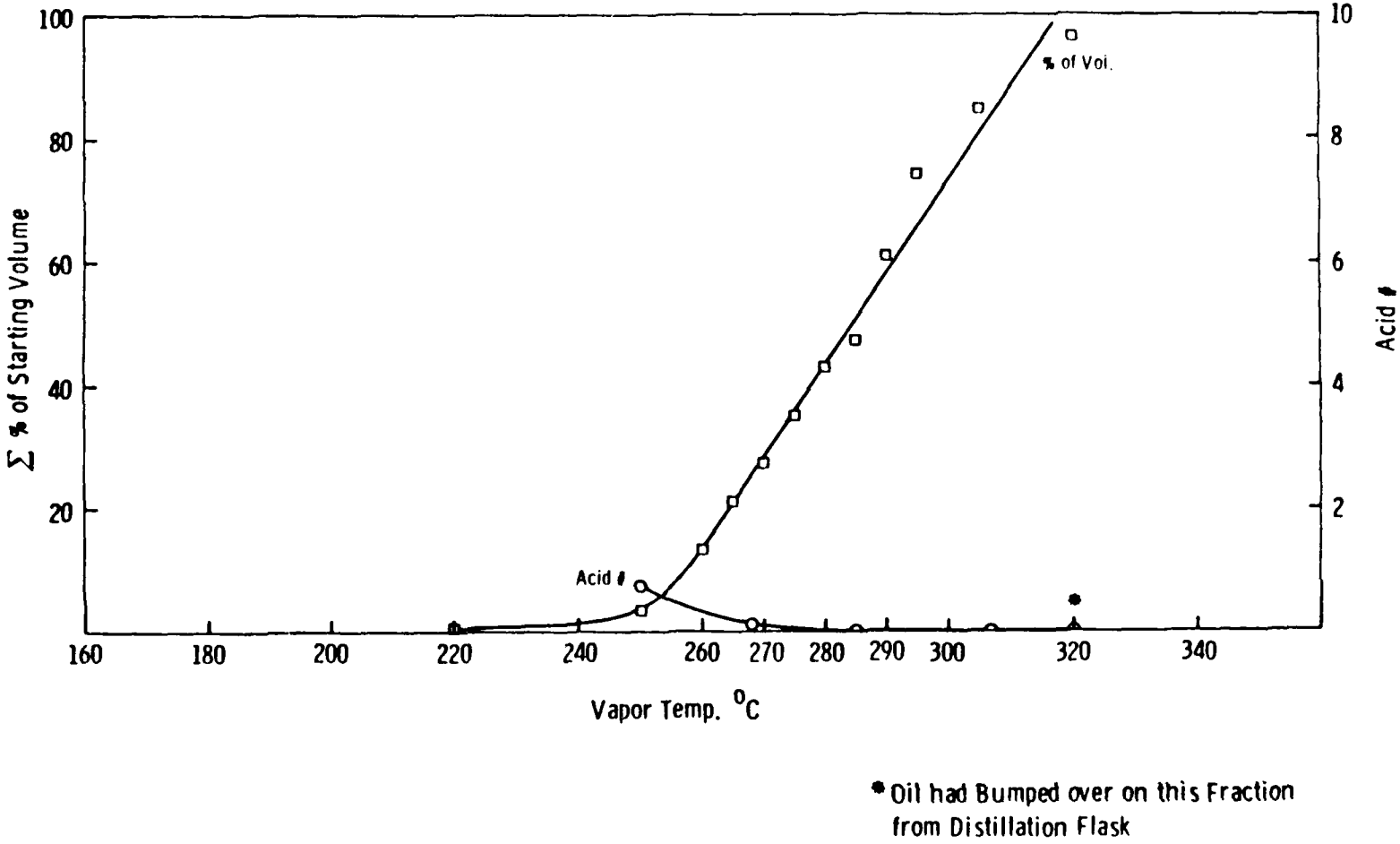


Figure 47. Sample 1732526, Royal Lube base stock with MRC proposed additive package, distilled with sufficient NaOH for neutralization.

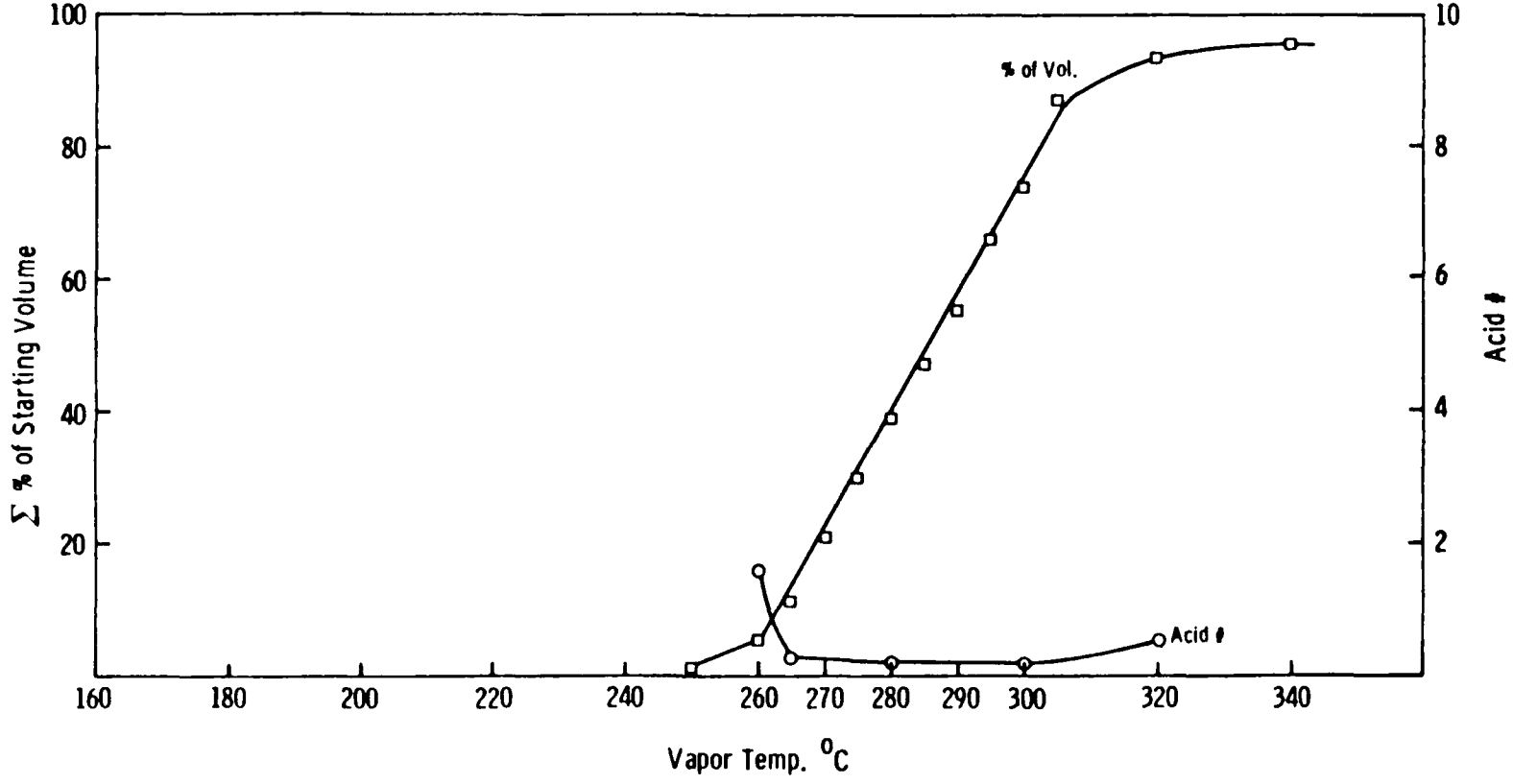


Figure 48. Sample 1732518, Royal Lube base stock with MRC proposed additive package.

3.4.5 Scaleup of Selected Distillations to 13-Liter Quantities

Results from previous small-scale (300-mL) distillations using various basic materials added to used oil warranted scaleup to 13-liter batch distillations with NaOH/methanol, 75/25 NaOH/ magnesium oxide in methanol, NaOH/isopropyl alcohol:water, and regular distillation. Tables 5 and 6 summarize the intended and actual distillation conditions of the four batches. The intended distillation conditions listed in Tables 5 and 6 should be compared to prior distillation studies shown in Figures 34, 35, 40, and 41. Intended conditions were chosen by expected acid numbers and quantity recovered. The differences between the two sets of conditions were partially brought about by unfamilarity with the larger scale equipment, and by the extension of the distillation time (2 hr to 7-9 hr) caused by distilling larger oil quantities. Even with scaleup, and startup difficulties, NaOH distillation still improves acid numbers.

From these experiments, we produced sufficient quantities of distilled oil for adsorbent and other studies.

3.5 USE OF CALCIUM HYDROXIDE TO LOWER ACID NUMBERS

Our reclamation process will have to lower acid numbers to a level in which attapulgus clay is most effective in meeting MIL-L-7808H specifications. Data from earlier work in this program, described in Section 3.6, suggested the use of a slurry with sufficient $Ca(OH)_2$ to neutralize the acid number, but data from distillations with acid numbers around 0.50 or less did not support this idea. Results of a study initiated to determine the necessary level of $Ca(OH)_2$ are presented in Table 7. The data generally indicate 0.2% by weight $Ca(OH)_2$ as the minimum amount, unless a high acid number requires a larger amount for neutralization. In some instances treated samples of the same distillate do not have comparable acid numbers, but the numbers are still sufficiently lowered for reformulation. For our process, we will use a minimum of 0.3% by weight of $Ca(OH)_2$; The maximum will be the amount necessary to neutralize larger acid numbers. The time required to slurry the $Ca(OH)_2$ will be determined individually for each batch reclaimed in the pilot plant, and the resultant data will enable us to determine a minimum slurry time.

3.6 ADSORPTION TREATMENT STUDY

Another part of our reclamation process will be an adsorption treatment to remove surfactants, trace metals, and other material that distilled over and was not removed during $Ca(OH)_2$ treatment.

TABLE 5. LARGE-SCALE (13-LITER) DISTILLATION CONDITIONS

Batch identify	Intended distillation conditions[a]			Actual distillation conditions[a]		
	Distillation temperature, °C	Percent original volume	Estimated acid number	Distillation temperature, °C	Percent original volume	Acid number[b]
	1732588 with NaOH/methanol					
1st Fraction Precollection	200 PT[c]-240 VT[d]	16.0	1.5+	200 PT-240 VT	5.1	3.73
2nd Fraction Collected for intended reformulation	240 VT-285 VT	73.0	0.25	240 VT-285 VT	70.5	0.45
3rd Fraction Postcollection	285+ VT	6.0	1.5+	285 VT-305 VT	9.0	1.58
	1732591 with 75/25 NaOH/magnesium oxide in methanol					
1st Fraction Precollection	200 PT-250 VT	11.0	1.5+	200 PT-250 VT	7.0	2.57
2nd Fraction Collected for intended reformulation	250 VT-285 VT	76.0	0.40	250 VT-282 VT	65.5	0.32
3rd Fraction Postcollection	285+ VT	8.0	1.5+	282 VT-314 VT	20.0	1.31

[a] 14-15 mm pressure.

[b] Original acid number for oil before distillation = 2.45.

[c] PT = pot temperature.

[d] VT = vapor temperature.

TABLE 6. LARGE-SCALE (13-LITER) DISTILLATION CONDITIONS

Batch identity	Intended distillation conditions[a]			Actual distillation conditions[a]		
	Distillation temperature, °C	Percent original volume	Estimated acid number	Distillation temperature, °C	Percent original volume	Acid number[b]
	NaOH/isopropyl alcohol/water					
1st Fraction Precollection	200 PT[c]-240 VT[d]	15	1.5+	200 PT-240 VT	1.7	8.95
2nd Fraction Collected for intended reformulation	240-290 VT	71	0.3	240-290 VT	65.5	2.10
3rd Fraction Postcollection	290+ VT	5	1.5+	290-335 VT	27.2	1.70
	Regular distillation (without treatment)					
1st Fraction Precollection	200 PT-255 VT	19	3.4+	200 PT-260 VT	9.2	23.88
2nd Fraction Collected for intended reformulation	255-320 VT	71	1.0	260-310 VT	82	16.3
3rd Fraction Postcollection	320+ VT	2	2.7+	310-330 VT	5.2	7.3

[a] 14-15 mm pressure.

[b] Original acid number for oil before distillation = 2.69.

[c] PT = pot temperature.

[d] VT = vapor temperature.

TABLE 7. RESULTS OF VARYING $Ca(OH)_2$ TREATMENT LEVEL

Reference number	Treatment level of $Ca(OH)_2$	Acid number	Percent by weight $Ca(OH)_2$ to oil
1830378	Amount to neutralize acid number in 1732591-2[a]	0.39	0.04
1830378	5 times amount to neutralize acid number in 1732591-2	<.018	0.2
1830364	12.5 times amount to neutralize acid number in 1732591-2	.032	0.5
1830358	12.5 times amount to neutralize acid number in 1732591-2	.045	0.5
1830352	12.5 times amount to neutralize acid number in 1732591-2	.090	0.5
1830380	Amount to neutralize acid number in 1732588-4[b]	<.018	0.2
1830380	4 times amount to neutralize acid number in 1732588-4	<.018	0.8
1830380	12.5 times amount to neutralize acid number in 1732588-4	<.018	2.6
1830382	Amount to neutralize acid number in 1732588-3[c]	.40	0.06
1830382	4 times amount to neutralize acid number in 1732588-3	<.018	0.24
1830382	8 times amount to neutralize acid number in 1732588-3	<.018	0.48
1830385	5 times amount to neutralize acid number in 1732588-3	.090	0.3

[a]Main fraction of caustic:MgO/methanol distillation, acid number of 0.39.

[b]Post fraction of caustic/methanol distillation, acid number of 1.58.

[c]Main fraction of caustic/methanol distillation acid number of 0.45.

3.6.1 Slurry Treatment

Oil samples from the main distillate of the used oil distilled with NaOH/methanol (RF N/M = reformulation fraction NaOH/methanol distillation), were slurried at two different temperatures with eight adsorbents at 10% by weight to oil. Samples were also slurried with $Ca(OH)_2$ and magnesium oxide (MgO) at various concentrations to maximize lowering of the acid number. Oil slurried with 0.5% by weight $Ca(OH)_2$ was also slurried with bleaching clays for examination. Table 8 summarizes the adsorbent study scheme. Table 9 summarizes thin layer chromatography (TLC), high performance liquid chromatography (HPLC), and acid number analyses of the distillate treated with bleaching clays at two different temperatures (70°C and 150°C) to determine minimum treatment temperature. The data in Table 9 suggest the use of attapulgus or fuller's clay for additive/degradation product removal at 70°C.

Acid number lowering of the distillate with basic material was examined and the data are shown in Table 10. HPLC and TLC results indicate no differences in additive/degradation product removal by varying the basic material level or between $Ca(OH)_2$ and MgO. Data indicates use of $Ca(OH)_2$ as most effective in lowering the acid number.

In another study, the RF N/M distillate was treated with 0.5% by weight $Ca(OH)_2$ and then slurried with bleaching clays at 70°C. The data shown in Table 11 suggest the use of attapulgus or fuller's clay for treatment.

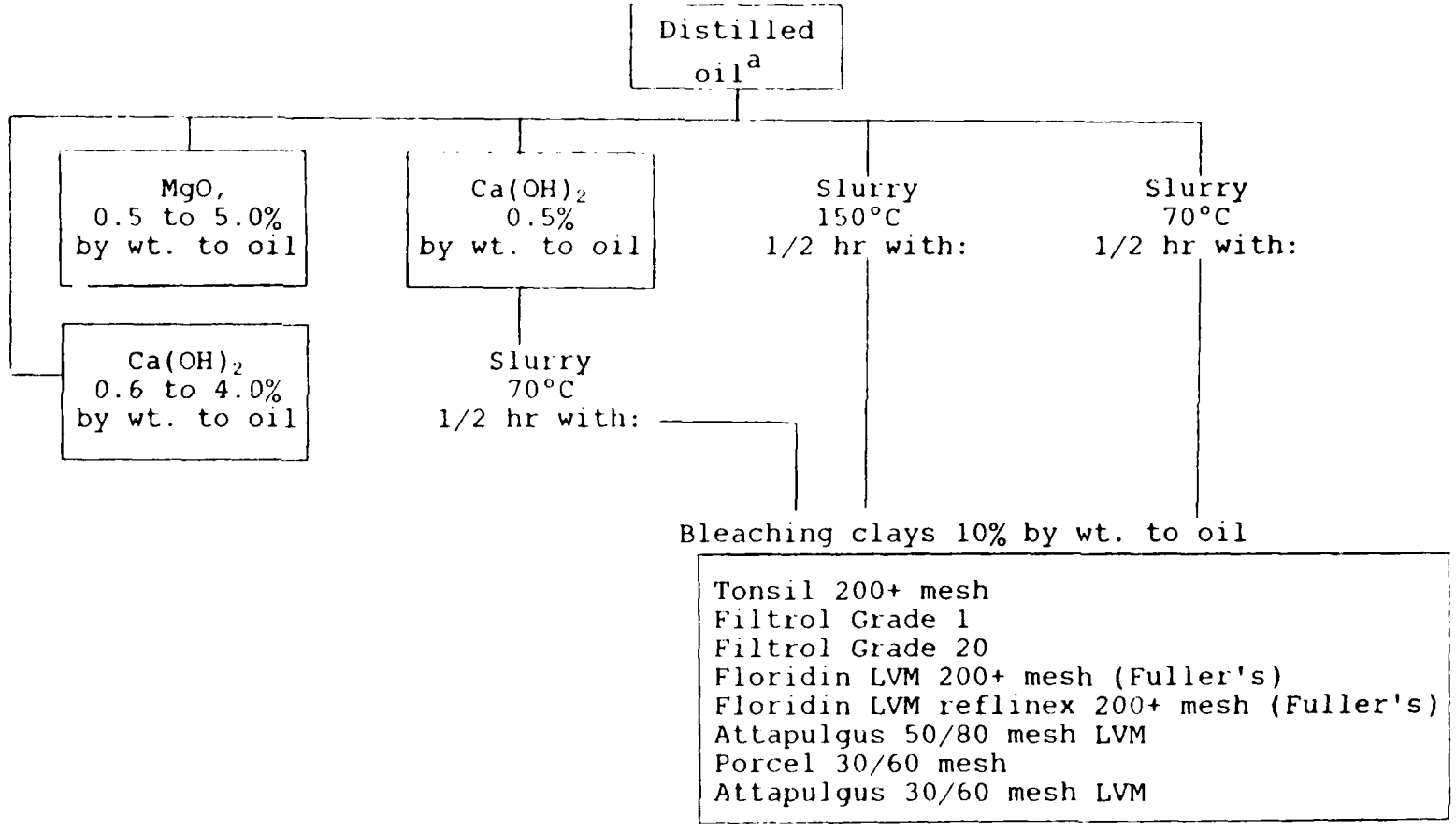
When acid number needs lowering, the data generated by this adsorbent study suggest a slurry at room temperature for 16 hr with the stoichiometric level of caustic to neutralize the acid number, followed by a slurry with fuller's or attapulgus bleaching clays at 70°C for 0.5 hour.

The adsorbent study scheme suggested by the data generated with the RF N/M distillate, which will be used with the distillates from the remaining three scaleup distillations is shown in Table 12.

Data presented in Table 13, for main distilled fraction (MDF) from $NaOH/H_2O$ in isopropyl alcohol (IPA) distillation, suggest the use of enough $Ca(OH)_2$ to neutralize the acid number, followed by treatment with fuller's earth. Data in Table 14 for MDF from NaOH/MgO distillation indicate the use of the stoichiometric level of $Ca(OH)_2$ to neutralize the acid number followed by treatment with either fuller's earth or attapulgus clay.

Data in Table 15 (MDF from no treatment) suggest slurry with the stoichiometric amount of $Ca(OH)_2$ to neutralize, followed by reslurry with fuller's earth.

TABLE 8. ADSORBENT STUDY SCHEME



[a]Main fraction distillate of NaOH/methanol distillation.

TABLE 9. SUMMARY OF DISTILLATE TREATED WITH VARIOUS BLEACHING CLAYS[a]

Bleaching Clays 10% by wt. to Oil	Acid No. Slurry heated to 70°C	Slurry heated to 150°C
Tonsil 200+ mesh	0.17	0.16
Filtrol Grade 1 200+ mesh	0.24	1.77
Filtrol Grade 20 200+ mesh	0.25	2.26
Floridin 200+ mesh (Fullers)	0.14	0.09
Floridin reflinex (Fullers) 200+ mesh	0.10	0.10
Attapulgus 50/80 mesh LVM	0.17	0.10
Porcel 30/60 mesh	0.10	0.15
Attapulgus 30/60 mesh LVM	0.11	0.18

Thin Layer Chromatography[b]

Slurry heated to 70°C		Slurry heated to 150°C	
Decreasing removal of material ↓			
Filtrol 1, Filtrol 20, Attapulgus 30/60	+	Tonsil, Floridin reflinex, Attapulgus 30/60	+
Tonsil, All Fullers, Attapulgus 50/80	0	Floridin 200+, Attapulgus 50/80	0
Porcel	-	Filtrol 1, Filtrol 20	0-
		Porcel	-

Clays rated + at 150°C are slightly better than clays rated + at 70°C.

High Pressure Liquid Chromatography[b]

Slurry heated to 70°C		Slurry heated to 150°C	
Decreasing removal of material ↓			
6 clays	0	Attapulgus 50/80, Porcel	+
Filtrol 1, Filtrol 20	-	4 Clays	0
		Filtrol 1, Filtrol 20	-

Comparison of clays rated + at 150°C are equivalent to clays rated 0 at 70°C.

[a]Main fraction from NaOH/methanol distillation.

[b]Subjective test, with + meaning the greatest removal of material.

TABLE 10. USE OF BASIC MATERIAL TO LOWER ACID NUMBER AFTER DISTILLATION[a]

$Ca(OH)_2$ as a base		MgO as a base	
Level of addition	Resulting acid number of the treated oil[b]	Level of addition	Resulting acid number of the treated oil[b]
Stoichiometric amount for 0.0 acid number (0.06% by wt. to oil)	0.02	Stoichiometric amount for 0.0 acid number (3.0% by wt. to oil)	0.48
0.5% by wt. to oil	0.06	0.5% by wt. to oil	0.35
2.0% by wt. to oil	0.02	2.0% by wt. to oil	0.14
4.0% by wt. to oil	0.03	4.0% by wt. to oil	0.10

[a]Slurry for 16 hours at room temperature; main fraction from NaOH/methanol distillation.

[b]Original acid number before treatment: 0.52.

TABLE 11. SUMMARY OF DISTILLATES[a] SLURRIED WITH 0.5% BY WEIGHT $Ca(OH)_2$ AND THEN WITH VARIOUS BLEACHING CLAYS

Bleaching Clays Slurried at 70°C	Acid No.
No Bleaching Clay treatment	<0.09
Porcel	<0.09
Attapulgus 30/60	<0.09
Attapulgus 50/80	0.00
Floridin (Fullers) 200+	<0.09
Floridin reflinex	<0.09
Tonsil	<0.09
Filtrol Grade 1	<0.08
Filtrol Grade 20	<0.09

High Pressure Liquid Chromatography[b]

Decreasing removal of material

Attapulgus 50/80
Attapulgus 30/60 } almost equal
Floridin 200+ (Fullers)

Remaining clays equal to or worse than oil before treatment.

Thin Layer Chromatography

Tonsil	+
Floridin reflinex	
Floridin 200+ mesh	0+
Attapulgus 30/60	
Attapulgus 50/80	
Filtrol Grade 1	0
Filtrol Grade 20	
Porcel	

[a]Main fraction from NaOH/methanol distillation.

[b]Subjective test, with + meaning greatest removal of material.

TABLE 12. ADSORBENT STUDY SCHEME TO USE WITH DISTILLATES OF THE REMAINING 3 SCALE-UP DISTILLATIONS

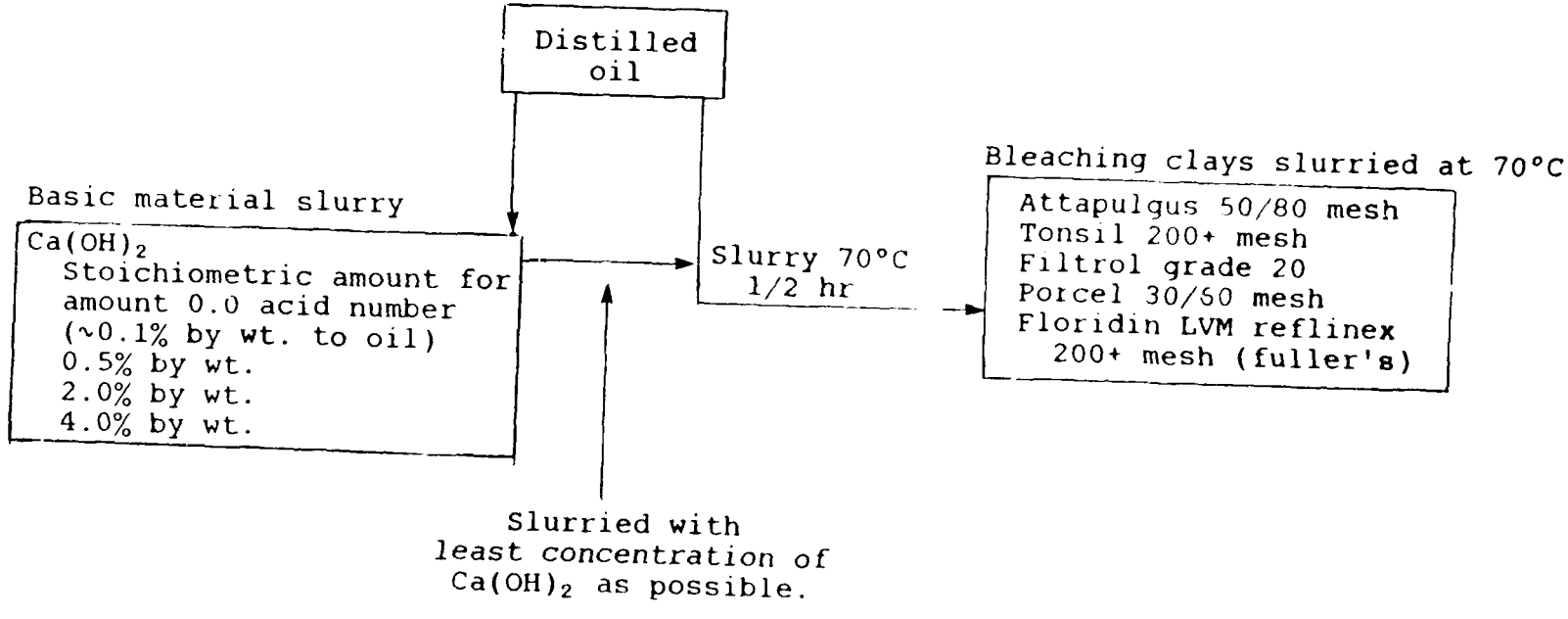


TABLE 13. SUMMARY OF MAIN FRACTION DISTILLATE FROM NaOH/ WATER: ISOPROPYL ALCOHOL DISTILLATION TREATED WITH VARIOUS BLEACHING CLAYS

Bleaching clays 10% by weight slurried at 70°C	Acid number oil treated with clay	Acid number of oil treated with 0.5% by weight $Ca(OH)_2$ and then with clay
No clay treatment	2.18	[illegible]
Tonsil	1.47	0.09 0.18
Attapulgas	1.66	0.09
Fullers (Floridin-reflinex)	1.18	<0.09
Filtrol 20	1.83	<0.09

Lowering of acid number with $Ca(OH)_2$	Acid number
Quantity to neutralize acid no. (0.28%)	<0.09
0.5% by weight $Ca(OH)_2$	<0.06
2.0% by weight $Ca(OH)_2$	<0.09

No difference in removal by changing $Ca(OH)_2$ treatment level by HPLC and TLC.

Thin Layer Chromatography

	Oil + adsorbents	Oil + $Ca(OH)_2$ + adsorbents
Decreasing removal of material ↓	Filtrol 20	Tonsil
	Tonsil	Fullers (reflinex)
	Attapulgas	Filtrol 20
	Fullers (reflinex)	Attapulgas

$Ca(OH)_2$ + bleaching clays better than bleaching clays only.

High Pressure Liquid Chromatography

	Oil + adsorbents	Oil + $Ca(OH)_2$ + adsorbents
Decreasing removal of material ↓	Attapulgas	Tonsil
	Tonsil	Fullers (reflinex)
	Fullers (reflinex)	Filtrol 20
	Filtrol 20	Attapulgas

$Ca(OH)_2$ + Tonsil better than attapulgas alone.

TABLE 14. SUMMARY OF MAIN FRACTION DISTILLATE FROM 75/25 NaOH/MgO IN METHANOL, DISTILLATION TREATED WITH VARIOUS BLEACHING CLAYS

Bleaching clays 10% by weight slurried at 70°C	Acid number oil treated with clay	Acid number of oil treated with 0.5% by weight $Ca(OH)_2$ and then with clay
No clay treatment	0.35	<0.08
Tonsil	0.12	<0.09
Filtrol 20	0.18	0.11
Fullers (reflinex)	0.09	<0.09
Attapulgas	0.13	<0.09

Lowering of acid number with $Ca(OH)_2$	Acid number
Quantity to neutralize acid no. (0.042%)	<0.080
0.5% by weight	<0.080
2.0% by weight	<0.085

No difference in removal by changing $Ca(OH)_2$ treatment level by HPLC and TLC.

Thin Layer Chromatography[a]

	Oil + adsorbents		Oil + $Ca(OH)_2$ + adsorbents	
Decreasing removal of material ↓	Tonsil	same	Tonsil	+
	Attapulgas		Fullers	+
	Fullers (reflinex)		Attapulgas	0
	Filtrol 20	-	Filtrol 20	-

Treatment with $Ca(OH)_2$ + clay better than clay alone.

High Pressure Liquid Chromatography[a]

	Oil + adsorbents	Oil + $Ca(OH)_2$ + adsorbents
Decreasing removal of material ↓	Fullers (reflinex)	Fullers (reflinex)
	Tonsil	Attapulgas
	Attapulgas	Tonsil
	Filtrol 20	Filtrol 20

Treatment with $Ca(OH)_2$ + clay slightly better than clay alone.

[a]Subjective test, with + meaning greatest removal of material.

TABLE 15. SUMMARY OF MAIN FRACTION DISTILLATE FROM DISTILLATION TREATED WITH NO BASIC MATERIAL, TREATED WITH VARIOUS BLEACHING CLAYS

Bleaching clays 10% by weight slurried at 70°C	Acid number oil treated with clay	Acid number of oil treated with 3% by weight $Ca(OH)_2$ and then with clay
No bleaching clay treatment	1.84	0.17
Tonsil 200+ mesh	1.24	0.24
Porcel	0.60	0.22
Fullers (Floridin-reflinex)	0.73	0.22
Attapulgas 50/80	1.40	0.17
Filtrol grade 20	1.43	0.17

Lowering of acid number with $Ca(OH)_2$	Acid number
0.5% by weight	0.22
2% by weight	0.17
Quantity to neutralize acid no. (2.1%)	0.17
3% by weight	0.17
4% by weight	0.16

No difference in removal by changing $Ca(OH)_2$ treatment level by HPLC and TLC.

Thin Layer Chromatography[a]

	Oil + adsorbents		Oil + $Ca(OH)_2$ + adsorbents	
Decreasing removal of material ↓	Filtrol grade 20	+	Tonsil	+
	Fullers (reflinex)	0+	Filtrol grade 20	0+
	Tonsil		Fullers (reflinex)	
	Attapulgas	0	Attapulgas	0
	Porcel	-	Porcel	-

Oil + $Ca(OH)_2$ + adsorbents better than oil + adsorbents.

High Pressure Liquid Chromatography[a]

	Oil + adsorbents		Oil + $Ca(OH)_2$ + adsorbents	
Decreasing removal of material ↓	Tonsil	+	Fullers	+
	Fullers	0	Tonsil	
	Attapulgas		Filtrol 20	0
	Filtrol 20	-	Attapulgas	
	Porcel		Porcel	-

Oil + $Ca(OH)_2$ + adsorbents = oil + adsorbents.

[a]Subjective test, with + meaning greatest removal of material.

A comparison of acid number, HPLC, and TLC data for the reclaimed oil from the four large-scale distillations suggests the following decreasing order of effectiveness for the additive/degradation removal processes:

1. NaOH/MeOH - $Ca(OH)_2$ slurry - attapulgus clay slurry

2. NaOH:MgO/MeOH - $Ca(OH)_2$ slurry - fuller's or attapulgus clay slurry

3. Either NaOH/H_2O:IPA - $Ca(OH)_2$ slurry - fuller's earth slurry, or use of no basic material - $Ca(OH)_2$ slurry - fuller's earth slurry

We selected NaOH/MeOH - $Ca(OH)_2$ slurry - attapulgus clay slurry as a viable reclamation procedure on which to base our continuing studies.

Previous studies had shown that attapulgus clay slurried in oil at approximately 70°C was the ideal treatment for additive/degradation product removal. We further defined the optimum slurry temperature through the use of HPLC, TLC, and acid number. Results are presented in Table 16. The data show a wide range of temperatures that could be used, but to keep energy costs low and oil viscosity down (to aid in filtration), we chose to clay treat at 50°C for 30 min to 1 hr.

The attapulgus clay-to-oil ratio were determined individually for each batch processed in the pilot plant; these results were then used to determine a general clay-to-oil ratio.

TABLE 16. VARYING ATTAPULGUS CLAY TREATMENT TEMPERATURE

Conditions (slurry for 30 min)	Acid number	TLC[a]	HPLC[a]
90-105°C	<0.018	0^+	0^-
70-77°C	<0.018	0	0
50-55°C	<0.018	0	0
30-40°C	<0.018	0	0

1830373 - Main fraction NaOH:MgO/ methanol distillate treated with $Ca(OH)_2$ and 10% by weight clay to oil.

[a]Subjective test, with + meaning the greatest removal of material.

3.6.2 Chromatographic Adsorbent Studies

We examined the use of a column (93 cm x 1 cm) packed with 50/80 mesh attapulgus clay heated from 70-100°C for comparison with the 10% by weight clay-to-oil slurry procedure. The MDF from NaOH/MeOH treated with 0.5% by weight $Ca(OH)_2$ was used, to provide a direct comparison with the slurry studies. Acid number data presented in Figure 49 for both procedures show that the slurry treatment is more efficient; HPLC and TLC data bear out this conclusion.

We extended this study by varying column temperature and oil retention time. Figures 50, 51, and 52 present sample acid number vs. total volume through column by varying column conditions. The data suggest that a ≦7.5-min retention time and a column temperature of 65-80°C will treat (determined by HPLC and TLC) a greater volume of oil than a slurry of 10% by weight clay to oil. Nevertheless, the 10% by weight clay presently used for slurrying is probably an excess, and column adsorption is not as attractive as the data suggest because of the extra work involved in packing columns on a large scale and the need for high pressure pumps. We will therefore use the slurry adsorption procedure in our reclamation process.

3.7 USE OF ACTIVATED CHARCOAL AS AN ADSORBENT

The original reclamation process included an activated charcoal treatment step of questionable value. We again examined the use of charcoal in this program. A sample of a distillate treated with $Ca(OH)_2$ and attapulgus clay was treated with 6% by weight charcoal to oil for 30 min at 70°C and examined by HPLC and TLC. The only perceptible change to the oil was a very slight color improvement, which may not improve the total reclamation process.

3.8 RECLAIMED BASE STOCK EVALUATION

In the earlier work on this contract we concentrated on identifying a distillation procedure, selecting a clay adsorbent for additive/degradation removal with a treatment procedure, and identifying the level of $Ca(OH)_2$ treatment required to lower the acid number. The next step was to reclaim a small quantity of oil with our modified procedure and reformulate it with additives to perform a few selective tests to verify our overall process. Table 17 presents our reclaimed oil test scheme.

To determine if precut, main cut, and postcut distillates could be combined as reclaimed base stock, distillates from NaOH/methanol distillation were combined, reformulated and foam tested. Table 18 lists the resulting foam test volumes, which suggest that it may be possible to combine all fractions for recovery. To further verify the reclamation process and the idea of combining the distillate

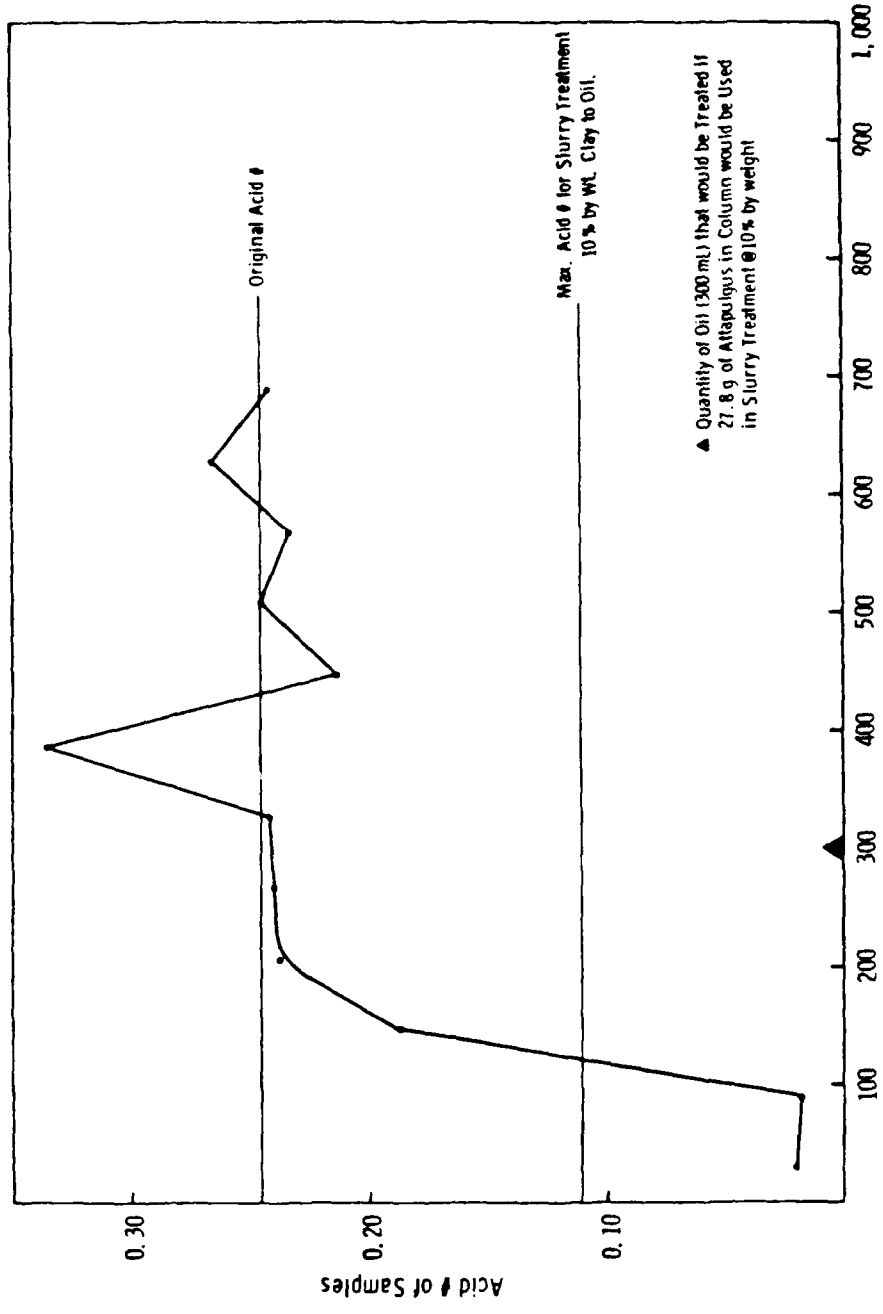


Figure 49. Main fraction from NaOH/MeOH distillation, pretreated with $Ca(OH)_2$, followed by chromatographic treatment with attapulgus clay.

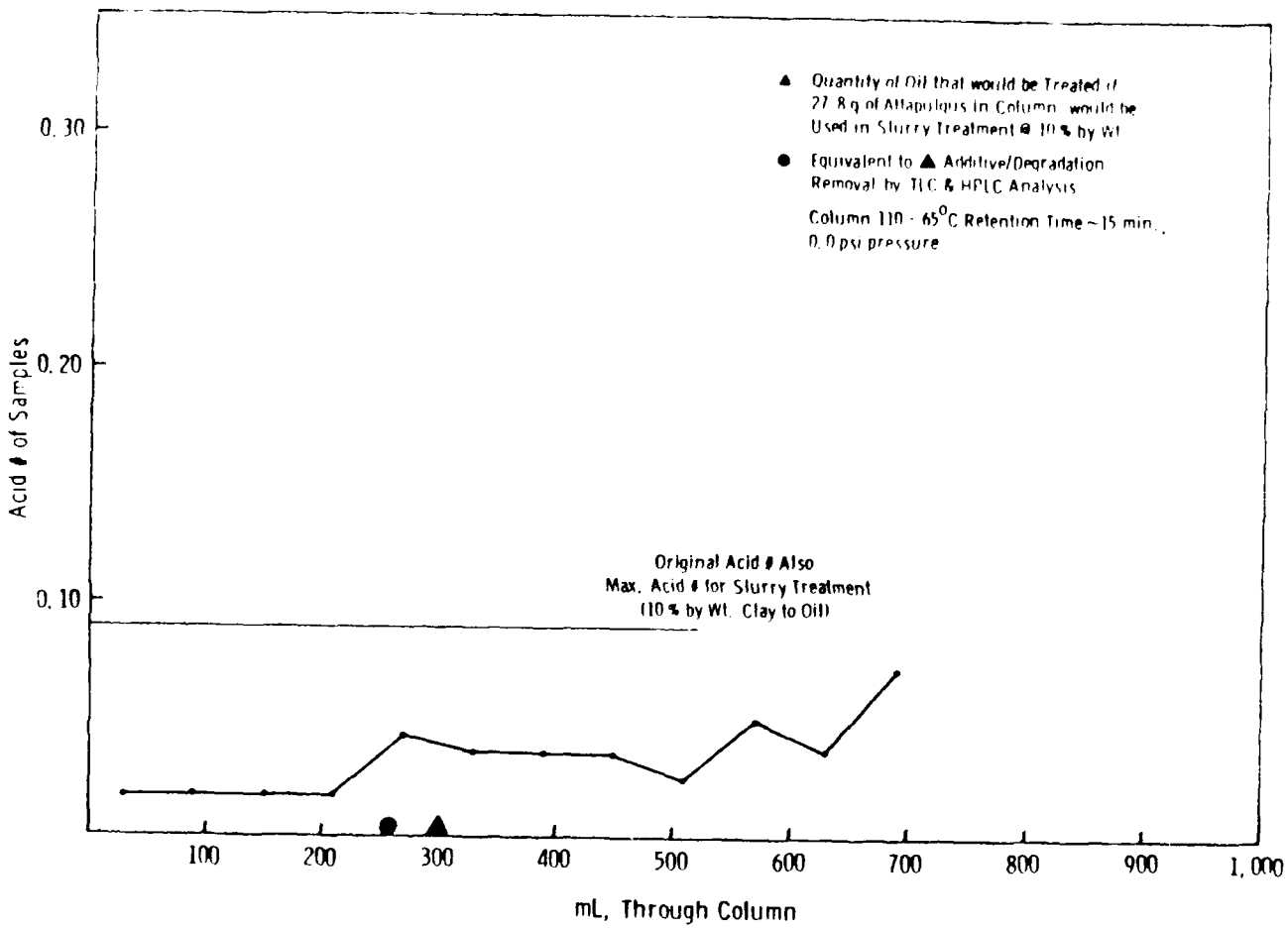


Figure 50. Main fraction from NaOH:MgO/MeOH distillation, pretreated with $Ca(OH)_2$, followed by chromatographic treatment with attapulgus clay.

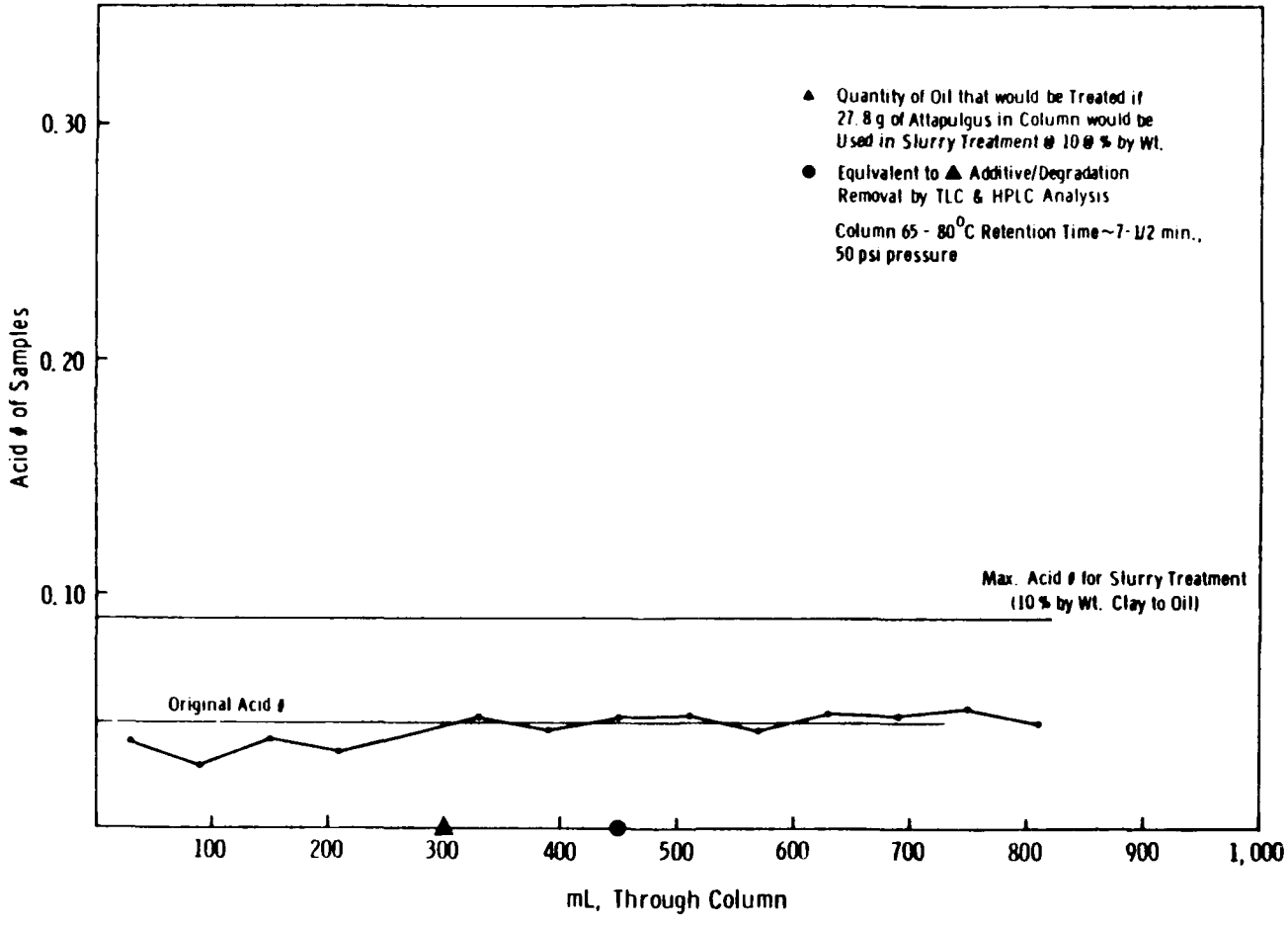


Figure 51. Main fraction from NaOH:MgO/MeOH distillation, pretreated with $Ca(OH)_2$, followed by chromatographic treatment with attapulgus clay.

