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U.S. ARMY/ENVIRONMENTAL PROTECTION AGENCY RE-REFINED ENGINE OIL--ETC(U)
MAY 78 E A FRAME, T C BOWEN DAAK70-78-C-0001
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**U.S. ARMY/ENVIRONMENTAL
PROTECTION AGENCY
RE-REFINED ENGINE
OIL PROGRAM**

AFLRL REPORT No. 98

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JUL 22 1978
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by

Edwin A. Frame

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Southwest Research Institute
San Antonio, Texas**

and

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Re-refined base oils were obtained and analyzed. Based on the analyses, six oils were formulated to MIL-L-46152 quality level using the same concentration of a single additive package. The formulated oils were tested against the requirements of MIL-L-46152. One oil passed all the engine tests. Vehicles from City of San Diego which operated on re-refined oil were disassembled and inspected for deposits.		

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FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL), located at Southwest Research Institute, San Antonio, Texas, under contract DAAG53-76-C-0003 during the period October 1976 through September 1977 and contract DAAK70-78-C-001 through May 1978. The U.S. Environmental Protection Agency participated in the program under Interagency Agreement EPA- IAG-D6-0957. The EPA project officer was Mr. H.B. Kaufman, while Mr. T.C. Bowen, Jr. of the U.S. Army Mobility Equipment Research and Development Command (USAMERADCOM, DRDME-GL) was the Army project officer. Mr. F.W. Schaekel of USAMERADCOM, DRDME-GL was the contract monitor.

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I. INTRODUCTION

A. Background

Over two billion gallons of lubricating oils are used annually in the United States. Of this amount, about one-half is consumed or otherwise lost in use. The other one billion gallons remain as waste products and require disposal.^{(1)*} When improper methods such as uncontrolled burning or dumping are used for disposal, these materials represent an environmental hazard. For example, the uncontrolled burning of used crankcase lubricant results in harmful emissions which pollute the atmosphere. Also, when dumped on land, this material can contaminate groundwater through leaching or waterways by runoff.

These waste products need not represent a hazard, but rather can be a significant potential resource. With the proper processing, waste oil can be used as fuel. Thus, energy is recovered which would otherwise be lost. Also, these materials can be used in cement manufacturing, asphalt road applications, or re-refined for reuse as base stock components of lubricating oils. The later re-refining option appears to be an efficient method of extending the useful life of petroleum products.

Based on the potential environmental benefits to be derived from re-refining waste oil, the Environmental Protection Agency (EPA) became interested in a program to investigate the feasibility of using re-refined base stock in engine lubricants of the type covered by Military Specification MIL-L-46152⁽²⁾, i.e., oils meeting the American Petroleum Institute (API) performance classifications SE and CC.⁽³⁻⁵⁾ This interest led to an interagency agreement, EPA-IAG-D6-0957, with the Department of Defense (DoD) covering the joint "EPA-DoD Re-Refined Engine Oil Program" being reported herein.

B. Program Objectives and Methodology

The objectives of the program were:

- (1) To demonstrate the feasibility of producing MIL-L-46152 quality lubricants using re-refined base oils and suitable additive treatment.
- (2) To generate technical data which will increase the understanding of re-refined lubricants.

Accomplishment of these objectives employed the following methodology. First, re-refined stocks were obtained and analyzed using standard laboratory techniques. Based on the analyses, six stocks were selected and each was formulated with the same additives to provide a finished engine oil. The finished lubricants were then subjected to the engine dynamometer tests used to define oil performance under the MIL-L-46152 specification and API classification system. In addition to this effort, the field performance of a re-refined product was evaluated through engine teardown inspections.

*Superscript numbers in parentheses refer to the List of References at the end of this report.

II. RE-REFINED BASE OILS

Representative re-refined base oils were obtained with the assistance of the Association of Petroleum Re-Refiners (APR). APR contacted 36 member and nonmember companies requesting them to consider participating in the re-refined oil program. A copy of this request letter is shown in Appendix A. Of the companies contacted, 17 participated in the program. These companies furnished 30 samples, 17 re-refined base stocks, 12 feedstocks, and a finished product.