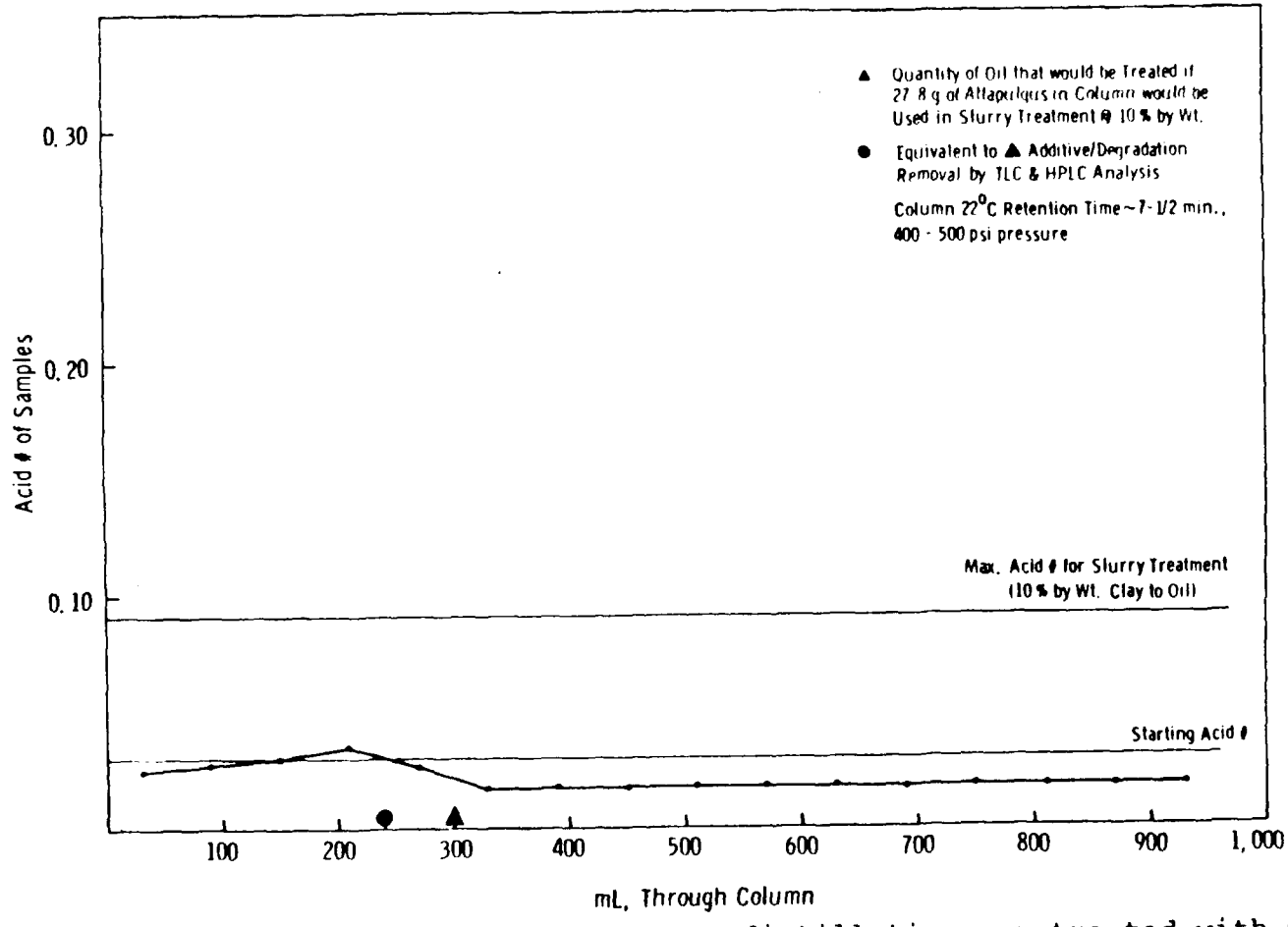
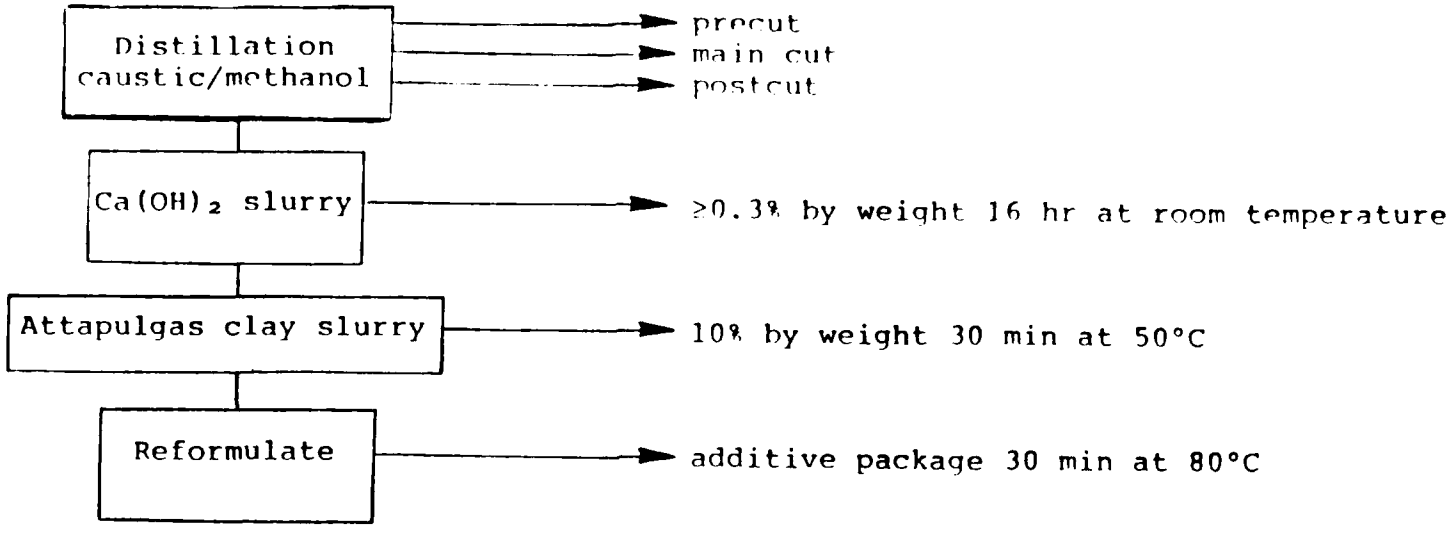


Figure 52. Main fraction from NaOH:MgO/MeOH distillation, pretreated with $Ca(OH)_2$, followed by chromatographic treatment with attapulgus clay.

TABLE 17. RECLAIMED OIL TEST SCHEME



Foam test 3 formulated samples - main cut
- main cut + appropriate percent of precut
- main cut + appropriate percent of pre- and postcut

Alcor testing of 2-2.6 liter samples - main cut + precut
- main cut + precut and postcut

Tests:

Acid number
Viscosities at 210°F and -65°F
Static foaming
Lead corrosion, 1 hr @ 325°F
Silver and bronze corrosion, 50 hr @ 450°F
Deposition number test
Corrosion and oxidation 48 hr @ 392°F

TABLE 18. REFORMULATED BASE STOCK FOAM VOLUMES

Sample	Distilled oil sample numbers	Foam volume
1	Reclaimed 1732588-3 (main cut)	10 mL
2	Reclaimed 1732588-3+ 6% (1732588-2) (precut)	15 mL
3	Sample 2 reformulated	15 mL
4	Sample 3 + reformulated 1732588-4 (postcut)	15 mL

fractions, two 2.6-liter samples had been reclaimed. They were reformulated with additives and sent to Alcor Testing Laboratory for tests outlined in our reclaimed oil test scheme.

The two reclaimed samples of combined NaOH/methanol distillates had the vapor temperature ranges shown in Table 19.

TABLE 19. VAPOR TEMPERATURE RANGES OF COLLECTED DISTILLATES IN THE ALCOR TESTED SAMPLES

Sample	Vapor temperature range
1830396-C/M-23	135-285°C
1830397-C/M-234	135-305°C

The results of the selected MIL-L-7808H tests by Alcor are included in Appendix B.

In general, the reclaimed/reformulated (RR) samples performed as well as the formulated virgin base stocks from various manufacturers used earlier to verify the additive package. We have shown by experimentation in the laboratory that the 100-120 mL foam volumes of the RR samples were due to inadvertent addition of excess (3%) PANA additive. In the corrosion and oxidation stability test, the RR samples do not meet the MIL-L-7808H requirements with respect to percent change in viscosity and total acid number change. However, this is attributed to the probable presence of MIL-L-7808G oils in the samples which do not meet these requirements even as new oils. This deficiency could be corrected by dilution with a MIL-L-7808 virgin base stock if necessary. In conclusion, we feel that the developed reclamation process is viable.

3.9 LARGE SCALE RECLAMATION - PILOT PLANT SCALE

In this section we'll discuss scale-up of the reclamation process from laboratory to pilot plant scale. Also included in this section are laboratory studies that were initiated as difficulties arose.

3.9.1 Pilot Plant Distillations (25 gallon scale)

Figure 53 shows a schematic diagram of the distillation set-up in the plant. The equipment was cleaned with soap, water, acetone and a chlorinated solvent. Virgin basestock was distilled in the equipment for final clean out. After each distillation, the still was cleaned with acetone, water and then acetone to remove still bottoms and NaOH. The initial batches distilled encountered problems with bump-over, so we decreased the distillation rate. Appendix G and I contains the data for each batch and procedure sheets for a typical batch distilled. No problems were encountered with the 0-79- series of used oils, though some were distilled 2 times, due to foaming caused by $Ca(OH)_2$ treatment. Distillates from the 0-82- series had considerable problems with high foam test volumes.

3.9.1.1 Distillate Foam Test Failure

Foam test failure of distillates from the 0-82- series of used oil batches have been uncorrectable. Table I-1 (page 227) lists data from those distillations. The data compared with earlier 0-79- used oil samples processed, are considerably different. There is significantly more still bottoms and still toppings/pre-cut material The earlier cuts in the distillations contain kerosene types of material.

Table 20 presents data from a foam test study involving addition of JP-4 jet fuel and hydraulic fluid to a low foaming formulated oil. The data suggests that the used oils maybe contaminated by these fluids and their associated additives. Treatment of the distillates with $Ba(OH)_2 \cdot H_2O$ and fullers earth did not lower the foam test volume.

Metal analysis of these oils, indicate that used oil 0-82-6 maybe contaminated with motor oil. This oil has very high levels of lead (25 ppm) and zinc (76 ppm), not normally, found in 7808H oils.

Use of HPLC, TLC and IR as a screening method for contamination was unsuccessful. Appendix J contains the chromatograms for HPLC, GC and IR. As can be seen the HPLC and IR resemble the other used oil samples, the GC chromatograms do show a significant difference. Comparison of chromatograms (180-350°C program) with the 0-79- used oils show additional peaks buried in the initial solvent peak. Comparing the chromatograms with their distillates, show the removal of the extra peaks. The GC program was changed to 100-350°C for better separation of the peaks buried under the initial solvent peak, these chromatogram are also in Appendix J. Also included are samples of JP-4 and hydraulic fluids. GC appears to be a good way to screen each used oil sample before processing.

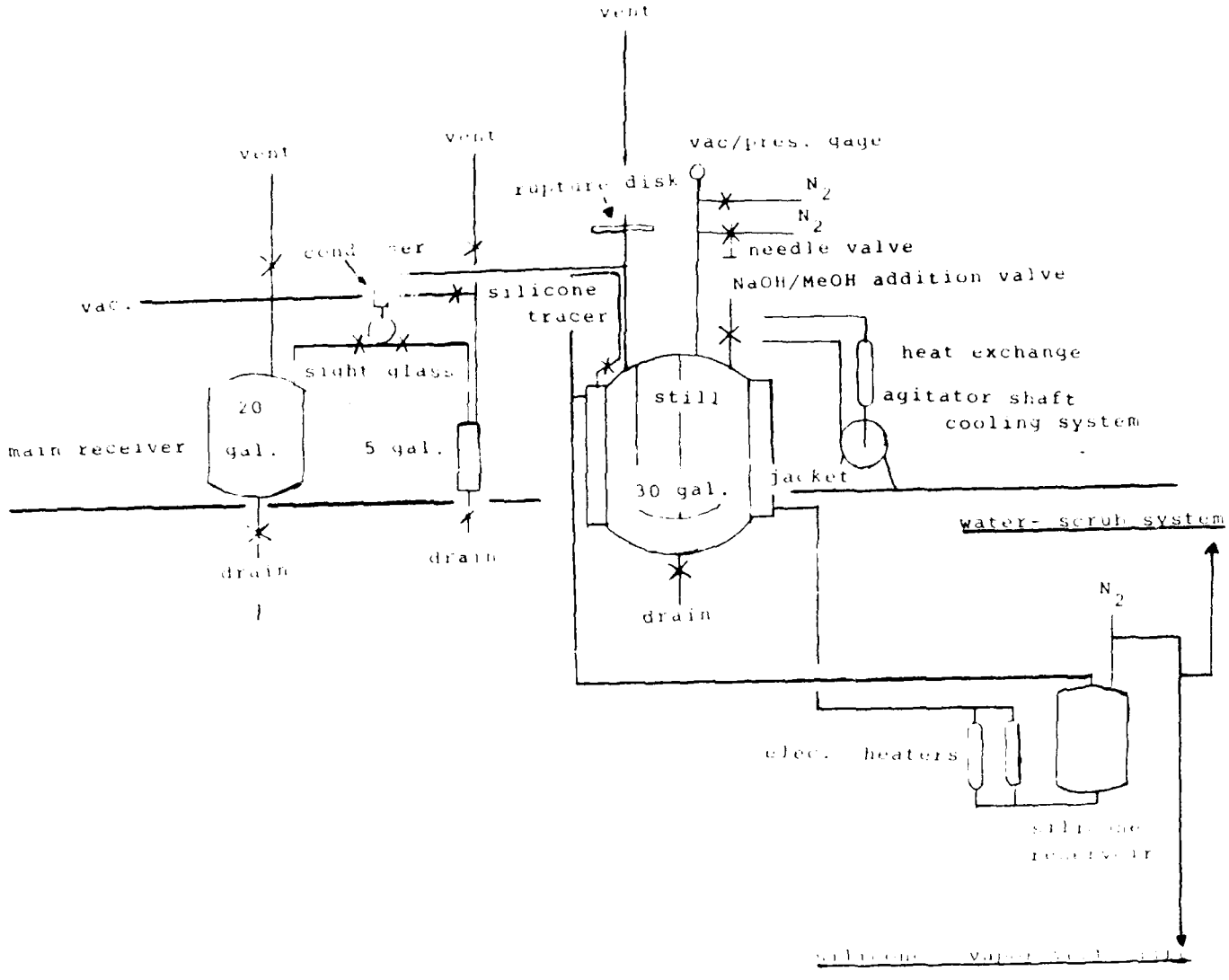


Figure 53. Plant distillation equipment.

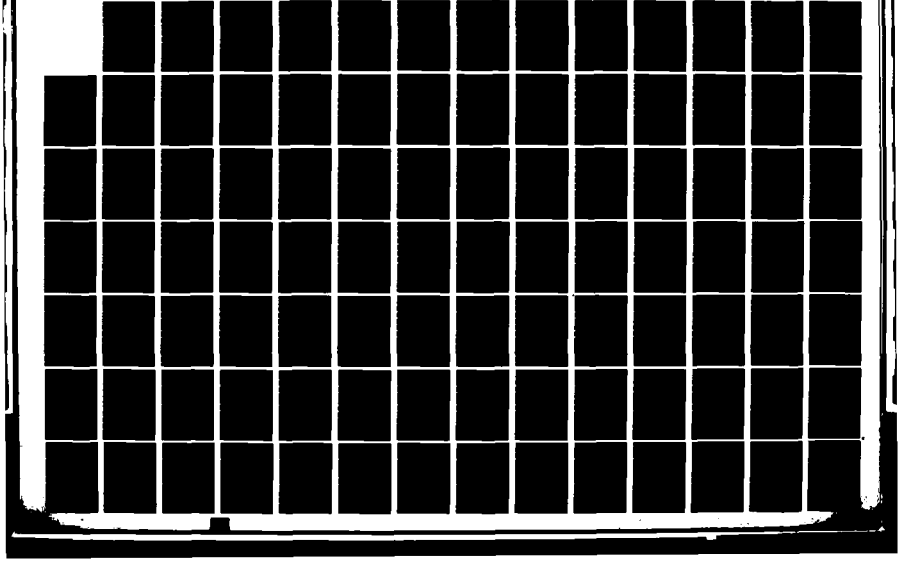
TABLE 20. FOAM TEST RESULTS WITH JP-4 AND HYDRAULIC FLUIDS[2]

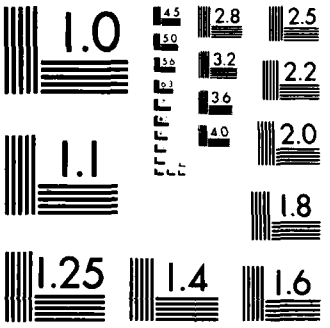
NB#	Treated samples	Foam test mL before treatment	Foam test mL after treatment
2776315A	2% JP-4	50	50
2776315B	Extra JP-4 additives (2%)	50	50
2776315C[1]	Oil from 2276315B heated to 320°C for 7 hr.	50	225
2276316A	Ten drops each of 2 different hydraulic fluids - ester based.	45	50
2276316B	2276316A plus additional 10 drops each of the hydraulic fluids.	50	9[illegible]

1. Simulate distillation conditions.
2. In 200 mL of low foaming formulated Emery basestock.

AD-A135 926 TURBINE ENGINE LUBRICANT RECLAMATION(U) MONSANTO RESEARCH CORP DAYTON OHIO DAYTON LAB R J BRUNS ETAAL. JUN 83 MRC-DA-1120 AFWAL-TR-83-2042 F33615-79-C-2052 2/3

UNCLASSIFIED F/G 11/8 NL





Used oil 0-79-2 was also unreclaimable, a GC chromatogram, Figure J-20, does not indicate any contamination. Since the used oil is only one out of 15 that does not pass the foam test the sample in a plant processing situation could easily be diluted with others.

Used oil 0-79-2 was also distilled without caustic (stoichiometric amount to neutralize the acid no.) to show that caustic does indeed lower, or decrease the acid number compared to no treatment. The acid number of the caustic treated oil is almost 1/2 that of the non-treated oil. It appears that the most dramatic effects of acid number lowering occurs on oils having starting acid numbers of 0.75 to 1.0 or greater (see Appendix I).

Also included in Appendix J are the IR and HPLC chromatograms of JP-4 and the hydraulic fluids.

3.9.2 Use of Basic Material to Lower Acid No.

Figure 54 shows a schematic diagram of the adsorbent treatment in the plant. The equipment was prepared similar to the distillation equipment. The cotton filter bag (for the bag filter) was washed 2 times and rinsed several times, with the last 3 rinses being deionized water.

Initially we used $Ca(OH)_2$ to lower the acid number and started getting inconsistant and poor foam test results. The problem was traced to contamination from the equipment and also, $Ca(OH)_2$ itself was found to cause foaming. Treatment with attapulgas clay was not beneficial. Lab studies, using $Ba(OH)_2 \cdot H_2O$ in place of $Ca(OH)_2$ to lower the acid number in our reclamation process, were consistently successful. The use of $Ba(OH)_2 \cdot H_2O$ resulted in 0.00 acid numbers compared to 0.02 to 0.2 with $CA(OH)_2$ treatment. The foam test results after $Ba(OH)_2 \cdot H_2O$ treatment were comparable to foam volumes prior to treatment. We determined that the minimum level of $Ba(OH)_2 \cdot H_2O$ for treatment was 0.77% by weight or the stoichiometric amount to neutralize the acid number.

Most oils after $Ba(OH)_2 \cdot H_2O$ [also $Ca(OH)_2$] treatment had after 1 day of standing, turned from a crystal clear to a cloudy liquid with some precipitate. By raising our treatment temperature from 20°C to 50°C we were able to increase the precipitation (ppt) rate so it could be removed along with the $Ba(OH)_2 \cdot H_2O$ during filtration. The acid number remained at zero after increased temperature treatment.

The earlier processed oils that had developed high foam test volumes due to $Ca(OH)_2$ treatment and equipment contamination were successfully lowered by redistillation and treatment with $Ba(OH)_2 \cdot H_2O$.

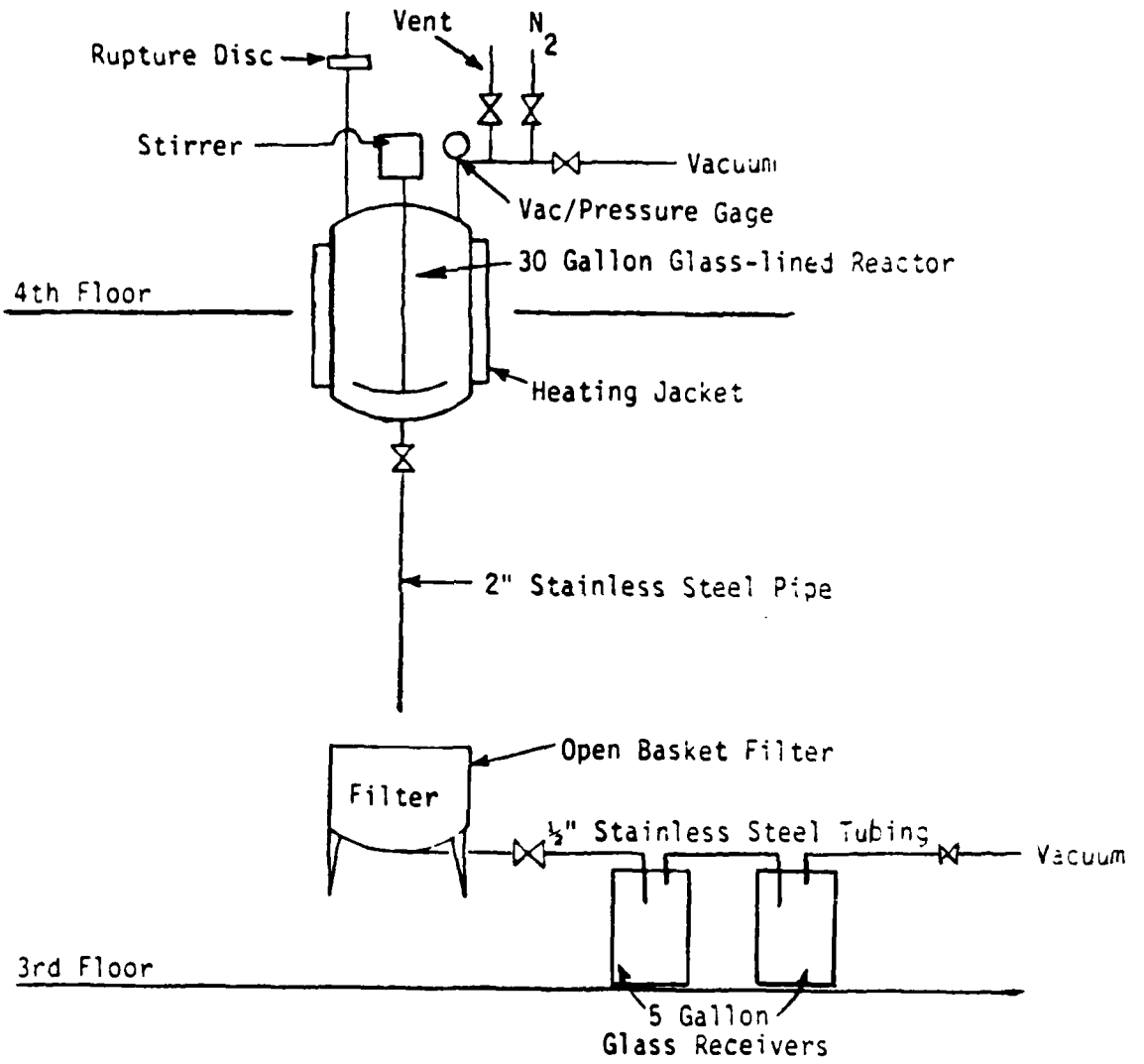


Figure 54. Equipment design for $Ba(OH)_2 \cdot H_2O$ adsorbent treatment in Pilot Plant.

Two reclaimed basestocks 0-79-7 and -14 and to some extent 0-79-12 and-10 had developed precipitate after a couple of months storage prior to reformulation. Various samples were prepared from 0-79-14 to solve the problem. Table 21 describes the various treatments and their results. In the plant we first filtered the batches and then heated them to 60°C prior to reformulation.

TABLE 21. SAMPLES PREPARED TO SOLVE PRECIPITATE PROBLEMS IN RECLAIMED BASESTOCK 0-79-14 (1997651)

Sample No.	Treatment	Storage results*
2000251A	Control - only filtered	ppt
2000251B	Held at 50°C for 16 hours/filter	No ppt
2000251C	Held at 60°C for 16 hours/filter	No ppt
2000251D	50°C for 16 hours with $Ba(OH)_2 \cdot H_2O$/filter	ppt
2000251E	60°C for 16 hours with $Ba(OH)_2 \cdot H_2O$/filter	ppt
2000251F	Fullers earth @ 50°C for 1/2 hour/filter	ppt

*Samples stored for approximately 1-1/2 months, except F-only 1 month.

3.9.3 Adsorbent Treatment

Two used oils 0-79-11 and 0-79-9 (first group sent out for testing) were formulated without attapulgas clay treatment, sent to Alcor Testing Labs for MIL-L-7808H testing. It was possible that we would be able to exclude clay treatment from the process. Originally the clay was used to remove trace metals and surfactants, but trace metals are lowered through distillation and $Ba(OH)_2 \cdot H_2O$ treatment. Attapulgus clay also has been shown not to remove troublesome surfactants in the oil. Later it was found that the clay also threw the accelerated storage stability test out of specifications. The results of the MIL-L-7808H testing indicated no need for clay treatment, so none of the reclaimed oils in the plant were clay treated.

We also had looked at ion exchange resins in the lab [Rexyn 101(H), Amberlyst 15, and Amberlite 200] to remove surfactants that may have caused foaming. However, no beneficial effects were found.

3.9.4 Reformulation

Figure 55 shows a schematic diagram of the reformulation equipment in the plant. The equipment was prepared similar to the distillation equipment. The Filterite inline cotton filter was washed with 5 gallon of virgin basestock. The first quantity of virgin

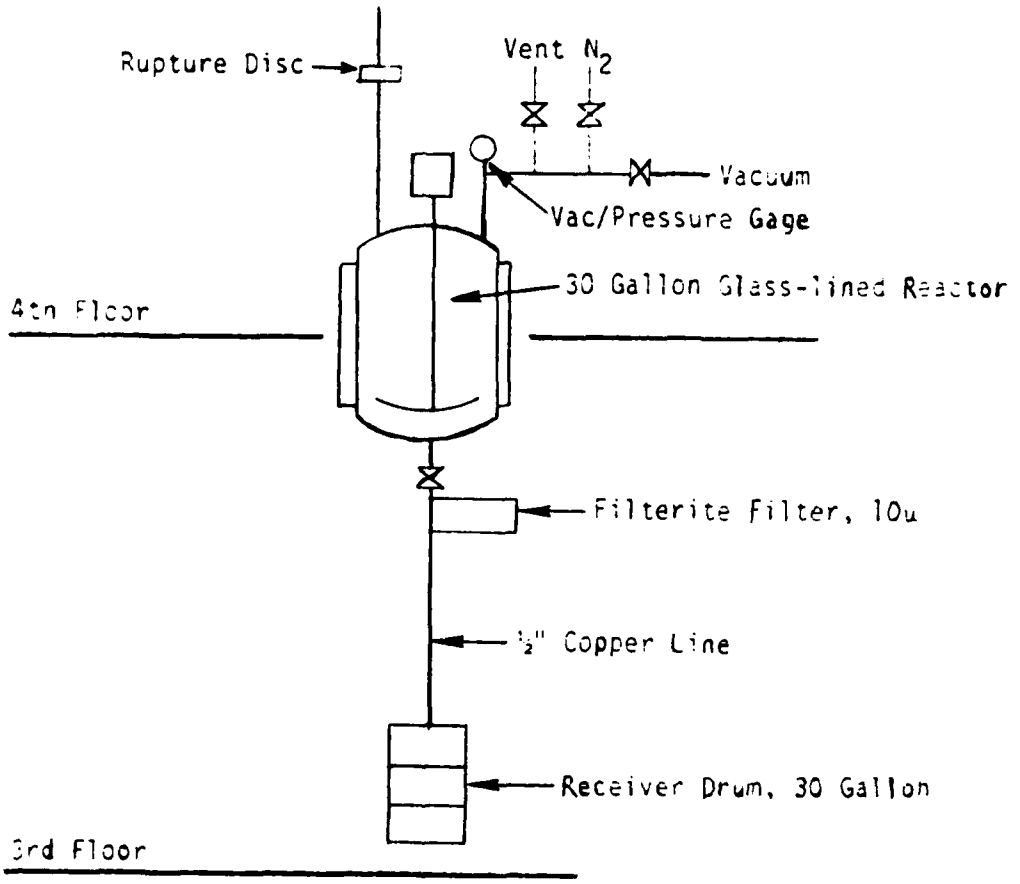


Figure 55. Equipment design for oil reformulation.

oil through the filter had a high foam test volume, while a later sample had a low volume. The unlined steel drums were washed with acetone and dried with a nitrogen stream.

Each reclaimed basestock was analyzed prior to reformulation by HPLC for DODPA, PANA, and TCP, major additives in the 7808H oils. Appendix C lists the analytical conditons for the analysis. The major additive levels in the oils were taken into consideration for the reformulation. Table 22 lists the additive and their level of addition. The lower level additives are assumed at a low concentration in the reclaimed oil.

Prior to reformulating a batch in the plant, we did a reformulation in the laboratory for foam testing and acid number determination. We found that there was a correlation between the constituent level of the major additives and foam test level. This suggested that TCP maybe interacting with PANA to form an amide which is known to lower surface tension and act as a surfactant. We finally did reformulation studies on each batch to maintain a low foam volume. Tables 23 and 24 bring out the difficulty in balancing different additive levels. This difficulty is a result of not knowing all the additives and their levels in the oil. We finally maintained a 2% antioxidant level with DODPA (no PANA addition) and did not necessarily add TCP. By this time we had some test results back on load bearing tests and had satisfactory results at lower levels of TCP.

We found it unnecessary to add virgin base stock for viscosity correction to any batch.

In the plant the additives were dissolved at 80°C/1 hr.

3.9.5 MIL-L-7808H Test Results

The 7808H test results for the 10 batches of used oil reclaimed in the pilot plant are in Appendix E. Table 25 summarizes the major problems encountered with these batches.

Initially foaming results were high. High test volumes were traced to major additive levels as discussed in Section 3.9.4.

TABLE 22. ADDITIVE LEVELS ADDED TO THE RECLAIMED BASESTOCKS

Additives	Percent by weight
Tricresyl phosphate (TCP)	2.0
4,4'-Dioctyldiphenylamine (DODPA)	1.0
Phenyl-α-naphthylamine (PANA)	1.0
Benzotriazole	0.1
Triphenyl phosphite	0.1
Quinizarin	0.05
Antioxidant 703	0.2

TABLE 23. FOAM TEST RESULTS OF REFORMULATION STUDIES

Percent[b]	0-79-15 (2000247)							0-79-8 (2000248)						0-79-8[c] 1997645		0-79-15[c] 1997650	
	#1	#2	#3	#4	#5	#6	#7[a]	#1	#2	#3	#4	#5[a]	#6	#1	#2	#1	#2
TCP	1.32	0	0	0.66	0	0.4	0.8	1.0	0.4	0.4	0	0.4	1.0	1.0	1.6	0.7	1.2
DODPA	0.2	0.2	0.2	0.2	0.5	0.5	0.5	0.26	0.26	0.26	0.49	0.49	0.49	1.24	1.24	1.3	1.3
PANA	0.3	0.3	0.3	0.3	0	0	0	0.23	0.23	0.23	0	0	0	0.27	0.27	0.2	0.2
Benzotriazole	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1				
TPP	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1				
Quinizarin	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05				
Antioxidant 703	0.2	1.5	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.2	0.2	0.2	0.2				
Foam test level	>200	45	20	70	10	15	65	65	80	80	10	20	95				

[a]Formulation used in pilot plant batch.

[b]Level of additives added to samples, % wt to oil wt.

[c]Major additive level in basestock before reformulation. Number one samples were results of first analysis, number two-results of the second.

TABLE 24. FOAM TEST RESULTS OF REFORMULATION STUDIES

Percent[b]	0-74-7 2000267 Additive level before formulation	#1[a]	0-74-14 2000268 Additive level before formulation	#1	#2	#3	#4	#5[a]	#6	0-79-12 2000275 Additive level before formulation	#1	#2	#3[a]	#4	0-79-12 2000274 Additive level before formulation	#1	#2[a]
TSP	0.8	1.20	1.00	0.5	0	0	0.5	0	0.5	1.19	0.81	0	0	0.4	1.24	0	0
EDTA	1.32	0.18	1.20	0.3	0	0.3	0	0.54	0.54	1.29	0.21	0.21	0.35	0.35	1.46	0	0.24
NTA	0.27	0.23	0.26	0.24	0	0.24	0	0	0	0.36	0.14	0.14	0	0	0.03	0.2	0
Benzotriazole	0	0.1	0	0.1	0.1	0.1	0.1	0.1	0.1	0	0.1	0.1	0.1	0.1	0	0.1	0.1
TPP	0	0.1	0	0.1	0.1	0.1	0.1	0.1	0.1	0	0.1	0.1	0.1	0.1	0	0.1	0.1
Colorization	0	0.05	0	0.05	0.05	0.05	0.05	0.05	0.05	0	0.05	0.05	0.05	0.05	0	0.05	0.05
Antioxidant 500	0	0.2	0	0.2	0.2	0.2	0.2	0.2	0.2	0	0.2	0.2	0.2	0.2	0	0.2	0.2
Foam test level, ml	0	15	0	60	20	25	75	20	60	0	20	75	25	65	0	65	50

[a] Formulation used in Pilot Plant batch.

[b] Level of additive added to sample, % wt to oil wt.

TABLE 25. SUMMARY OF MIL-L-7808H TEST PROBLEMS

Test groups	Treatments	Problems
1st Group of 2		
1997659 (0-79-9)	Distilled	Some foaming - traced to higher level of TCP & PANA, shown in lab to cause foaming
1997658 (0-79-11)	$Ba(OH)_2 \cdot H_2O$	Elastomer results slightly off
2nd Group of 2		
1997695 (0-79-6)	Distilled	Foaming - OK
1997693 (0-79-13)	$Ba(OH)_2 \cdot H_2O$	Elastomer results are better (lower level of TCP)
	Attapulgus clay Also 0-79-13 has 2 x's level of quinizarin	Accelerates storage stability test ~288 normal additive level - ~190 with 2 times quinizarin
Group of 6-1 liter[3]		
Sample for accelerated storage	Assorted	Attapulsus clay appears to adversely increase accelerated storage test results
3rd Group of 2^2		
2000247 (0-79-15)[1]	Distilled	Some static foam problems
2000248 (0-79-8)[1]	$Ba(OH)_2 \cdot H_2O$	
4th Group of 2^2		
2000267 (0-79-7)	Distilled	Accelerated storage stability test for 0-79-14
2000268 (0-79-14)[1]	$Ba(OH)_2 \cdot H_2O$	Both batches had heavy precipitate prior to reformulation
5th Group of 2^2		
2000275 (0-79-12)[1]	Distilled	Both batches had light precipitate prior to reformulation
2000273 (0-79-10)	$Ba(OH)_2 \cdot H_2O$	

[1]Oil distilled two times.
[2]Contains two times the amount of Ethyl 703.
[3]See Table 26.

Some elastomer results were high on the earlier batches. In the later batches the TCP level was lowered and there was a significant improvement.

The corrosion and oxidation results of some batches at 96 hours are higher than expected. The data is similar to results from a diester basestock provided by Rohm and Haas. MIL-L-7808G (di & triester) oils would not be expected to have the same stability as the MIL-L-7808H oils.

The accelerated storage stability results had been inconsistent throughout this program. It was learned after the completion of the work phase, that miscalculations by an outside testing laboratory had resulted in erroneously high test results. These results led to studies to improve the storage results. Table 25 displays the various treatments and results of the reclaimed batches. Also, we did a modified accelerated storage test of a sample consisting of 3 of the 5 formulated virgin basestocks used to verify MRC's additive package earlier in this program. The results are listed below:

Accelerated storage test on sample 1 1/2 years in storage	Specification	Results
0.0 hr		0.3
168 hr	150 max.	69.9

We also worked up 6 samples by various treatments for accelerated storage testing. Table 26 summarizes the treatments and results of these samples. The samples indicate that the attapulgus clay we had used to treat the 2nd group (Table 25) in the pilot plant may have been instrumental in increasing the accelerated storage test results.

Still working with erroneous test results, we then processed the remaining batches in the plant with no clay treatment and doubled the ethyl antioxidant 703 additive level. We then went back at the end of the program and added additional antioxidant 703 (to bring the level to 0.20%) to the attapulgus clay treated batches (see Table 25) to correct their storage results. Table 27 contains the results of retesting. As can be seen, the test results were improved.

We conclude from the revised data, the attapulgus clay treated batches would have passed the accelerated storage test without clay treatment, we then have 90% passing on the test.

TABLE 26. ACCELERATED STORAGE TEST SAMPLES/RESULTS

NBP	Treatment	Test Results 25 max. 48 hrs.	Test Results 150 max. 168 hrs.
2000227A	Treated with attapulgus clay previously dried @ 500°C, formulated with standard additive package.	2.1	42.1
2000227B	Treated with attapulgus clay as received and used in Pilot Plant formulated with standard additive package.	21.7	106.4
2000227C	Treated with Fullers earth previously dried @ 500°C as in earlier contract (AFAPL-TR-78-50), formulated with standard additive package.	8.3	17.6
2000227D	Treated with Fullers earth as received, formulated with standard additive package.	0.4	50.5
2000227E	No clay treatment, 2 times normal level of Ethyl Antioxidant 703 (storage stability additive).	0.5	47.6
2000227F	No clay treatment, 2 times normal level of quinizarin (anti-lead corrosion additive).	3.0	48.1

TABLE 27. ACCELERATED STORAGE STABILITY TEST RESULTS

Addition of Extra Ethyl 703 to Attapulgus Clay Treated Batches

Comments	NBP	48 hrs	168 hrs
Original	1997693(0-79-13)	12.7	190.7
	1997695(0-79-06)	57.8	288.5
10 months later, extra Ethyl 703 added and retested	1997693(0-79-13)	3.0	85.7
	1997695(0-79-06)	30.6	136.6

The ten reclaimed oil batches were blended together in pairs for 5 bearing deposition tests, to lower testing costs. Table 28 lists the pairs blended and reasoning for the combinations. Generally all results were within specifications, a few were 4 to 6% over, but we feel no reason for concern (see Appendix E). Groups 4 and 5 had significantly higher total sludge during testing then the other groups. The inlet/outlet screens in the test equipment had a sludge similar to the precipitates formed in the plant. There was no precipitate prior to testing. 90-96% of the sludge was removed on the 1st screen weighings. Examination of plant data on these oils, show that they were hydroxide treated below 45°C. We believe that the sludge formation would have been less if the hydroxide treatment temperature would have be[illegible] higher.

TABLE 28. BATCH BLENDING FOR BEARING D[illegible]OSITION TESTS

Group	NBP	Used oil	C[illegible]ents
1	2000248 2000247	(0-79-08) (0-79-15)	Good batches.
2	2000267 2000268	(0-79-07) (0-79-14)	Batches had developed heavy precipitation on sitting prior to reformulation.
3	2000273 2000275	(0-79-10) (0-79-12)	Batches had developed light precipitation on sitting prior to reformulation.
4	1997693 1997695	(0-79-13) (0-79-06)	Poor accelerated storage results; batches were attapulgus clay treated.
5	1997659 1997658	(0-79-09) (0-79-11)	

3.9.6 The Optimized Process

The following discussion details the process, as developed and optimized in the pilot plant. The operation discussed follow those outlined in Figure 56, Section 5. The materials used are those specified in Appendix H. Not all the operations discussed were necessary for the oils reclaimed in this program, i.e., phase separation, but they are included to keep the process as general as can be envisioned being required.

It is assumed that the used oils will arrive at the processing plant in 55 gallon drums. A 500 mL sample should be removed. A acid number and a GC/IR analysis should be run on the sample.

A acid no. >10.0 or a unusual GC or IR pattern would constitute rejection of the drum. Next, a lab distillation of the sample, followed by a foam test. If after 2 distillations a sample does not pass the foam test, discard the drum. NaOH will not need to be added to the sample for the lab distillations.

The next step will be to remove solid insoluble materials present (e.g., wear metals, sludges), by pumping through a 100 μm cartridge (cotton) type filter, into the still. These materials are present in very low amount, usually less than 0.2% by weight.

The oil is allowed to sit overnight and then a small quantity is removed from the bottom of the still for identification of a insoluble liquid phase. Next a stoichiometric addition of NaOH/methanol (usually 100 g NaOH/½ gallon methanol) solution to neutralize the acid no. (see Appendix F) is added to the still (short path distillation). The oil is distilled at 15 mm. The forerun is collected up to vapor temperature of 135°C and discarded. The main cut is collected from a vapor temperature of 135°C to a pot temperature of 325°C. There is an average of 85% recovery after distillation.

The distillate (main cut) is foam tested and a acid no. determined. A foam test over 20 mL indicate the distillate should be redistilled.

The oil is then treated with $Ba(OH)_2 \cdot H_2O$ at 55-60°C for 15-18 hrs. The amount of hydroxide added is 0.77% by wt (see Appendix F), unless if a larger amount is necessary to stoichiometrically neutralize the acid no. The oil is filtered to remove the hydroxide; filter aid maybe necessary to prevent a slimy and slow filtering cake. Losses are usually 5%.

At this point the reclaimed base stock should have a acid number of 0.0, but if over 0.1, retreat with the hydroxide. A foam test >35 mL indicates the oil should be redistilled. A value >35 mL at this point would probably result in a >100 mL after reformulation.

Next a HPLC analysis of the oil for major additive levels. A sample should be reformulated in the lab and foam tested. Viscosities should also be determined.

The plant batch can then be reformulated and heated to 80°C for 1 hour, filtered through a 10 μm inline cotton filter and pumped into a storage tank. A poor foam test or acid no. >0.3 indicates need for a redistillation. Viscosities out of specs. can be corrected through use of virgin oil.

Another assumption is the reclaimed oil will be kept in a storage tank until results of MIL-L-7808H tests. The oil will then be packaged into sealed containers for delivery.

4. DISPOSAL

In this section we'll discuss the disposal of material generated by the reclamation process.

4.1 NON-RECLAIMABLE OIL

Oils that do not pass the initial screening or prove at a later time unreclaimable, can be sold to other reclaimers for use as a plasticizer.

4.2 STILL BOTTOMS

The majority of the additives distill over, leaving a very low additive level in the still bottoms. The bottoms are similar to materials the asphalt producers use.

4.3 STILL TOPPINGS, WASTE ACETONE

Still toppings consisting of toluene, kerosene, low molecular weight oil and other similar material, can be mixed with fuel oil for on site use. The acetone from still clean-outs could also be disposed of, in the same manner.

4.4 $Ba(OH)_2 \cdot H_2O$/FILTER AID

Filter aid mixed with $Ba(OH)_2 \cdot H_2O$ will need to be buried in an EPA approved land fill.

5. COST ANALYSIS - ENGINEERING STUDY

Two cost analyses were to be performed. One based on a process intended for maximum yield and one for maximum cost effectiveness.

The present process operates at maximum yield and maximum cost effectiveness. The pre-cut range was selected for removal of low boiling contaminants. The still bottom consists of undistillable/ unrecoverable material.

The process developed in the earlier contract had included clay and charcoal treatment. These materials are ideal for recycling to lower costs, but has since been found unnecessary. Therefore, only one cost analysis is presented.

A cost analysis of the reclamation process was carried out to estimate the cost of reclaiming used oils in batch size of 1,000, 2,500, 5,000, and 10,000 gallons. The operations involved in the processing are shown in Figure 56.

The capital cost estimates for installation of a process in an existing plant were made using a 1,000 gallon batch size as the basic unit and escalating the costs to other sizes using an escalation factor of 0.6, an average value used frequently in the types of calculation [1]. The standard equation for such types of calculation, where $cost_o$ is the cost

$$cost = cost_o \left(\frac{S}{S_o}\right)^x$$

for 1,000 gallon scale, S is the size factor and x the escalation factor, was used. The capital cost data and assumptions are presented in Tables 29 and 30.

Installation costs can vary considerably depending on several factors, e.g., level of instrumentation, amount of piping required, etc. Consequently, the capital costs can vary.

The cost analysis was made based on the assumption that a plant was set up and operating continuously over a year period. This assumes an adequate used oil supply. No cost is included for collection and delivery of used oil to reclamation site.

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[1] Peters, M. S., and Timmerhaus, K. D., "Plant Design and Economics for Chemical Engineers," 2nd edition, 1968 McGraw Hill Book Company, New York, NY.

Popper, H., editor, "Modern Cost-Engineering Techniques," 1970, McGraw-Hill Book Company, New York, NY.

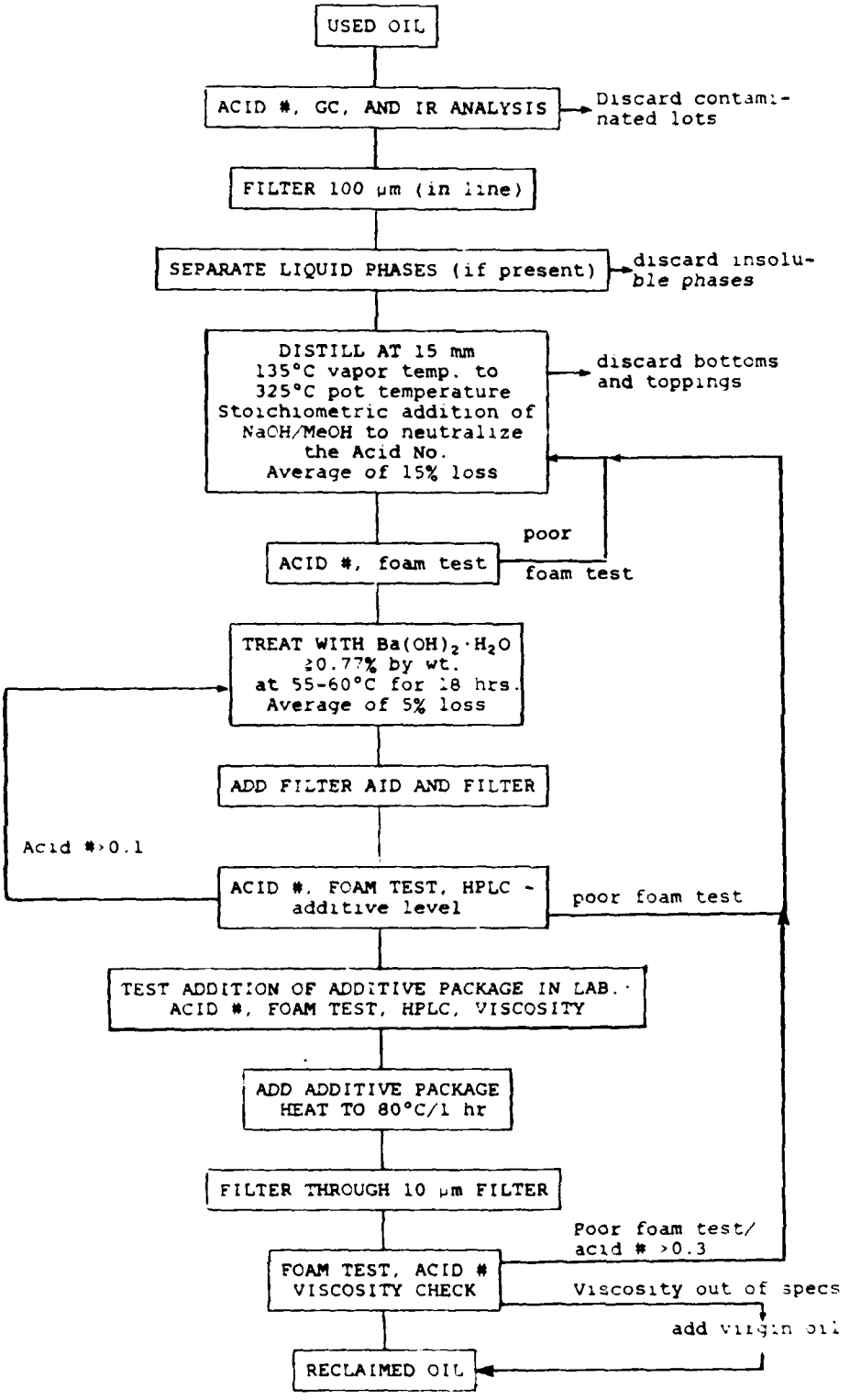

Figure 56. Reclamation process.

TABLE 29. CAPITAL COST DATA
(1,000 gal scale, 1 shift, 40 hr/wk)

PLANT

Equipment requirements (1,000gallons)		Cost, $ est. 1982	Installation, $
1	Filter (bag)	2,500	1,000
1	Gas still	75,000	75,000
1	Vacuum jet	18,000	3,000
2	Jacketed agitator vessel	30,000	120,000
1	Condensor	8,000	-*
1	Receiver	10,000	5,000
1	Bottling equipment	18,000	2,500
28	Product storage tanks	98,000	84,000
		249,500	285,500

*Installation included in with jacketed agitation vessels

TOTAL PLANT $550,000

LAB

Equipment requirements	Cost, $ est. 1982
HPLC	12,000
Auto sampler/GC	16,000
IR	12,000
Installation/other equipment	15,000
	55,000

The projected recovery efficiency for this process is 73%, other estimated costs are presented in Table 31, along with cost breakdown for the four batches sizes. The cost assigned to utilities is considered to be a very crude estimate and was derived by calculating the cost of energy required for distillation and multiplying the answer by 5 to cover the costs of the remaining operation cost for filtration, pumping materials, etc.

Not included in the above analysis is the cost incurred if virgin base stock must be added to adjust the viscosity. This is treated here as a separate case since this addition was found not to be necessary in our study.

TABLE 30. ASSUMPTIONS (1,000 gal. batches)

Waste oil arrives in 55 gal drums

Each drum is tested by GC, IR, acid # determination and lab distillation/foam test

Each plant batch requires 8 hrs to distill and 8 hrs to clean-up and recharge

$H_{\overline{V}}$ 50 g-cal/g

Specific gravity 0.93

2 Month turnaround time for commercial testing ∴ need 800 gal. storage tanks

40 hr work week - 1 shift

100 batches/yr, 2 days/batch, 20% downtime

$130,000 labor costs/yr

90% of processed batches will be good

85% recovery on each batch due to distillation

95% recovery on each batch due to $Ba(OH)_2 \cdot H_2O$ treatment

1,000 gal still can only contain 800 gal of actual oil

15 gal removed out of each batch for testing

56,790 actual gal processed each yr

Capital costs spread out over 3 years

Qualification tests $5,000 each

Qualified batches are packaged in pt or qt cans

TABLE 31. OPERATING COSTS ($/gallon of oil)

	1,000 gal scale (56,790 gal/yr)	2,500 gal scale (143,850 gal/yr)	5,000 gal scale (289,200 gal/yr)	10,000 gal scale (579,900 gal yr)
Labor: 1 shift/40 hr wk[a]	2.29	1.04	0.59	0.33
Materials				
$Ba(OH)_2 \cdot H_2O$	6.72×10^{-2}	→	→	→
Filteraid	8.6×10^{-3}	→	→	→
Additives[b]				
(0.3%) DODPA	5.95×10^{-2}	→	→	→
(0) PANA	-	→	→	→
(0.5%) TCP	6.64×10^{-2}	→	→	→
(0.1%) TPP	2.67×10^{-2}	→	→	→
(0.1%) Benzotriazole	7.11×10^{-2}	→	→	→
(0.05%) Quinizarin	3.54×10^{-2}	→	→	→
(0.2%) Ethyl 703	3.27×10^{-2}	→	→	→
Utilities	0.38	→	→	→
Capital related costs[c]	0.07	0.07	0.07	0.07
Capital costs[d]	3.55	2.32	1.76	1.33
Disposal [$Ba(OH)_2 \cdot H_2O$]	0.08	0.08	0.08	0.08
Qualification tests	8.80	3.48	1.73	0.86
Miscellaneous	1.00	1.00	1.00	1.00
No. of people	5	6	7	8
Total cost/gallon	16.54	8.74	5.98	4.42

Labor: 3 shifts/40 hr wk	170,370 gal/yr
No. of people	15
Total cost/gallon	16.97

[a] Labor costs split equally between plant and lab.

[b] Additive levels are based on what actually was added to successfully reclaimed oil.

[c] Maintenance.

[d] Spread over 3 years.

6. CONCLUSIONS

1. A feasible process for reclamation of synthetic ester turbine engine oils was developed in an earlier program. The main features of the process consist of distillation to recover base stock, treatment with adsorbents to remove degradation products, and addition of additives to upgrade the oil to meet specifications.

2. MRC's proposed additive package has been shown to be satisfactory in five different base stock formulations.

3. The use of NaOH during distillation helps lower the acid no., therefore decreasing the amount of $Ba(OH)_2 \cdot H_2O$ to use in the next step.

4. Treatment with $Ba(OH)_2 \cdot H_2O$ to lower the acid number depends on the following rule of thumb: $\geqq 0.77\%$ by weight $Ba(OH)_2 \cdot H_2O$ to oil. (A greater amount of $Ba(OH)_2 \cdot H_2O$ may be required to neutralize larger acid numbers.)

5. The use of bleaching clay and activated charcoal was found unnecessary.

6. An extensive used oil screening method had to be devised for identification of contaminated lots to eliminate those used oil samples not suitable for reclamation.

7. Extensive laboratory evaluation on each reclaimed basestock was necessary to determine optimum additive replenishment concentrations without causing high foam.

8. Ten 25 gallon batches of used oil were reclaimed. MIL-L-7808H test results on the batches were generally good. However, none of the ten batches completely passed all the tests.

9. A cost analysis of the process was carried out. Assuming a half million quarts of oil to be reclaimed each year, each gallon could be reclaimed for $8.74.

7. RECOMMENDATIONS

The process as now defined has been shown to be feasible for reclamation of used MIL-L-7808H oils. However, the following are areas in which it is recommended that further effort be placed for improvement and refinement of the process.

1. Additional 15-25 gallon plant reclamations for additional data.

    a. Additional MIL-L-7808H test results.

    b. Statistics on precipitation formation in processed oil.

    c. Correlate GC information of used oil with lab distillation/ foam test data to lower initial screening costs.

    d. To develop a better understanding between reformulation, foaming and accelerated storage stability results.

2. Two or three 500 gallon plant reclamations for additional scale-up information.

BIBLIOGRAPHY

Gunderson, R. C.; Hart, A. W.; et al. Synthetic Lubricants, New York, Reinhold Publishing Corp. 1962.

"Reclamation of Synthetic Turbine Engine Oil Mixtures," Technical Report AFAPL-TR-78-50, AF Aero Propulsion Lab, AF Wright Aeronautical Laboratories, AF Systems Command, Wright-Patterson Air Force Base, OH 45433.

Ronney, W. M. Lubricant Additives, Chemical Technology Review No. 104, Park Ridge, NJ, Noyes Data Corp. 1978.

Ronney, W. M. Synthetic Oils and Greases for Lubricants, Chemical Technology Review No. 72, Park Ridge, NJ, Noyes Data Corp. 1976.

"Turbine Engine Lubricant Reclamation," Technical Interim Report AFWAL-TR-81-2053, Aero Propulsion Laboratory, AF Wright Aeronautical Laboratories, AF Systems Command, Wright Patterson Air Force Base, OH 45433.

APPENDIX A

MIL-L-7808H TEST RESULTS FOR SELECTED FORMULATED VIRGIN BASE STOCKS

1732506 (Hatco base stock)

1732510 (Stauffer base stock)

1732511 (Rohm and Haas base stock) (Plexol)

1732508 (APL-furnished ATL 9148 base stock)

1732509 (APL-furnished ATL 9149 base stock)

ADDITIVE LEVELS USED IN VIRGIN BASE STOCKS

Additives	Percent by weight
Tricresyl phosphate	2.0
4,4'-Dioctyldiphenylamine	1.0
Phenyl-α-naphthylamine	1.0
Benzotriazole	0.1
Triphenyl phosphite	0.1
Quinizarin	0.05
Antioxidant 703	0.1

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SAMPLE Monsanto #1732511 Date 4/07/80

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number	0.30 Max.	0.14
Viscosity @ 210°F, cs	3.0 Min.	3.0
Viscosity @ 100°F, cs	Report	11.5
VISCOSITY STABILITY @ -65°F		
Original Oil, 35 Min., cs	17,000 Max.	11,341
After 3 Hours, cs	17,000 Max.	11,633
Viscosity Change, %	6.0 Max.	+2.6
FOAMING CHARACTERISTICS, STATIC		
176°F, Vol. after 30 Min. Aeration, ml	100 Max.	35
Collapse time, seconds	60 Max.	6.8
FOAMING CHARACTERISTICS, DYNAMIC		
176°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	24.6 sec.
Volume @ 1500 cc air, cc	150 Max.	20
Collapse time, minutes	60 Max.	38.9 sec.
Volume @ 2000 cc air, cc	Report	20
Collapse time, minutes	60 Max.	33.8 sec.
230°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	31.8 sec.
Volume @ 1500 cc air, cc	150 Max.	10
Collapse time, minutes	60 Max.	31.8 sec.
Volume @ 2000 cc air, cc	Report	20
Collapse time, minutes	60 Max.	35.5 sec.

Sample Monsanto #1732511

	Specification	Results
FA ELASTOMER COMPATIBILITY, 72 HOURS @ 347°F		
% Swell	2.0 - 25.0	+15.8
Tensile Strength, % Change	50 Max.	-14.0
Elongation, % Change	50 Max.	+15.6
Hardness, No. Change	20 Max.	-5
LEAD CORROSION, 1 Hour @ 325°F		
Weight Change, mg/in^2	6 Max.	0.0
SILVER & BRONZE CORROSION, 50 HOURS @ 450°F		
Silver Weight Change, mg/in^2	3.0 Max.	-0.1
Bronze AMS4616, mg/in^2	3.0 Max.	0.0
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max.	0.67
Viscosity Change, %	Report	55.6
TAN Change	Report	22.88
Oil Consumption	Report	175 cc
RYDER GEAR TEST (see attached data sheet)		
2 Determinations, ppi	2400 Min.	2631

Sample Monsanto # 1732511

	Specification	Results
CORROSION AND OXIDATION STABILITY, 96 HOURS @ 392°F		
Corrosion:		
Steel, mg/cm^2	Report	-0.030
Silver, mg/cm^2	Report	-0.148
Aluminum, mg/cm^2	Report	+0.010
Magnesium, mg/cm^2	Report	-12.247
Bronze AMS4616, mg/cm^2	Report	+0.053
Titanium, mg/cm^2	Report	+0.050
M50 Steel, mg/cm^2	Report	+0.060
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	Mg
Staining	Report	None

Oxidation:

		16	24	40	48	64	72	88	96
Viscosity @ 100°F, % Change	Report	+4.3	+5.2	+13.0	+24.3	+56.5	+74.8	+113.9	+129.6
Viscosity @ 210°F, % Change	Report								+73.3
Total Acid Number, Change	Report	0.64	1.16	5.58	12.26	24.30	27.46	38.26	40.74
Evaporation Loss, %	Report								5.9
Sludge, Volume, %	Report								0.2

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SAMPLE Monsanto #1732509 Date 4/07/80

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number	0.30 Max.	0.17
Viscosity @ 210°F, cs	3.0 Min.	3.5
Viscosity @ 100°F, cs	Report	14.8
VISCOSITY STABILITY @ -65°F		
Original Oil, 35 Min., cs	17,000 Max.	16,642
After 3 Hours, cs	17,000 Max.	16,954
Viscosity Change, %	6.0 Max.	+1.9
FOAMING CHARACTERISTICS, STATIC		
176°F, Vol. after 30 Min. Aeration, ml	100 Max.	115
Collapse time, seconds	60 Max.	7.4
FOAMING CHARACTERISTICS, DYNAMIC		
176°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	12.7 sec.
Volume @ 1500 cc air, cc	150 Max.	30
Collapse time, minutes	60 Max.	14.7 sec.
Volume @ 2000 cc air, cc	Report	30
Collapse time, minutes	60 Max.	18.9 sec.
230°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	13.1 sec.
Volume @ 1500 cc air, cc	150 Max.	20
Collapse time, minutes	60 Max.	14.2 sec.
Volume @ 2000 cc air, cc	Report	20
Collapse time, minutes	60 Max.	14.7 sec.

	Specification	Results
FA ELASTOMER COMPATIBILITY, 72 HOURS @ 347°F		
% Swell	2.0 - 25.0	+15.8
Tensile Strength, % Change	50 Max.	-11.4
Elongation, % Change	50 Max.	+35.9
Hardness, No. Change	20 Max.	0
LEAD CORROSION, 1 Hour @ 325°F		
Weight Change, mg/in^2	6 Max.	0.0
SILVER & BRONZE CORROSION, 50 HOURS @ 450°F		
Silver Weight Change, mg/in^2	3.0 Max.	0.0
Bronze AMS4616, mg/in^2	3.0 Max.	0.1
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max.	0.56
Viscosity Change, %	Report	112.8
TAN Change	Report	14.41
Oil Consumption	Report	150 cc
RYDER GEAR TEST (see attached data sheet)		
2 Determinations, ppi	2400	3282

Sample Monsanto #1735209

	Specification	Results

CORROSION AND OXIDATION STABILITY, 96 HOURS @ 392°F

Corrosion:		
Steel, mg/cm^2	Report	-0.024
Silver, mg/cm^2	Report	-0.059
Aluminum, mg/cm^2	Report	0.000
Magnesium, mg/cm^2	Report	-0.036
Bronze AMS4616, mg/cm^2	Report	+0.010
Titanium, mg/cm^2	Report	-0.020
M50 Steel, mg/cm^2	Report	+0.030
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	None
Staining	Report	None

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, % Change	Report	+6.1	+7.4	+9.5	+10.1	+12.2	+12.8	+14.2	+14.2
Viscosity @ 210°F, % Change	Report								+8.6
Total Acid Number, Change	Report	0.51	0.83	1.17	1.23	1.57	1.69	1.91	2.30
Evaporation Loss, %	Report								3.8
Sludge, Volume, %	Report								0.0

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SAMPLE Monsanto #1732510 Date 4/07/80

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number	0.30 Max.	0.25
Viscosity @ 210°F, cs	3.0 Min.	3.1
Viscosity @ 100°F, cs	Report	12.8
VISCOSITY STABILITY @ -65°F		
Original Oil, 35 Min., cs	17,000 Max.	13,925
After 3 Hours, cs	17,000 Max.	13,484
Viscosity Change, %	6.0 Max.	+3.1
FOAMING CHARACTERISTICS, STATIC		
176°F, Vol. after 30 Min. Aeration, ml	100 Max.	30
Collapse time, seconds	60 Max.	3.8
FOAMING CHARACTERISTICS, DYNAMIC		
176°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	9.1 sec.
Volume @ 1500 cc air, cc	150 Max.	10
Collapse time, minutes	60 Max.	9.3 sec.
Volume @ 2000 cc air, cc	Report	10
Collapse time, minutes	60 Max.	10.3 sec.
230°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	8.5 sec.
Volume @ 1500 cc air, cc	150 Max.	10
Collapse time, minutes	60 Max.	8.2 sec.
Volume @ 2000 cc air, cc	Report	10
Collapse time, minutes	60 Max.	8.9 sec.

	Specification	Results
FA ELASTOMER COMPATIBILITY, 72 HOURS @ 347°F		
% Swell	2.0 - 25.0	+17.6
Tensile Strength, % Change	50 Max.	-11.9
Elongation, % Change	50 Max.	+17.2
Hardness, No. Change	20 Max.	-5
LEAD CORROSION, 1 Hour @ 325°F		
Weight Change, mg/in^2	6 Max.	-0.1
SILVER & BRONZE CORROSION, 50 HOURS @ 450°F		
Silver Weight Change, mg/in^2	3.0 Max.	-0.1
Bronze AMS4616, mg/in^2	3.0 Max.	-0.2
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max.	0.40
Viscosity Change, %	Report	82.0
TAN Change	Report	14.85
Oil Consumption	Report	125 cc
RYDER GEAR TEST (see attached data sheet)		
2 Determinations, ppi	2400 Min.	2671

Sample Monsanto # 1732510

	Specification	Results

CORROSION AND OXIDATION STABILITY, 96 HOURS @ 392°F

Corrosion:		
Steel, mg/cm^2	Report	+0.069
Silver, mg/cm^2	Report	-0.040
Aluminum, mg/cm^2	Report	+0.016
Magnesium, mg/cm^2	Report	+0.030
Bronze AMS4616, mg/cm^2	Report	+0.115
Titanium, mg/cm^2	Report	+0.050
M50 Steel, mg/cm^2	Report	-0.028
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	None
Staining	Report	None

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, % Change	Report	+6.3	+7.0	+7.0	+9.4	+10.7	+11.7	+14.1	+14.8
Viscosity @ 210°F, % Change	Report								+9.7
Total Acid Number, Change	Report	0.71	0.87	1.43	1.61	1.77	2.05	2.33	2.89
Evaporation Loss, %	Report								5.2
Sludge, Volume, %	Report								0.0

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SAMPLE Monsanto #1732506 Date 4/07/80

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number	0.30 Max.	0.20
Viscosity @ 210°F, cs	3.0 Min.	3.0
Viscosity @ 100°F, cs	Report	12.5
VISCOSITY STABILITY @ -65°F		
Original Oil, 35 Min., cs	17,000 Max.	12,136
After 3 Hours, cs	17,000 Max.	12,561
Viscosity Change, %	6.0 Max.	3.5
FOAMING CHARACTERISTICS, STATIC		
176°F, Vol. after 30 Min. Aeration, ml	100 Max.	15
Collapse time, seconds	60 Max.	3.2
FOAMING CHARACTERISTICS, DYNAMIC		
176°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	7.9 sec.
Volume @ 1500 cc air, cc	150 Max.	10
Collapse time, minutes	60 Max.	8.8 sec.
Volume @ 2000 cc air, cc	Report	10
Collapse time, minutes	60 Max.	8.5 sec.
230°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	8.4 sec.
Volume @ 1500 cc air, cc	150 Max.	10
Collapse time, minutes	60 Max.	8.5 sec.
Volume @ 2000 cc air, cc	Report	10
Collapse time, minutes	60 Max.	7.6 sec.

	Specification	Results
FA ELASTOMER COMPATIBILITY, 72 HOURS @ 347°F		
% Swell	2.0 - 25.0	+12.4
Tensile Strength, % Change	50 Max.	+2.1
Elongation, % Change	50 Max.	+9.3
Hardness, No. Change	20 Max.	-5
LEAD CORROSION, 1 Hour @ 325°F		
Weight Change, mg/in^2	6 Max.	0.0
SILVER & BRONZE CORROSION, 50 HOURS @ 450°F		
Silver Weight Change, mg/in^2	3.0 Max.	0.0
Bronze AMS4616, mg/in^2	3.0 Max.	-0.2
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max.	0.52
Viscosity Change, %	Report	48.8
TAN Change	Report	13.10
Oil Consumption	Report	110 cc
RYDER GEAR TEST (see attached data sheet)		
2 Determinations, ppi	2400 Min.	2919

Sample Monsanto # 1732506

	Specification	Results

CORROSION AND OXIDATION STABILITY, 96 HOURS @ 392°F

Corrosion:		
Steel, mg/cm^2	Report	+0.010
Silver, mg/cm^2	Report	-0.069
Aluminum, mg/cm^2	Report	-0.030
Magnesium, mg/cm^2	Report	-0.022
Bronze AMS4616, mg/cm^2	Report	-0.032
Titanium, mg/cm^2	Report	-0.026
M50 Steel, mg/cm^2	Report	-0.020
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	None
Staining	Report	None

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, % Change	Report	+5.6	+6.4	+8.8	+8.8	+10.4	+11.2	+12.0	+12.8
Viscosity @ 210°F, % Change	Report								+16.7
Total Acid Number, Change	Report	0.24	0.42	1.14	1.38	1.82	2.04	2.44	2.91
Evaporation Loss, %	Report								3.8
Sludge, Volume, %	Report								0.0

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(512) 349-3771

SAMPLE Monsanto #1732508 Date 4/07/80

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number	0.30 Max.	0.22
Viscosity @ 210°F, cs	3.0 Min.	3.4
Viscosity @ 100°F, cs	Report	14.2
VISCOSITY STABILITY @ -65°F		
Original Oil, 35 Min., cs	17,000 Max.	14,462
After 3 Hours, cs	17,000 Max.	14,799
Viscosity Change, %	6.0 Max.	+2.3
FOAMING CHARACTERISTICS, STATIC		
176°F, Vol. after 30 Min. Aeration, ml	100 Max.	100
Collapse time, seconds	60 Max.	5.1
FOAMING CHARACTERISTICS, DYNAMIC		
176°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	18.9 sec.
Volume @ 1500 cc air, cc	150 Max.	20
Collapse time, minutes	60 Max.	16.4 sec.
Volume @ 2000 cc air, cc	Report	20
Collapse time, minutes	60 Max.	19.5 sec.
230°F		
Volume @ 1000 cc air, cc	100 Max.	10
Collapse time, minutes	60 Max.	12.9 sec.
Volume @ 1500 cc air, cc	150 Max.	10
Collapse time, minutes	60 Max.	15.2 sec.
Volume @ 2000 cc air, cc	Report	20
Collapse time, minutes	60 Max.	13.9 sec.

	Specification	Results
FA ELASTOMER COMPATIBILITY, 72 HOURS @ 347°F		
% Swell	2.0 - 25.0	+16.1
Tensile Strength, % Change	50 Max.	-12.1
Elongation, % Change	50 Max.	+12.5
Hardness, No. Change	20 Max.	-5
LEAD CORROSION, 1 Hour @ 325°F		
Weight Change, mg/in^2	6 Max.	0.0
SILVER & BRONZE CORROSION, 50 HOURS @ 450°F		
Silver Weight Change, mg/in^2	3.0 Max.	0.1
Bronze AMS4616, mg/in^2	3.0 Max.	0.1
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max.	0.51
Viscosity Change, %	Report	118.3
TAN Change	Report	11.96
Oil Consumption	Report	135 cc
RYDER GEAR TEST (see attached data sheet)		
2 Determinations, ppi	2400 Min.	2815

Sample Monsanto #1732508

	Specification	Results

CORROSION AND OXIDATION STABILITY, 96 HOURS @ 392°F

Corrosion:		
Steel, mg/cm^2	Report	+0.020
Silver, mg/cm^2	Report	-0.099
Aluminum, mg/cm^2	Report	-0.010
Magnesium, mg/cm^2	Report	-0.030
Bronze AMS4616, mg/cm^2	Report	-0.010
Titanium, mg/cm^2	Report	0.000
M50 Steel, mg/cm^2	Report	+0.030
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	None
Staining	Report	None

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, % Change	Report	+5.6	+6.3	+8.5	+8.5	+9.8	+11.2	+11.9	+11.9
Viscosity @ 210°F, % Change	Report								+5.9
Total Acid Number, Change	Report	0.42	0.46	0.74	0.84	1.08	1.24	1.36	1.60
Evaporation Loss, %	Report								4.6
Sludge, Volume, %	Report								0.0

APPENDIX B

MIL-L-7808H TEST RESULTS FOR LAB RECLAIMED OIL

1830396-C/M-23 (Reclaimed oil, NaOH/methanol, vapor temperature distillation 135-285°C)

1830397-C/M-234 (Reclaimed oil, NaOH/methanol, vapor temperature distillation 135-305°C)

ADDITIVE LEVELS USED IN RECLAIMED BASE STOCK

Additives	Percent by weight
Tricresyl phosphate	2.0
4,4'-Dioctyldiphenylamine (DODPA)	1.0
Phenyl-α-naphthylamine	1.0[a]
Benzotriazole	0.1
Triphenyl phosphite	0.1
Quinizarin	0.05
Antioxidant 703	0.10

[a]Both samples inadvertently had more (3%) than 1% PANA added.

ALCOR, INC.
10130 Jones Maltsberger Road
San Antonio, Texas
(512) 349-3771

CUSTOMER Monsanto Research Corporation Date 10/16/80

	Specification	Results 183039-6C/M-23	Results 183039-7C/M-234
PHYSICAL & CHEMICAL PROPERTIES			
Neutralization Number (T.A.N.)	0.30 Max.	0.22	0.20
Viscosity @ 210°F, cs	3.0 Min.	3.0	3.1
VISCOSITY STABILITY @ -65°F			
Original Oil, 35 Minutes, cs	17,000 Max.	9,515	10,428
After 3 Hours, cs	17,000 Max.	9,777	10,340
Viscosity Change, %	6.0 Max.	+2.8	-0.8
FOAMING CHARACTERISTICS, STATIC			
176°F, Volume after 30 Min. Aeration, ml	100 Max.	100	120
Collapse Time, seconds	60 Max.	4.2	5.1
LEAD CORROSION, 1 HOUR @ 325°F			
Weight Change, mg/in^2	6 Max.	+0.1	+0.1
SILVER & BRONZE CORROSION, 50 HOURS @ 450°F			
Silver Weight Change, mg/in^2	3.0 Max.	-0.1	-0.1
Bronze AMS4616, mg/in^2	3.0 Max.	-0.3	-0.5
DEPOSITION NUMBER (see attached data sheets)			
Deposit Number	1.5 Max.	0.53	0.41
Viscosity Change, %	Report	+119.3	+38.3
T.A.N. Change	Report	25.04	11.42
Oil Consumption	Report	100 cc	50 cc

	Specification	Results 183039-6C/M-23	183039-7C/M-23
CORROSION & OXIDATION STABILITY, 48 HOURS @ 392°F			
Corrosion:			
Steel, mg/cm^2	±0.2	-0.010	-0.099
Silver, mg/cm^2	±0.2	-0.139	-0.050
Aluminum, mg/cm^2	±0.2	-0.073	-0.040
Magnesium, mg/cm^2	±0.4	-0.050	-0.030
Bronze AMS4616, mg/cm^2	±0.4	-0.119	-0.010
Titanium, mg/cm^2	±0.2	-0.040	-0.080
M-50 Steel, mg/cm^2	±0.2	-0.080	-0.046
Appearance of Metal Specimens:			
Pitting	Report	None	None
Etching	Report	None	None
Corrosion	Report	None	None
Staining	Report	None	None
Oxidation:			
Viscosity @ 100°F, Initial	Report	11.4	12.0
Viscosity @ 100°F, % Change	-5 to +25	+25.4	+23.3
Viscosity @ 210°F, Initial	Report	3.0	3.1
Viscosity @ 210°F, % Change	Report	+13.3	+12.9
Total Acid Number, Initial	Report	0.22	0.20
Total Acid Number, Change	4.0 Max.	5.03	4.80
Evaporation Loss, %	Report	2.5	2.1
Sludge, Volume, %	Report	0.0	0.0

APPENDIX C

ANALYTICAL CONDITIONS

Gas Chromatography

High Performance Liquid Chromatography

Thin Layer Chromatography

ANALYTICAL CONDITIONS

GAS CHROMATOGRAPHY

Column:	3% Dexsil 300 on Chromasorb W 80/100 0.125 in. OD 12 ft length acid washed and silanized
Sample:	10% in cyclohexane
Sample size:	1 µL
Helium flow rate:	40 cc/min
Inlet temperature:	300°C
FID temperature:	400°C
Temperature programs:	180-350°C at 4°C/min 100-350°C at 4°C/min

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

General Conditions

Column:	Partisil PXS 10/25 PAC
Sample:	10% in cyclohexane
Sample size:	10 µL
Mobile phase:	100% isooctane TO 70/30 methylene chloride/isooctane, 2 mL/min.
Program:	Linear, 20 min
Detector:	UV @ 254 nm
Chart:	0.5 cm/min

Specific Conditions

PANA AND DODPA ANALYSIS

Program:	Program #7 on Water's Solvent Programmer, 7 min., 2 mL/min.
Mobil phase:	100% isooctane to 95/5 methylene chloride/isooctane

ANALYTICAL CONDITIONS (continued)

TCP ANALYSIS

Program:	Program #6 on Water's Solvent Programmer, 15 min., 2 mL/min.
Mobil phase:	100% isooctane to 90/10 methylene chloride/isooctane
Sample:	100 µL of 30% oil

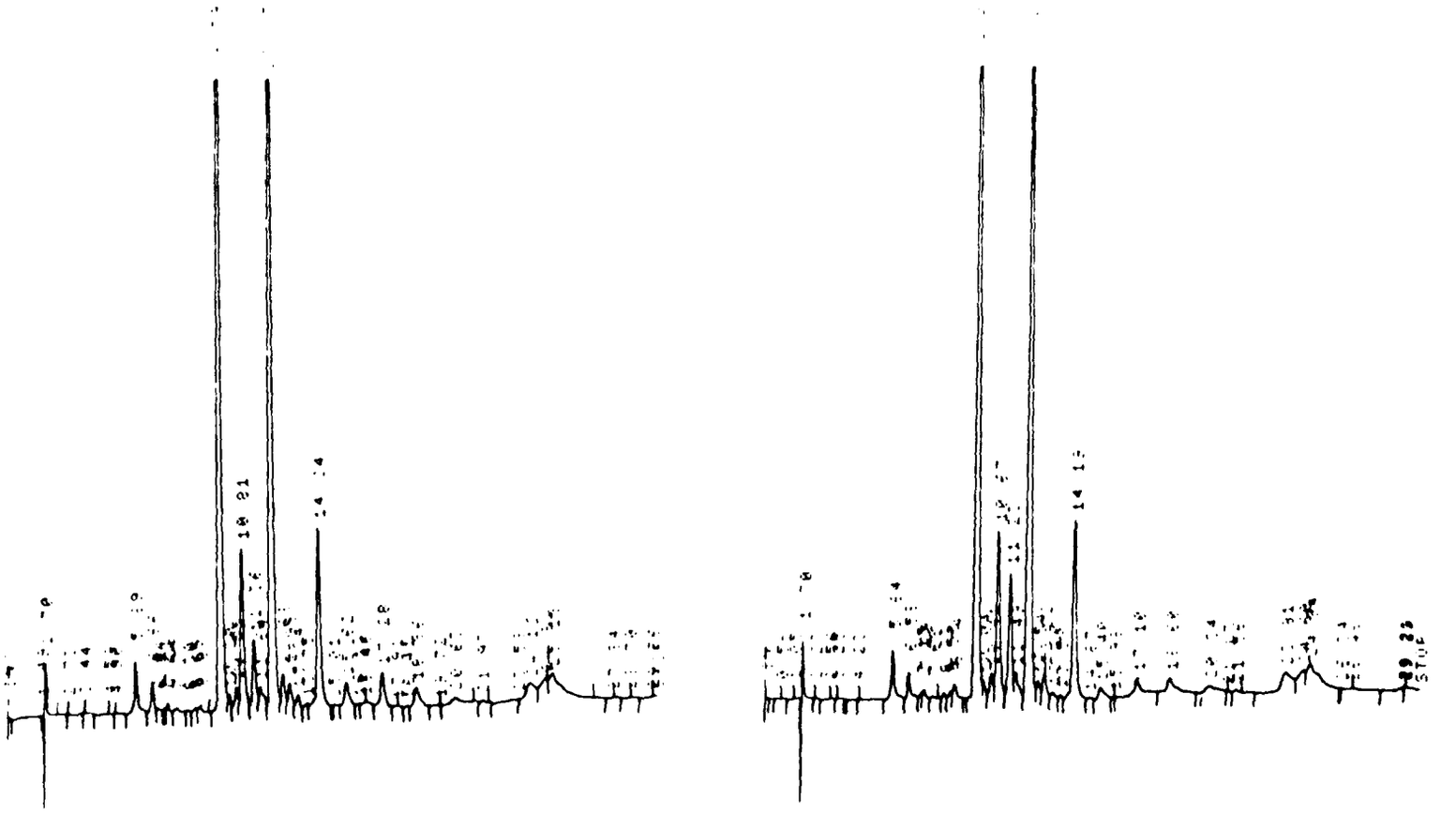
Area determination of peaks was found to be more accurate by calculating the areas by hand. More reproduceable data may be obtained with the use of second generation analytical software.

THIN LAYER CHROMATOGRAPHY

Plate:	Silica gel F-254 activated at 100°C for 1 hr
Sample:	30 µL 10% solution oil in cyclohexane
Mobile phase:	30/70 heptane/toluene
Lamp:	254 nm UV

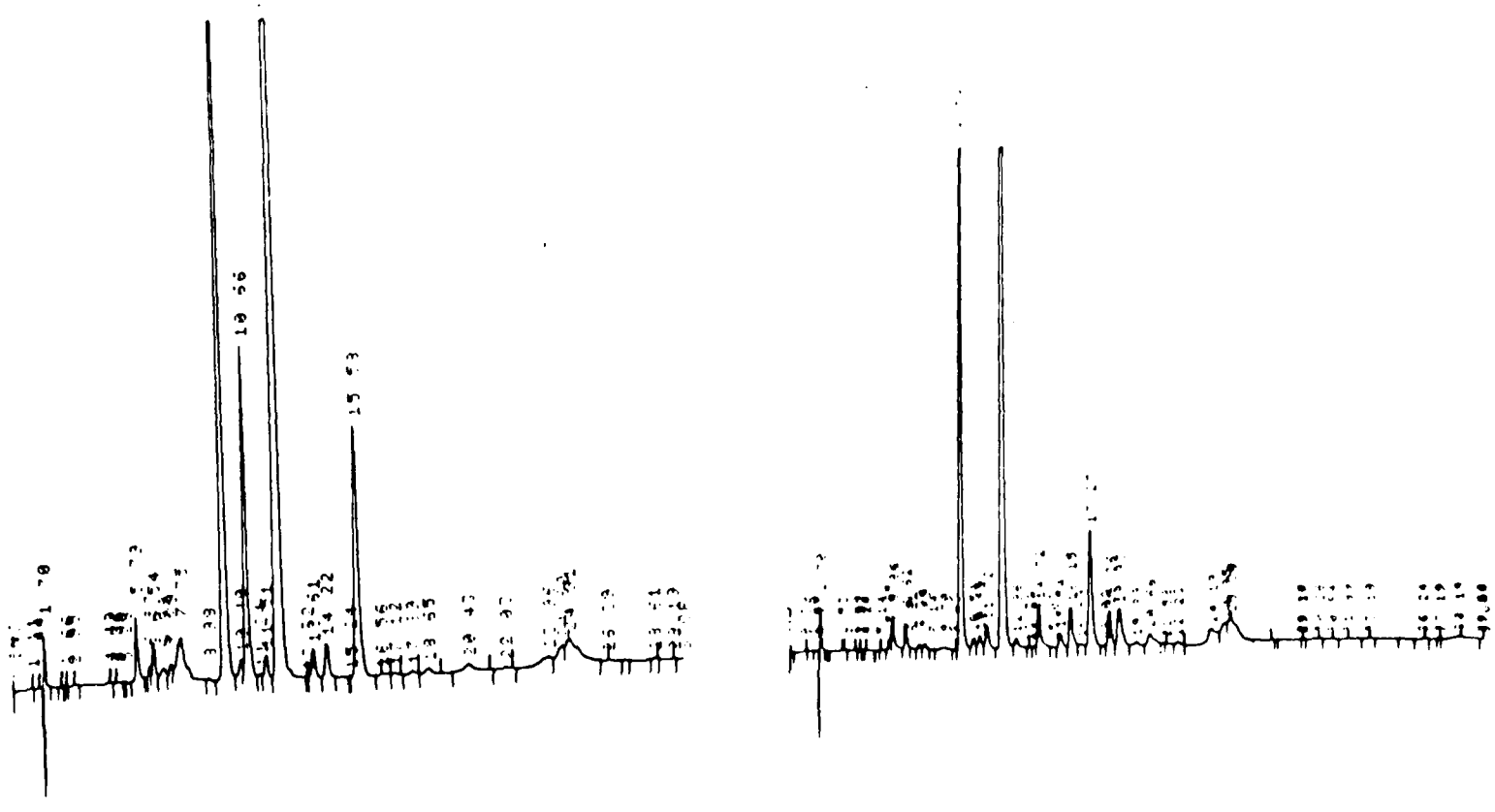
APPENDIX D

HIGH PERFORMANCE LIQUID CHROMATOGRAMS OF ORIGINAL 15 USED OILS AS RECEIVED



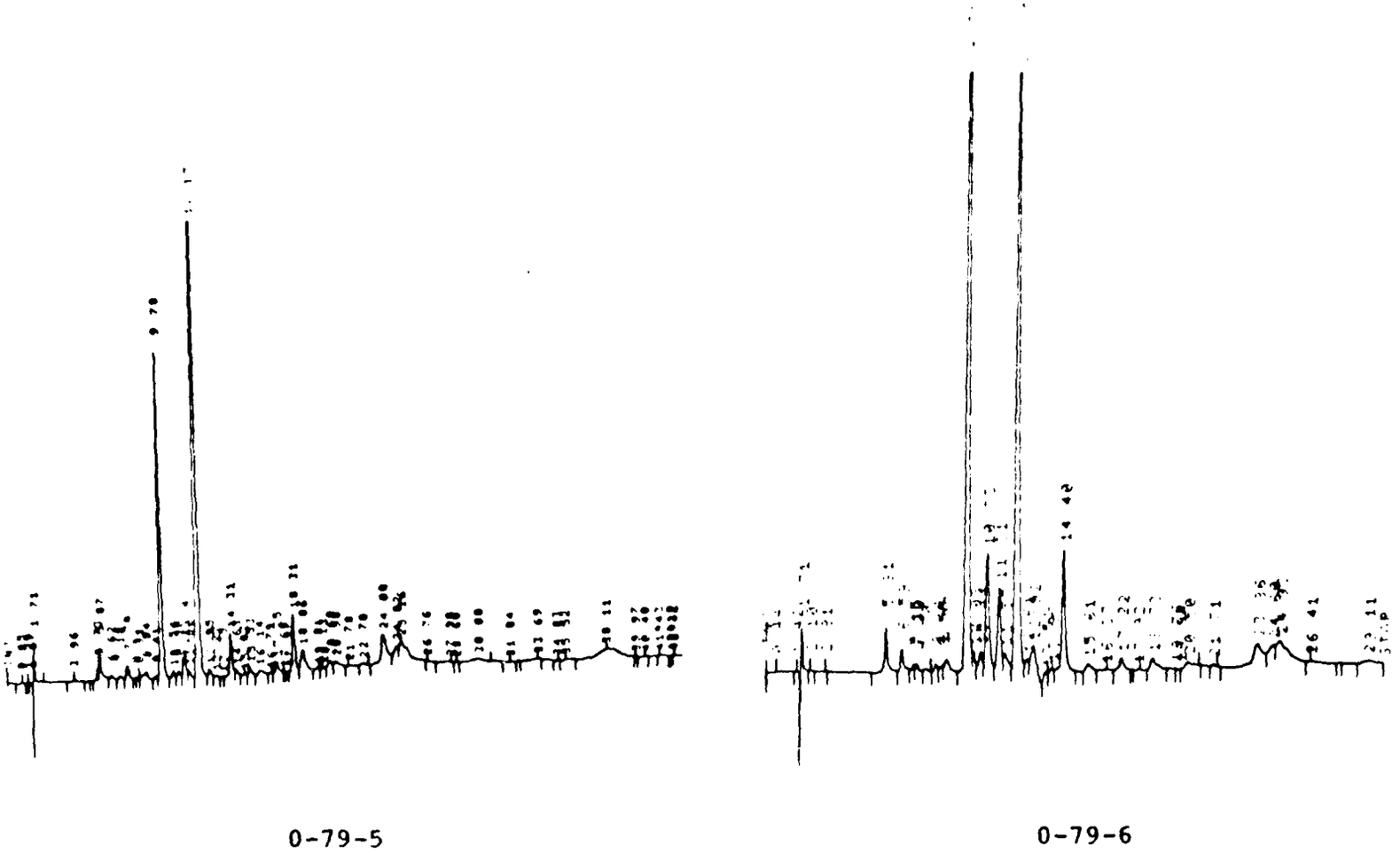
0-79-1 0-79-2

Figure D-1. High performance liquid chromatograms of used oil as received.



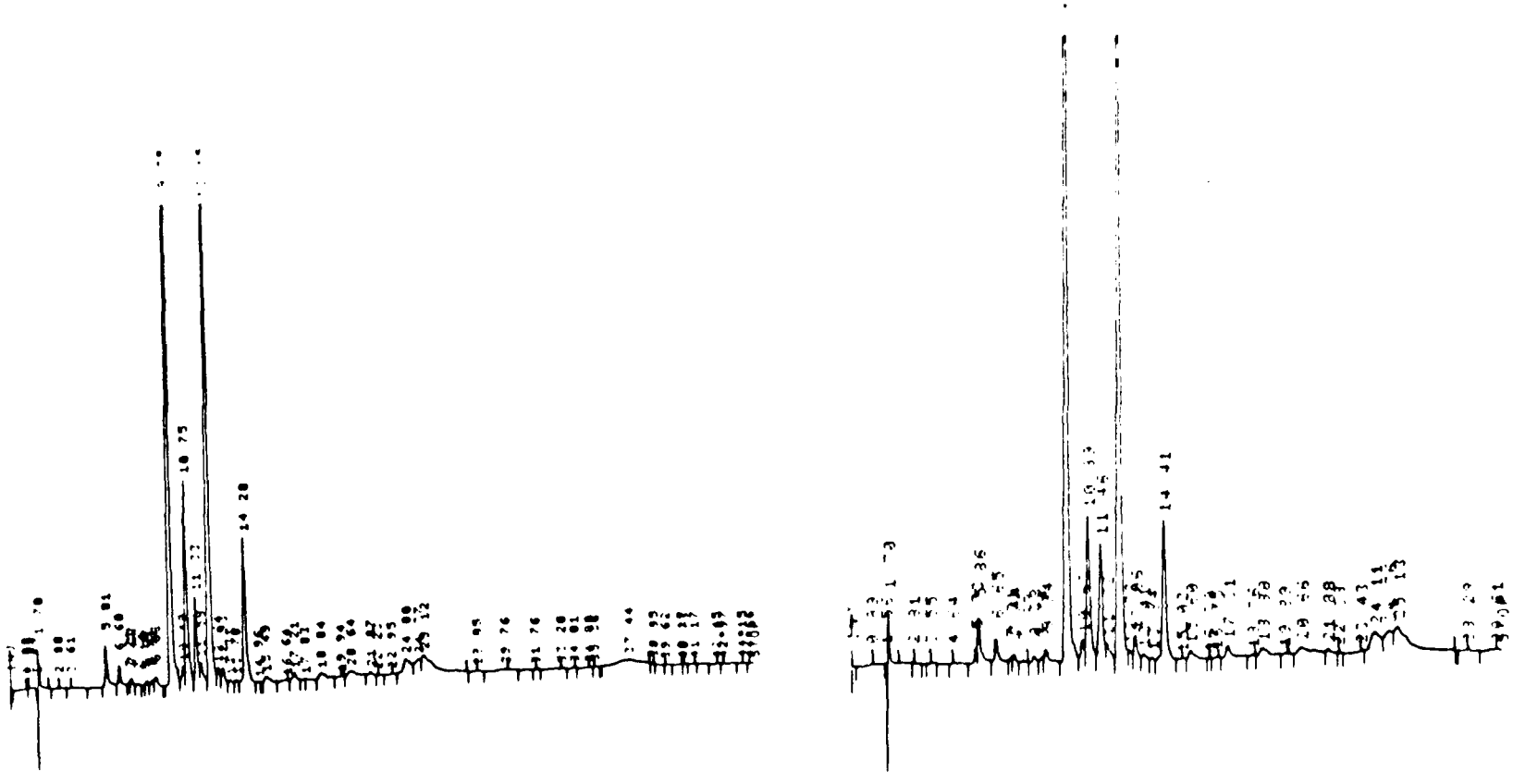
0-79-3 0-79-4

Figure D-2. High performance liquid chromatograms of used oil as received.