A. Analytical Characterization

The analytical characterization consisted of the following three major areas:

(1) Physical Properties

- Viscosity
- Gravity
- Pour Point
- Flash Point
- Color

(2) Re-Refining Level

- Total Acid Number
- Total Base Number
- Saponification Number
- Carbon Residue
- Total Ash
- Insolubles
- Copper Strip Corrosion
- Trace Metals

(3) Composition

- Aniline Point
- Boiling Point Distribution by Gas Chromatographic Technique
- Elemental Content
- Aromaticity
- Characteristic Groups (Column Chromatography)

Fifteen re-refined base oils were received in time to be analyzed and considered for selection to be blended to finished product. Some materials were late arrivals, and the analyses for these are given in Appendix C. Table 1 shows most of the analytical inspection data for the 15 re-refined base oils, and a virgin base oil denoted AL-6755 which AFLRL had obtained previously. It was later determined that the virgin stock was a hydrotreated base oil which explained some of its compositional characteristics such as very low sulfur content and very light color. Table 2 gives the characteristic groups' (D2007) determina-

TABLE 1
INSPECTION OF RE-REFINED BASE OILS

Sample I.D. AL No. IR Trace No.	ASTM No.	6688 992	6689 993	6690 994	6691 995	6692 996	6693 997	6694 998	6695 999	6696 1000	6697 1001	6698 1002	6699 1003	6700 1004	6701 1005	6702 1006	6755 1019
Inspection	D445	63.82	50.51	64.66	70.27	70.90	88.52	58.95	60.50	67.08	76.93	50.64	119.60	50.75	80.26	45.67	46.60
K. Viscosity @ 100° F, cSt		8.36	7.23	8.02	8.75	8.81	10.13	7.86	7.92	8.54	9.23	6.84	12.09	6.83	9.33	6.63	6.59
Viscosity Index	D2270	110	116	98	105	104	101	108	105	105	103	98	99	97	100	110	108
SAE Grade		30	20	20	20	20	30	20	20	20	20	20	30	20	20	20	20
API Gravity,	D287	29.7	30.4	29.1	29.6	29.4	29.1	30.0	30.5	29.6	29.5	29.8	28.7	29.6	28.5	28.8	31.6
ASTM Color	D1500	4.5	4.5	5.5	7.0	7.0	4.5	4.5	2.5	3.5	2.5	2.5	2.5	2.5	6.0	6.5	2.5
Carbon Residue, wt %	D524	0.14	0.13	0.13	0.09	0.15	0.24	0.06	0.05	0.05	0.08	0.06	0.10	0.06	0.14	0.34	0.07
Pour Point, °F	D97	-15	10	5	10	-25	10	15	5	10	10	15	15	15	0	-5	-5
Flash Point, °F	D92	425	380	425	410	440	430	385	415	415	435	440	465	450	435	365	440
Aniline Point, °F	D611	222	221	221	225	225	226	222	225	225	227	225	227	225	227	228	228
TAN	D664	0.09	0.16	0.16	0.05	0.11	0.22	0.05	0.17	0.10	0.08	0.04	0.04	0.09	0.11	0.17	0.03
TBN	D2896	0.00	0.06	0.00	0.00	0.06	0.00	0.00	0.00	0.00	0.06	0.00	0.27	0.06	0.00	0.00	0.16
Copper Cor., 3hr @ 212°F	D130	1B	1B	1B	1B	1B	1B	1B	1B	1B	1B	1B	1B	1B	1B	1B	1A
Chlorine, ppm	XRF	<100	<100	<100	<100	<100	1250	<100	<100	<100	<100	<100	<100	<100	<100	1010	<100
Sulfur, wt %	XRF	0.21	0.16	0.31	0.18	0.34	0.19	0.14	0.16	0.12	0.15	0.16	0.14	0.15	0.24	0.29	0.05
Phosphorus, ppm	D1091	7	24	3	25	65	25	30	33	5	17	14	9	5	5	70	7
Elemental, ppm	MOD.																
Calcium	AA	10	10	<5	10	111	10	<5	15	<5	44	<5	16	<5	75	<5	<5
Barium	AA	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Zinc	AA	<5	15	<2	11	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2
Lead	AA	<1	<1	<1	<1	47	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Magnesium	AA	<1	12	<1	3	<1	<1	<1	<1	2	<1	<1	<1	<1	11	<1	<1
Copper	AA	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Chromium	AA	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Iron	AA	6	13	<2	13	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2
Sodium	AA	<2	<2	<2	9	<2	<2	<2	<2	<2	<2	4	5	5	5	<2	<2
Silicon	AA	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Aluminum	AA	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Saponification No.	D94	0.43	0.37	0.49	0.36	0.52	0.7	0.35	0.39	0.24	0.21	0.25	0.22	0.38	0.45	4.1	ND
Total Ash, wt %	D482	<0.001	0.014	<0.001	<0.001	0.036	0.004	<0.001	0.005	0.002	0.009	0.001	0.004	0.001	0.022	0.004	ND
Nitrogen, wt %	PE	0.03	0.01	0.01	0.01	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02
Oxygen, wt %	PE	0.66	0.51	0.70	0.80	0.76	0.82	0.65	0.53	0.41	0.55	0.59	0.55	0.40	0.55	0.68	0.44
Benzene Insolubles (uncoagulated), wt %	D893	0.022	0.016	0.014	0.013	0.022	0.018	0.033	0.022	0.032	0.016	0.017	0.020	0.026	0.024	0.030	0.016
GC BP Distribution % Off @ °C																	
1		224	295	346	330	298	346	240	257	276	314	337	355	338	270	226	383
5		370	366	377	383	366	382	339	370	371	383	380	397	380	368	326	400
10		388	384	394	403	390	405	377	388	390	400	395	414	397	357	357	414
50		444	441	448	466	455	465	452	443	447	452	450	483	453	463	434	450
90		535	545	540	574	537	563	551	539	550	532	518	600	518	564	555	488
Residue, wt % > 600°C		5	5	5	6	3	5	5	3	4	1	<1	10	<1	5	5	0 (537 EP)