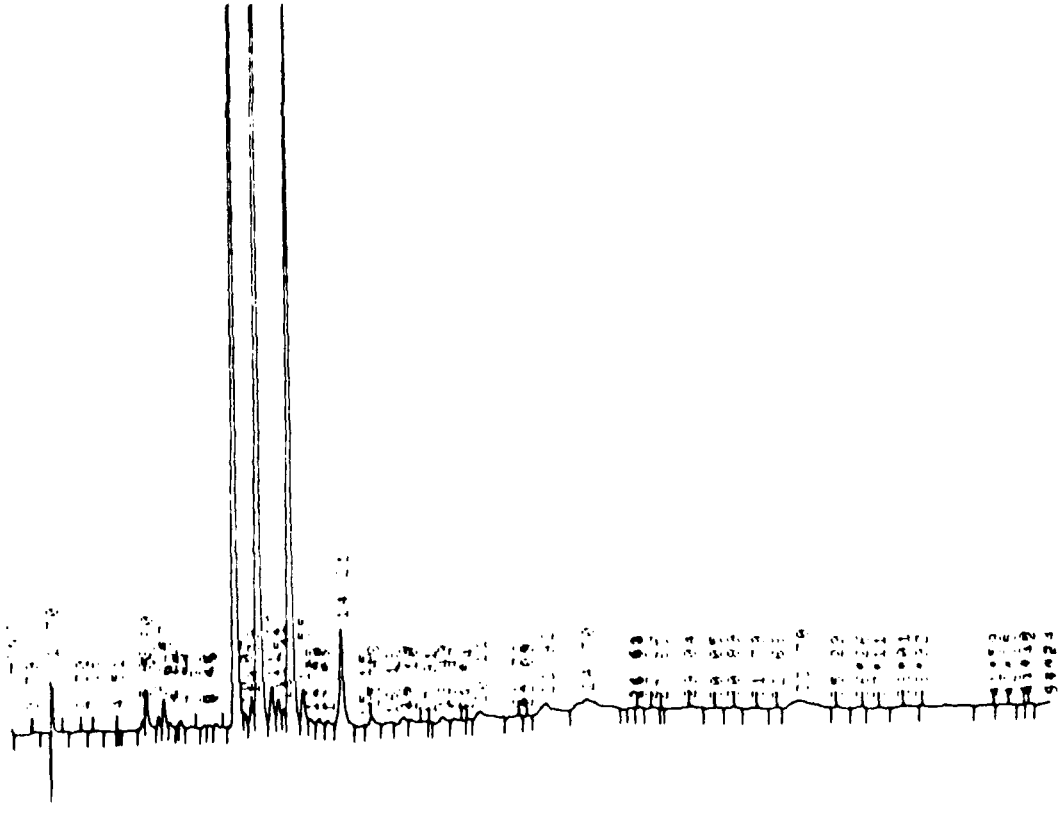
0-79-5 0-79-6

Figure D-3. High performance liquid chromatograms of used oil as received.



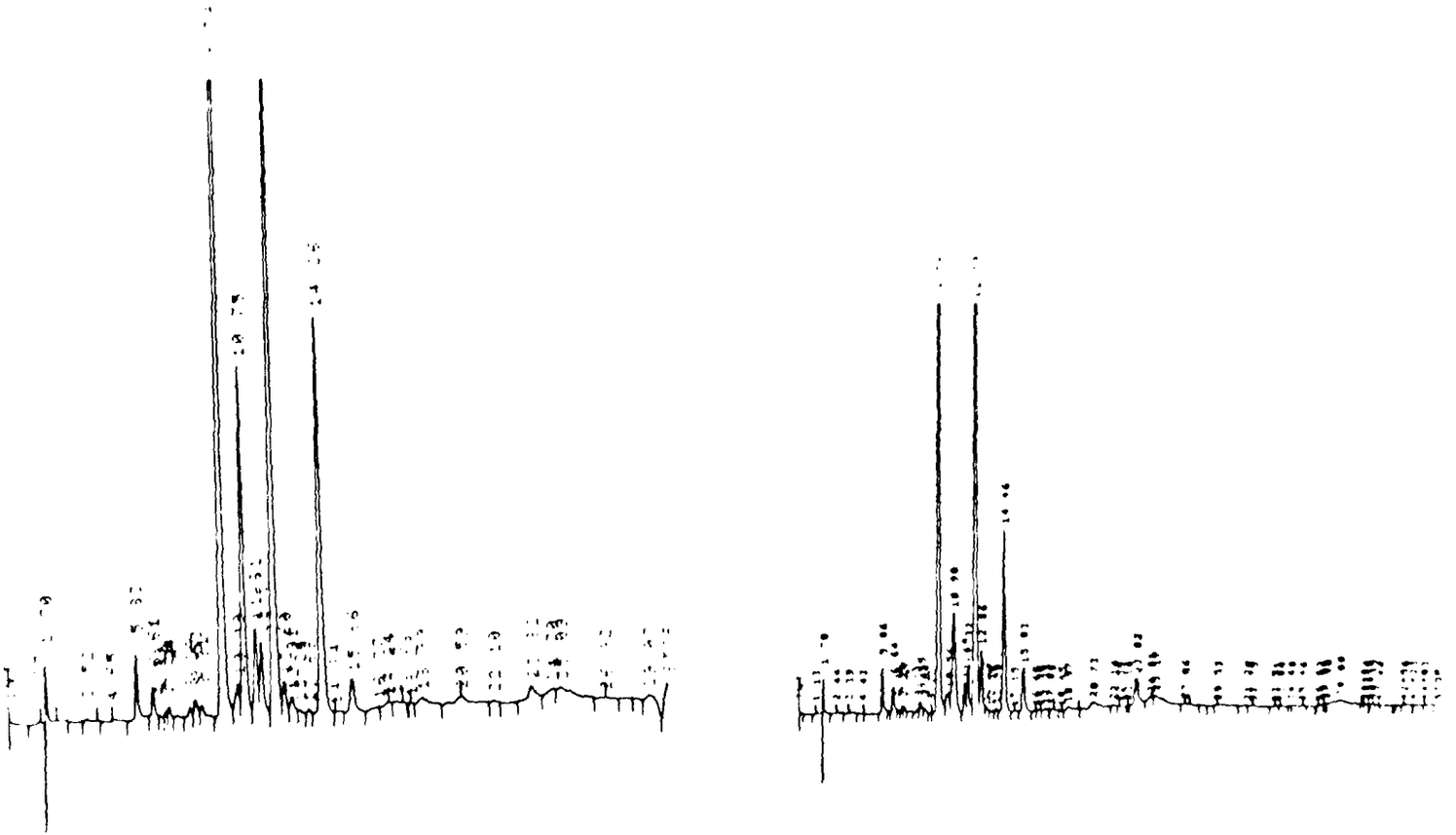
0-79-7 0-79-8

Figure D-4. High performance liquid chromatograms of used oil as received.



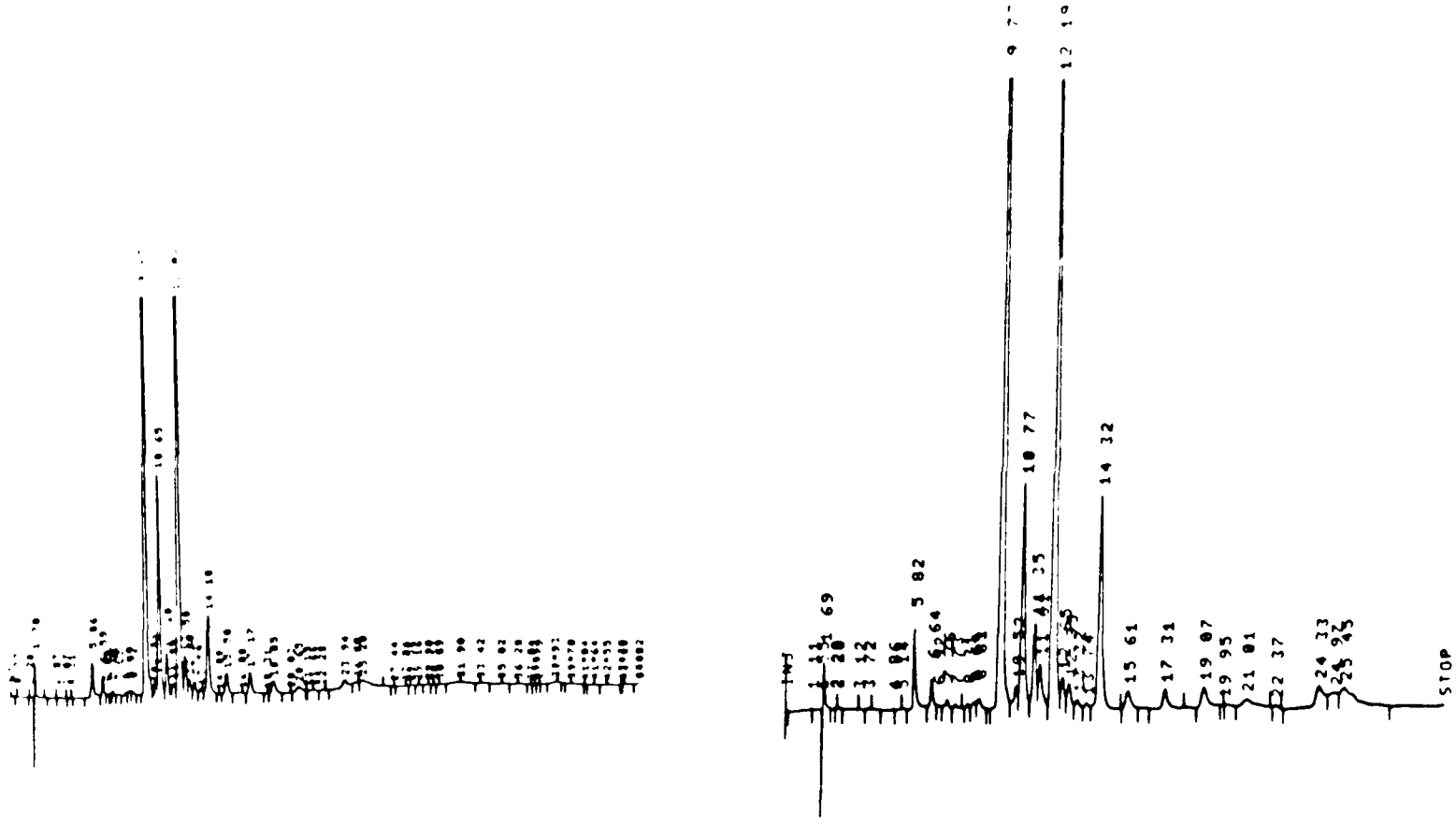
0-79-9

Figure D-5. High performance liquid chromatogram of used oil as received.



0-79-10 0-79-11

Figure D-6. High performance liquid chromatograms of used oil as received.



0-79-12 0-79-13

Figure D-7. High performance liquid chromatograms of used oil as received.

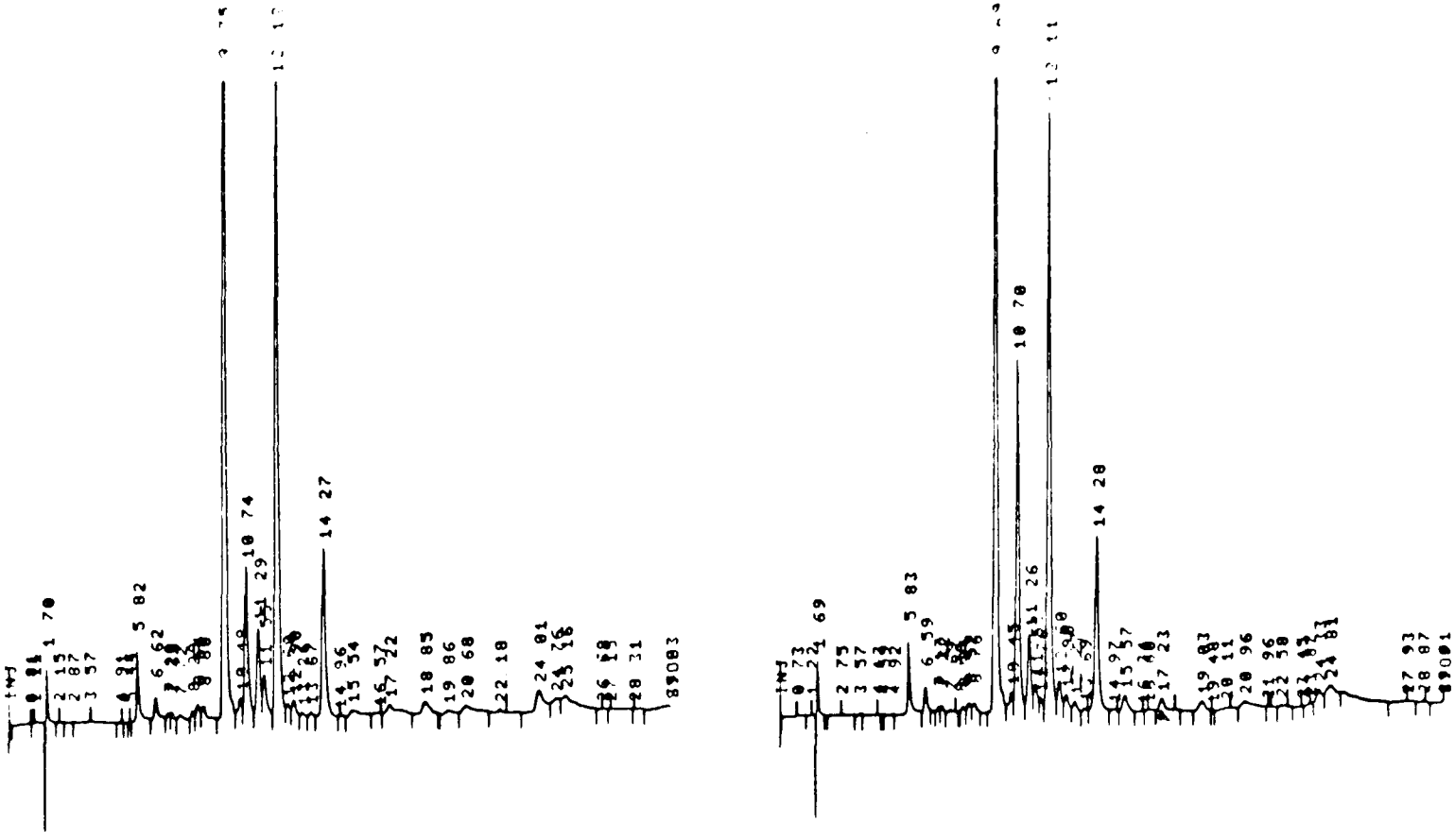


Figure D-8. High performance liquid chromatograms of used oil as received.

APPENDIX E

MIL-L-7808H TEST RESULTS FOR LARGE SCALE (25 GALLON) RECLAIMED OIL BATCHES

LEVEL OF ADDITION (% by wt), ADDED TO LARGE SCALE RECLAIMED OILS

	1997658 (0-79-11)		1997659 (0-79-9)		1997695 (0-79-6)		1997693 (0-79-13)		2000248 (0-79-8)	
	Added	Original level	Added	Original level	Added	Original level	Added	Original level	Added	Original level
Tricresyl phosphate	0.7	1.3	1.2	0.8	none	2.0	none	2.0	0.4	1.0
4,4'-dioctyldiphenylamine (DODPA)	none	1.4	0.4	1.01	0.25	1.24	0.15	1.35	0.49	1.24
Phenyl-alpha-naphthylamine (PANA)	0.2	0.31	0.2	0.19	0.25	0.25	0.25	0.25	none	0.27
Benzotriazole	0.1		0.1		0.1		0.1		0.1	
Triphenyl phosphite	0.1		0.1		0.1		0.1		0.1	
Quinizarin	0.05		0.05		0.05		0.1		0.05	
Antioxidant 703	0.1		0.1		0.1		0.1		0.1	

	2000247 (0-79-15)		2000267 (0-79-7)		2000268 (0-79-14)		2000273 (0-79-10)		2000275 (0-79-12)	
	Added	Original level	Added	Original level	Added	Original level	Added	Original level	Added	Original level
Tricresyl phosphate	0.8	0.68	1.2	0.8	none	1.0	none	1.24	none	1.19
4,4'-dioctyldiphenylamine (DODPA)	0.5	1.3	0.18	1.32	0.57	1.2	0.24	1.46	0.23	1.29
Phenyl-alpha-naphthylamine (PANA)	none	0.2	0.23	0.27	none	0.26	none	0.3	none	0.36
Benzotriazole	0.1		0.1		0.1		0.1		0.1	
Triphenyl phosphite	0.1		0.1		0.1		0.1		0.1	
Quinizarin	0.05		0.05		0.05		0.05		0.05	
Antioxidant 703	0.2		0.2		0.2		0.2		0.20	

GEAR LOAD CARRYING RATING

Method ASTM D1947
(one gear, two determinations)
(Ryder Gear)

NB number	Used oil	Test (PPI)	Specifications
1997693	(0-79-13)	2935	2400 PPI min.
1997695	(0-79-6)	2697	
1997659	(0-79-9)	2364	
1997658	(0-79-11)	2816	
2000248	(0-79-8)	2587	
2000247	(0-79-15)	2407	
2000275	(0-79-12)	2787	
2000273	(0-79-10)	3092	
2000267	(0-79-7)	2967	
2000268	(0-79-14)	2813	

BEARING DEPOSITION TEST SUMMARY

Bearing Deposition Test (see enclosed copies)	2000248 (0-79-8) & 2000247 (0-79-15) Test No. 48-312	2000267 (0-79-7) & 2000268 (0-79-14) Test No. 48-313	2000273 (0-79-10) & 2000275 (0-79-12) Test No. 48-314	1997693 (0-79-10) & 1997695 (0-79-6) Test No. 48-316	1997659 (0-79-9) & 1997658 (0-79-11) Test No. 48-317
Overall rating, max. 60	47.1	63.9	33.0	39.7	46.6
Viscosity, % change, max. 25%	+7.3	+15.3	+8.1	+9.7	+8.7
TAN change, max. 2.5	2.66	2.63	1.32	1.93	1.77
Oil consumption	4.6	3.5	7.1	13.8	2.1
Total sludge, max. 2 grams	0.598	0.666	0.418	2.132	1.337

BEARING DEPOSITION TEST SUMMARY

Test No. 48-316

MONSANTO RESEARCH CORPORATION

Sample No. 1997693 (0-79-13) & 1997695 (0-79-6)

(Rig No. 1)

DEPOSITS

End Cover	0.0
Spacer & Nut	14.0
Heater Front	99.6
Heater Rear	86.4
Seal Plate	10.0
Bearing	28.0
OVERALL RATING	39.7

SLUDGE

Inlet Screen	.762
Outlet Screen	1.370
TOTAL	2.132

OIL CONSUMPTION, ml/hr 13.8

SUMP*

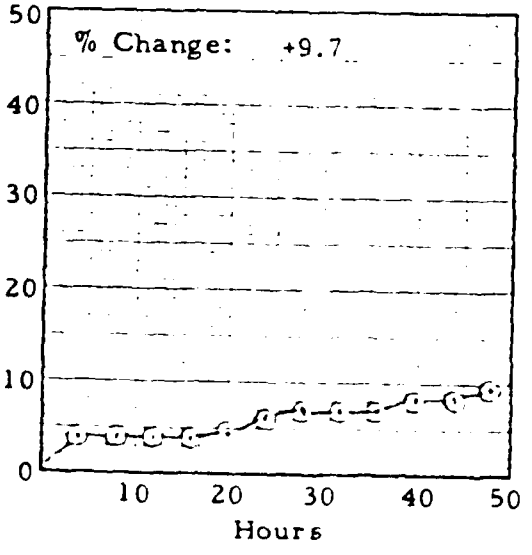
Sides	Lt. Varnish
Bottom	Lt. Varnish

* Not included in the overall rating above.

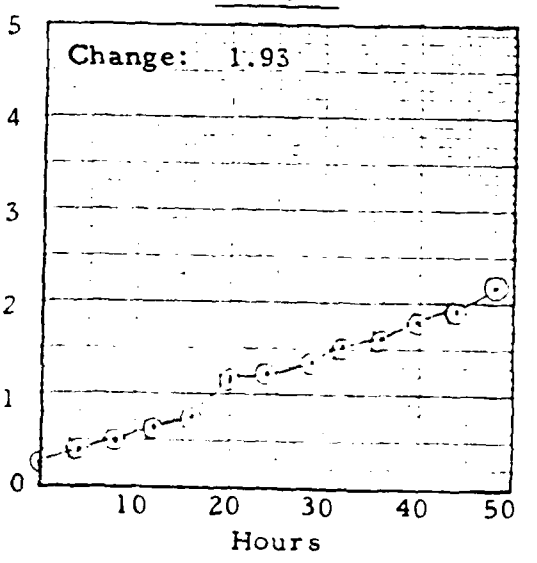
METALS

Aluminum	-0.020
Silver	0.000
Bronze	-0.020
Steel	+0.040
M-50 Steel	-0.040
Waspaloy	+0.059
Titanium	+0.020

VISCOSITY



T. A. N.



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BEARING DEPOSITION TEST SUMMARY

Test No. 48-317

MONSANTO RESEARCH CORPORATION

Sample No. 1997659 (0-79-9) & 1197658 (0-79-11)

(Rig No. 1)

DEPOSITS

End Cover	1.2
Spacer & Nut	14.0
Heater Front	102.3
Heater Rear	119.1
Seal Plate	0.0
Bearing	43.0
OVERALL RATING	46.6

SLUDGE

Inlet Screen	.392
Outlet Screen	.945
TOTAL	1.337

OIL CONSUMPTION, ml/hr 2.1

SUMP*

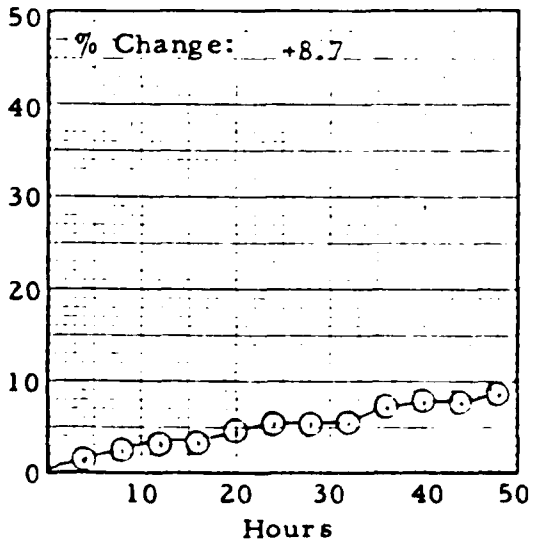
Sides	Lt. Varnish
Bottom	Lt. Varnish

* Not included in the overall rating above.

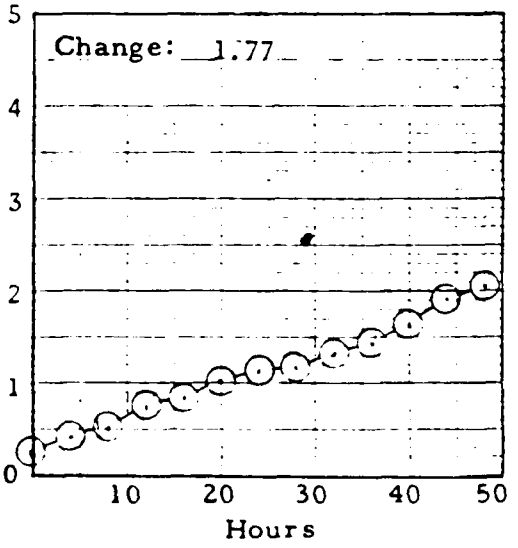
METALS

Aluminum	+0.020
Silver	+0.040
Bronze	0.000
Steel	-0.040
M-50 Steel	0.000
Waspaloy	+0.020
Titanium	0.000

VISCOSITY



T. A. N.



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BEARING DEPOSITION TEST SUMMARY

MONSANTO RESEARCH CORPORATION
Test No. 48-313
Sample No. 2000267 (0-79-7) & 2000268 (o-79-14)
(Rig No. 2)

DEPOSITS	
End Cover	1.2
Spacer & Nut	29.0
Heater Front	151.2
Heater Rear	102.9
Seal Plate	11.0
Bearing	88.0
OVERALL RATING	63.9

SLUDGE	
Inlet Screen	.356
Outlet Screen	.310
TOTAL	.666

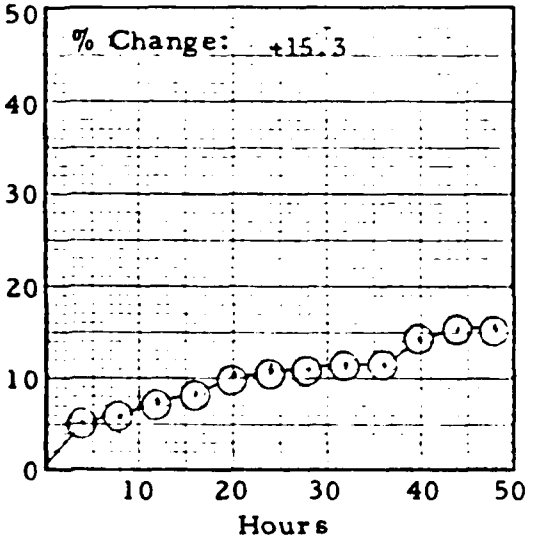
OIL CONSUMPTION, ml/hr 3.5

SUMP*	
Sides	Lt. Varnish
Bottom	Lt. Varnish

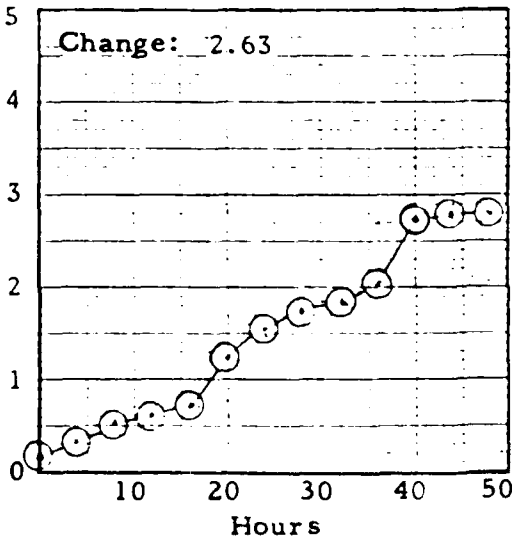
* Not included in the overall rating above.

METALS	
Aluminum	0.000
Silver	-0.040
Bronze	+0.020
Steel	0.000
M-50 Steel	-0.020
Waspaloy	0.000
Titanium	+0.020

VISCOSITY



T. A. N.



(This sheet may be detached for filing)

BEARING DEPOSITION TEST SUMMARY

Test No. 48-312

MONSANTO RESEARCH CORPORATION

Sample No. 2000248 (0-79-8) & 2000247 (0-79-15)

(Rig No. 1)

DEPOSITS	
End Cover	1.5
Spacer & Nut	14.0
Heater Front	77.1
Heater Rear	142.2
Seal Plate	10.0
Bearing	37.5
OVERALL RATING	47.1

SLUDGE	
Inlet Screen	.262
Outlet Screen	.336
TOTAL	.598

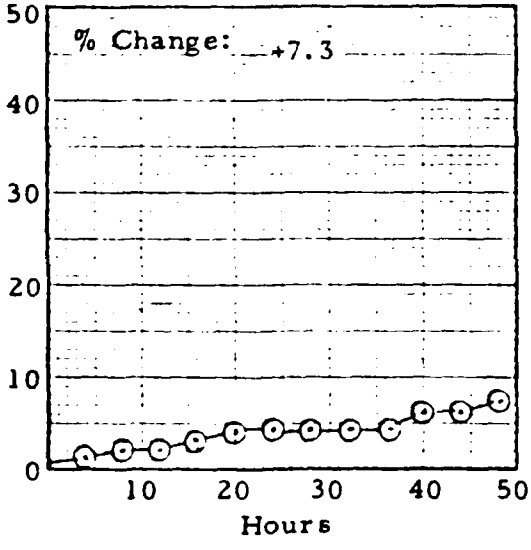
OIL CONSUMPTION, ml/hr 4.6

SUMP*	
Sides	Lt. Sludge
Bottom	Lt. Varnish

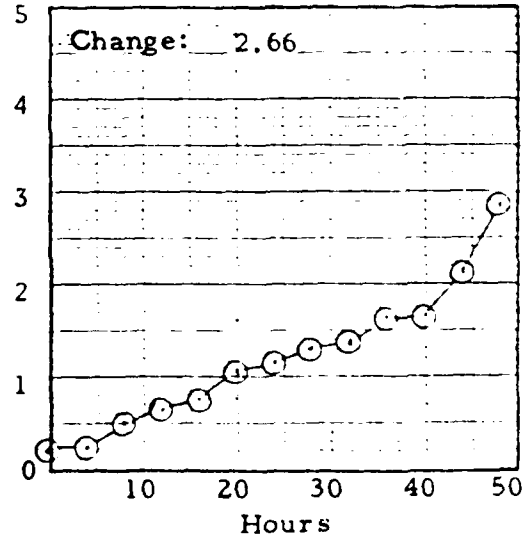
* Not included in the overall rating above.

METALS	
Aluminum	0.000
Silver	+0.040
Bronze	+0.040
Steel	0.000
M-50 Steel	0.000
Waspaloy	+0.020
Titanium	0.000

VISCOSITY



T. A. N.



(This sheet may be detached for filing)

BEARING DEPOSITION TEST SUMMARY
MONSANTO RESEARCH CORPORATION
Test No. 48-314
Sample No. 2000273 (0-79-10) & 2000275 (0-79-12)
(Rig No. 1)

DEPOSITS	
End Cover	0.0
Spacer & Nut	14.0
Heater Front	82.2
Heater Rear	64.2
Seal Plate	10.0
Bearing	27.5
OVERALL RATING	33.0

SLUDGE	
Inlet Screen	.183
Outlet Screen	.235
TOTAL	.418

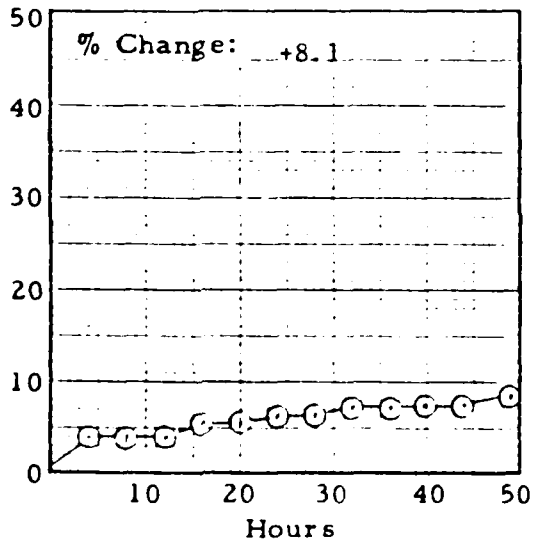
OIL CONSUMPTION, ml/hr 7.1

SUMP*	
Sides	Lt. Varnish
Bottom	Lt. Varnish

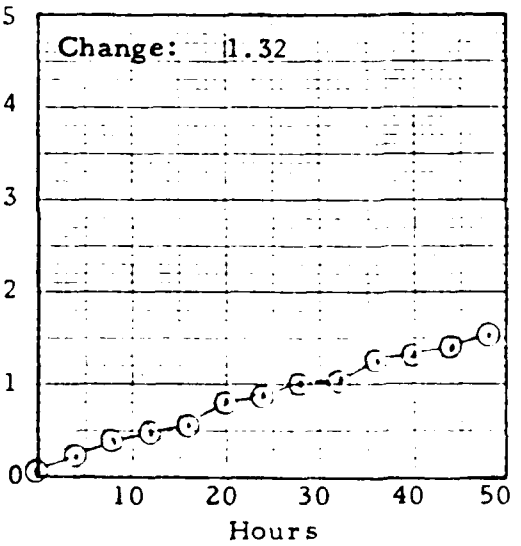
* Not included in the overall rating above.

METALS	
Aluminum	-0.020
Silver	-0.020
Bronze	+0.079
Steel	-0.020
M-50 Steel	+0.079
Waspaloy	-0.040
Titanium	-0.040

VISCOSITY



T.A.N.



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ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE August 19, 1981

SAMPLE 1997659, Batch 0-79-9

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.)	0.30 Max	0.17
Viscosity @ 210°F, cs	3.0 Min	3.1
Viscosity @ 100°F, cs	Report	12.8
Flash Point (COC), °F	400.0 Min	435
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %	30.0 Max	18.2
TRACE SEDIMENT		
Precipitation, ml/200 ml	0.005 Max	0.000
PARTICULATE CONTAMINATION		
Contamination, mg/ltr	10 Max	0.01
FOAMING CHARACTERISTICS, STATIC		
176°F, Volume after 30 min. aeration, ml	100 Max	35 ml
Collapse time, sec.	60 Max	8.1 sec
FOAMING CHARACTERISTICS, DYNAMIC		
176°F, Volume at 1000 cc air, cc	100 Max	10 ml
Collapse time, min.	60 Max	61.7 sec
Volume @ 1500 cc air, cc	150 Max	10 ml
Collapse time, min.	60 Max	61.3 ml
Volume @ 2000 cc air, cc	Report	20 ml
Collapse time, min.	60 Max	58.5 sec
230°F, Volume @ 1000 cc air, cc	100 Max	10 ml
Collapse time, min.	60 Max	50.9 sec
Volume @ 1500 cc air, cc	150 Max	15 ml
Collapse time, min.	60 Max	55.6 sec
Volume @ 2000 cc air, cc	Report	30 ml
Collapse time, min.	60 Max	51.6 sec
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in²	6 Max	0.0
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in²	3.0 Max	- 0.1
Bronze, AMS 4640, mg/in²	3.0 Max	- 0.5

MIL-L-7808H QUALITY CONFORMANCE

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
% Swell	12.0-35.0	+28.5
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
% Swell	2.0-25.0	+16.4
Tensile Strength, % Change	50 Max	- 1.6
Elongation, % Change	50 Max	+24.6
Hardness, No. Change	20 Max	-10
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2-30	+ 7.4
Tensile Strength, % Change	50 Max	-59.2
Elongation, % Change	50 Max	-34.3
Hardness, No. Change	20 Max	- 5
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2-25 Max	+ 5.2
Tensile Strength, % Change	50 Max	-58.9
Elongation, % Change	50 Max	-30.4
Hardness, No. Change	20 Max	- 5
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max	0.16
Viscosity Change, %	Report	73.4
TAN Change	Report	15.65
Oil Consumption	Report	125
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs	25 Max	~~-1.4~~ (+ 0.7)*
168 Hrs	150 Max	~~-257.0~~ (-125.8)*
VISCOSITY STABILITY, 3 Hrs @ -65°F		
Original Oil, cst	17,000 Max	12,805
3 Hrs, cst	17,000 Max	12,691
Viscosity Change, %	6.0 Max	0.9
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original Oil, cst	17,000	12,805
72 Hrs, cst	17,000	11,039
Viscosity Change, %	6.0 Max	13.8
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE August 19, 1981

SAMPLE 1997659, Batch 0-79-9

	Specification	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F		
Corrosion:		
Steel, mg/cm^2	Report	0.000
Silver, mg/cm^2	Report	-0.010
Aluminum, mg/cm^2	Report	0.000
Magnesium, mg/cm^2	Report	-15.940
Bronze, AMS4616, mg/cm^2	Report	-0.059
Titanium, mg/cm^2	Report	0.000
M-50 Steel, mg/cm^2	Report	-0.010
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	Magnesium
Staining	Report	All Metals

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial	Report								12.8
Viscosity @ 100°F, % Change	Report	+7.0	+7.8	+13.3	14.1	+18.0	20.3	23.4	24.2
Viscosity @ 210°F, Initial	Report								3.1
Viscosity @ 210°F, % Change	Report								19.4
Total Acid Number, Initial	Report								0.17
Total Acid Number, Change	Report	0.51	1.17	2.35	2.75	3.71	3.75	3.85	3.83
Evaporation Loss, %	Report								2.1
Sludge, Volume, %	Report								0.0

MIL-L-7808 DEPOSITION TEST

ALCOR INC.

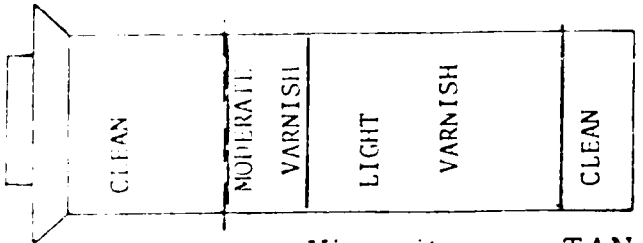
TEST LUBRICANT DESIGNATION Monsanto, Sample 1997659, Batch 0-79-9

TEST NO 4464 DATE 8/3/81 OPERATOR Pavlicek, Trawick

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES, °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP	CAB AIR 4				
START	1915	300	455	590	540	116	185	2.5	0	1.0
	45	300	450	590	550	116	185	2.5	0	1.0
1	2000	300	450	590	550	116	185	2.5	0	1.0
	15	300	450	590	555	116	185	2.5	0	1.0
2	2115	300	445	590	555	116	185	2.5	0	1.0
	45	300	450	590	555	116	185	2.5	0	1.0
3	2200	300	450	590	550	116	185	2.5	0	1.0
	45	300	445	590	555	116	185	2.5	0	1.0
4	2300	300	445	590	550	116	185	2.5	0	1.0
	45	300	445	590	545	116	185	2.5	0	1.0
5	2400	300	445	590	545	116	185	2.5	0	1.0
	15	300	445	590	540	116	185	2.5	0	1.0
6	0015	300	445	590	545	116	185	2.5	0	1.0
	45	300	455	590	550	116	190	2.5	0	1.0
7	0100	300	455	590	550	116	190	2.5	0	1.0
	15	300	455	590	550	116	190	2.5	0	1.0
8	0215	300	455	590	550	116	190	2.5	0	1.0
	45	300	450	590	555	116	190	2.5	0	1.0
9	0300	300	455	590	550	116	190	2.5	0	1.0
	45	300	450	590	555	116	190	2.5	0	1.0
10	0415	300	455	590	550	116	190	2.5	0	1.0
	45	300	455	590	550	116	190	2.5	0	1.0
11	0515	300	455	590	555	116	190	2.5	0	1.0
	45	300	455	590	550	116	190	2.5	0	1.0
12	0615	300	455	590	550	116	190	2.5	0	1.0

OIL LEVEL



Test Section A Tube No. 2

Oil Cons., cc 125 Cooler Sludge Clean

Coking Tube, gm .015

Filter Sludge, gm .013

DEPOSIT RATING .16

	Viscosity	TAN
Before	12.8	.17
After	22.2	15.82
Change	9.4	15.65

150

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE August 19, 1981

SAMPLE: 1997658, Batch 0-79-11

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.)	0.30 Max	0.17
Viscosity @ 210°F, cs	3.0 Min	3.2
Viscosity @ 100°F, cs	Report	12.5
Flash Point (COC), °F	400.0 Min	435
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %	30.0 Max	17.8
TRACE SEDIMENT		
Precipitation, ml/200 ml	0.005 Max	0.000
PARTICULATE CONTAMINATION		
Contamination, mg/ltr	10 Max	0.02
FOAMING CHARACTERISTICS, STATIC		
176°F, Volume after 30 min. aeration, ml	100 Max	30 ml
Collapse time, sec.	60 Max	4.5 sec.
FOAMING CHARACTERISTICS, DYNAMIC		
176°F, Volume @ 1000 cc air, cc	100 Max	10 ml
Collapse time, min.	60 Max	30.5 sec.
Volume @ 1500 cc air, cc	150 Max	10 ml
Collapse time, min.	60 Max	51.0 sec.
Volume @ 2000 cc air, cc	Report	10 ml
Collapse time, min.	60 Max	31.6 sec.
230°F, Volume @ 1000 cc air, cc	100 Max	10 ml
Collapse time, min.	60 Max	28.9 sec.
Volume @ 1500 cc air, cc	150 Max	10 ml
Collapse time, min.	60 Max	50.4 sec.
Volume @ 2000 cc air, cc	Report	10 ml
Collapse time, min.	60 Max	19.7 sec.
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2	6 Max	0.0
SILVER & BRONZE CORROSION, 50 Hrs @ 450 F		
Silver Wt. Change, mg/in^2	3.0 Max	-0.1
Bronze, AMS4616, mg/in^2	3.0 Max	0.0

MIL-L-7808H QUALITY CONFORMANCE

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
% Swell	12.0-35.0	+26.8
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
% Swell	2.0-25.0	+16.3
Tensile Strength, % Change	50 Max	- 4.8
Elongation, % Change	50 Max	+58.5
Hardness, No. Change	20 Max	-10
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2-30 Max	+ 8.9
Tensile Strength, % Change	50 Max	-42.5
Elongation, % Change	50 Max	-25.7
Hardness, No., Change	20 Max	- 5
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2-25 Max	+ 5.8
Tensile Strength, % Change	50 Max	-46.1
Elongation, % Change	50 Max	-26.1
Hardness, No., Change	20 Max	- 5
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max	0.48
Viscosity Change, %	Report	95.2
TAN Change	Report	21.77
Oil Consumption	Report	150
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs	25 Max	- 2.8 (1.4)*
168 Hrs	150 Max	-275.3 (132.7)*
VISCOSITY STABILITY @ 3 Hrs @ -65°F		
Original Oil, cst	17,000 Max	11,417
Viscosity Change, %	6.0 Max	0.3
3 Hrs, cst	17,000 Max	11,381
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original Oil, cst	17,000 Max	11,417
Viscosity Change, %	6.0 Max	11.5
72 Hrs, cst	17,000 Max	10,100
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE August 19, 1981

SAMPLE: 1997658, Batch 0-79-11

	Specification	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F		
Corrosion:		
Steel, mg/cm²	Report	-0.010
Silver, mg/cm²	Report	-0.040
Aluminum, mg/cm²	Report	0.000
Magnesium, mg/cm²	Report	-8,822
Bronze, AMS4616, mg/cm²	Report	0.000
Titanium, mg/cm²	Report	+0.010
M-50 Steel, mg/cm²	Report	0.000
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	Magnesium
Staining	Report	All Metals

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial	Report								12.5
Viscosity @ 100°F, % Change	Report	+6.4	+7.2	+11.2	+12.0	+24.0	+39.2	+71.2	+84.8
Viscosity @ 210°F, Initial	Report								3.2
Viscosity @ 210°F, % Change	Report								+46.9
Total Acid Number, Initial	Report								0.17
Total Acid Number, Change	Report	0.83	1.17	2.47	2.81	9.65	17.11	23.71	25.67
Evaporation Loss, %	Report								3.0
Sludge, Volume, %									0.0

MIL-L-7808 DEPOSITION TEST

Falcon Inc.
10130 JONES MALTSBERGER ROAD P.O. BOX 32516
SAN ANTONIO, TEXAS 78284 512-349-3771

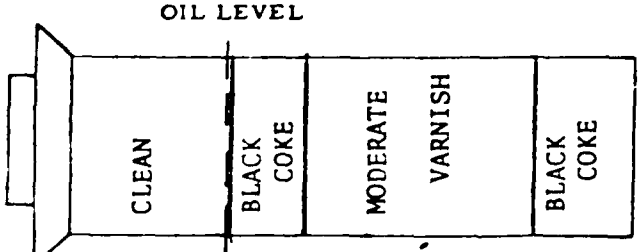
TEST LUBRICANT DESIGNATION Monsanto, Sample 1997658, Batch 0-79-11

TEST NO. 4465 DATE 8/4/81 OPERATOR Pavlicek, Revet, Trawick

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES, °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP	CAB AIR 4				
START	1345	300	435	590	545	116	190	2.5	0	.90
	1415	300	450	590	550	116	190	2.5	0	.90
1	45	300	450	590	550	116	190	2.5	0	.90
	1515	300	440	590	550	116	190	2.5	0	.90
2	45	300	440	590	555	116	190	2.5	0	.90
	1615	300	445	590	560	116	190	2.5	0	.90
3	45	300	445	590	560	116	190	2.5	0	.90
	1715	300	445	590	560	116	190	2.5	0	.90
4	45	300	450	590	565	116	190	2.5	0	.90
	1815	300	450	590	560	116	190	2.5	0	.90
5	45	300	445	590	555	116	190	2.5	0	.90
	1915	300	445	590	550	116	190	2.5	0	.90
6	45	300	440	590	545	116	190	2.5	0	.90
	2015	300	445	590	555	116	190	2.5	0	.90
7	45	300	450	590	560	116	190	2.5	0	.90
	2115	300	450	590	560	116	190	2.5	0	.90
8	45	300	460	590	550	116	190	2.5	0	.90
	2215	300	455	590	560	116	190	2.5	0	.90
9	45	300	450	590	560	116	195	2.5	0	.90
	2315	300	455	590	560	116	195	2.5	0	1.00
10	45	300	450	590	565	116	195	2.5	0	1.00
	2415	300	455	590	560	116	195	2.5	0	1.00
11	45	300	450	590	565	116	195	2.5	0	1.00
	0115	300	450	590	560	116	195	2.5	0	1.0
12	45	300	450	590	560	116	195	2.5	0	1.0

OIL LEVEL



Test Section D Tube No. 3

Oil Cons., cc 150 Cooler Sludge Light

Coking Tube, gm .046

Filter Sludge, gm .022

DEPOSIT RATING .48

	Viscosity	TAN
Before	12.5	.17
After	24.4	21.94
Change	11.9	21.77
% Change	+95.2	

154

AFCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE November 4, 1981

SAMPLE 1997693, Batch 0-79-13

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.)	0.30 Max	0.25
Viscosity @ 210°F, cs	3.0 Min	3.3
Viscosity @ 100°F, cs	Report	12.5
Flash Point (COC), °F	400.0 Min	440
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %	30.0 Max	5.7
TRACE SEDIMENT		
Precipitation, ml/200 ml	0.005 Max	0.0
PARTICULATE CONTAMINATION		
Contamination, mg/ltr	10.0 Max	0.0
FOAMING CHARACTERISTICS, STATIC		
176°F, Volume after 30 min. aeration, ml	100.0 Max	10 ml
Collapse time, sec.	60.0 Max	1
FOAMING CHARACTERISTICS, DYNAMIC		
176°F, Volume @ 1000 cc air, cc	100.0 Max	10 ml
Collapse time, min.	60.0 Max	40.3 sec.
Volume @ 1500 cc air, cc	150.0 Max	10 ml
Collapse time, min.	60.0 Max	49.5 sec.
Volume @ 2000 cc air, cc	Report	10 ml
Collapse time, min.	60.0 Max	41.9 sec.
230°F, Volume @ 1000 cc air, cc	100.0 Max	10 ml
Collapse time, min	60.0 Max	48.6 sec.
Volume @ 1500 cc air, cc	150.0 Max	10 ml
Collapse time, min.	60.0 Max	48.2 sec.
Volume @ 2000 cc air, cc	Report	10 ml
Collapse time, min.	60.0 Max	52.1 sec.
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2	6.0 Max	0.4
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2	3.0 Max	0.0
Bronze, AMS4616, mg/in^2	3.0 Max	-0.4

MIL-L-7808H QUALITY CONFORMANCE

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
% Swell	12.0-35.0	+27.6
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
% Swell	2.0-25.0	+13.7
Tensile Strength, % Change	50.0 Max	- 0.8
Elongation, % Change	50.0 Max	+13.8
Hardness, No., Change	20.0 Max	- 5
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-30.0	+ 5.2
Tensile Strength, % Change	50.0 Max	-56.9
Elongation, % Change	50.0 Max	-34.3
Hardness, No., Change	20.0 Max	- 5
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-25.0	+ 5.3
Tensile Strength, % Change	50.0 Max	-51.3
Elongation, % Change	50.0 Max	-40.6
Hardness, No., Change	20.0 Max	- 5
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max	.80
Viscosity Change, %	Report	107.2
TAN Change	Report	23.09
Oil Consumption	Report	125
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs	25.0 Max	~~26.2~~ (12.7)*
168 Hrs	150.0 Max	~~395.7~~ (190.7)*
VISCOSITY STABILITY @ 3 Hrs @ -65°F		
Original Oil, cst	17,000 Max	10,946
Viscosity Change, %	6.0 Max	2.0
3 Hrs, cst	17,000 Max	10,718
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original Oil, cst	17,000 Max	10,946
Viscosity Change, %	6.0 Max	17.2
72 Hrs, cst	17,000 Max	9,066
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3711

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE November 4, 1981

SAMPLE 1997693, Batch 0-79-13

	Specification	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F		
Corrosion:		
Steel, mg/cm²	Report	+0.010
Silver, mg/cm²	Report	0.000
Aluminum, mg/cm²	Report	-0.010
Magnesium, mg/cm²	Report	-10.554
Bronze, AMS-4616, mg/cm²	Report	0.000
Titanium, mg/cm²	Report	0.000
M-50 Steel, mg/cm²	Report	-0.010
Appearance of Metal Specimens:		
Pitting	Report	Mg.
Etching	Report	None
Corrosion	Report	None
Staining	Report	M-50, Bz, Ag, Steel

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial	Report								12.5
Viscosity @ 100°F, % Change	Report	+6.4	+8.0	+12.8	+15.2	+18.4	+20.0	+40.0	+43.2
Viscosity @ 210°F, Initial	Report								3.3
Viscosity @ 210°F, % Change	Report								21.2
Total Acid Number, Initial	Report								0.25
Total Acid Number, Change	Report	0.47	1.15	2.95	3.51	5.03	7.51	14.67	14.79
Evaporation Loss, %	Report								3.2
Sludge, Volume,	Report								0.0

MIL-L-7808 DEPOSITION TEST

Alcor Inc.

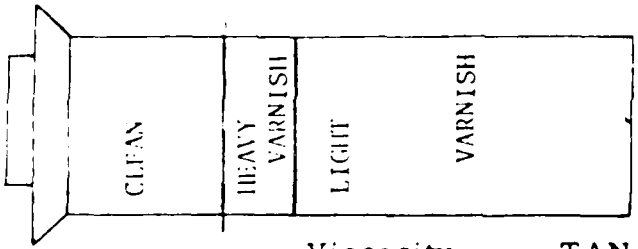
TEST LUBRICANT DESIGNATION: Monsanto Research, Sample 1997693, Batch 0-79-13

TEST NO 4472 DATE 9/22/81 OPERATOR Trawick, Revet, Pavlicek

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	HEATER TEMPERATURES °F: COKING TUBE 1	3	2	VAP SP	CAP AIR 4	HEATER VOLTS	AIR ROTOMETER	OIL LEVEL	FILTER PRESSURE
START	0930	[illegible]	[illegible]	[illegible]	560	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	1100	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
1	30	[illegible]	440	[illegible]	565	116	[illegible]	2.5	C	1.0
	1100	[illegible]	[illegible]	590	560	116	190	2.5	C	1.0
2	30	[illegible]	455	590	570	116	190	2.5	C	1.0
	1200	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	190	2.5	C	1.0
3	30	[illegible]	455	[illegible]	[illegible]	[illegible]	190	2.5	C	1.0
	[illegible]	[illegible]	[illegible]	590	570	116	190	2.5	C	1.0
4	[illegible]	[illegible]	450	[illegible]	570	116	190	2.5	C	1.0
		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	190	2.5	C	1.0
5		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2.5	C	1.0
		[illegible]	450	590	[illegible]	116	190	2.5	C	1.0
6		[illegible]	[illegible]	[illegible]	[illegible]	116	190	2.5	C	1.0
		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
7		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2.5	C	1.0
	710	[illegible]	[illegible]	[illegible]	[illegible]	116	190	2.5	C	1.0
8	[illegible]	[illegible]	450	590	570	116	190	2.5	C	1.0
	[illegible]	[illegible]	[illegible]	[illegible]	570	116	190	2.5	C	1.0
9	[illegible]	[illegible]	450	590	570	116	190	2.5	C	1.0
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2.5	C	1.0
10	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	116	190	2.5	C	1.0
	[illegible]	[illegible]	450	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
11	[illegible]	[illegible]	450	[illegible]	[illegible]	116	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
12	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2.5	C	1.0

OIL LEVEL



Test Section A Tube No. 1

Oil Cons., cc 125 Cooler Sludge Light

Coking Tube, gm .076

Filter Sludge, gm .035

DEPOSIT RATING .80

	Viscosity	TAN 158
Before	12.5	.25
After	25.9	23.34
Change	13.4	23.09

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE November 4, 1981

SAMPLE 1997695, Batch G-79-6

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.)	0.30 Max	0.22
Viscosity @ 210°F, cs	3.0 Min	3.3
Viscosity @ 100°F, cs	Report	12.4
Flash Point (COC), °F	400.0 Min	445
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %	30.0 Max	5.8
TRACE SEDIMENT		
Precipitation, ml/200 ml	0.005 Max	0.00
PARTICULATE CONTAMINATION		
Contamination, mg/ltr	10.0 Max	0.00
FOAMING CHARACTERISTICS, STATIC		
176°F, Volume after 30 min. aeration, ml	100.0 Max	10 ml
Collapse time, sec.	60.0 Max	2
FOAMING CHARACTERISTICS, DYNAMIC		
176°F, Volume @ 1000 cc air, cc	100.0 Max	10
Collapse time, min.	60.0 Max	7.8 sec.
Volume @ 1500 cc air, cc	150.0 Max	10
Collapse time, min.	60.0 Max	8.8 sec.
Volume @ 2000 cc air, cc	Report	10
Collapse time, min.	60.0 Max	8.5
230°F, Volume @ 1000 cc air, cc	100.0 Max	10
Collapse time, min	60.0 Max	8.9 sec.
Volume @ 1500 cc air, cc	150.0 Max	10
Collapse time, min.	60.0 Max	10.1
Volume @ 2000 cc air, cc	Report	10
Collapse time, min.	60.0 Max	10.5
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2	6.0 Max	0.0
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2	3.0 Max	+0.2
Bronze, AMS4616, mg/in^2	3.0 Max	-0.1

MIL-L-7808H QUALITY CONFORMANCE

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
% Swell	12.0-35.0	+26.0
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
% Swell	2.0-25.0	+12.6
Tensile Strength, % Change	50.0 Max	0.0
Elongation, % Change	50.0 Max	+13.8
Hardness, No., Change	20.0 Max	- 5
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-30.0	+ 5.3
Tensile Strength, % Change	50.0 Max	-41.9
Elongation, % Change	50.0 Max	-20.7
Hardness, No., Change	20.0 Max	- 5
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-25.0	+ 5.2
Tensile Strength, % Change	50.0 Max	-48.2
Elongation, % Change	50.0 Max	-40.6
Hardness, No., Change	20.0 Max	- 5
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max	.45
Viscosity Change, %	Report	113.7
TAN Change	Report	26.44
Oil Consumption	Report	75
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs	25.0 Max	~~119.9~~ (57.8)*
168 Hrs	150.0 Max	~~598.7~~ (288.5)*
VISCOSITY STABILITY @ 3 Hrs @ -65°F		
Original Oil, cst	17,000 Max	10,351
Viscosity Change, %	6.0 Max	-0.2
3 Hrs, cst	17,000 Max	10,331
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original Oil, cst	17,000 Max	10,351
Viscosity Change, %	6.0 Max	-0.6
72 Hrs, cst	17,000 Max	10,289
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
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MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Corporation DATE November 4, 1981

SAMPLE 1997695, Batch 0-79-6

	Specification	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F		
Corrosion:		
Steel, mg/cm²	Report	+0.119
Silver, mg/cm²	Report	-0.069
Aluminum, mg/cm²	Report	+0.020
Magnesium, mg/cm²	Report	-7.950
Bronze, AMS-4616, mg/cm²	Report	+0.050
Titanium, mg/cm²	Report	0.000
M-50 Steel, mg/cm²	Report	+0.108
Appearance of Metal Specimens:		
Pitting	Report	Mg.
Etching	Report	None
Corrosion	Report	Mg
Staining	Report	M-50, Bz, Steel, Mg

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial	Report								12.4
Viscosity @ 100°F, % Change	Report	+6.5	+7.3	+12.1	+14.5	+29.0	+35.5	+45.2	+47.5
Viscosity @ 210°F, Initial	Report								3.3
Viscosity @ 210°F, % Change	Report								+27.3
Total Acid Number, Initial	Report								0.22
Total Acid Number, Change	Report	0.90	1.74	3.54	4.66	12.70	15.26	15.50	16.06
Evaporation Loss, %	Report								2.9
Sludge, Volume, %	Report								0.0

MIL-L-7808 DEPOSITION TEST

Algert Inc.

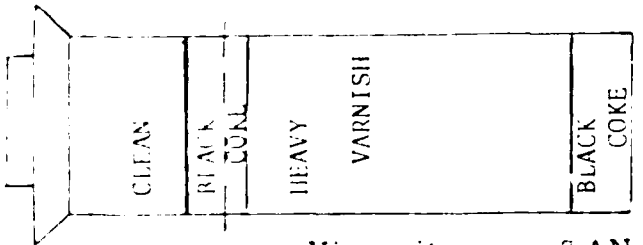
TEST LUBRICANT DESIGNATION: Monsanto Research, Sample 1997695, Batch 0-79-6

TEST NO. 4475 DATE 9/23/81 OPERATOR Trawick, Revel, Pavlicek

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HRS	TIME	TEMPERATURES °F: OIL IN 1	OIL OUT 3	2	VAT SP	AP 4	HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
START			550	570	570	15		2.5		
		300				115	185	2.5	0	1.0
1			300	590	590	115	185	2.5	0	1.0
			300					2.5	0	0
2								2.5	0	
						115	185	2.5	0	
3		300				115	185	2.5	0	1.0
							185	2.5	0	
4							185	2.5		
								2.5		
			570			115	185	2.5	0	
7						115	185		0	
			570	590	590	115	185	2.5	0	1.0
8									0	
		300	550	590	590	115	185	2.5	0	1.0
9										
10										
11										
12										

OIL LEVEL



Test Section D Tube No. 3

Oil Cons., cc 75 Cooler Sludge Clean

Coking Tube, gm .044

Filter Sludge, gm .014

DEPOSIT RATING .45

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	Viscosity	TAN
Before	12.4	.22
After	26.5	26.66
Change	14.1	26.44

ALCOR Inc.
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(512) 349-5771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Company DATE 3/9/82

SAMPLE 2000248, Batch 0-79-8

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.)	0.30 Max	0.08
Viscosity @ 210°F, cs	3.0 Min	3.2
Viscosity @ 100°F, cs	Report	12.5
Flash Point (COC), °F	400.0 Min	435
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %	30.0 Max	15.1
TRACE SEDIMENT		
Precipitation, ml/200 ml	0.005 Max	0.000
PARTICULATE CONTAMINATION		
Contamination, mg/ltr	10.0 Max	0.0
FOAMING CHARACTERISTICS, STATIC		
176°F, Volume after 30 min. aeration, ml	100.0 Max	90
Collapse time, sec.	60.0 Max	26.4 sec
FOAMING CHARACTERISTICS, DYNAMIC		
176°F, Volume @ 1000 cc air, cc	100.0 Max	30
Collapse time, min.	60.0 Max	59.3 sec
Volume @ 1500 cc air, cc	150.0 Max	45
Collapse time, min.	60.0 Max	63.0 sec
Volume @ 2000 cc air, cc	Report	25
Collapse time, min.	60.0 Max	65.6 sec
230°F, Volume @ 1000 cc air, cc	100.0 Max	45
Collapse time, min	66.0 Max	47.6 sec
Volume @ 1500 cc air, cc	150.0 Max	50
Collapse time, min.	60.0 Max	48.3 sec
Volume @ 2000 cc air, cc	Report	50
Collapse time, min.	60.0 Max	61.8 sec
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2	6.0 Max	+ 0.1
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2	3.0 Max	- 0.1
Bronze, AMS4616, mg/in^2	3.0 Max	0.0

MIL-L-7808H QUALITY CONFORMANCE

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
% Swell	12.0-35.0	+26.1
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
% Swell	2.0-25.0	+16.2
Tensile Strength, % Change	50.0 Max	-11.8
Elongation, % Change	50.0 Max	+10.9
Hardness, No., Change	20.0 Max	-10
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-30.0	+14.6
Tensile Strength, % Change	50.0 Max	-19.5
Elongation, % Change	50.0 Max	-14.0
Hardness, No., Change	20.0 Max	- 5
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-25.0	+ 5.1
Tensile Strength, % Change	50.0 Max	-50.4
Elongation, % Change	50.0 Max	-42.4
Hardness, No., Change	20.0 Max	-10
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max	1.0
Viscosity Change, %	Report	+85.6
TAN Change	Report	23.04
Oil Consumption	Report	125
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs	25.0 Max	~~+ 3.6~~ (+ 1.7)*
168 Hrs	150.0 Max	~~-68.1~~ (32.8)*
VISCOSITY STABILITY @ 3 Hrs @ -65°F		
Original Oil, cst	17,000 Max	10,914
Viscosity Change, %	6.0 Max	- 1.6
3 Hrs, cst	17,000 Max	10,738
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original Oil, cst	17,000 Max	10,914
Viscosity Change, %	6.0 Max	- 1.6
72 Hrs, cst	17,000 Max	10,741
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
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San Antonio, Texas 78284
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MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Company DATE 3/9/82

SAMPLE 2000248, Batch 0-79-8

	Specification	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F		
Corrosion:		
Steel, mg/cm^2	Report	-0.024
Silver, mg/cm^2	Report	-0.-59
Aluminum, mg/cm^2	Report	-0.001
Magnesium, mg/cm^2	Report	-6.903
Bronze, AMS4616, mg/cm^2	Report	-5.409
Titanium, mg/cm^2	Report	-0.010
M-50 Steel, mg/cm^2	Report	+0.063
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	Bz, Mg
Staining	Report	None

Oxidation:		16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial	Report								12.5
Viscosity @ 100°F, % Change	Report	+5.6	+5.6	+9.6	+10.4	+32.8	+51.2	+91.2	+104.8
Viscosity @ 210°F, Initial	Report								3.2
Viscosity @ 210°F, % Change	Report								+ 62.5
Total Acid Number, Initial	Report								0.08
Total Acid Number, Change	Report	1.32	2.04	3.12	3.80	16.52	20.40	26.08	30.00
Evaporation Loss, %	Report								6.9
Sludge, Volume, %	Report								0.0

MIL-L-7808 DEPOSITION TEST

Alcor inc.

10130 JONES MALTSBERGER ROAD P.O. BOX 32516
SAN ANTONIO, TEXAS 78216 (512) 349-3771

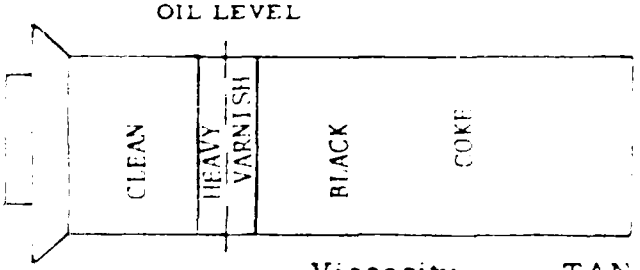
TEST LUBRICANT DESIGNATION Monsanto Research, Sample 2000248, Batch 0-79-8

TEST NO. 4503 DATE 2/18/82 OPERATOR Revet, Trawick, Pavlicek

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES, °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP	CAB AIR 4				
START	1330	300	425	590	535	116	185	2.3	0	0.9
	1400	300	440	590	545	116	190	2.3	0	1.0
1	30	300	430	590	550	116	190	2.3	0	1.0
	1500	300	430	590	550	116	190	2.3	0	1.0
2	30	300	430	590	555	116	190	2.3	0	1.0
	1600	300	430	590	555	116	190	2.3	0	1.0
3	30	300	430	590	555	116	190	2.3	0	1.0
	1700	300	430	590	555	116	190	2.3	0	1.0
4	30	300	430	590	555	116	190	2.3	0	1.0
	1800	300	430	590	555	116	190	2.3	0	1.0
5	30	300	430	590	555	116	190	2.3	0	1.0
	1900	300	430	590	555	116	190	2.3	0	1.0
6	30	300	430	590	550	116	190	2.3	0	1.0
	2000	300	430	590	550	116	190	2.3	0	1.0
7	30	300	430	590	570	116	190	2.3	0	1.0
	2100	300	430	590	570	116	190	2.3	0	1.0
8	30	300	430	590	570	116	190	2.3	0	1.0
	2200	300	430	590	570	116	190	2.3	0	1.0
9	30	300	430	590	560	116	190	2.3	0	1.0
	2300	300	430	590	550	116	190	2.3	0	1.0
10	30	300	430	590	550	116	190	2.3	0	1.0
	2400	300	430	590	555	116	190	2.3	0	1.0
11	30	300	430	590	550	116	190	2.3	0	1.0
	0100	300	430	590	550	116	190	2.3	0	1.0
12	30	300	430	590	550	116	190	2.3	0	1.0

OIL LEVEL



Test Section D Tube No. 4

Oil Cons., cc 125 Cooler Sludge Light

Coking Tube, gm .092

Filter Sludge, gm .079

DEPOSIT RATING 1.00

	Viscosity	TAN
Before	12.5	.08
After	23.2	23.12
Change	10.7	23.04
% Change	+85.6	

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ALCOR Inc.
10130 Jones Maltsberger Road
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MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Company DATE 3/9/82

SAMPLE 2000247, Batch 0-79-15

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.)	0.30 Max	0.06
Viscosity @ 210°F, cs	3.0 Min	3.2
Viscosity @ 100°F, cs	Report	12.4
Flash Point (COC), °F	400.0 Min	435
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %	30.0 Max	17.5
TRACE SEDIMENT		
Precipitation, ml/200 ml	0.005 Max	0.000
PARTICULATE CONTAMINATION		
Contamination, mg/ltr	10.0 Max	+0.01
FOAMING CHARACTERISTICS, STATIC		
176°F, Volume after 30 min. aeration, ml	100.0 Max	120
Collapse time, sec.	60.0 Max	29.1 sec
FOAMING CHARACTERISTICS, DYNAMIC		
176°F, Volume @ 1000 cc air, cc	100.0 Max	30
Collapse time, min.	60.0 Max	46 sec
Volume @ 1500 cc air, cc	150.0 Max	50
Collapse time, min.	60.0 Max	50.5 sec
Volume @ 2000 cc air, cc	Report	30
Collapse time, min.	60.0 Max	58.9 sec
230°F, Volume @ 1000 cc air, cc	100.0 Max	30
Collapse time, min	60.0 Max	43.5 sec
Volume @ 1500 cc air, cc	150.0 Max	35
Collapse time, min.	60.0 Max	43.7
Volume @ 2000 cc air, cc	Report	30
Collapse time, min.	60.0 Max	46.1
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change mg/in^2	6.0 Max	~~+0.2~~ (+0.1)*
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2	3.0 Max	-0.1
Bronze, AMS4616, mg/in^2	3.0 Max	0.0

*Corrected results

MIL-L-7808H QUALITY CONFORMANCE

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
% Swell	12.0-35.0	+27 8
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
% Swell	2.0-25.0	+15.0
Tensile Strength, % Change	50.0 Max	-25.3
Elongation, % Change	50.0 Max	- 9.4
Hardness, No., Change	20.0 Max	-10
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-30.0	+17.2
Tensile Strength, % Change	50.0 Max	- 9.2
Elongation, % Change	50.0 Max	- 2.9
Hardness, No., Change	20.0 Max	0
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
% Swell	2.0-25.0	+ 6.6
Tensile Strength, % Change	50.0 Max	-31.9
Elongation, % Change	50.0 Max	-25.8
Hardness, No., Change	20.0 Max	-10
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number	1.5 Max	.71
Viscosity Change, %	Report	+79.8
TAN Change	Report	23.38
Oil Consumption	Report	125
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs	25.0 Max	~~11.9~~ (5.7)*
168 Hrs	150.0 Max	~~88.5~~ (42.7)*
VISCOSITY STABILITY @ 3 Hrs @ -65°F		
Original Oil, cst	17,000 Max	11,213
Viscosity Change, %	6.0 Max	- 3.5
3 Hrs, cst	17,000 Max	10,823
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original Oil, cst	17,000 Max	11,213
Viscosity Change, %	6.0 Max	-19.0
72 Hrs, cst	17,000 Max	9,085
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
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MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Company DATE 3/9/82

SAMPLE 2000247, Batch 0-79-15

	Specification	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F		
Corrosion:		
Steel, mg/cm²	Report	+0.018
Silver, mg/cm²	Report	-0.024
Aluminum, mg/cm²	Report	+0.007
Magnesium, mg/cm²	Report	-11.702
Bronze, AMS4616, mg/cm²	Report	-1.510
Titanium, mg/cm²	Report	+0.008
M-50 Steel, mg/cm²	Report	+0.024
Appearance of Metal Specimens:		
Pitting	Report	None
Etching	Report	None
Corrosion	Report	Bz,Mg
Staining	Report	None

Oxidation:	Specification	16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial	Report								12.4
Viscosity @ 100°F, % Change	Report	+6.5	+6.5	+9.7	+10.5	+25.6	+40.3	+75.8	+87.1
Viscosity @ 210°F, Initial	Report								3.2
Viscosity @ 210°F, % Change	Report								+50.0
Total Acid Number, Initial	Report								0.06
Total Acid Number, Change	Report	1.18	1.68	2.84	3.42	12.34	16.32	20.88	25.42
Evaporation Loss, %	Report								6.1
Sludge, Volume, %	Report								0.0

MIL-L-7808 DEPOSITION TEST

Alcor inc.
10130 JONES MALTSBERGER ROAD P O BOX 32516
SAN ANTONIO TEXAS 78284 512 349-3771

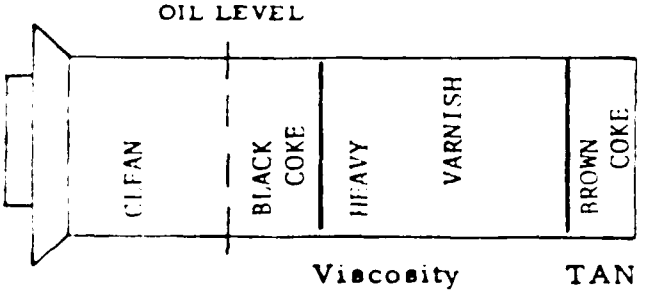
TEST LUBRICANT DESIGNATION Monsanto Research, Sample 2000247, Batch 0-79-15

TEST NO. 4502 DATE 2/17/82 OPERATOR Revet, Trawick, Pavlicek

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES, °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP	CAB AIR 4				
START	1435	300	430	590	530	116	195	2.3	0	1.0
	1505	300	435	590	535	116	190	2.3	0	1.0
1	35	300	430	590	530	116	190	2.3	0	1.0
	105	300	435	590	535	116	190	2.3	0	1.0
2	35	300	430	590	530	116	190	2.3	0	1.0
	205	300	430	590	530	116	190	2.3	0	1.0
3	35	300	430	590	530	116	190	2.3	0	1.0
	305	300	430	590	530	116	190	2.3	0	1.0
4	35	300	430	590	530	116	190	2.3	0	1.0
	1405	300	430	590	530	116	190	2.3	0	1.0
5	35	300	430	590	530	116	190	2.3	0	1.0
	1505	300	435	590	550	116	190	2.3	0	1.0
6	35	300	435	590	550	116	190	2.3	0	1.0
	2105	300	435	590	550	116	190	2.3	0	1.0
7	35	300	435	590	550	116	190	2.3	0	1.0
	2205	300	435	590	550	116	190	2.3	0	1.0
8	35	300	435	590	525	116	190	2.3	0	1.0
	2305	300	435	590	525	116	190	2.3	0	1.0
9	35	300	1135	590	530	116	190	2.3	0	1.0
	2405	300	435	590	530	116	190	2.3	0	1.0
10	35	300	435	590	530	116	190	2.3	0	1.0
	0105	300	435	590	530	116	190	2.3	0	1.0
11	35	300	435	590	530	116	190	2.3	0	1.0
	0205	300	435	590	525	116	190	2.3	0	1.0
12	35	300	435	590	525	116	190	2.3	0	1.0

OIL LEVEL



Test Section C Tube No. 3

Oil Cons., cc 125 Cooler Sludge Clean

Coking Tube, gm .067

Filter Sludge, gm .037

DEPOSIT RATING .71

	Viscosity	TAN 170
Before	12.4	.06
After	22.3	23.44
Change	9.9	23.38
% Change	+79.8	

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Company DATE 7/8/82

SAMPLE 2000273 (0-79-10)

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.), max	0.30	0.11
Viscosity @ 210°F, cSt, min	3.0	3.1
Viscosity @ 100°F, cSt	Report	12.1
Flash Point (COC), °F, min	400.0	420
Evaporation, 6.5 Hrs @ 400, Wt. Loss, %, max	30.0	18.9
TRACE SEDIMENT		
Precipitation, ml/200 ml, max	0.005	0.000
PARTICULATE CONTAMINATION		
Contamination, mg/ltr, max	10.0	0.1
FOAMING CHARACTERISTICS - Static		
176°F		
Volume after 30 min aeration, ml, max	100.0	65.0
Collapse time, sec, max	60.0	17.7 sec
FOAMING CHARACTERISTICS - Dynamic		
176°F		
Volume @ 1000 cc air, cc, ml, max	100.0	40.0
Collapse time, min	60.0 Max	34.0 sec
@ 1500 cc air, cc, ml, max	150.0	80.0
Collapse time, min	60.0 Max	59.9 sec
@ 2000 cc air, cc, ml	Report	90.0
Collapse time, min	60.0 Max	63.2 sec
230°F		
Volume @ 1000 cc air, cc, ml, max	100.0	30.0
Collapse time, min	60.0 Max	54.1 sec
@ 1500 cc air, cc, ml, max	150.0	60.0
Collapse time, min	60.0 Max	55.3 sec
@ 2000 cc air, cc, ml	Report	60.0
Collapse time, min, min	60.0 Max	56.5 sec
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2, max	6.0	~~2.0~~ (1.0)*
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2, max	3.0	-0.1
Bronze, AMS4616, mg/in^2, max	3.0	-0.1

*Corrected results

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
Swell, % , max	12.0 to 35.0	+28.0
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
Swell, %, max	2.0 to 25.0	+14.9
Tensile Strength, % Change, max	50.0	+32.0
Elongation, % Change, max	50.0	+45.0
Hardness, No., Change, max	20.0	- 5.0
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
Swell, %, max	2.0 to 30.0	+12.9
Tensile Strength, % Change, max	50.0	-45.0
Elongation, % Change, max	50.0	-14.3
Hardness, No., Change, max	20.0	-10.0
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
Swell, %, max	2.0 to 25.0	+ 6.2
Tensile Strength, % Change, max	50.0	-52.8
Elongation, % Change, max	50.0	-42.0
Hardness, No., Change, max	20.0	- 5.0
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number, max	1.5	.33
Viscosity Change, %	Report	+76.9
Total Acid Number (T.A.N.)	Report	18.59
Oil Consumption, cc	Report	90
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2, max		
48 Hrs, max	25.0	~~11.8~~ (5.7)*
168 Hrs, max	150.0	~~257.2~~ (123.9)*
VISCOSITY STABILITY, 3 Hrs @ -65°F		
Original, cSt, max	17,000	11,041
Viscosity Change, %, max	6.0	+1.6
3 Hrs, cSt	17,000	11,218
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original, cSt, max	17,000	11,041
Viscosity Change, %, max	6.0	-13.4
72 Hrs, cSt, max	17,000	9,737
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Company DATE 7/8/82

SAMPLE 2000273 (-0-79-10)

CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F	Results
Corrosion:	
Steel, mg/cm^2	-0.006
Silver, mg/cm^2	-0.020
Aluminum, mg/cm^2	+0.006
Magnesium, mg/cm^2	-14.121
Bronze, AMS4616, mg/cm^2	0.018
Titanium, mg/cm^2	-0.006
M-50 Steel, mg/cm^2	0.000
Appearance:	
Pitting	None
Etching	None
Corrosion	Mg
Staining	None

Oxidation:	16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial								12.1
% Change	+6.6	+7.4	+11.6	+16.5	+42.1	+60.3	+94.2	+104.1
Viscosity @ 210°F, Initial								3.1
% Change								+ 61.3
Total Acid Number, Initial								0.11
Change	0.73	1.75	3.15	4.83	14.53	21.09	25.49	27.49
Evaporation Loss, %	5.1							
Sludge, Volume, %	0.0							

MIL-L-7808 DEPOSITION TEST

Alcor inc.

10100 JONES MALTSBERGER ROAD P.O. BOX 32516
SAN ANTONIO TEXAS 78284 512 349 3771

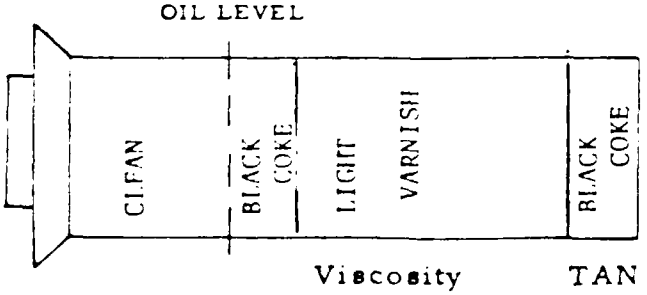
TEST LUBRICANT DESIGNATION Monsanto Research, Sample: 2000273 (0-79-10)

TEST NO 4513 DATE 6/8/82 OPERATOR Trawick, Pavlicek

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES, °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP	CAB AIR 4				
START	1800	300	440	590	545	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
1	1900	300	440	590	555	116	190	2.3	0	1.0
	30	300	445	590	550	116	190	2.3	0	1.0
2	2000	300	440	590	545	116	190	2.3	0	1.0
	30	300	445	590	540	116	190	2.3	0	1.0
3	2100	300	445	590	545	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
4	2200	300	435	590	545	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
5	2300	300	440	590	550	116	190	2.3	0	1.0
	30	300	445	590	555	116	190	2.3	0	1.0
6	2400	300	445	590	550	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
7	0100	300	440	590	550	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
8	0200	300	440	590	550	116	190	2.3	0	1.0
	30	300	445	590	555	116	190	2.3	0	1.0
9	0300	300	440	590	550	116	190	2.3	0	1.0
	30	300	445	590	555	116	190	2.3	0	1.0
10	0400	300	440	590	550	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
11	0500	300	440	590	550	116	190	2.3	0	1.0
	30	300	440	590	550	116	190	2.3	0	1.0
12	0600	300	440	590	550	116	190	2.3	0	1.0

OIL LEVEL



	Viscosity	TAN
Before	12.1	.11
After	21.4	18.7
Change	9.3	18.59
% Change	+76.9	

174

Test Section C Tube No. 3

Oil Cons., cc 90 Cooler Sludge Clean

Coking Tube, gm .031

Filter Sludge, gm .021

DEPOSIT RATING .33

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Company DATE July 7, 1982

SAMPLE 2000268 (0-79-14)

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.), max	0.30	0.14
Viscosity @ 210°F, cSt, min	3.0	3.1
Viscosity @ 100°F, cSt	Report	11.7
Flash Point (COC), °F, min	400	395
Evaporation, 6.5 Hrs @ 400°F, Wt. Loss, %, max	30.0	17.5
TRACE SEDIMENT		
Precipitation, ml/200 ml, max	0.005	0.000
PARTICULATE CONTAMINATION		
Contamination, mg/ltr, max	10.0	0.0
FOAMING CHARACTERISTICS - Static		
176°F		
Volume after 30 min aeration, ml, max	100	35
Collapse time, sec, max	60	21.3
FOAMING CHARACTERISTICS - Dynamic		
176°F		
Volume @ 1000 cc air, cc, ml, max	100	30
Collapse time, min	60 Max	38.0 sec
@ 1500 cc air, cc ml, max	150	50
Collapse time, min	60 Max	45.9 sec
@ 2000 cc air, ml	Report	60
Collapse time, min	60 Max	57.5 sec
230°F		
Volume @ 1000 cc air, cc, ml, max	100	10
Collapse time, min	60 Max	36.3 sec
@ 1500 cc air, cc, ml, max	150	30
Collapse time, min	60 Max	40.1 sec
@ 2000 cc air, ml	Report	30
Collapse time, min	60 Max	41.0 sec
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2, max	6.0	~~0.9~~ (0.5)*
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2, max	3.0	0.0
Bronze, AMS4616, mg/in^2, max	3.0	-0.1

*Corrected results

	Specification	Results	
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F			
Swell, %, max	12.0 to 35.0	+26.5	
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F			
Swell, %, max	2.0 to 25.0	+13.7	
Tensile Strength, % Change, max	50.0	-22.9	
Elongation, % Change, max	50.0	-12.5	
Hardness, No., Change, max	20.0	-10.0	
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F			
Swell, %, max	2.0 to 30.0	+17.9	
Tensile Strength, % Change, max	50.0	-34.0	
Elongation, % Change, max	50.0	+ 5.7	
Hardness, No., Change, max	20.0	-10.0	
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F			
Swell, %, max	2.0 to 25.0	+ 6.9	
Tensile Strength, % Change, max	50.0	-55.4	
Elongation, % Change, max	50.0	-28.8	
Hardness, No., Change, max	20.0	-10.0	
DEPOSITION NUMBER (see attached data sheet)			
Deposit Number, max	1.5	1.14	
Viscosity Change, %	Report	42.7	
Total Acid Number (TAN)	Report	14.0	
Oil Consumption, cc	Report	125	
ACCELERATED STORAGE STABILITY @ 230°F			
Lead Wt. Loss, mg/in^2			
48 Hrs, max	25.0	~~14.5~~	(7.0)*
168 Hrs, max	150.0	~~468.5~~	(225.7)*
VISCOSITY STABILITY - 3 Hrs @ -65°F			
Original, cSt, max	17,000	9,868	
Viscosity Change, %, max	6.0	-0.4	
3 Hrs, cSt, max	17,000	9,825	
VISCOSITY STABILITY, 72 Hrs @ -65°F			
Original, cSt, max	17,000	9,868	
Viscosity Change, %, max	6.0	- 7.5	
72 Hrs, cSt, max	17,000	9,126	
WORKMANSHIP			
Clear, Transparent	Report	Clear	

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Company DATE July 7, 1982

SAMPLE: 2000268 (0-79-14)

	Results
CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F	
Corrosion:	
Steel, mg/cm^2	+0.008
Silver, mg/cm^2	-0.016
Aluminum, mg/cm^2	-0.012
Magnesium, mg/cm^2	-15.764
Bronze, AMS4616, mg/cm^2	0.000
Titanium, mg/cm^2	-0.007
M-50 Steel, mg/cm^2	-0.004
Appearance:	
Pitting	None
Etching	None
Corrosion	Mg
Staining	None

Oxidation:	16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial								11.7
% Change	+8.5	+12.0	+19.7	+23.9	+40.2	+47.9	+60.7	+63.2
Viscosity @ 210°F, Initial								3.1
% Change								-35.4
Total Acid Number, Initial								0.14
Change	1.82	2.56	6.64	7.72	11.80	13.54	16.46	16.46
Evaporation Loss, %	2.3							
Sludge, Volume, %	0.0							

MIL-L-7808 DEPOSITION TEST

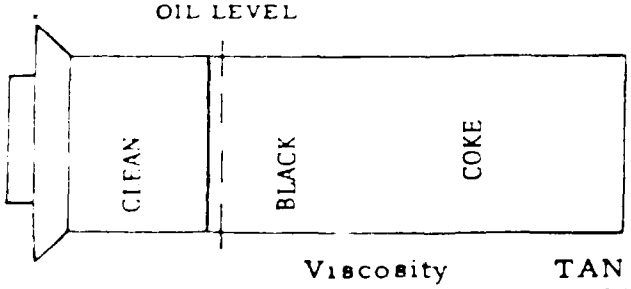
Alcor inc.
10130 JONES MALTSBERGER ROAD P.O. BOX 32516
SAN ANTONIO TEXAS 78284 512/349-3771

TEST LUBRICANT DESIGNATION Monsanto Research, Sample: 2000268 (0-79-14)

TEST NO 4511 DATE 6/2/82 OPERATOR Revet, Trawick, Pavlicek

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP	CAB AIR 4				
START	[illegible]	300	400	590	545	115	195	2.3	0	1.0
	[illegible]	300	425	590	565	115	190	2.3	0	1.0
1	[illegible]	300	420	590	560	115	190	2.3	0	1.0
	[illegible]	300	420	590	560	115	190	2.3	0	1.0
2	[illegible]	300	420	590	565	115	190	2.3	0	1.0
	[illegible]	300	420	590	560	115	190	2.3	0	1.0
3	[illegible]	300	420	590	565	115	190	2.3	0	1.0
	[illegible]	300	420	590	565	115	190	2.3	0	1.0
4	[illegible]	300	420	590	555	115	190	2.3	0	1.0
	[illegible]	300	420	590	560	115	190	2.3	0	1.0
5	[illegible]	300	420	590	560	115	190	2.3	0	1.0
	[illegible]	300	425	590	560	115	190	2.3	0	1.0
6	[illegible]	300	425	590	560	115	190	2.3	0	1.0
	[illegible]	300	425	590	560	115	190	2.3	0	1.0
7	[illegible]	300	425	590	570	115	190	2.3	0	1.0
	[illegible]	300	425	590	550	115	190	2.3	0	1.0
8	[illegible]	300	425	590	555	115	190	2.3	0	1.0
	[illegible]	300	425	590	560	115	190	2.3	0	1.0
9	[illegible]	300	420	590	550	115	190	2.3	0	1.0
	[illegible]	300	420	590	550	115	190	2.3	0	1.0
10	[illegible]	300	425	590	555	115	190	2.3	0	1.0
	[illegible]	300	425	590	550	115	190	2.3	0	1.0
11	[illegible]	300	420	590	550	115	190	2.3	0	1.0
	[illegible]	300	425	590	550	115	190	2.3	0	1.0
12	[illegible]	300	425	590	550	115	190	2.3	0	1.0



Test Section C Tube No. 4

Oil Cons., cc 125 Cooler Sludge Clean

Coking Tube, gm .113

Filter Sludge, gm .014

DEPOSIT RATING 1.14

	Viscosity	TAN 178
Before	11.7	.14
After	16.7	14.14
Change	5.0	14.00

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Company DATE 7/8/82

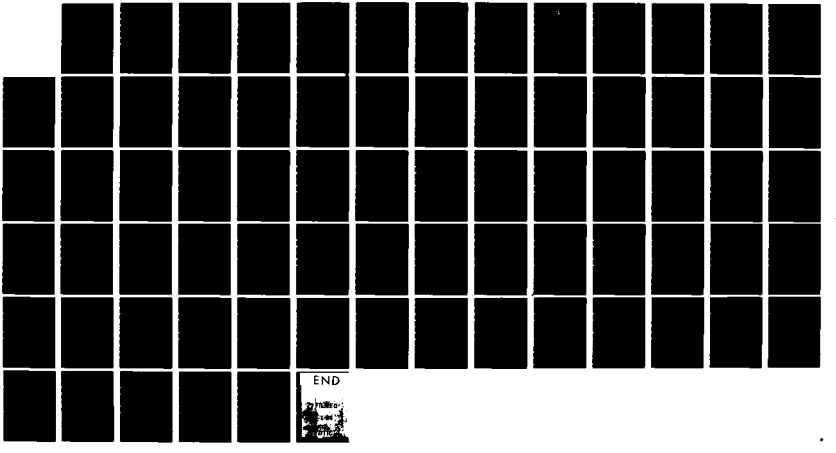
SAMPLE 2000275 (0-79-12)

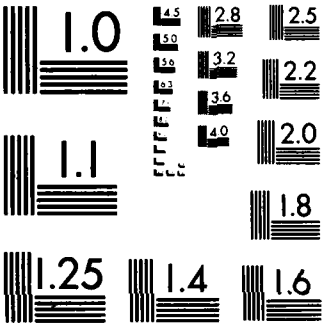
	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.), max	0.30	0.11
Viscosity @ 210°F, cSt, min	3.0	3.2
Viscosity @ 100°F, cSt	Report	13.3
Flash Point (COC), °F, min	400.0	430
Evaporation Loss, 6.5 Hrs @ 400°F, Wt. Loss, %, max	30.0	16.7
TRACE SEDIMENT		
Precipitation, ml/200 ml, max	0.005	0.000
PARTICULSTE CONTAMINATION		
Contamination, mg/ltr, max	10.0	0.0
FOAMING CHARACTERISTICS - Static		
176°F		
Volume after 30 min aeration, ml, max	100.0	60.0
Collapse time, sec, max	60.0	24.6
FOAMING CHARACTERISTICS - Dynamic		
176°F		
Volume @ 1000 cc air, cc, ml, max	100.0	10.0
Collapse time, min	60.0 Max	37.7 sec
@ 1500 cc air, cc, ml, max	150.0	30.0
Collapse time, min	60.0 Max	61.5 sec
@ 2000 cc air, cc, ml	Report	30.0
Collapse time, min	60.0 Max	67.2 sec
230°F		
Volume @ 1000 cc air, cc, ml, max	100.0	10.0
Collapse time, min	60.0 Max	41.6 sec
@ 1500 cc air, cc, ml, max	150.0	30.0
Collapse time, min	60.0 Max	61.5 sec
@ 2000 cc air, cc, ml	Report	30.0
Collapse time, min	60.0 Max	66.8 sec
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in^2, max	6.0	0.4 *
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in^2, max	3.0	- 0.2
Bronze, AMS4616, mg/in^2, max	3.0	0.0

*Corrected results

AD-A135 926 TURBINE ENGINE LUBRICANT RECLAMATION(U) MONSANTO RESEARCH CORP DAYTON OHIO DATTON LAB R J BRUNS ETAAL. JUN 83 MRC-DA-1120 AFWAL-TR-83-2042 F33615-79-C-2052 3/3

UNCLASSIFIED F/G 11/8 NL





	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
Swell, %, max	12.0 to 35.0	+ 27.8
J ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
Swell, %, max	2.0 to 25.0	+ 14.8
Tensile Strength, % Change, max	50.0	- 27.7
Elongation, % Change, max	50.0	+ 34.5
Hardness, No., Change, max	20.0	5.0
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
Swell, %, max	2.0 to 30.0	+ 9.9
Tensile Strength, % Change, max	50.0	- 45.0
Elongation, % Change, max	50.0	- 20.0
Hardness, No., Change, max	20.0	- 10.0
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
Swell, %, max	2.0 to 25.0	+ 6.0
Tensile Strength, % Change, max	50.0	- 52.8
Elongation, % Change, max	50.0	- 47.8
Hardness, No., Change, max	20.0	- 5.0
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number, max	1.5	.37
Viscosity Change, %	Report	+218.9
Total Acid Number (T.A.N.)	Report	20.21
Oil Consumption, cc	Report	75
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs, max	25.0	~~1.3~~ (0.6)*
168 Hrs, max	150.0	~~245.4~~ (118.3)*
VISCOSITY STABILITY, 3 Hrs @ -65°F		
Original, cSt, max	17,000	12,612
Viscosity Change, %, max	6.0	+ 0.2
3 Hrs, cSt, max	17,000	12,643
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original, cSt, max	17,000	12,612
Viscosity Change, %, max	6.0	- 5.4
72 Hrs, cSt, max	17,000	11,925
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Company DATE July 8, 1982

SAMPLE 2000275 (0-79-12)

CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F	Results
Corrosion:	
Steel, mg/cm^2	-0.010
Silver, mg/cm^2	-0.012
Aluminum, mg/cm^2	0.000
Magnesium, mg/cm^2	-10.798
Bronze, AMS4616, mg/cm^2	-0.026
Titanium, mg/cm^2	-0.008
M-50 Steel, mg/cm^2	-0.006
Appearance:	
Pitting	None
Etching	None
Corrosion	Mg
Staining	None

Oxidation:	16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial								13.3
% Change	+1.5	+4.5	+8.3	+10.5	+12.8	+15.8	+40.6	+48.9
Viscosity @ 210°F, Initial								3.2
% Change								+37.5
Total Acid Number, Initial								0.11
Change	0.67	1.47	2.63	3.03	4.05	5.93	16.61	18.41
Evaporation Loss, %	3.4							
Sludge, Volume, %	0.0							

MIL-L-7808 DEPOSITION TEST

Alcor inc.
10130 JONES MALTSBERGER ROAD P O BOX 32516
SAN ANTONIO TEXAS 78216 512 349-3771

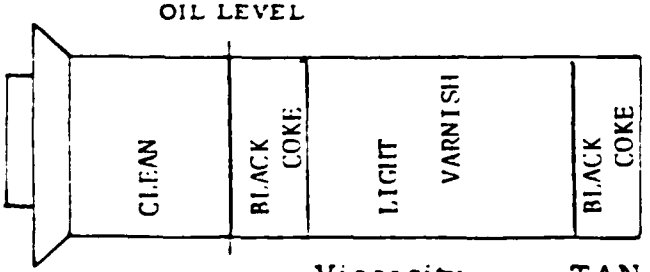
TEST LUBRICANT DESIGNATION Monsanto Research, Sample: 2000275 (0-79-12)

TEST NO 4514 DATE 6/9/82 OPERATOR Revet, Pavlicek, Trawick

Coking Tube 590°F · Oil In 300°F · Oil Flow 300 cc/min · Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP.	CAB AIR 4				
START	[illegible]	300	445	590	555	115	180	2.3	0	1.0
	[illegible]	[illegible]	440	590	560	115	180	2.3	0	1.0
1	[illegible]	300	440	590	560	115	180	2.3	0	1.0
	[illegible]	300	445	590	570	115	180	2.3	0	1.0
2	[illegible]	300	445	590	570	115	180	2.3	0	1.0
	[illegible]	300	445	590	570	115	180	2.3	0	1.0
3	[illegible]	300	435	590	570	115	180	2.3	0	1.0
	[illegible]	300	445	590	565	115	185	2.3	0	1.0
4	[illegible]	300	445	590	570	115	185	2.3	0	1.0
	[illegible]	300	450	590	570	115	185	2.3	0	1.0
5	[illegible]	300	450	590	580	115	185	2.3	0	1.0
	[illegible]	300	450	590	570	115	185	2.3	0	1.0
6	[illegible]	300	450	590	570	115	185	2.3	0	1.0
	[illegible]	300	450	590	570	115	185	2.3	0	1.0
7	[illegible]	300	450	590	570	115	185	2.3	0	1.0
	[illegible]	300	450	590	570	115	185	2.3	0	1.0
8	[illegible]	300	445	590	560	115	185	2.3	0	1.0
	[illegible]	300	445	590	560	115	185	2.3	0	1.0
9	[illegible]	300	445	590	560	115	185	2.3	0	1.0
	[illegible]	300	445	590	560	115	185	2.3	0	1.0
10	[illegible]	300	445	590	560	115	185	2.3	0	1.0
	[illegible]	300	445	590	560	115	185	2.3	0	1.0
11	[illegible]	300	445	590	565	115	185	2.3	0	1.0
	[illegible]	300	450	590	565	115	185	2.3	0	1.0
12	[illegible]	300	450	590	565	115	185	2.3	0	1.0

OIL LEVEL



Test Section D Tube No. 2

Oil Cons., cc 75 Cooler Sludge Lt. Sludge

Coking Tube, gm .035

Filter Sludge, gm .024

DEPOSIT RATING .37

	Viscosity	TAN
Before	12.7	.11
After	40.5	20.32
Change	27.8	20.21
% Change	+218.9	

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ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

MIL-L-7808H QUALITY CONFORMANCE

CUSTOMER Monsanto Research Company DATE 7/7/82

SAMPLE 2000267 (0-79-7)

	Specification	Results
PHYSICAL & CHEMICAL PROPERTIES		
Neutralization Number (T.A.N.), max	0.30	0.11
Viscosity @ 210°F, cSt, min	3.0	3.1
Viscosity @ 100°F, cSt	Report	12.1
Flash Point COC, cSt, °F, min	400	400
Evaporation Loss, 6.5 Hrs @ 400°F, Wt. Loss, %, max	30.0	14.0
TRACE SEDIMENT		
Precipitation, ml/200 ml, max	0.005	0.0
PARTICULATE CONTAMINATION		
Contamination, mg/ltr, max	10.0	0.0
FOAMING CHARACTERISTICS - Static		
176°F		
Volume after 30 min aeration, ml, max	100	50
Collapse time, sec, max	60	12.0
FOAMING CHARACTERISTICS - Dynamic		
176°F		
Volume @ 1000 cc air, cc, max	100	10
Collapse time, min	60 Max	17.9 sec
@ 1500 cc air, cc, max	150	10
Collapse time, min	60 Max	31.5 sec
@ 2000 cc air, cc	Report	20
Collapse time, min	60 Max	48.6 sec
230°F		
Volume @ 1000 cc air, cc, max	100	10
Collapse time, min	60 Max	33.2 sec
@ 1500 cc air, cc, max	150	10
Collapse time, min	60 Max	40.6 sec
@ 2000 cc air, cc,	Report	20
Collapse time, min	60 Max	74.6 sec
LEAD CORROSION, 1 Hr @ 325°F		
Wt. Change, mg/in², max	6.0	~~1.4~~ (+0.3)
SILVER & BRONZE CORROSION, 50 Hrs @ 450°F		
Silver Wt. Change, mg/in², max	3.0	0.0
Bronze, AMS4616, mg/in², max	3.0	0.4

*Corrected results

	Specification	Results
H ELASTOMER COMPATIBILITY, 168 Hrs @ 158°F		
Swell, %, max	12.0 to 35.0	+27.3
FA ELASTOMER COMPATIBILITY, 72 Hrs @ 347°F		
Swell, % , max	2.0 to 25.0	+12.6
Tensile Strength, % Change, max	50.0	+17.1
Elongation, % Change, max	50.0	+20.3
Hardness, No., Change, max	20.0	5.0
QVI ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
Swell, %, max	2.0 to 30.0	+16.0
Tensile Strength, % Change, max	50.0	-35.5
Elongation, % Change, max	50.0	+ 5.7
Hardness, No., Change, max	20.0	15
FS ELASTOMER COMPATIBILITY, 72 Hrs @ 302°F		
Swell, %, max	2.0 to 25.0	+ 5.0
Tensile Strength, % Change, max	50.0	-62.7
Elongation, % Change, max	50.0	-43.9
Hardness, No., Change, max	20.0	-10.0
DEPOSITION NUMBER (see attached data sheet)		
Deposit Number, max	1.5	.32
Viscosity Change, %	Report	+116.5
Total Acid Number	Report	26.97
Oil Consumption, cc	Report	100
ACCELERATED STORAGE STABILITY @ 230°F		
Lead Wt. Loss, mg/in^2		
48 Hrs, max	25.0	~~6.2~~ (3.0)*
168 Hrs, max	150.0	~~194.0~~ (93.5)*
VISCOSITY STABILITY, 3 Hrs @ -65°F		
Original, cSt, max	17,000	10,566
Viscosity Change, %, max	6.0	+ 0.4
3 Hrs, cSt, max	17,000	10,612
VISCOSITY STABILITY, 72 Hrs @ -65°F		
Original, cSt, max	17,000	10,566
Viscosity Change, %, max	6.0	-11.7
72 Hrs, cSt, max	17,000	9,326
WORKMANSHIP		
Clear, Transparent	Report	Clear

*Corrected results

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Company DATE July 7, 1982

SAMPLE 2000267 (0-79-7)

CORROSION & OXIDATION STABILITY, 96 Hrs @ 392°F	Results
Corrosion:	
Steel, mg/cm^2	-0.007
Silver, mg/cm^2	-0.069
Aluminum, mg/cm^2	-0.007
Magnesium, mg/cm^2	-6.774
Bronze, AMS4616, mg/cm^2	+0.059
Titanium, mg/cm^2	-0.006
M-50 Steel, mg/cm^2	+0.020
Appearance:	
Pitting	None
Etching	None
Corrosion	Mg
Staining	Bz

Oxidation:	16	24	40	48	64	72	88	96
Viscosity @ 100°F, Initial								12.1
% Change	+7.4	+9.9	+15.7	+19.0	+23.9	+37.2	+57.0	+63.6
Viscosity @ 210°F, Initial								3.1
% Change								+38.7
Total Acid Number, Initial								0.11
Change	1.19	1.79	3.87	4.27	8.69	14.03	20.53	22.01
Evaporation Loss, %	1.3							
Sludge, Volume, %	0.0							

MIL-L-7808 DEPOSITION TEST

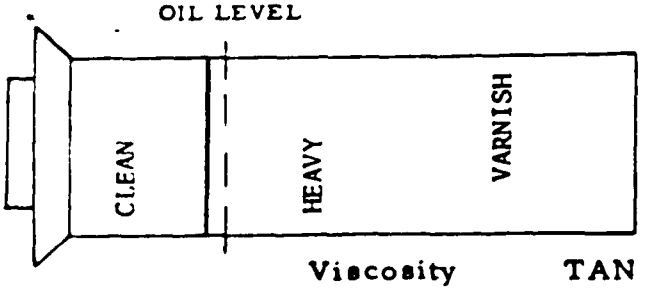
Alcor inc.
10130 JONES MALTSBERGER ROAD P O BOX 32516
SAN ANTONIO TEXAS 78284 - 512/349-3771

TEST LUBRICANT DESIGNATION Monsanto Research, Sample: 2000267 (0-79-7)

TEST NO. 4510 DATE 6/1/82 OPERATOR Revet, Trawick, Pavlicek

Coking Tube 590°F • Oil In 300°F • Oil Flow 300 cc/min • Air Flow 300 cc/min

HOURS	TIME	TEMPERATURES, °F					HEATER VOLTS	AIR ROTO-METER	OIL LEVEL	FILTER PRESSURE
		OIL IN 1	OIL OUT 3	COKING TUBE 2	COKING TUBE VAP SP.	CAB AIR 4				
START	[illegible]	[illegible]	445	[illegible]	540	[illegible]	[illegible]	[illegible]	[illegible]	1.0
	45	300	445	590	550	116	190	2.3	0	1.0
1	[illegible]	300	445	590	550	116	[illegible]	2.3	0	1.0
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
2	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	300	440	590	550	[illegible]	190	2.3	[illegible]	1.0
3	[illegible]	300	440	590	545	116	190	2.3	0	1.0
	[illegible]	300	445	590	550	116	190	2.3	0	1.0
4	[illegible]	300	440	590	545	116	190	2.3	0	1.0
	[illegible]	300	440	590	550	116	190	2.3	0	1.0
5	[illegible]	300	440	590	545	116	190	2.3	0	1.0
	[illegible]	300	440	590	540	116	190	2.3	0	1.0
6	[illegible]	[illegible]	440	590	540	116	190	2.3	0	1.0
	[illegible]	300	440	590	545	116	190	2.3	0	1.0
7	[illegible]	300	445	590	540	116	195	2.3	0	1.0
	[illegible]	300	460	590	560	116	195	2.3	0	1.0
8	[illegible]	[illegible]	460	590	560	116	195	2.3	0	1.0
	[illegible]	[illegible]	460	[illegible]	560	116	195	2.3	[illegible]	1.1
9	[illegible]	[illegible]	460	[illegible]	560	116	195	2.3	[illegible]	1.1
	[illegible]	[illegible]	460	[illegible]	560	116	195	2.3	[illegible]	1.1
10	[illegible]	[illegible]	460	[illegible]	560	116	195	2.3	[illegible]	1.1
	[illegible]	[illegible]	455	[illegible]	560	116	195	2.3	[illegible]	1.1
11	[illegible]	[illegible]	455	[illegible]	560	116	195	2.3	[illegible]	1.1
	[illegible]	[illegible]	460	[illegible]	565	116	195	2.3	[illegible]	1.1
12	[illegible]	[illegible]	460	[illegible]	565	116	195	2.3	[illegible]	1.1



Test Section D Tube No. 1

Oil Cons., cc 100 Cooler Sludge Light

Coking Tube, gm .030

Filter Sludge, gm .018

DEPOSIT RATING .32

	Viscosity	TAN
Before	12.1	.11
After	26.2	27.08
Change	14.1	26.97

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Alcor inCo

September 23, 1982

Mr. Richard Bruns
Monsanto Research Company
1515 Nicholas Road
Dayton, OH 45407

SAMPLE: 1997659 (0-79-9), 1997658 (0-79-11), 19976693 (0-79-13), 1997695 (0-79-6)

Richard, following are the test results obtained from your sample as requested with the 703 added per your letter of August 23, 1982.

	Results			
	1997659 (0-79-9)	1997658 (0-79-11)	1997693 (0-79-13)	1997695 0-79-6)
ACCELERATED STORAGE				
Lead Wt. Loss, mg/in^2	(95.5)*	(102.4)*	(3.0)*	(30.6)*
48 Hrs	198.2	212.6	6.2	63.5
168 Hrs	293.4	729.5	178.0	283.4
	(141.4)*	(351.5)*	(85.7)*	(136.6)*

Please let us know if we may be of further service or answer any questions you might have regarding these results. Best regards.

Sincerely,

Phillip O. Fruin

PHILLIP O. FRUIN
Manager, Testing Services

POF:b
Enclosures

*Corrected results

10130 JONES-MALTSBERGER ROAD ☐ P.O. BOX 32516 ☐ SAN ANTONIO, TEXAS 78284 ☐ 512/349-3771 ☐ 767470 ALCOR SNT

ALCOR Inc.
10130 Jones Maltsberger Road
San Antonio, Texas 78284
(512) 349-3771

CUSTOMER Monsanto Research Corporation DATE December 29, 1981

SAMPLE 2000227A, B, C, D, E, F

	Specification	Results					
		2000227 A	2000227 B	2000227 C	2000227 D	2000227 E	2000227 F
STORAGE STABILITY							
Lead Weight Loss, mg/in^2							
48 Hours	25 mg Max	(+ 2.1)* ~~+ 4.4~~	(21.7)* ~~45.0~~	(+8.3)* ~~+ 17.2~~	(+0.4)* ~~+ 0.8~~	(0.5)* ~~1.0~~	(3.0)* ~~6.3~~
168 Hours	150 mg Max	~~87.3~~ (42.1)*	~~220.8~~ (106.4)*	~~36.6~~ (17.6)*	~~104.7~~ (50.5)*	~~98.7~~ (47.6)*	~~99.7~~ (48.1)*

*Corrected results

APPENDIX F

SAMPLE CALCULATIONS
NaOH Addition
$Ba(OH)_2 \cdot H_2O$ Treatment

NaOH CALCULATION FOR DISTILLATION

Acid # of 1.3 is defined as milligrams of potassium hydroxide, that is required to neutralize all acidic constituents in 1 g sample or 0.0013 g KOH/g of oil.

$$\frac{0.0013\ g\ KOH}{g\text{-}oil} \times \frac{40.1\ g\text{-}mole\ NaOH}{56.1\ g\text{-}mole\ KOH} \times \frac{0.932\ g\text{-}oil}{mL} \times \frac{3{,}785.3\ mL}{gal} \times \frac{gal}{7.77\ \#\ oil} \times$$

N(number of lbs of used oil in batch) = 0.422 g of NaOH t dd to batch

$Ba(OH)_2 \cdot H_2O$ CALCULATION

$$\frac{0.0013\ g\ KOH}{g\text{-}oil} \times \frac{189\ g\text{-}mole\ Ba(OH)_2 \cdot H_2O}{56.1\ g\text{-}mole\ KOH} \times \frac{0.932\ g\ oil}{oil} \times \frac{3{,}785.3\ mL}{gal} \times \frac{gal}{7.77\ \#\ oil} \times$$

N(number of lbs of used oil in batch) = 1.989 g of $Ba(OH)_2 \cdot H_2O$ to add to batch
but the minimum amount is 0.77% by wt.

APPENDIX G

SELECTED LARGE SCALE DISTILLATION DATA

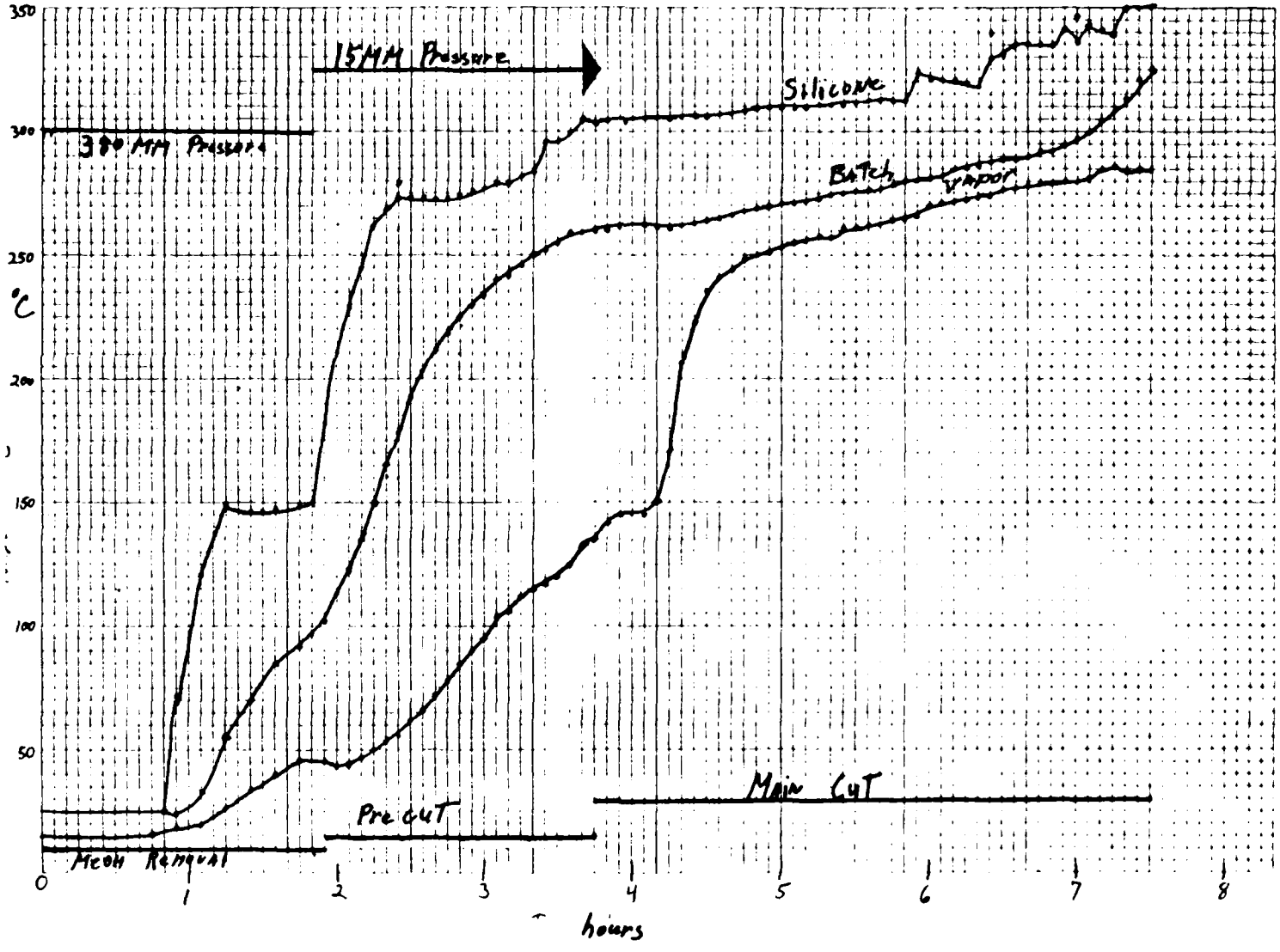
LARGE SCALE DISTILLATION DATA

Date distilled	NBP	Used oil	Comments
2/17/81	1833517	Virgin basestock	
2/24/81	1833520	0-79-01	
2/26/81	1833523	0-79-02	(No NaOH used in distillation)
3/02/81	1833526	0-79-02	
3/04/81	1833529	0-79-02	
3/06/81	1833534	0-79-03	
3/10/81	1833536	0-79-08	
3/12/81	1833543	0-79-07*	
3/17/81	1833546	0-79-11*	
3/19/81	1833548	0-79-12	
3/30/81	1833550	0-79-14	
4/01/81	1833552	0-79-09*	
4/03/81	1833555	0-79-04	
4/07/81	1833557	0-79-15	
4/09/81	1833560	0-79-06*	
6/18/81	1997632	0-79-13*	
6/15/81	1997633	0-79-10*	
6/23/81	1997634	0-79-15*	Redistilled from above
6/25/81	1997636	0-79-12*	Redistilled from above
6/30/81	1997641	0-79-08*	Redistilled from above
7/02/81	1997644	0-79-14*	Redistilled from above
7/22/82	2000294A	0-82-06	
8/02/82	2000295B	0-82-06	Redistilled from above
7/27/82	2000296	0-82-09	
8/04/82	2000298	0-82-07	No NaOH used in distillation
8/16/82	2276303	0-82-04	

*Distillate totally reclaimed and Mil-L-7808H tested.

K·E 10 X 10 TO THE INCH 46 0703

1833552 (0-79-9)



BATCH 183552 (1-79-9)
DATE 4/16/81
ACID NO. _____

DISTILLATION

INITIALS	DATE		
_____	_____	1.	Turn on vacuum jet.
_____	_____	2.	Close valve, bottom of 2F. Pull vacuum.
_____	_____	3.	Check to see if filter (50 micron) is in, inline filter on drum suction tube.
_____	_____	4.	Place drum on scale, suck into 2F 155 lbs of used oil (20 gals).

oil + container (initial)
- _____ oil + container (final)
oil weight

_____	_____	5.	Let oil sit over night in 2F with no agitation, open sample valves for vent.
_____	_____	6.	Vent vacuum lines.
_____	_____	7.	Turn off vacuum jet.
_____	_____	8.	Check batch temperature, should be aroun 20°C. Draw 1/2 gallon oil out of bottom of 2F, if no phase separation, return oil to 2F.
_____	_____	9.	Start agitator on 2F, make sure batch temperature is below 30°C before adding methanol, add gallon of alcoholic NaOH (see supervisor).
_____	_____	10.	Let NaOH/oil stir for 1/2 hour without heat.
_____	_____	11.	Turn on vacuum jet only on main receiver for vacuum check if greater than 10 mm consult supervisor.

BATCH _____

DATE _____

ACID NO. _____

DISTILLATION - (Cont'd)

INITIALS	DATE		
_____	_____	12.	Vent main receiver.
_____	_____	13.	Turn on cold water to condenser.
_____	_____	14.	Open distillate line to small receiver.
_____	_____	15.	Turn on N_2 flow on full, into 2F and adjust vacuum to 15 inches with vent on small receiver.
_____	_____	16.	After 1/2 hour hold, start silicone fluid through 2F jacket. Set heater to 150-160°C (300°F on dials).
_____	_____	17.	Turn on 2F shaft collar coolant pump, and water to its heat exchanger.
_____	_____	18.	Turn on silicone tracer on distillate line.
GSM RB	_____	19.	Maintain 15 inches vacuum until MeOH is removed (Batch temperature approximately 100°C).
GSM RB	_____	20.	Pull vacuum slowly until 27 inches, break vacuum by closing vacuum lines and letting N_2 fill to atmospheric pressure.
GSM RB	_____	20a.	Turn silicone heaters off, maintain silicone flow.
GSM RB	_____	21.	Drain MeOH from small receiver.
GSM RB	_____	22.	Pull vacuum on entire system plus 2 receivers @ 15 mm. Use N_2 bleed through needle valve on 2F to maintain 15 mm @ 5 gallon south receiver.
GSM RB	_____	23.	Turn heater on silicone heating fluid to maintain the fluid outlet at 280°C (525°F on dials).

BATCH _____

DATE _____

ACID NO. _____

DISTILLATION - (Cont'd)

INITIALS	DATE		
GSM	_____	24.	When vapor temperature @ 135°C, drain double valved distillate sample line and switch to main receiver. Maintain 13 1/2 - 15 mm vacuum in main receiver.
GSM	_____	25.	Turn off silicone tracer line on distillate line.
GSM	_____	26.	Once batch temperature is 240°C, start maintaining a difference of 40°C between the silicone and batch temperature by increasing the silicone oil temperature.
RB	_____	27.	Watch for increase in flow (distillation rate) shut off silicone to 2F if rate becomes too high.
RB	_____	28.	When batch temperature at 300°C, increase silicone oil temperature to 345-355°C.
RB	_____	29.	Distill to 325°C batch temperature and 15 mm vacuum in main receiver.
RB	_____	30.	Turn off vacuum to entire system, break vacuum in system with N_2 through needle valve on 2F. Turn off silicone heaters and pump.
RB	_____	31.	When pot pressure @ 0, open vent.
RB	_____	32.	Turn off agitator.
RB	_____	33.	Immediately drain (VERY HOT!) still bottoms into a N_2 blanket on 5 gallon can. The can, will be VERY HOT!! Approximately 700°F!
RB	_____	34.	Let 2F cool down for clean-up and recharging.

*Corrected results

BATCH _____
DATE _____
ACID NO. _____

DISTILLATION - (Cont'd)

INITIALS	DATE		
NS	_____	35.	Turn off cooling water to 2F condenser.
NS	_____	36.	Turn off N_2 flow into silicone vent line.
RS	_____	37.	Shut down vacuum jet, first open drain valve on 1st floor manifold, and then shut down.
_____	_____	38.	Drain south 5 gallon receiver and weigh, place liquid into solvent scrap drum, after removing 200 ml sample.
_____	_____	39.	Drain main receiver into previously cleaned 20 gallon drum and weigh. Mark drum. "Batch _____ Product Reclaim Oil".
_____	_____	40.	When batch temperature in 2F is at 200°C turn off 2F shaft collar coolant pump, and water to its heat exchanger.
_____	_____	41.	Turn off N_2 flow into 2F.
_____	_____	42.	Consult with supervisor for clean-up of 2F.

BATCH 1833-52 (C-74-9)

DATE 4/11/81

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. ~~Inches~~ mm 2F	Remarks
7:15	—	24	—			ATM	added Cond. Switch to TC#3 to HEAT ON
7:45	—	24	1	—	—	15 inch	VAC applied TC#3 & heat on next to 300°F
7:55	72	24	19			15"	
8:05	121	34	20			15"	
0815	149	55	27			15"	
0825	146	70	33			15"	
0835	147	85	40			15"	
0845	149	92	46			15"	HEAT OFF DRAINED PRODUCT RECEIVER
08:50	150	97	45	~~15mm~~	15 mm		HEAT ON
08:55	182	102	43	~~14mm~~	14 mm		
09:00	211	113	43		15		
09:05	229	123	45		15		
:10	245	135	47		15		
:15	262	150	50		15		
:20	268	165	53°		15 mm		
:25	273	179	56		15 mm		
:30	274	193	62		15		STARTED TO GET VAPORS & SMALL DRIPS
:35	273	203	66		15		
:40	273	212	72		15		
:45	273	219	78		16		
:50	274	225	85		15 mm		
:55	276	230	90		15 mm		
10:00	277	234	95		15.5		
:05	279	240	104		16mm		
:10	279	241	106		15		

BATCH _____

DATE _____

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
10:15	282°C	246°C	112°C		16 mm		
:20	284	250	115		14 mm		
:25	296	252	117		15 mm		
:30	296	255	120		14 mm		
:35	299	259	125		15.5 mm		
:40	305	259	132		15 5mm		
:45	303	260	135		16 mm		SWITCHED RECEIVERS
:50	304	260	141	15 mm			
:55	303	262	145	16			
11:00	~~305~~	~~263~~	~~145~~	~~17~~			
:05	305	263	145	17			
:10	305	262	150	16			
:15	305	261	171	15 mm			
:20	306	262	207	15mm			
:25	306	263	222	15			
:30	306	264	235	14½			
:35	307	265	241	15			
:40	307	267	244	15			
:45	306	268	248	15½			
:50	309	269	250	15			
:55	309	270	252	15½			
12:00	309	271	253	16			
12:05	309	271	255	16			
12:10	310	272	256	16			
	311	273	257	16.5			

BATCH _____

DATE _____

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
12.20	311°C	274°C	257°C	15 mm			
:25	311°C	275°C	261°C	14.5 mm			
:30	312°	276°	261°C	14 mm			
:35	313	276	262	14.5			
:40	313	277	262	14			
:45	313	278	264	14			
:50	313	280	265	14			
:55	324	280	266	14			
1300	321	282	270	14			
:05	320	283	271	14.5			
:10	320	285	271	14½			
:15	319	285	272	14.5			
:20	318	286	273	14.5			
:25	330	287	274	14			
:30	322	288	277	14.5			
135	325	288	277	14.5			
:40	325	290	278	14			
:45	325	293	279	14.5			
:50	325	293	279	14			
:55	331	295	280	14			heater set to 625°F
1400	336	297	280	14½			heat set to 650°F
14:05	343	300	282	14			
14:10	341	305	285	15			
14:15	339	308	286	14			
14:20	355	314	284	15			

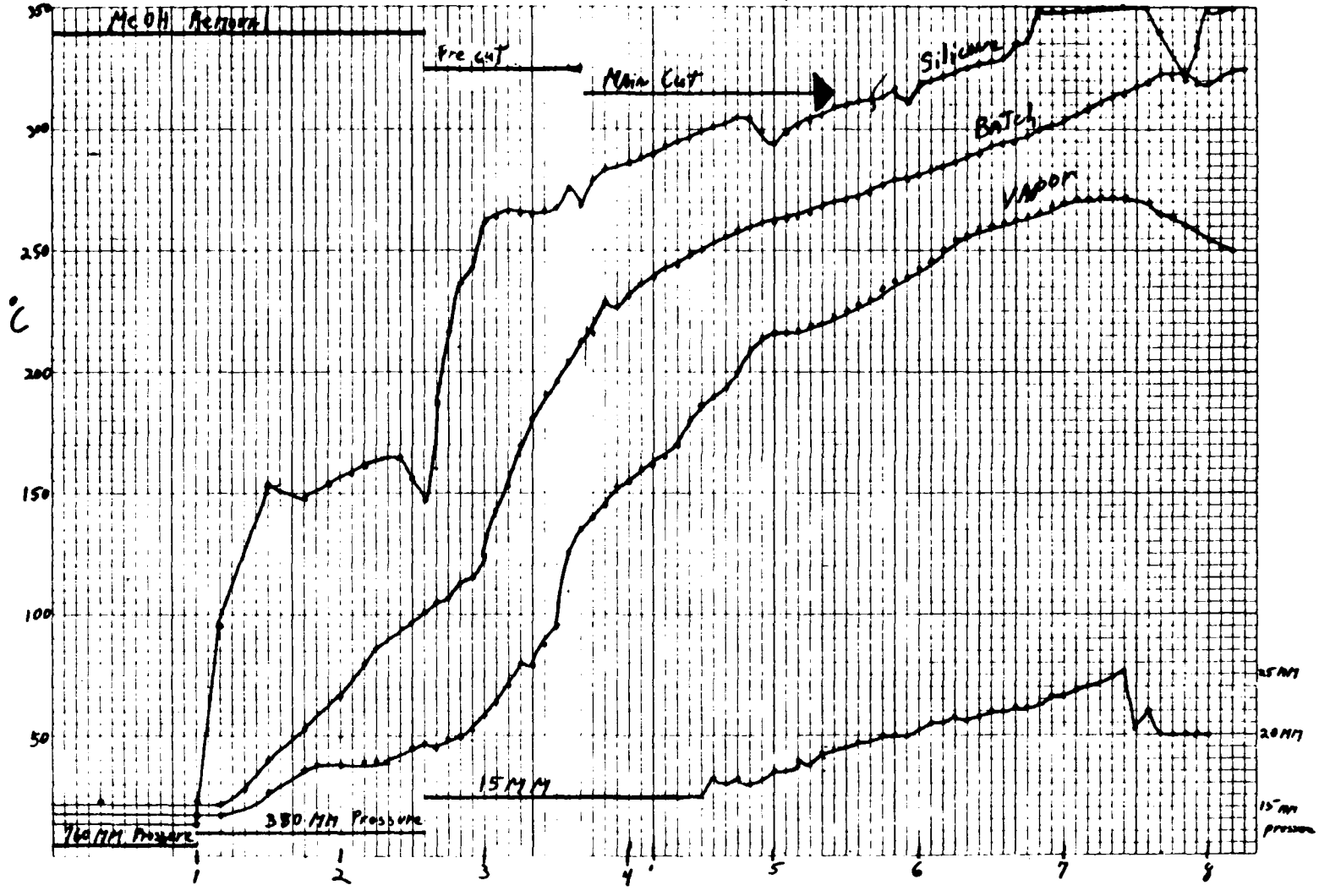
BATCH _____

DATE _____

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
2:25	357	324	285	15			
2:30		325			Finished		

1833555 (0-79-4)



BATCH 833555 (4-24-4)
DATE 5/3/8
ACID NO. _____

DISTILLATION

INITIALS	DATE		
_____	_____	1.	Turn on vacuum jet.
_____	_____	2.	Close valve, bottom of 2F. Pull vacuum.
_____	_____	3.	Check to see if filter (50 micron) is in, inline filter on drum suction tube.
_____	_____	4.	Place drum on scale, suck into 2F 155 lbs of used oil (20 gals).

oil + container (initial)
- _____ oil + container (final)
139 oil weight

_____	_____	5.	Let oil sit over night in 2F with no agitation, open sample valves for vent.
_____	_____	6.	Vent vacuum lines.
_____	_____	7.	Turn off vacuum jet.
_____	_____	8.	Check batch temperature, should be aroun 20°C. Draw 1/2 gallon oil out of bottom of 2F, if no phase separation, return oil to 2F.
_____	_____	9.	Start agitator on 2F, make sure batch temperature is below 30°C before adding methanol, add gallon of alcoholic NaOH (see supervisor).
_____	_____	10.	Let NaOH oil stir for 1/2 hour without heat.
_____	_____	11.	Turn on vacuum jet only on main receiver for vacuum check if greater than 10 mm consult supervisor.

BATCH _____

DATE _____

ACID NO. _____

DISTILLATION - (Cont'd)

INITIALS	DATE		
		12.	Vent main receiver.
		13.	Turn on cold water to condenser.
		14.	Open distillate line to small receiver.
		15.	Turn on N_2 flow on full, into 2F and adjust vacuum to 15 inches with vent on small receiver.
		16.	After 1/2 hour hold, start silicone fluid through 2F jacket. Set heater to 150-160°C (300°F on dials).
		17.	Turn on 2F shaft collar coolant pump, and water to its heat exchanger.
		18.	Turn on silicone tracer on distillate line.
		19.	Maintain 15 inches vacuum until MeOH is removed (Batch temperature approximately 100°C).
		20.	Pull vacuum slowly until 27 inches, break vacuum by closing vacuum lines and letting N_2 fill to atmospheric pressure.
		20a.	Turn silicone heaters off, maintain silicone flow.
		21.	Drain MeOH from small receiver.
		22.	Pull vacuum on entire system plus 2 receivers @ 15 mm. Use N_2 bleed through needle valve on 2F to maintain 15 mm @ 5 gallon south receiver.
		23.	Turn heater on silicone heating fluid to maintain the fluid outlet at 280°C (525°F on dials).

BATCH _____

DATE _____

ACID NO. _____

DISTILLATION - (Cont'd)

INITIALS	DATE		
CB	_____	24.	When vapor temperature @ 135°C, drain double valved distillate sample line and switch to main receiver. Maintain 13 1/2 - 15 mm vacuum in main receiver.
[illegible]	_____	25.	Turn off silicone tracer line on distillate line.
[illegible]	_____	26.	Once batch temperature is 240°C, start maintaining a difference of 40°C between the silicone and batch temperature by increasing the silicone oil temperature.
[illegible]	_____	27.	Watch for increase in flow (distillation rate) shut off silicone to 2F if rate becomes too high.
[illegible]	_____	28.	When batch temperature at 300°C, increase silicone oil temperature to 345-355°C.
[illegible]	_____	29.	Distill to 325°C batch temperature and 15 mm vacuum in main receiver.
[illegible]	_____	30.	Turn off vacuum to entire system, break vacuum in system with N_2 through needle valve on 2F. Turn off silicone heaters and pump.
CB	_____	31.	When pot pressure @ 0, open vent.
[illegible]	_____	32.	Turn off agitator.
[illegible]	_____	33.	Immediately drain (VERY HOT!) still bottoms into a N_2 blanket on 5 gallon can. The can, will be VERY HOT!! Approximately 700°F!
[illegible]	_____	34.	Let 2F cool down for clean-up and recharging.

BATCH _____

DATE _____

ACID NO. _____

DISTILLATION - (Cont'd)

INITIALS	DATE		
_____	_____	35.	Turn off cooling water to 2F condenser.
_____	_____	36.	Turn off N_2 flow into silicone vent line.
_____	_____	37.	Shut down vacuum jet, first open drain valve on 1st floor manifold, and then shut down.
_____	_____	38.	Drain south 5 gallon receiver and weigh, place liquid into solvent scrap drum, after removing 200 ml sample.
_____	_____	39.	Drain main receiver into previously cleaned 20 gallon drum and weigh. Mark drum. "Batch _____ Product Reclaim Oil".
_____	_____	40.	When batch temperature in 2F is at 200°C turn off 2F shaft collar coolant pump, and water to its heat exchanger.
_____	_____	41.	Turn off N_2 flow into 2F.
_____	_____	42.	Consult with supervisor for clean-up of 2F.

BATCH 1833555 (0-74-4)

DATE

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
7:20	—	23	—			ATM	Cooler/fresh added no heat on
8:00	13	23	16			15"	heat on set for 300°F
8:10	96	22	16			14½	MeOH coming off
8:20		28	27 (crossed out)				
8:30	153	40	27			14"	
8:45	148	53	36			14"	
9:00	154	67	38			18"	
9:10	159	80	39			17"	
9:15	162	87	40			17"	
9:20	165	97	45				Starting to draw 25" vac heat off
9:30	166	101	47			29"	MeOH still coming off
9:40	147	105	45		15MM		heat back on [illegible] to 325°F MeOH still coming off
9:45	187	107	48		14MM		
9:50	217	113	50		15MM		Low boilers or MeOH still coming off
9:55	236	120	53		14½MM		
10:00	243	129	58		15		
10:05	262	143	64		15		
10:10	269	153	71		15		
10:15	271	170	80		16		
10:20	270	181	79		15		
10:25	270	191	87		15		
10:30	271	197	95		15½		
10:35	272	204	120		17		[illegible] off
10:40	276	213	135		15½		[illegible] right now

BATCH _____

DATE _____

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
10:45	269	216	140	16			
10:50	274	229	145	15			
10:55	283	227	153	13.5			
11:00	286	232	155	15 0			
11:05	288	237	160	16 5			
11:10	290	239	162	18 ½			
11:15	294	243	170	13			Start glass cleaner as enough to see distillate - steam
11:20	295	244	175	14			
11:25	297	248	180	14½			
11:30	299	250	186	15½			
11:35	302	254	190	16½			
11:40	303	256	194	16			
11:45	305	258	200	16½			
11:50	305	260	209	16			
11:55	299	262	214	16¼			½ into 2F shut off
12:00	294	262	216	17			
12:05	298	263	216	17			
12:10	302	264	217	18			
12:15	304	266	219	17½			turned heaters to start
12:20	306	268	220	18½			
12:25	309	270	223	18 ¾			Turned heat to 57%
12:30	310	271	227	19			
12:35	312	273	228	19½			
12:40	313	274	230	19½			
12:45	314	277	235	20			

BATCH _____

DATE _____

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
12:50	317	279	237	20 MM			
12:55	311	280	239	20			Turned heaters to 600°F
13:00	318	282	243	20½			
13:05	320	284	246	21			
13:10	322	285	250	21			
13:15	324	287	254	21½			
13:20	325	288	256	21¼			
13:25	327	290	258	21½			
1:30	328	293	260	22			
1:35	329	294	261	22			
1:40	333	295	262	22¼			heater set to 650°F
1:45	337	297	264	22½			
1:50	350	300	266	22½			
1:55	348	302	268	23¼			
2:00	348	304	270	23¼			
2:05	348	306	272	23½			
2:10	349	308	272	24			
2:15	350	311	272	24¼			
2:20	350	313	272	24¾			
2:25	352	315	272	25¼			able to pump liquid out
2:30		318	-	15½			of line
2:35	352	319	270	17			heater off
2:40	340	323	265	15			closed line again
2:45		323	264	15			
2:50	322	321	260	15			closed line again

BAT. II _____

DATE _____

DATA SHEET

Time	Silicone Oil Temp. Outlet	Batch Temp.	Vapor Temp.	Vac. mm Main Receiver	Vac. mm Small Receiver	Vac. mm 2F	Remarks
2'55	334	317	258				Last Load in
3.00	350	319	254	15			
3.05	349	321	251				
3.10	350	324	250				
3.15		325		10	Finished		

APPENDIX H

PRODUCT SPECIFICATIONS FOR ADDITIVES AND ADSORBANTS

Dioctyldiphenylamine (DODPA)
Tricresyl Phosphate (TCP)
Quinizarin
Benzotriazole
Ethyl Antioxidant 703
Triphenyl Phosphite
Emery Base Stocks
Phenyl-α Naphthylamine (PANA)
Barium Hydroxide-Monohydrate

Product data sheets were obtained, where possible, for the additives and adsorbents used in the optimized reclamation process. The available data sheets are included here to identify the source and quality of the materials used. The following discussion identifies the source and quality of those materials for which a specification sheet was not available.

Quinizarin was obtained from GAF Corporation. It is identified only as purified 1,4-dihydroxyanthroqinone with no physical properties listed.

Tricresyl phosphate was obtained from FMC Corporation as Kronitex AA.

Triphenyl phosphite was obtained from Eastman Kodak Company. It has a boiling point of 360°C and a density at 25°C of 1.184.

Barium hydroxide monohydrate tech., obtained from Barium & Chemical, Inc., Steubenville, OH 43952.

The filter aid used was Hyflo Super Cel from Johns-Manville Corporation. This material is a Celite diatomite, mainly aluminum silicates, having a specific gravity of 2.30 and the following chemical analysis: SiO_2, 89.6%; Al_2O_3, 4.0%; Fe_2O_3, 1.5%; P_2O_5, 0.2%; TiO_2, 0.2%; CaO, 0.5%; MgO, 0.6%, Na_2O and K_2O, 3.3%.

R.T. Vanderbilt Company, Inc.

TECHNICAL DATA SHEET

PETROLEUM DEPARTMENT VL81-1

VANLUBE® 81

ASHLESS HIGH-TEMPERATURE ANTIOXIDANT and CORROSION INHIBITOR

Typical Properties

Composition:	p,p'-dioctyldiphenylamine
Physical State:	Powder
Color:	Off-white
Specific Gravity:	1.01
Melting Point:	95C
Distillation Range:	490-500F/0.25-0.75 mm Hg
Ash:	<0.01%
Heating Loss:	<0.50%
Solubility:	Soluble in silicones, silanes, siloxanes, diesters, petroleum oils. Insoluble in water.

VANLUBE 81 is a specially purified grade of p,p' dioctyldiphenylamine. It was developed commercially after extensive tests showed it to be an effective high-temperature oxidation and corrosion inhibitor in synthetic lubricants based on silane, siloxane, diester, and silicone fluids. In these fluids, VANLUBE 81 at concentrations of 0.5% to 2.0% is an effective oxidation and corrosion inhibitor at temperatures of 400-500F. It is also an effective high-temperature oxidation inhibitor in suitable petroleum base stocks.

In high-temperature lubricating greases, both petroleum and synthetic base, VANLUBE 81 shows good antioxidant properties in ASTM D-942 oxidation tests and in high-speed spindle tests. Siloxane greases containing 2% VANLUBE 81 have given outstanding results in bearing performance tests at 350F.

VANLUBE 81 has good solubility in a variety of synthetic and petroleum base lubricants and is color stable when exposed to light. It can be used as a general-purpose additive in a number of petroleum lubricants which require a stable, ashless, high-temperature oxidation inhibitor.

7410

R.T. Vanderbilt Company, Inc.

MATERIAL SAFETY DATA SHEET

SECTION I

CHEMICAL NAME AND SYNONYMS: p,p'-Dioctyldiphenylamine

EMERGENCY TELEPHONE NO: 203-853-1400

CHEMICAL FAMILY: Organic secondary amine

FORMULA: $C_{28}H_{43}N$

TRADE NAME AND SYNONYMS: VANLUBE 81

SECTION II HAZARDOUS INGREDIENTS

MATERIAL	%	TLV UNITS
p,p'-Dioctyldiphenylamine		Acute oral LD_{50}
		>5,000 mg/kg rats

SECTION III PHYSICAL DATA

BOILING POINT °F	SPECIFIC GRAVITY (H_2O=1)
VAPOR PRESSURE mm Hg	PERCENT VOLATILE BY VOLUME %
VAPOR DENSITY (AIR=1)	EVAPORATION RATE (=1)
SOLUBILITY IN WATER: negligible	Density Mg/m^3: 1.01

APPEARANCE AND ODOR: White powder

SECTION IV FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (METHOD USED)

FLAMMABLE LIMITS LEL UEL

EXTINGUISHING MEDIA: CO_2, foam, dry chemical

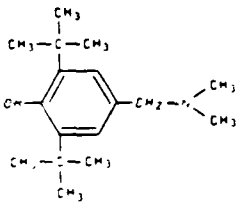
SPECIAL FIRE FIGHTING PROCEDURES

UNUSUAL FIRE AND EXPLOSION HAZARDS

703

"ETHYL" ANTIOXIDANT 703

2, 6-Di-tert butyl-α-dimethylamino-p-cresol



TYPE

[illegible] phenolic antioxidant.

TYPICAL PROPERTIES

Form	Crystalline powder	Melting point	94°C (201°F)
Color	Light yellow	Boiling point	179°C (354°F) at 40 mm
Molecular weight	263.4	Flash point (COC)	>200°F

APPLICATIONS

Oxidation inhibitor in natural and synthetic rubbers, polyolefin plastics, resins, adhesives, petroleum oils and waxes.

SOLUBILITY (Wt. %) at 20°C.

Toluene	22	Water	< 0.0007
Ethyl Alcohol	28	10% NaOH	< 0.002

TOXICITY

The acute oral L.D. for "Ethyl" Antioxidant 703 is 1030 mg/kg of body weight.

ETHYL CORPORATION
INDUSTRIAL CHEMICALS DIVISION

Ordering and Shipping Information

Refer to product as:

"ETHYL" Antioxidant 703

Container sizes:

100 lb net 24 gal non-returnable drum
25 lb net 8 gal non-returnable drum
5 lb net 2 gal non-returnable drum

Weight Information

Drum Size:	100 lb	25 lb	5 lb
Net contents, gal (nominal)	24	8	2
Net contents, lb	100	25	5
Tare, lb approx	9	5	1.5
Dimensions, diameter, in	16	11.5	8.5
Dimensions, height, in	30.75	21.25	9.75
Volume, cu ft	4.59	1.63	0.42

Rail Shipments:

Minimum carload lots 325 drums, 24 gallon size

Truck Shipments:

Minimum truckload lots 271 drums, 24 gallon size

Mail or wire orders:

Ethyl Corporation
Industrial Chemicals Division
Baton Rouge, La. 70821

Telephone orders:

Baton Rouge, La. 504-344-5222

Shipping point:

Orangeburg, South Carolina

IN CANADA

Orders should be placed with Ethyl Corporation Limited, 48 St. Clair Avenue West, Toronto

[illegible]

ETHYL CORPORATION
INDUSTRIAL CHEMICALS DIVISION
ETHYL TOWER 451 FLORIDA
BATON ROUGE LA 70801

CHICAGO • CLEVELAND • HOUSTON • LOS ANGELES • NEW YORK • TULSA

BENZOTRIAZOLE - Photo Grade

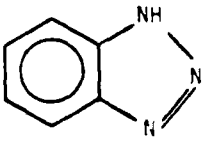
Code: BT-PG Order Entry No.: X19 BT 5589

Synonym: 1,2,3-Benzotriazole

sherwin williams chemicals

a division of THE SHERWIN WILLIAMS CO

technical bulletin 142



$C_6H_5N_3$

M.W. 119.12

PROPERTIES

Appearance — White crystalline needles.

	Specifications*	Typical Analysis
Assay	98.0% min	99.5
Appearance of 1% Solution	Essentially clear	Essentially clear
Residue After Ignition	0.5% max.	0.08
Volatile Matter at 70°C	0.5% max.	0.08
Identity Melting Point	98-99°C	98-99°C

*Meets requirements of American National Standard Institute specification PH4.204-1972. Methods of analysis are given in the specification.

USES

Photographic chemical--developer, anti-fogging agent and restrainer in gelatin emulsions. Also in other areas where light sensitive substances are useful. (See References).

TOXICITY

The oral LD_{50} in white rats is 560 mg per kg. Chemicals with values of this order are generally considered moderately toxic. Tests made on intact and abraded skin of rabbits showed no skin irritation.

SAFE HANDLING

Benzotriazole and its solutions are not serious industrial hazards provided that workers are properly instructed and adequately supervised in handling procedures. Where adequate ventilation is not available, approved respiratory and eye protection are required in dust laden areas.

AVAILABILITY

Readily available from stock. Package sizes, prices and other details of sale are stated in our latest Chemicals Price List.

REFERENCES

Battaglia, Photogr. Sci. Eng. 1970, 14 (4), 275; CA 73, 50708.

Brit. 1,173,426 (1969); CA 73, 40477.

Fr. 1,542,505 (1968); CA 71, 66055.

Ger. Offen. 2,028,214 (1971); (To Western Electric); CA 74, 118423.

Sahyun, Photogr. Sci. Eng. 1970, 14(3), 192; CA 73, 20423. Ibid, 1971 15(1), 48; CA 74, 48046.

Sheberstov and Borokova, Zh. Nauch. Prikl. Fotogr. Kinematogr., 1969, 14(4), 292; CA 71, 96868.

U.S. 3,547,635 (1970) (To Eastman Kodak); CA 74, 149194.

Zyuskin and Braichevskaya, Zh. Nauch. Prikl. Fotogr. Kinematogr. 1968, 13(5), 326; CA 70, 16012.

Zyuskin and Glinskaya, Zh. Nauch. Prikl. Fotogr. Kinematogr. 1969, 14(6), 470 (Russ); CA 72, 61365.

sherwin williams chemicals

P.O.Box 6520, Cleveland, O. 44101

TABLE I

SPECIFICATIONS OF EMERY LUBRICANT BASES

Specification	Emery 2910	Emery 2911	Emery 2957	Emery 2958	Emery 2960	Emery 2934	Emery 2932
Acid Value, Max.	0.2	0.2	0.2	0.2	0.02	0.08	0.10
Hydroxyl Value, Max.	3.0	2.0	2.0	2.0	3.0	3.0	3.0
Iodine Value, Max	- -	2.0	2.0	2.0	1.0	- -	- -
Flash Point, °F, Min.	400	300	400	400	430	480	460
Fire Point, °F, Min.		350	450	450	- -	- -	- -
Cloud Point, °F, Max.	-40	-40	-40	-40	-40	- -	-40
Pour Point, °F, Max.	-70	-90	-50	-75	-75	-65	-75
Viscosity, Cs.							
210°F, Min.	2.60	1.60	3.20	2.85	4.3	4.65-4.85	4.0
100°F, Max.	- -	- -	- -	- -	- -	22.0-24.0	- -
-40°F, Max.	- -	- -	1800	- -	- -	- -	5000
-65°F, Max.	6500	800	- -	7000	- -	- -	- -
*SOD Lead Corrosion, Mg/sq. in.							
1 Hour, Max.	±4	- -	±4	±4	±4	No negative** values	No negative** values
*Oxidation Stability, 347°F Hours, Min.	100	- -	100	100	- -	- -	- -

*Contains 0.5% purified phenothiazine
0.1% Ethyl anti-oxidant 703

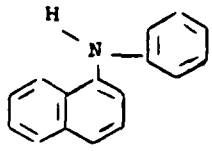
**Contains 0.5% purified phenothiazine

UNIROYAL CHEMICAL
Division of UNIROYAL Inc
Naugatuck Connecticut 06770

Naugatuck® Chemicals

PRELIMINARY DATA SHEET

NAUGARD® PANA for Synthetic



Phenyl-Alpha-Naphthylamine

Naugard PANA is an aromatic amine antioxidant for synthetic lubricants, lubricating greases, industrial oils, and railroad diesel engine oils. It offers excellent antioxidant activity at an economical cost. Naugard PANA is particularly useful in high temperature applications. It provides effective protection in synthetic lubricants at temperatures that at times reach 600-700°F (315-371°C)

Naugard PANA is also an important antioxidant in soap thickened greases as well as those made with non-soap thickeners. In addition to its many applications in petroleum, Naugard PANA is used as a rubber antioxidant. It is the primary antioxidant for neoprene rubber. Recommended concentrations for this product vary from 0.2-1.0% depending on the application.

Typical Properties

Chemical Name	N-phenyl-1-naphthylamine
Physical Form	Tan to purple crushed solid
Specific Gravity @ 25°C	1.23
Melting Point	131°F (55°C)
Ash	0.1%
Flash Point (Penske Marten)	417°F (214°C)
Solubility	Soluble in acetone, alcohol, benzene, carbon tetrachloride and chloroform. Insoluble in water.

Storage Stability

Naugard PANA has excellent storage stability. Long exposure to air and light will cause the product to darken but will not affect antioxidant activity.

Toxicity and Handling Precautions

PANA has been used for many years without any known serious health problems. As it has a reported oral LD_{50} (rats) of 1625 mg/kg it should be treated as a "toxic" substance. Human experience has indicated some skin irritation potential.

Trace amounts of alpha-naphthylamine (400-800 ppm) and beta-naphyl amine (0-80 ppm) have been detected in Naugard PANA. These substances have been designated as carcinogens by OSHA and when present at minimum levels of 10,000 ppm for alpha-naphthylamine and 1,000 ppm for beta-naphthylamine are subject to OSHA Regulations Numbers 1910.1004 and 1910.1009.

In the handling of PANA we advise taking appropriate precautions to avoid ingestion, contact with the skin, and breathing of dust or vapors.

UNIROYAL CHEMICAL
Division of UNIROYAL, Inc.
Naugatuck, Connecticut 06770

Naugatuck® Chemicals

TENTATIVE SALES SPECIFICATION

NAUGARD PANA

(Phenyl Alpha Naphthylamine)

FOR Petroleum & Synthetic Turbine Oils

TEST	MINIMUM	MAXIMUM	TEST METHOD
Assay, %	99	-	GLC-31QC
Ash, %	-	0.05	G-68-G
Toluene Ins., %	-	0.03	G-68-G
Moisture, %	-	0.1	G-24
Appearance	Lt. tan to purple crushed solid or solid cast in drums.		

JAS/GJH:lmh
Revised 7-28-77

UNIROYAL

APPENDIX I

PROCESS HISTORY FOR EACH USE OIL LOT

0-79-1 - After distillation treated with $Ca(OH)_2$, oil developed a foam test volume over 300^+ mL. Terminated further work up.

0-79-2 - Distilled one sample without NaOH as a control, had a fairly high foam test volume.

Distilled a 2nd sample with NaOH which had a very high foam test volume. We redistilled the sample and the sample still had a high foam test volume, so terminated further work up. A expanded GC of the oil does not indicate similarity to 0-82- used oils which also had foaming problems after distillation.

0-79-3 - After distillation, treated with $Ca(OH)_2$, oil developed a high test volume. Terminated further processing.

0-79-4 - Oil had left thick charred residue in still. Treat oil with $Ca(OH)_2$, oil developed a high test volume. Terminated further processing.

0-79-5 - Oil had a high acid number, assumed that a residue similiar to 0-79-4 would develop upon distillation, so did not attempt distillation.

0-79-6 - The distillate was treated with $Ba(OH)_2 \cdot H_2O$ overnight at 43°C, then treated with attapulgus clay. The sample was reformulated and sent for MIL-L-7808H testing.

0-79-7 - The distillate was treated with $Ba(OH)_2 \cdot H_2O$ overnight at 43°C (treatment temperature was to be 50°C). Precipitate (ppt) developed in oil upon sitting for a month, refiltered. Nine months after filtering, additional ppt in oil. Refiltered the oil, then heat treated to 60°C overnight and then reformulated. The sample was then MIL-L-7808H tested.

0-79-8 - The distillate was treated with $Ca(OH)_2$, oil developed a high foam test volume. Redistilled the distillate and treated with $Ba(OH)_2 \cdot H_2O$ @ 50°C overnight. The oil was then reformulated and sent out for MIL-L-7808H testing.

0-79-9 - The distillate was treated with $Ba(OH)_2 \cdot H_2O$ overnight at 43°C. The sample was formulated and sent for MIL-L-7808H testing.

0-79-10 - The distillate was treated with $Ba(OH)_2 \cdot H_2O$ overnight at 45°C. The sample contained a small amount of ppt, which was filtered out by final filtration after reformulation. Sample sent out for MIL-L-7808H testing.

0-79-11 - The distillate was treated with $Ba(OH)_2 \cdot H_2O$ overnight at 45°C. The sample was reformulated and sent for MIL-L-7808H testing.

0-79-12 - The distillate was treated with $Ca(OH)_2$, oil developed a high foam test volume. Redistilled the distillate and treated with $Ba(OH)_2 \cdot H_2O$ @ 45-50°C overnight. The oil was then reformulated and sent out for MIL-L-7808H testing. The sample had previously contained small amount of ppt, which was filtered out by final filtration after reformulation.

0-79-13 - The distillate was treated with $Ba(OH)_2$ overnight at 45°C, then treated with attapulgus clay. The sample was reformulated and sent for MIL-L-7808H testing.

0-79-14 - The distillate was treated with $Ca(OH)_2$, oil developed a high foam test volume. Redistilled the distillate and treated with $Ba(OH)_2 \cdot H_2O$ @ 42°C (treatment temperature was to be 50°C). Precipitate had developed in oil upon sitting for a month, refiltered. Nine months after the refiltering, additional ppt in oil. Refiltered the oil, then heat treated to 60°C overnight and then reformulated. The sample was then MIL-L-7808H tested.

0-79-15 - The distillate was treated with $Ca(OH)_2$, oil developed a high foam volume. Redistilled the distillate and treated with $Ba(OH)_2 \cdot H_2O$ overnight at 50°C. The sample was reformulated and sent for MIL-L-7808H testing.

0-82-4 - The distillate failed the foam test, further processing terminated.

0-82-5 - The oil had a strong kerosene small, stronger then 0-82-4, did not process this sample.

0-82-6 - Dis llate had a high foam test volume, redistillation of the disti..ate did not improve the test results.

0-82-7 - Distillate failed the foam test, further processing terminated.

0-82-8 - The oil had a strong kerosene small, stronger then 0-82-4, did not process this sample.

0-82-9 - Distillate failed the foam test, further processing terminated.

0-82-10 - Lab distillation for foam testing of distillate. Did pass foam test, but the foam volume was higher than would like for further processing.

0-82-11 - Lab distillation for foam testing of distillate, did not pass foam test.

0-82-12 - Lab distillation for foam testing of distillate, did not pass foam test.

TABLE 1-1. DISTILLATION DATA FOR EACH RECEIVED USED OIL

NB	Used oil	Still toppings/ pre-cut, %	Main cut, % (recovery)	Still bottoms, %	Loss[1], %	Starting acid #	Final acid #	Main cut[a] collections Vl to Bl	NaOH used in distillation	Distillate foam test ml	Density	Distillation time hours
1833520	0-79-01	1.0	90.2	9.0	-	0.48	1.17	135-325	Yes[e]	20	0.93	4.75
1833523	0-79-02	1.3	89.5	7.3	1.9	0.45	1.59	135-325	No	75	0.93	4.50
1833526	0-79-02	1.8	90.3	6.4	1.5	0.45	0.83	135-325	Yes	300[f]	0.93	5.25
1833529	0-79-02[d]	3.0	91.5	5.5	-	0.83	0.37	135-325	Yes	245	0.93	8.5
1833534	0-79-03	6.6	78.8	10.0	-	0.63	1.16	135-325	Yes	75	0.94	9.75
1833555	0-79-04[b]	5.0	65.5	23.4	6.1	15.52	6.29	135-325	Yes	80	0.93	8.0
-	0-79-05[b]	-	-	-	-	23.8	-	-	-	-	0.93	-
1833560	0-79-06[f]	5.1	82.5	11.5	0.9	3.14	1.03	135-325	Yes	10	0.92	7.5
1833543	0-79-07[f]	2.6	82.0	11.3	4.1	3.69	0.85	135-325	Yes	10	0.92	7.5
1833536	0-79-08[d,f]	2.4	83.3	10.8	3.5	2.17	0.78	135-325	Yes	20	0.92	8.0
1997641	0-79-08[f]	1.9	92.5	5.0	0.6	0.00	0.52	135-325	Yes	10	0.92	5.5
1833552	0-79-09[f]	1.6	91.0	3.5	4.1	0.93	0.70	135-325	Yes	5	0.93	7.5
1997633	0-79-10[f]	1.3	88.5	5.1	5.1	1.30	1.36	135-325	Yes	10	0.92	8.5
1833546	0-79-11[f]	2.0	90.0	6.8	1.2	1.36	0.73	135-325	Yes	10	0.93	8.0
1833548	0-79-12[d,f]	1.6	88.5	5.8	4.1	0.42	1.01	135-325	Yes	15	0.92	8.0
1997636	0-79-12[f]	1.6	96.1	2.6	-	0.22	0.50	135-325	Yes	15	0.92	7.0
1997632	0-79-13[f]	1.9	88.5	7.1	2.5	0.74	0.92	135-325	Yes[g]	10	0.92	5.75
1833550	0-79-14[d,f]	4.5	82.9	8.7	3.9	2.51	0.89	135-325	Yes	15	0.92	7.0
1997644	0-79-14[f]	3.8	89.2	4.4	2.6	0.00	0.44	135-325	Yes	15	0.92	6.75
1833557	0-79-15[d,f]	2.0	84.5	7.7	5.8	3.0	0.75	135-325	Yes	10	0.92	8.0
1997634	0-79-15[f]	2.6	93.0	2.6	1.8	0.0	0.38	135-325	Yes	10	0.92	6.75
2276303	0-82-04[b]	12.0	61.0	32.0	-	0.21	1.68	200-325[h]	Yes	150	0.90	9.5
--	0-82-05[b]	-	-	-	-	0.21	-	-	-	-	0.91	-
2000294A	0-82-06	5.0	53.0	36.0	6.0	0.17	2.3	135-325[h]	Yes	300[f]	0.94	9.0
2000295B	0-82-06	13.0	68.0	19.0	-	2.3	0.91	200-325[h]	Yes	300[f]	0.94	9.0
2000296	0-82-07[b]	16.0	54.0	27.0	3.0	-	1.8	200-325[h]	No	300[f]	0.94	9.0
--	0-82-08[b]	-	-	-	-	0.18	-	-	-	-	0.92	-
2000296	0-82-09[c]	22.0	80	13.5	-	0.08	1.35	200-325[h]	Yes	300[f]	0.92	8.5
2276319	0-82-10[c]	15.0	74.0	5.0	6.0	-	-	200-325	No	75	-	1.0
2276318	0-82-11[c]	5.3	86.5	5	3.2	-	-	200-325	No	175	-	2.0
2276320	0-82-12[c]	39.0	32.0	26.0	3.0	-		200-325	No	300[f]	-	2.0

[a]Main cut consists of distillates collected from vapor temperature °C to batch temperature °C.

[b]Not processed

[c]Distilled only in lab

[d]Redistilled distillate from above.

[e]Insufficient NaOH used

[f]Sample was [illegible] processed for BTU & ASOH testing

[g]Excess NaOH

[h]Run from boiled batch temperature of 200°C

[i]Acid not considered into original oil

APPENDIX J

IR, GC, AND HPLC CHROMATOGRAMS OF USED OIL LOTS RECEIVED LATER IN PROGRAM TO DEVELOP ADDITIONAL SCREENING METHODS

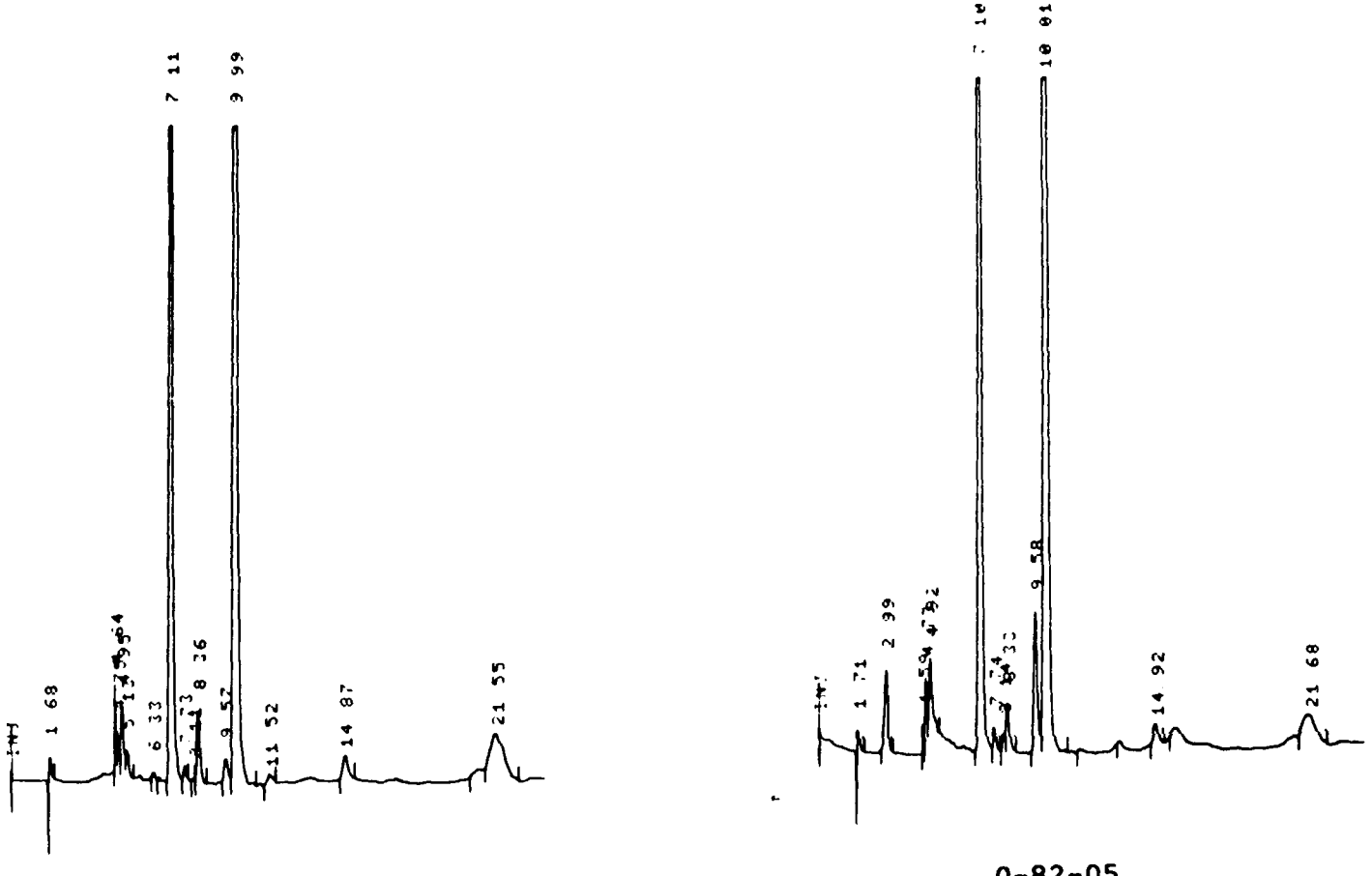
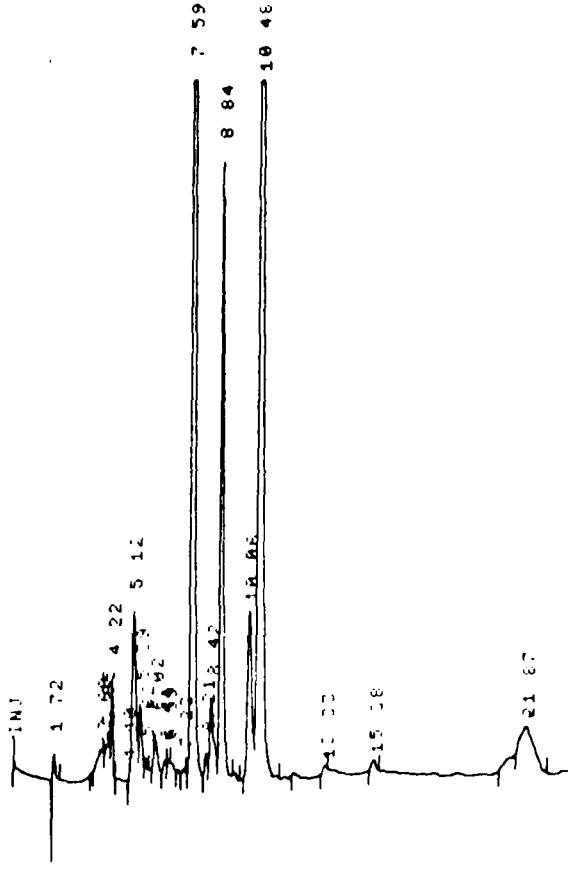


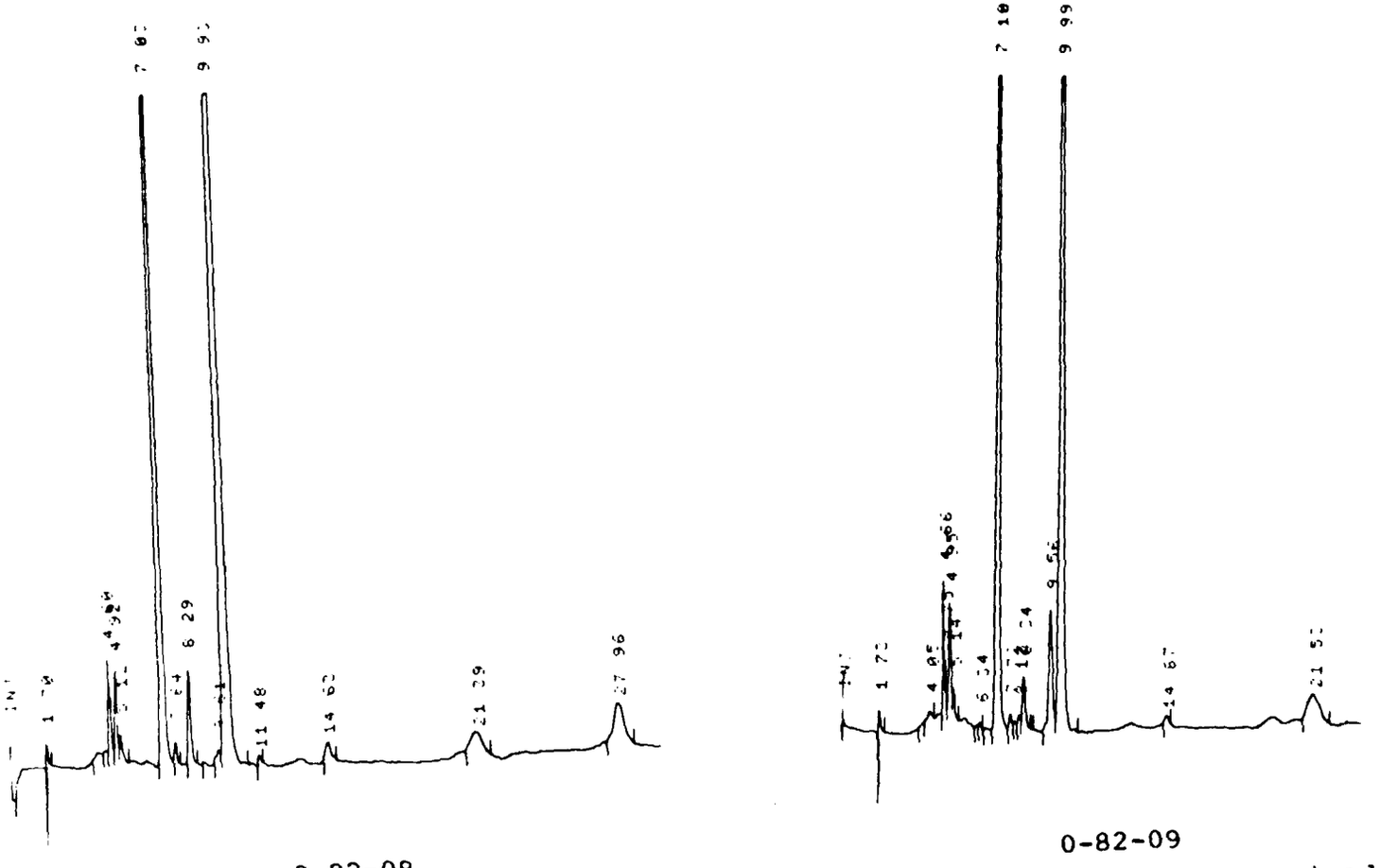
Figure J-1. High performance liquid chromatograms of used oil as received.

0-82-06



0-82-07

Figure J-2. High performance liquid chromatograms of used oil as received.



0-82-09

0-82-08

Figure J-3. High performance liquid chromatograms of used oil as received.

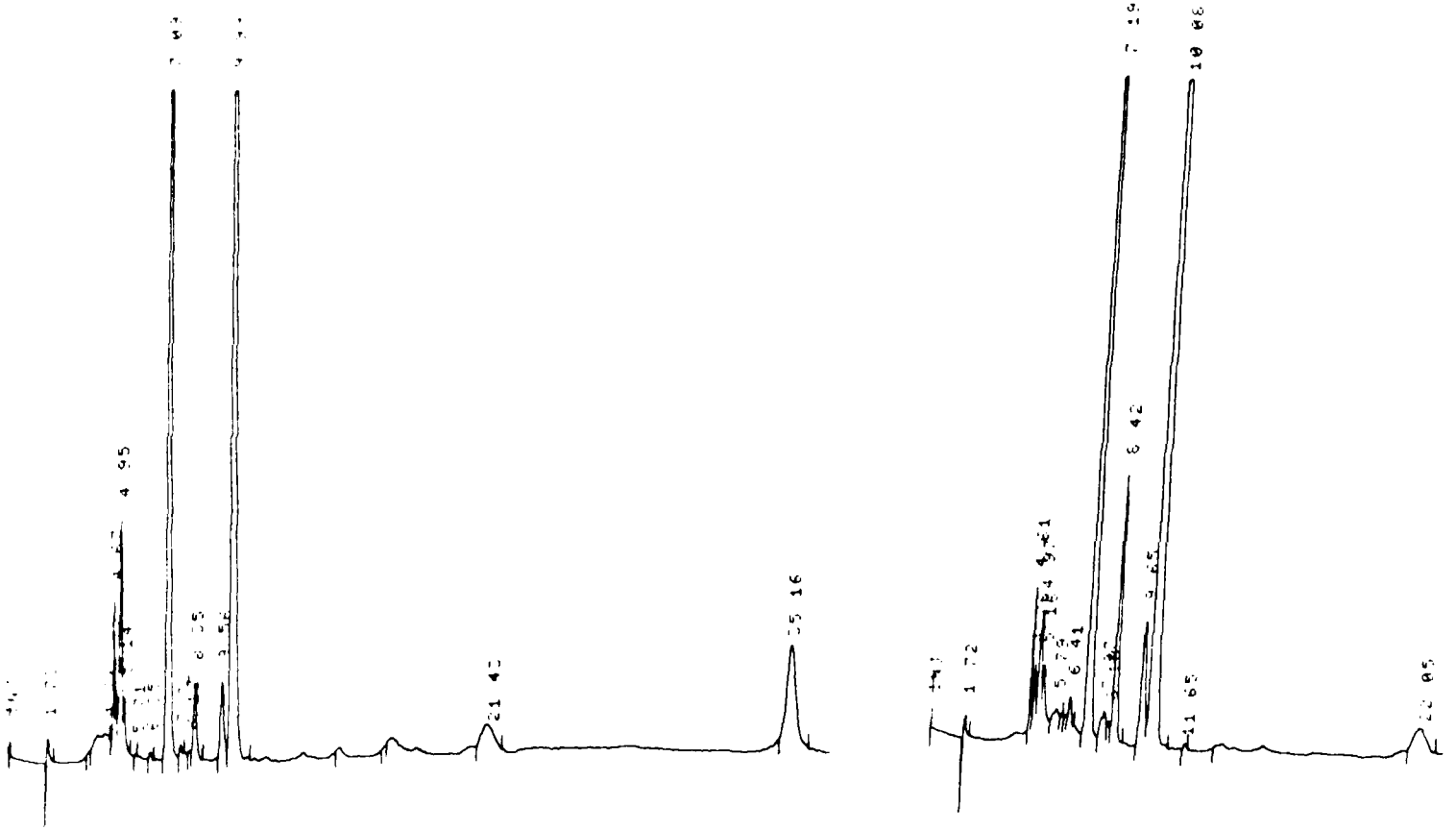
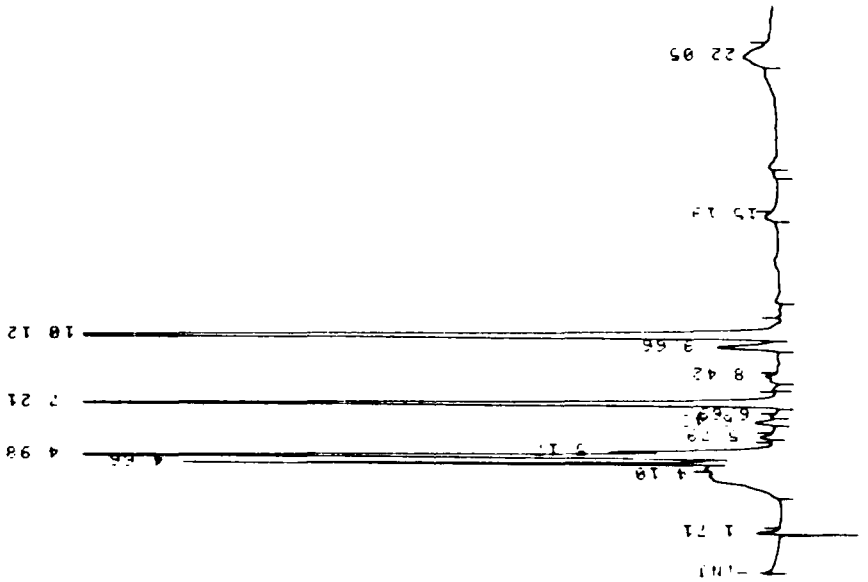


Figure J-4. High performance liquid chromatograms of used oil as received.



0-82-12

Figure J-5. High performance liquid chromatograms of used oil as received.

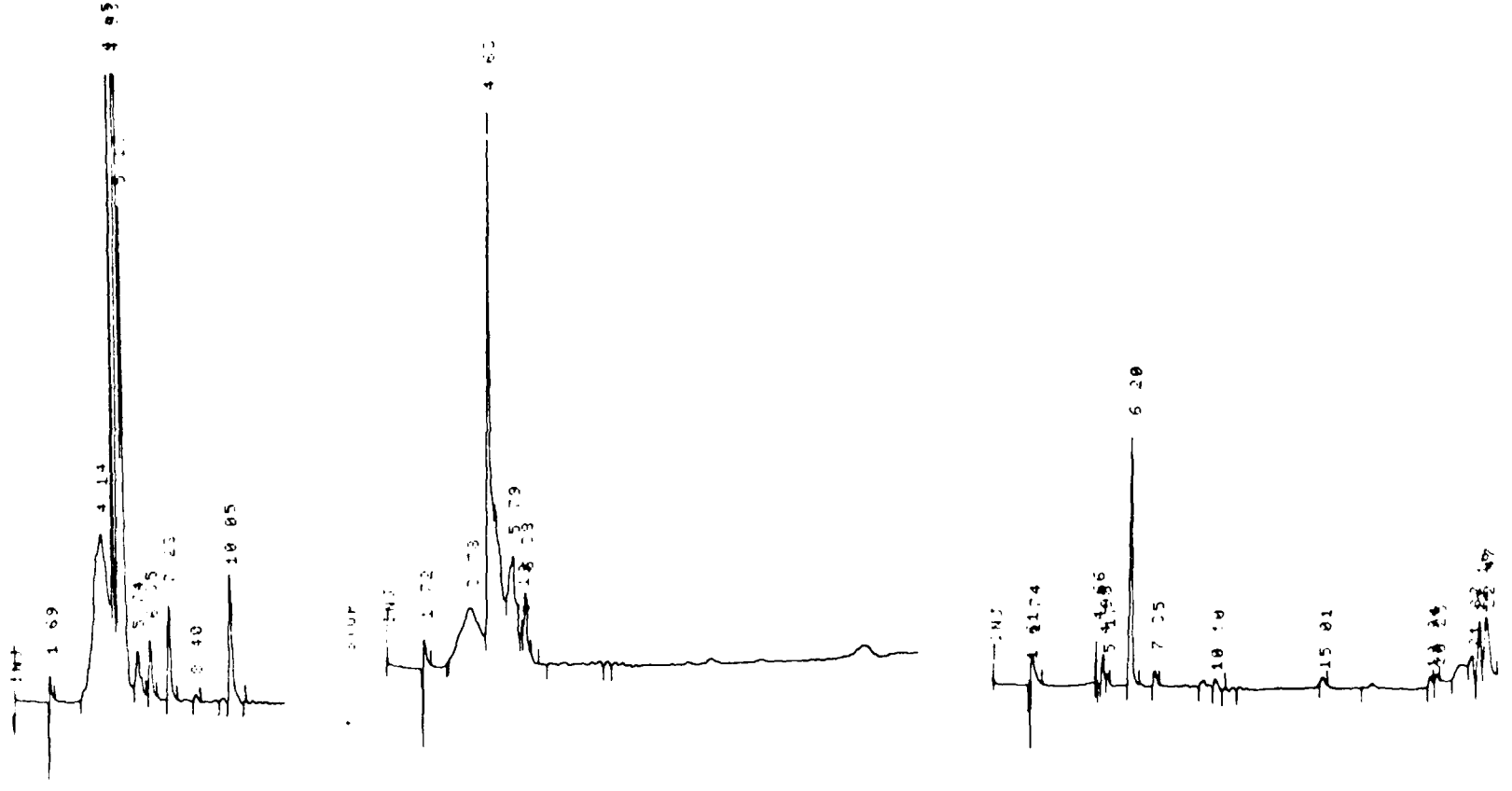
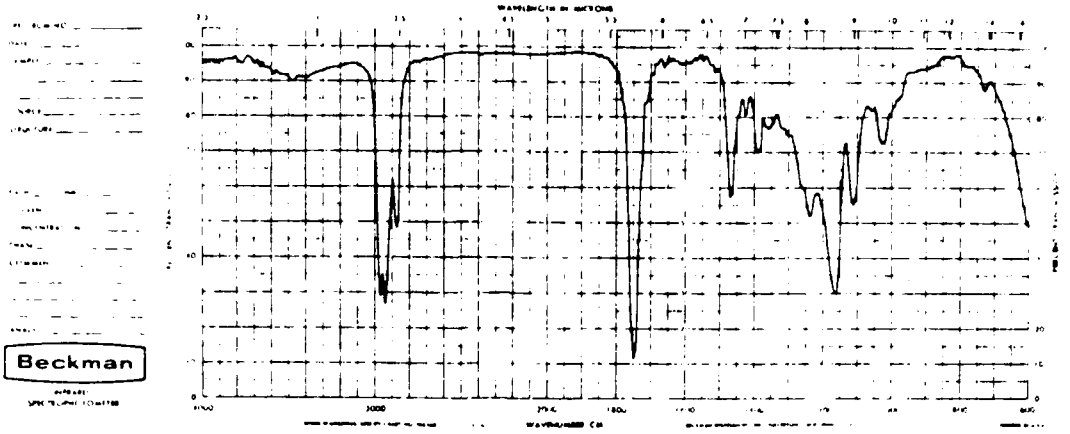


Figure J-6. HPLC of JP4 and 2 hydraulic fluids.



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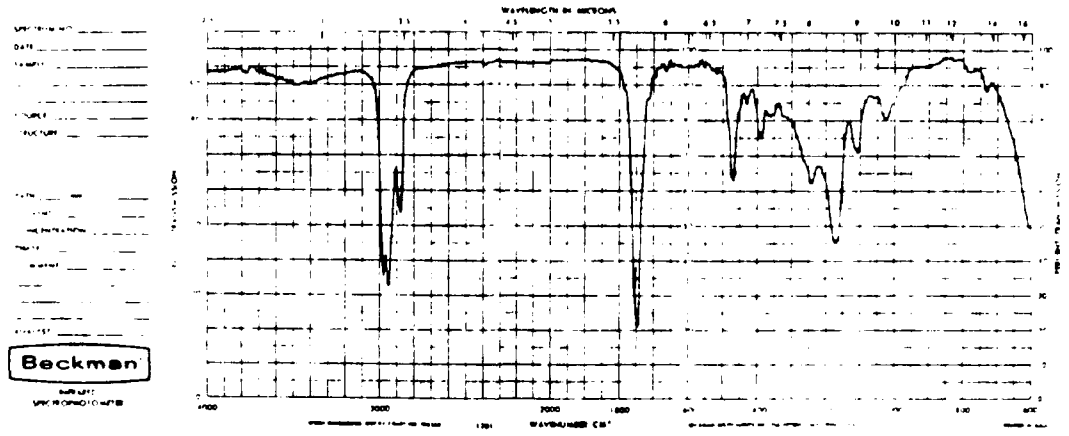
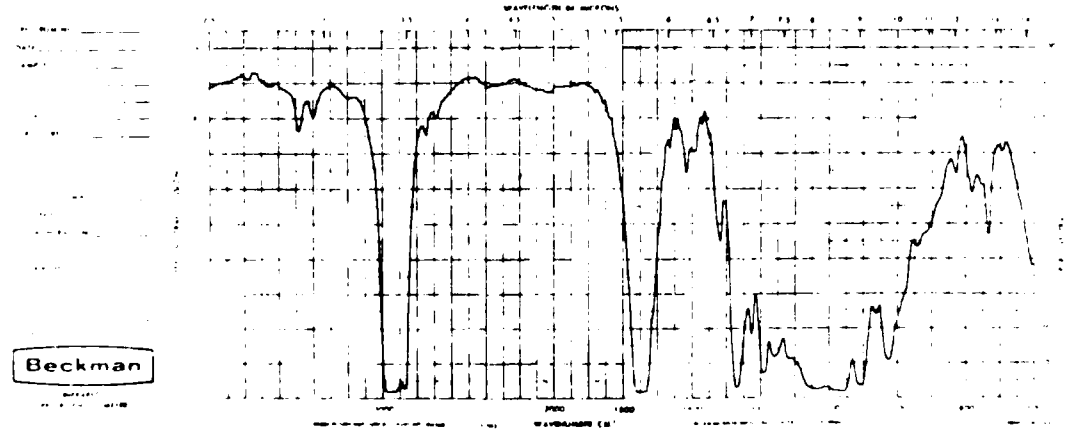
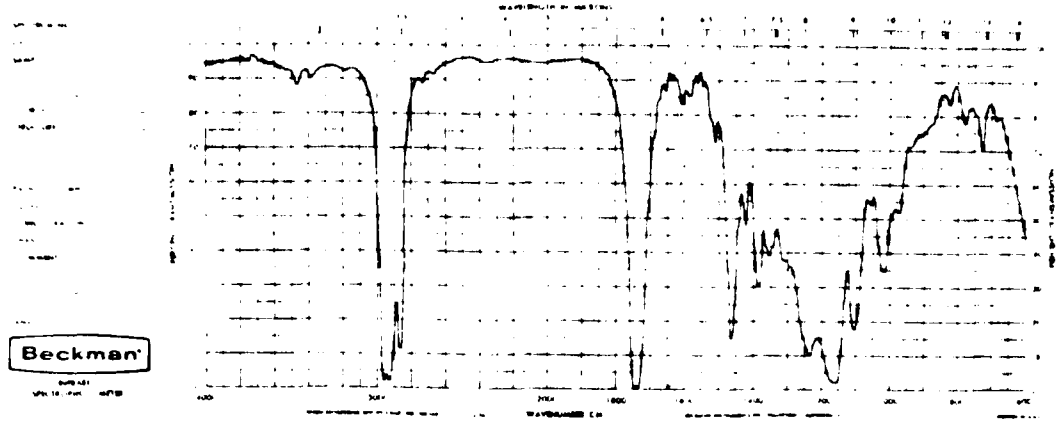


0-82-05



0-82-06

Figure J-7. Infrared spectrum of used oils.



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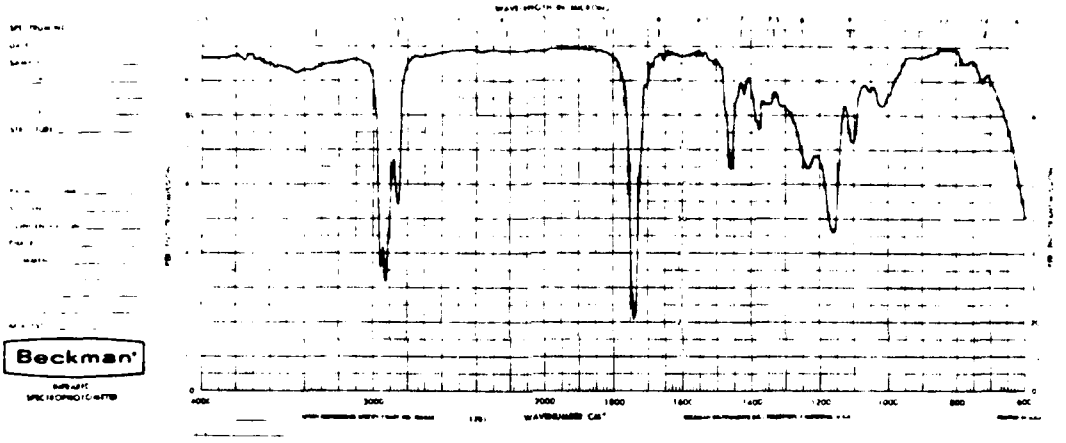

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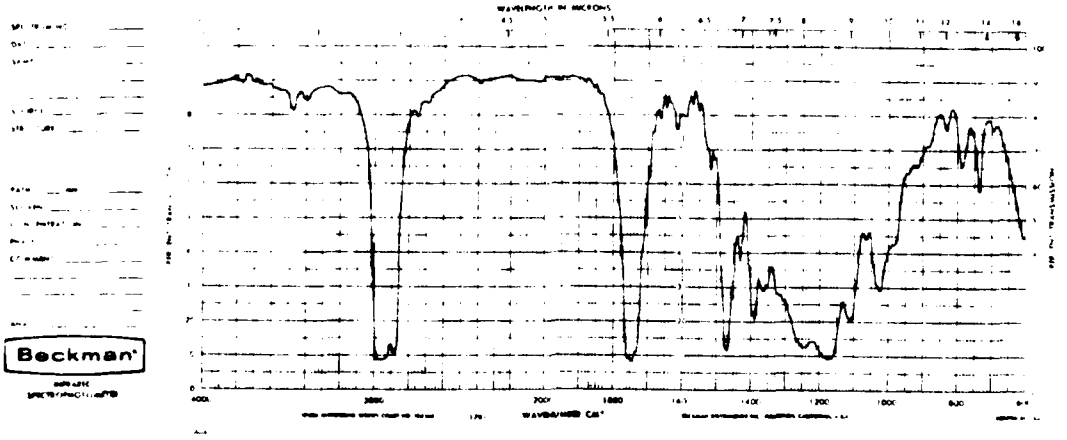
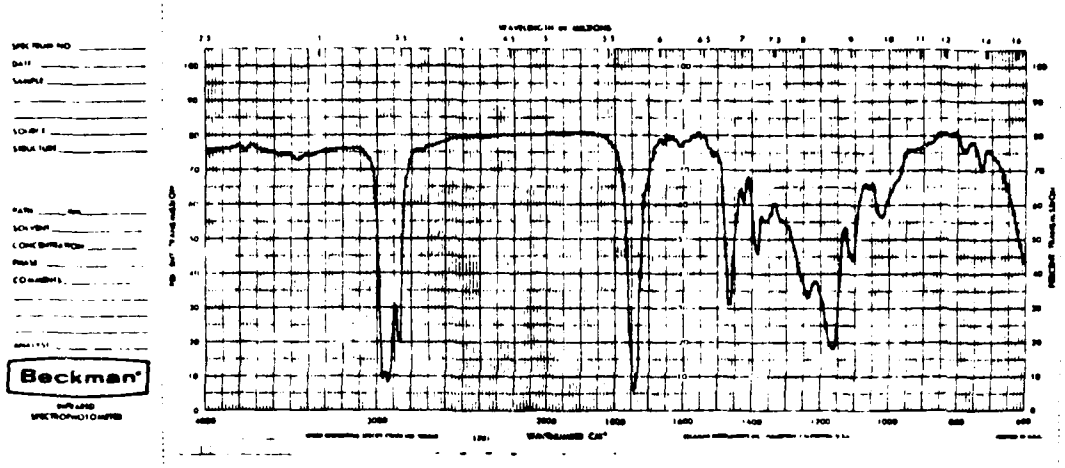


0-82-09

Figure J-8. Infrared spectrum of used oils.



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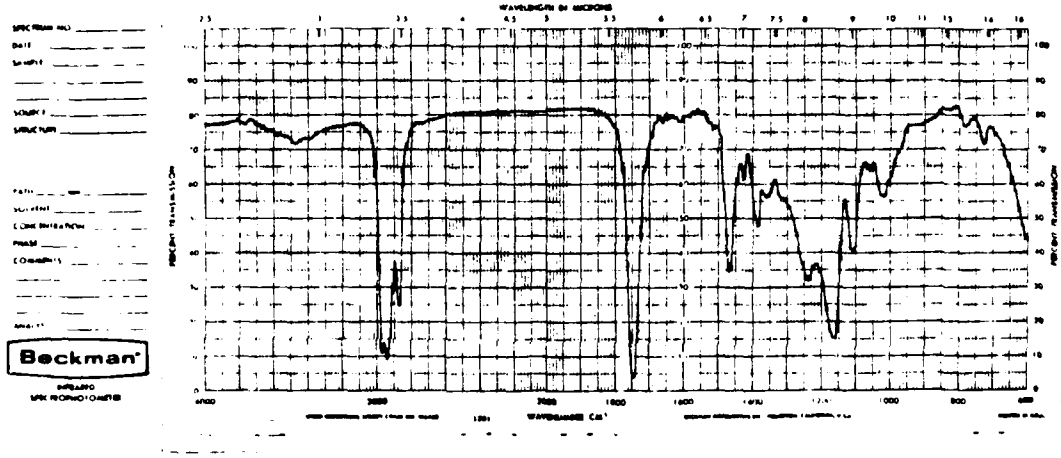
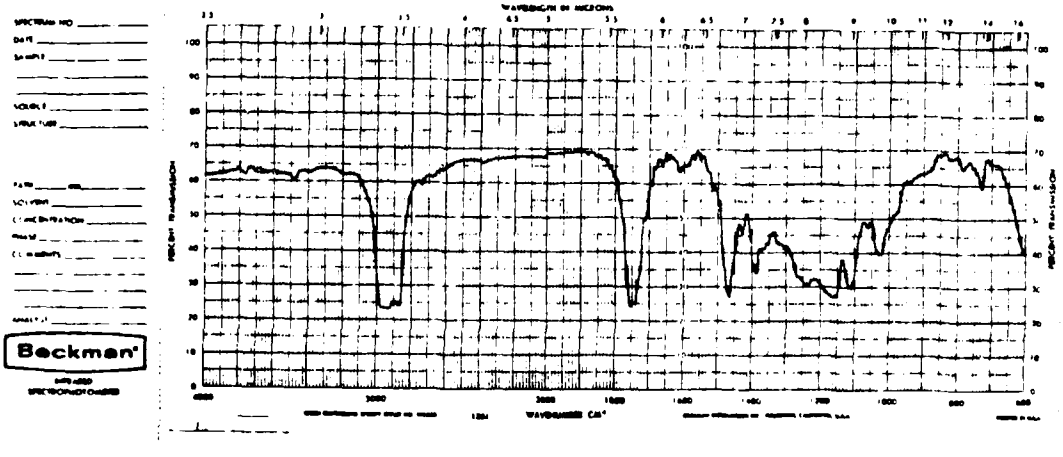
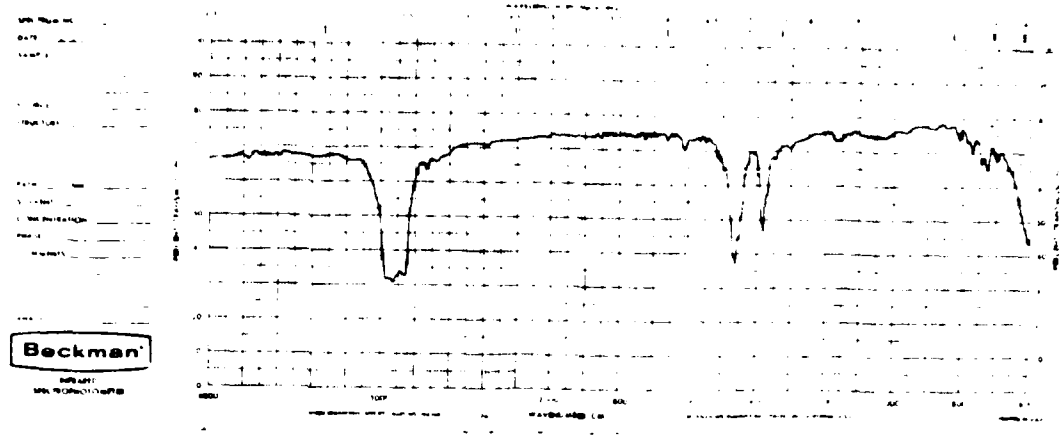


0-82-11

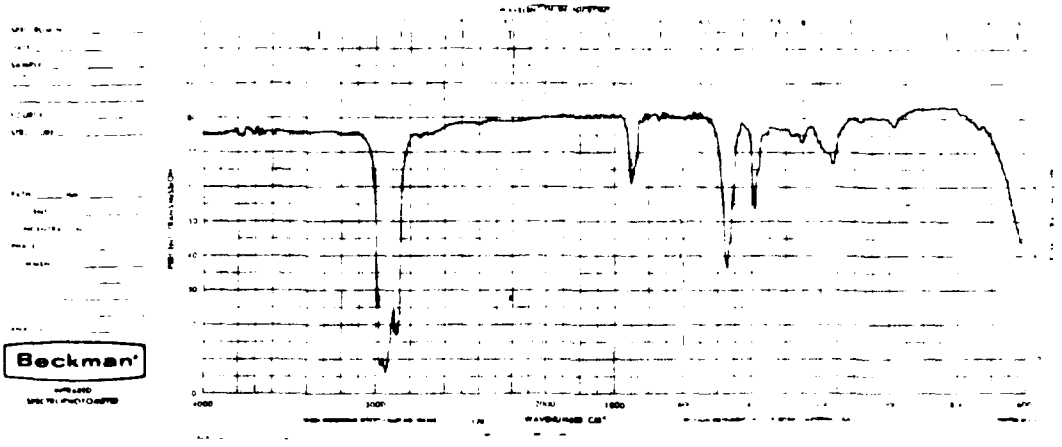


0-82-12

Figure J-9. Infrared spectrum of used oils.



JP4



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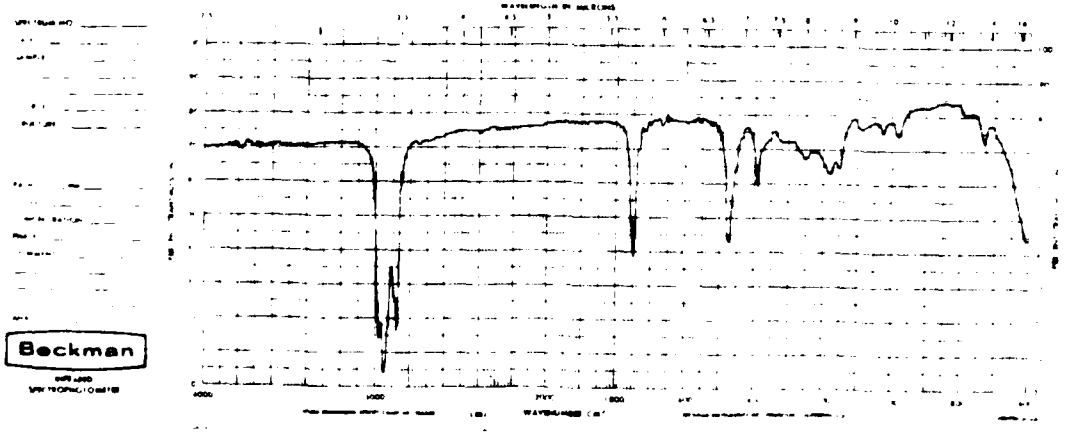

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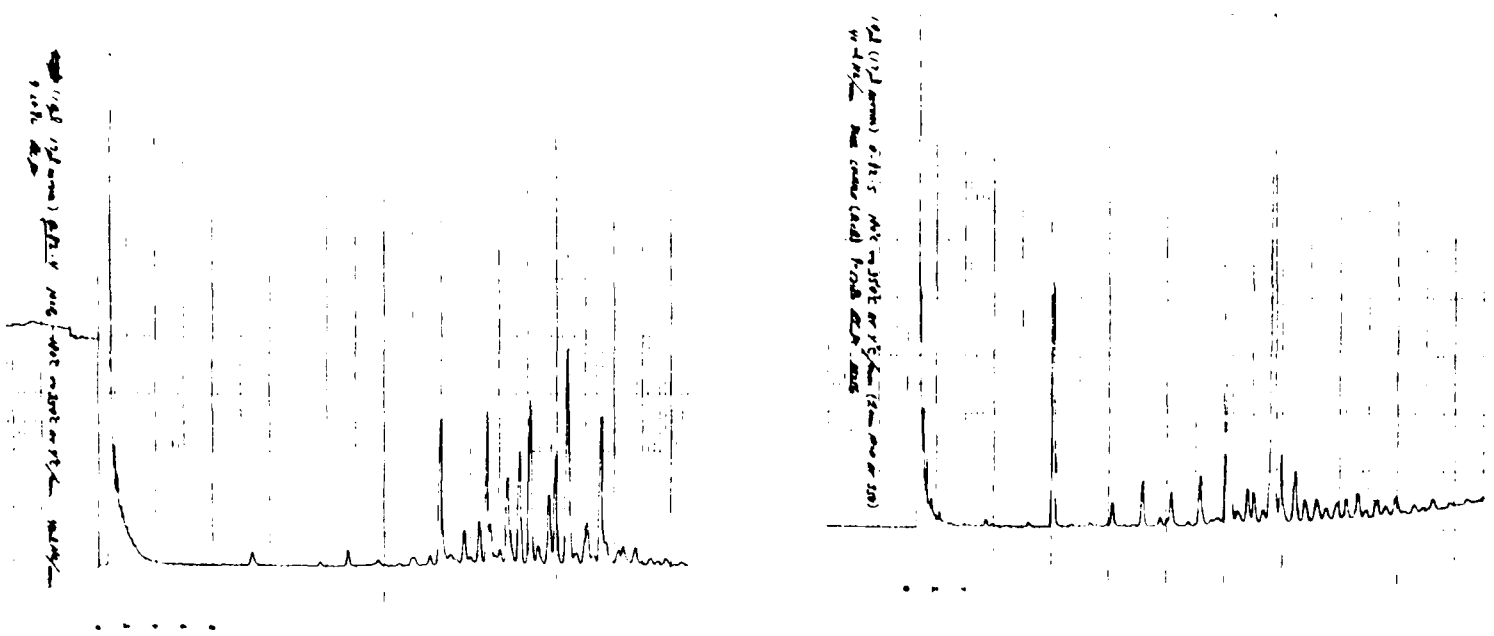
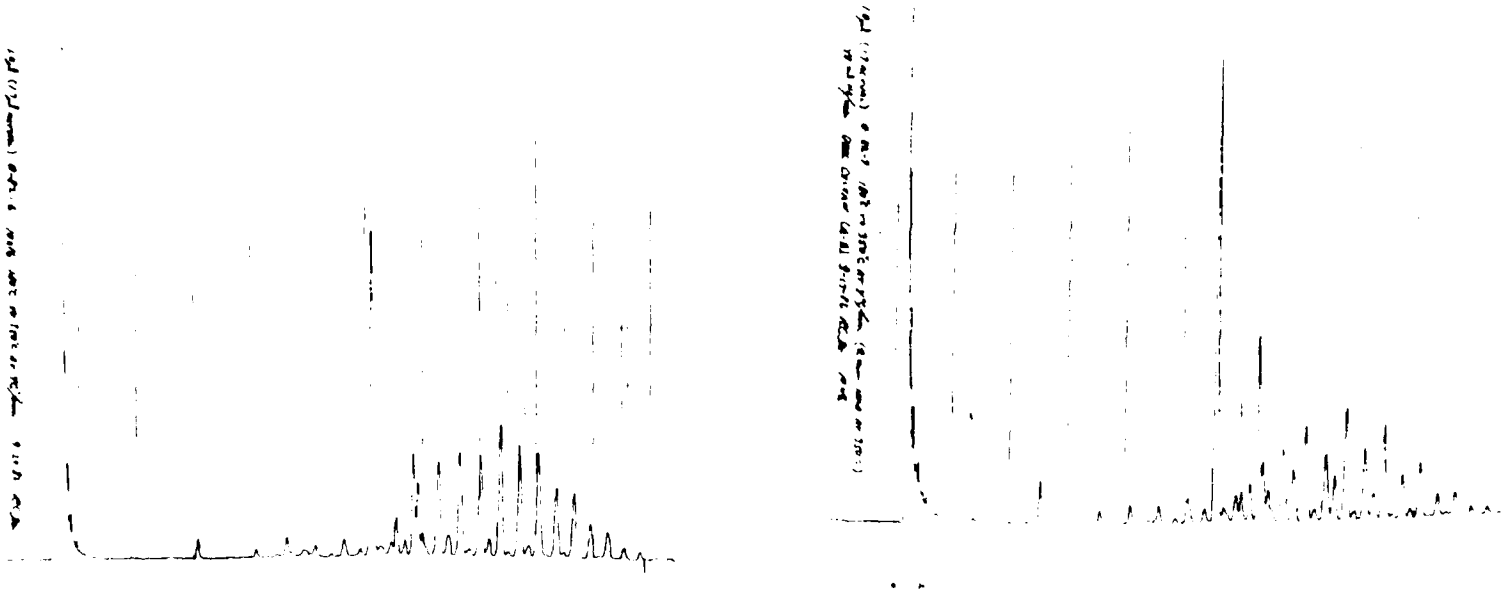
Figure J-10. Infrared spectrum of JP4 and 2 hydraulic fluids.



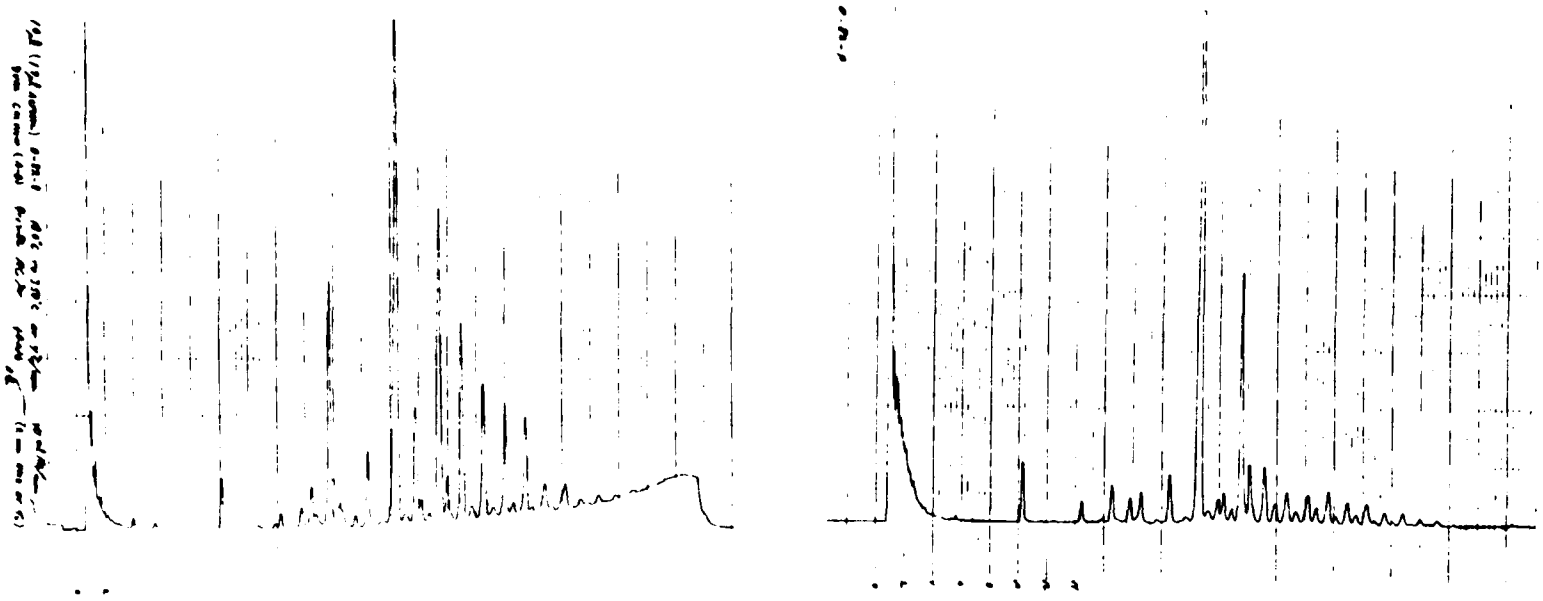
0-82-04 0-82-05

Figure J-11. Gas chromatograms of used oils as received; GC conditions 180-350°C.



0-82-06 0-82-07

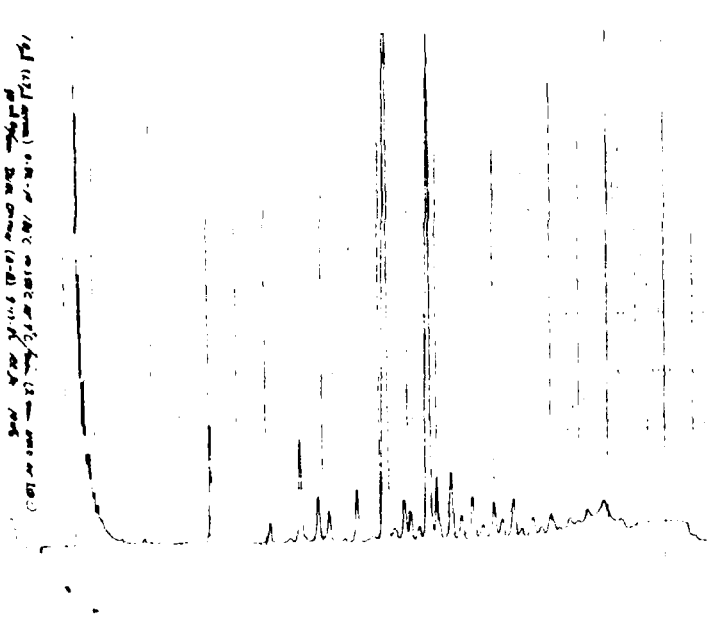
Figure J-12. Gas chromatograms of used oils as received; GC conditions 180-350°C.



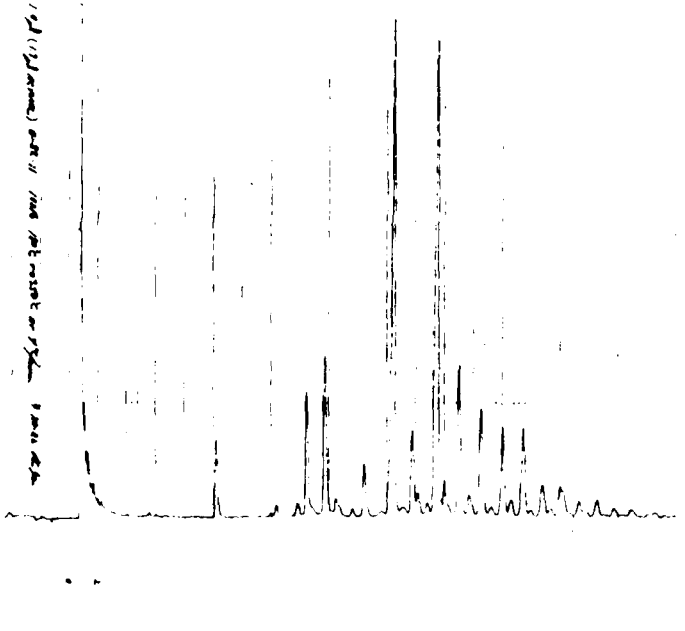
0-82-08

0-82-09

Figure J-13. Gas chromatograms of used oils as received; GC conditions 180-350°C.

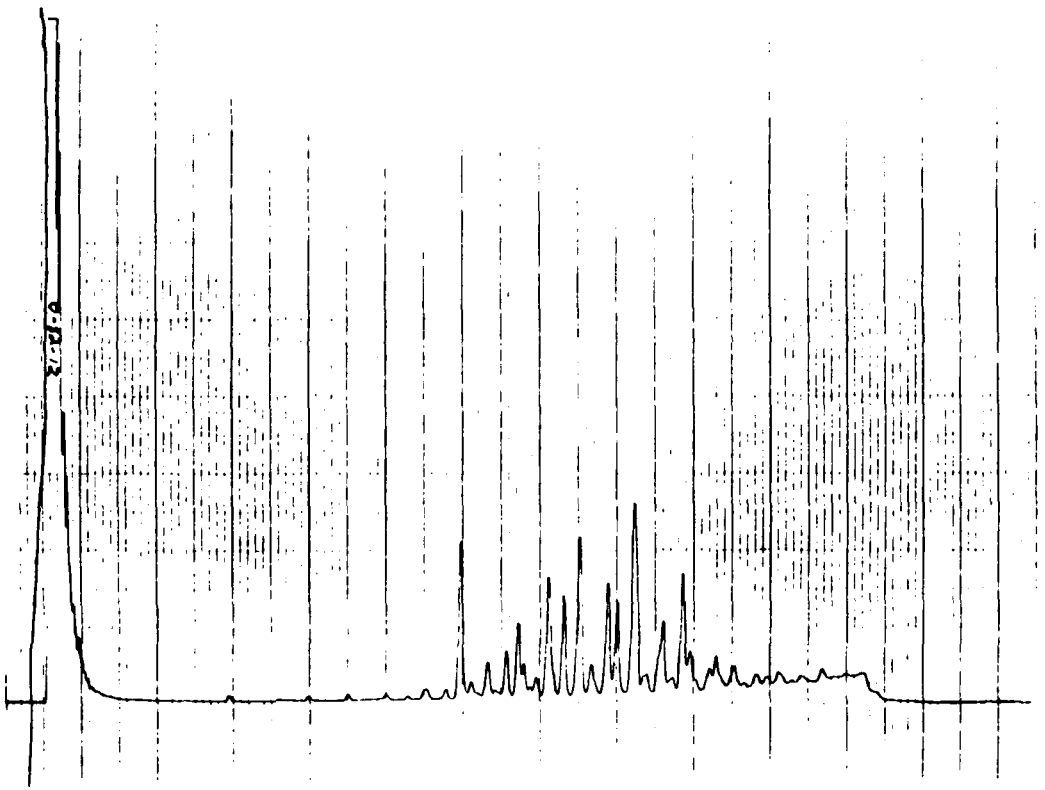


0-82-10



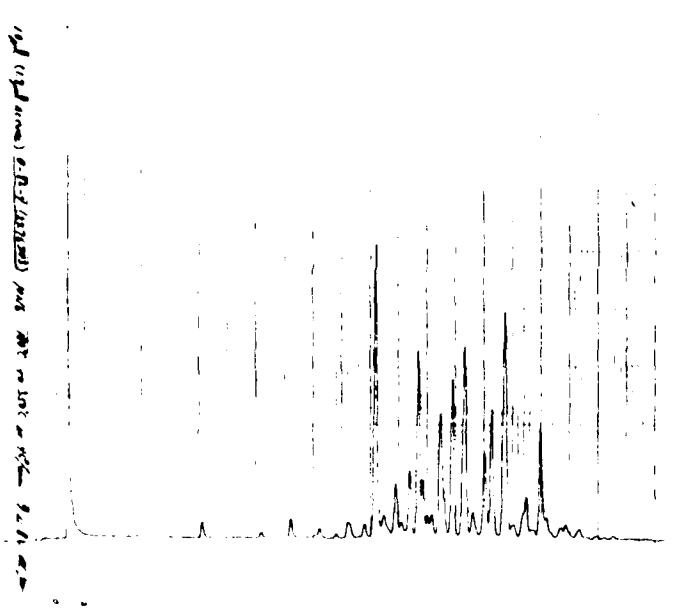
0-82-11

Figure J-14. Gas chromatograms of used oils as received; GC conditions 180-350°C.

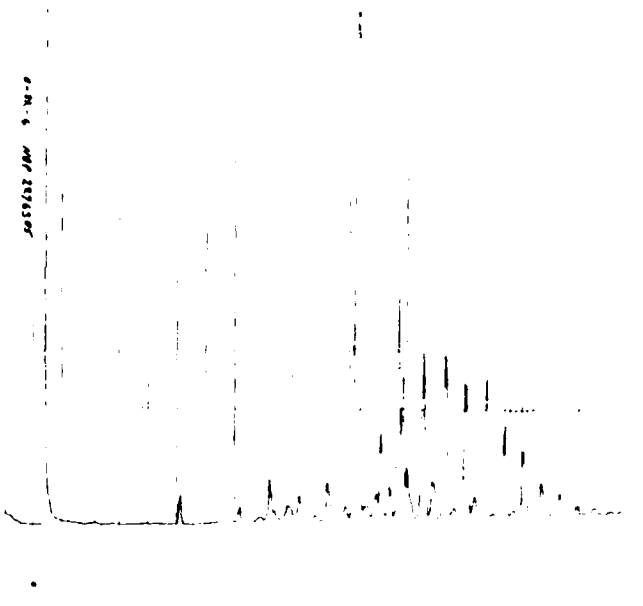


0-82-12

Figure J-15. Gas chromatograms of used oils as received; GC conditions 180-350°C.

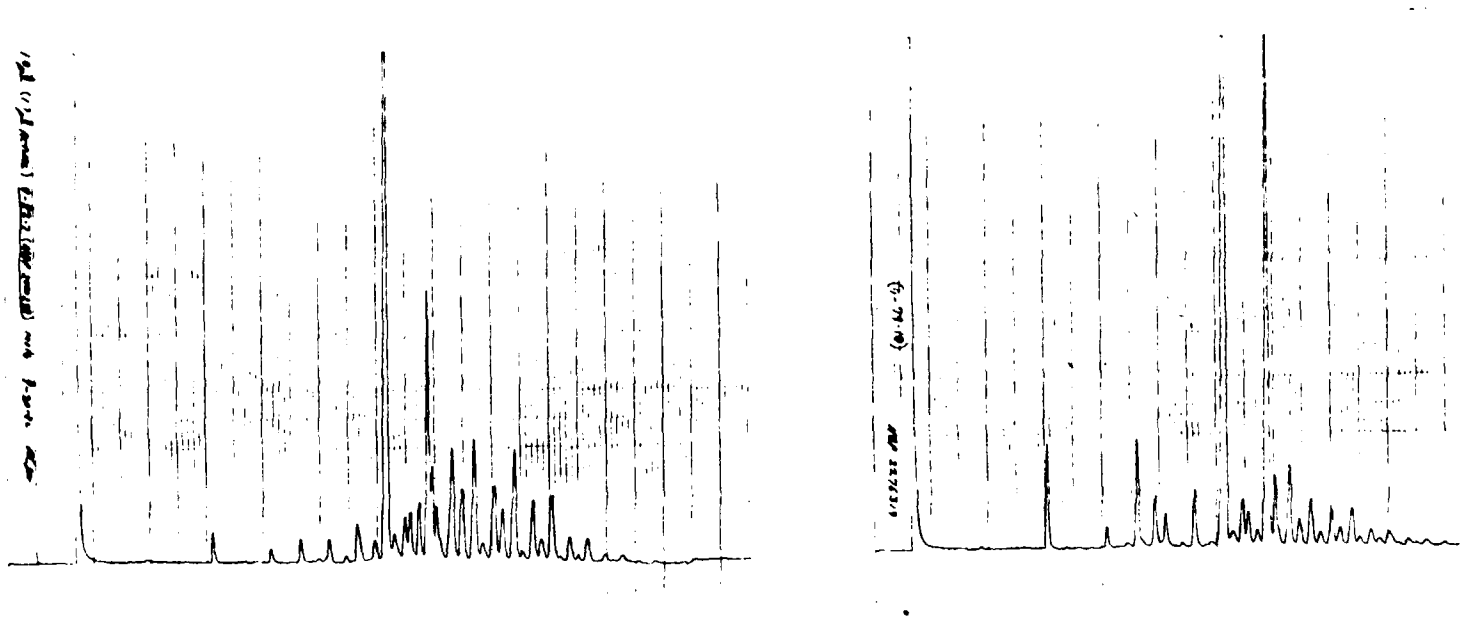


2276303
0-82-04



2276305
0-82-06

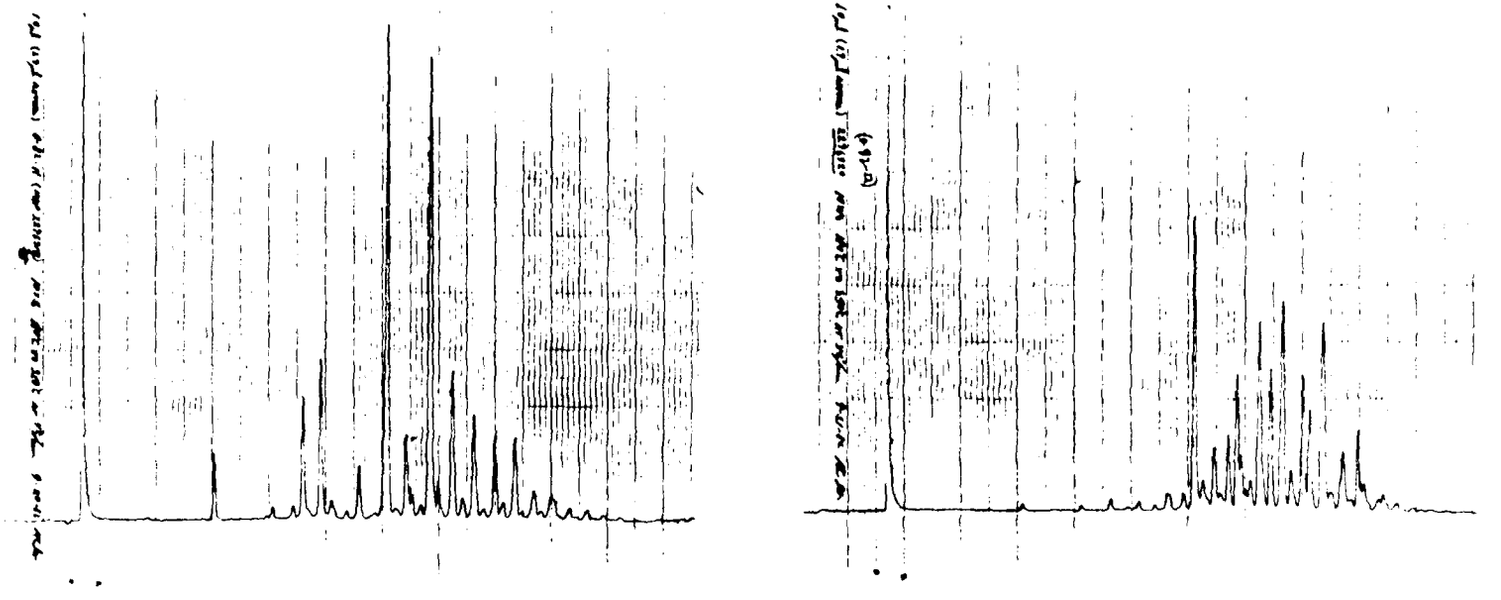
Figure J-16. Gas chromatograms of distillates, of selected used oils; GC condition 180-350°C.



2000298
0-82-07

2276319
0-79-10

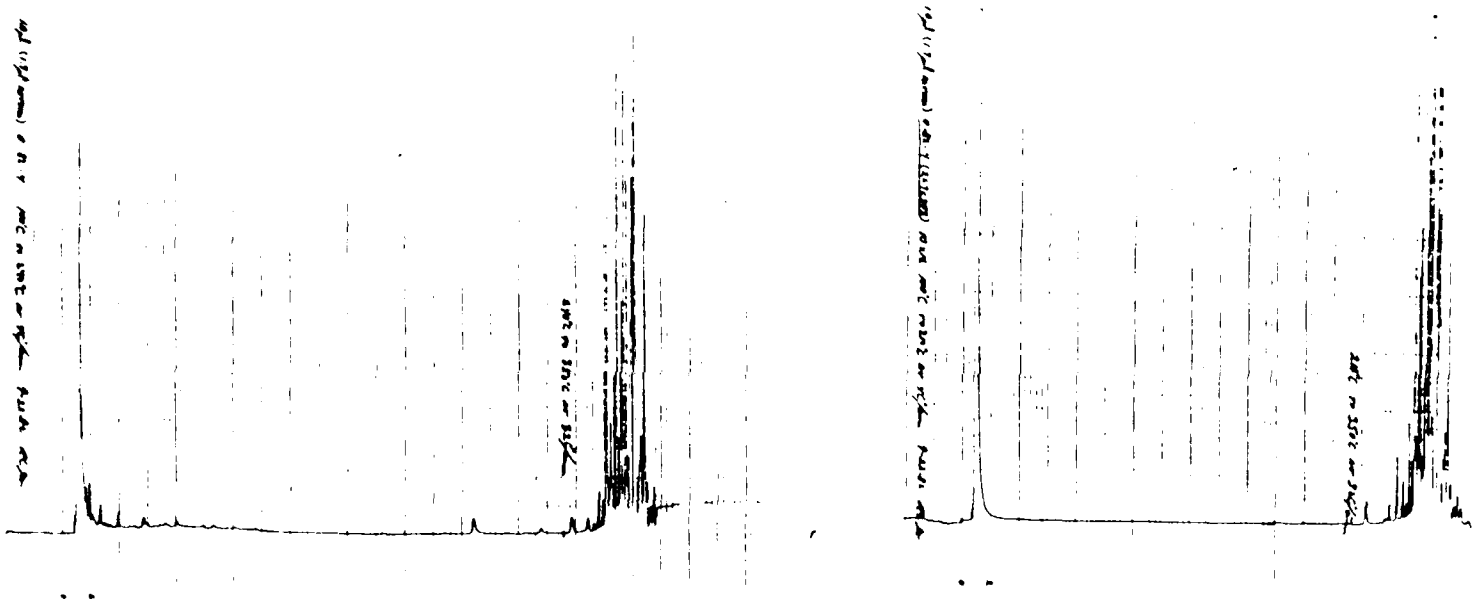
Figure J-17. Gas chromatograms of distillates, of selected used oils; GC condition 180-350°C.



2276318
0-82-11

2276320
0-82-12

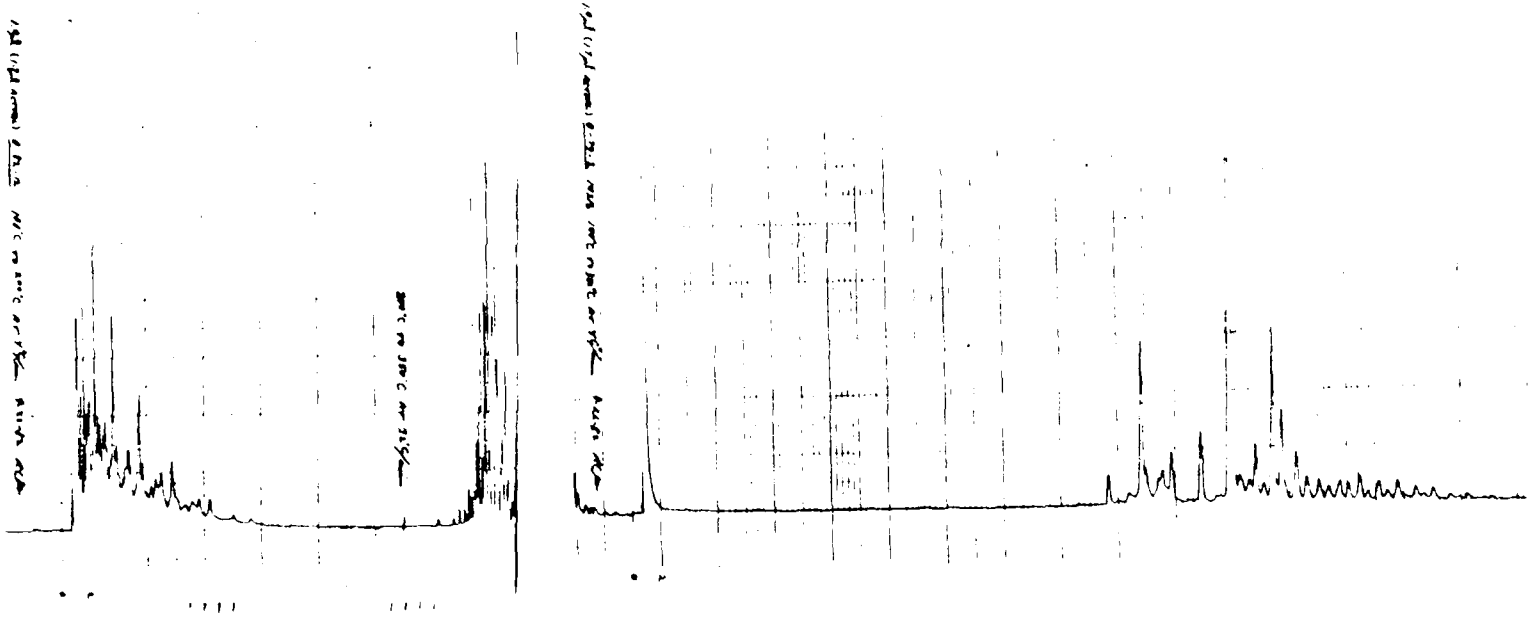
Figure J-18. Gas chromatograms of distillates, of selected used oils; GC condition 180-350°C.



0-82-04
as
received
100-210°C @ 4°/min

2276303
(0-82-04)
distillate
100-200°C @ 4°C/min
210-350°C @ 32°/min

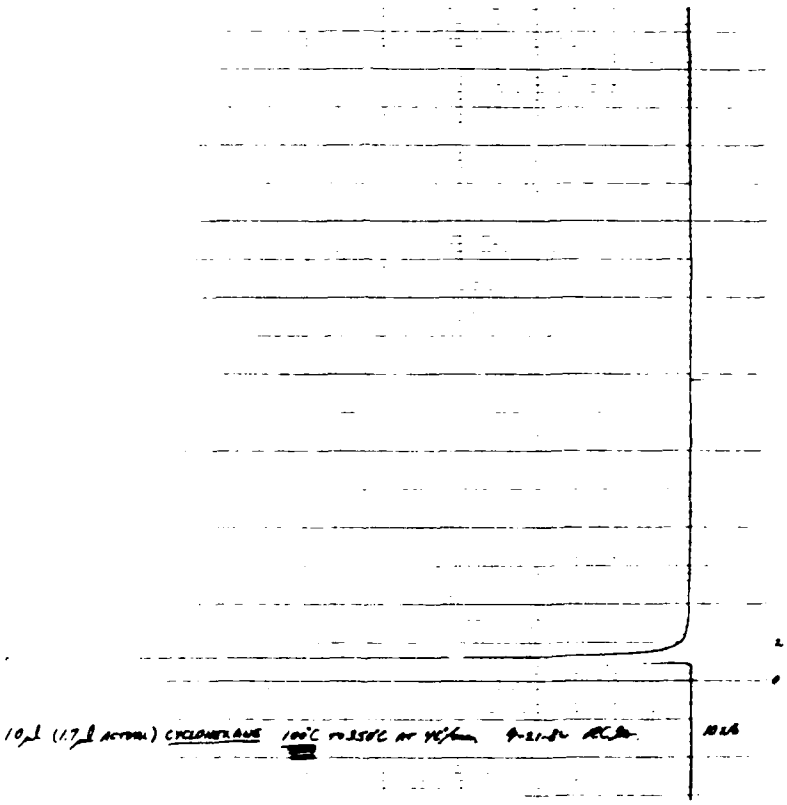
Figure J-19. Gas chromatograms of various samples of used oils.



0-82-12
100-200°C @ 4°C/min
200-350°C @ 32°C/min

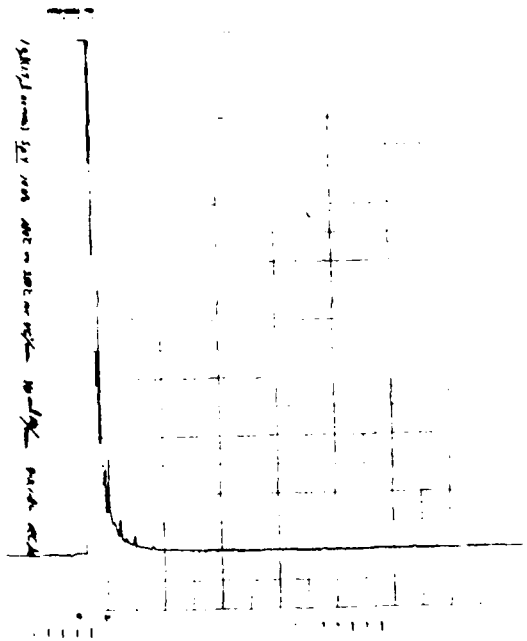
0-79-02
100-350°C @ 4°C/min

Figure J-20. Gas chromatogram of used oils.

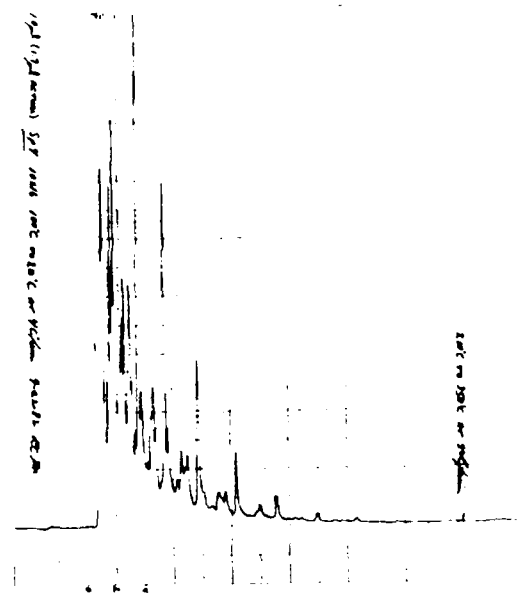


Cyclohexane
100-350°C @ 4°C/min

Figure J-21. Gas chromatogram of cyclohexane.

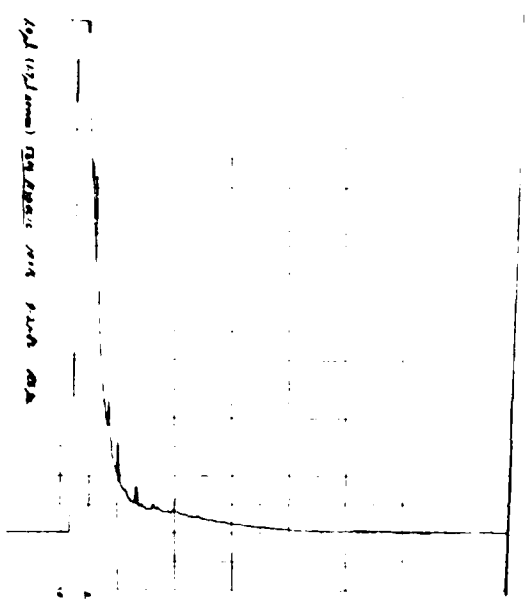


JP4
180-350°C @ 4°C/min

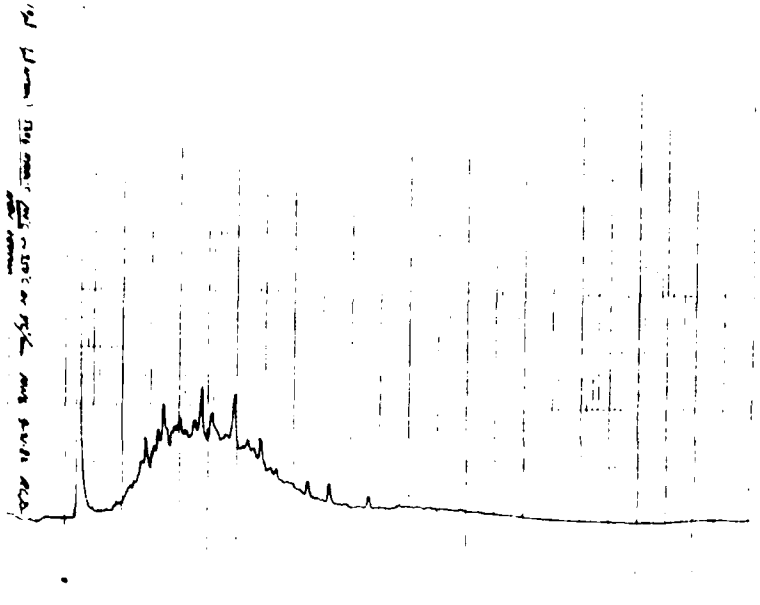


JP4
100-210°C @ 4°C/min
210-350°C @ 32°C/min

Figure J-22. Gas chromatogram of jet fuel JP4.



5606
180-350°C @ 4°C/min



5606
100-350°C @ 4°C/min

Figure J-23. Gas chromatogram of hydraulic fluid 5606.

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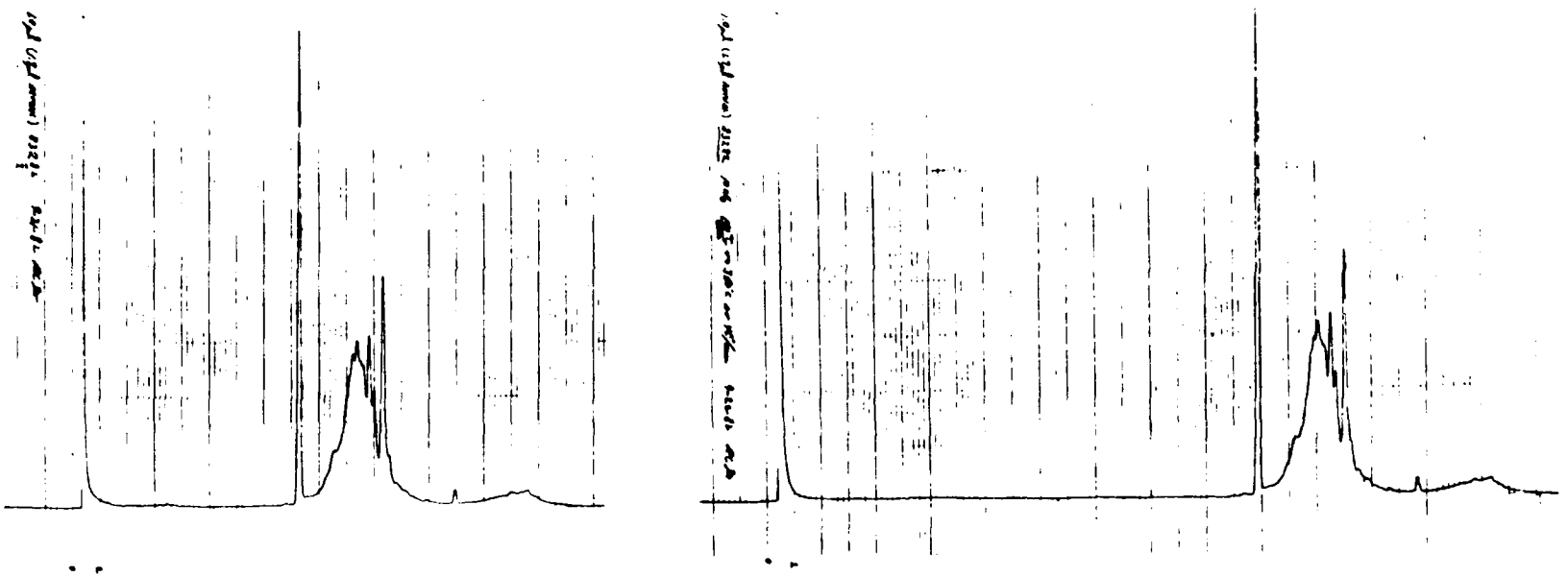

83282
180-350°C @ 4°C/min

83282
100-350°C @ 4°C/min

Figure J-24. Gas chromatogram of hydraulic fluid 83282.

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