XRF = X-Ray Fluorescence.
AA = Atomic Absorption.
ND = Not Determined.
PE = Perkin Elmer.

tions, while Table 3 shows the aromaticity by ultraviolet spectroscopy data. The UV aromaticity procedure is shown in Appendix B.

TABLE 2
CHARACTERISTIC GROUPS ASTM D2007

Sample ID		Weight Percent		
AL-	CCL-	Saturates	Polars	Aromatics
6688	844	78.4	2.8	18.8
6689	845	88.8	2.5	8.7
6690	846	77.3	2.6	20.1
6691	847	79.7	10.9	9.4
6692	848	71.2	3.0	25.8
6693	849	84.4	7.0	8.6
6694	850	84.4	2.7	12.9
6695	851	82.9	2.6	14.5
6696	852	80.7	2.7	16.6
6697	853	74.9	2.5	22.6
6698	863	75.1	2.5	22.4
6699	864	82.0	9.8	8.2
6700	865	83.9	8.1	8.0
6701	873	79.8	1.5	18.7
6702	879	75.0	2.3	22.7
6755	-	88.7	2.0	9.3

TABLE 3
RE-REFINED BASE OILS

AL-	Aromaticity by UV Spectroscopy wt %, Ring Carbon		
	Mono-Ring	Di-Ring	Tri-Ring
6688	3.1	0.89	0.22
6689	3.0	1.0	0.23
6690	3.6	1.3	0.36
6691	3.2	1.1	0.32
6692	3.5	1.2	0.36
6693	3.0	0.86	0.21
6694	3.1	0.98	0.20
6695	2.7	0.67	0.10
6696	2.9	0.96	0.24
6697	3.0	0.85	0.19
6698	3.0	1.0	0.27
6699	2.8	0.85	0.20
6700	3.1	1.1	0.28
6701	3.2	1.0	0.24
6702	3.7	1.3	0.27
6755	2.2	0.33	0.02

Significant items from Table 1 relating to re-refined stocks include:

- (1) Two base oils had rather high chlorine contamination levels (AL-6693—1250 ppm and AL-6702—1010 ppm).
- (2) With the exception of two stocks, all base oils fell within a Society of Automotive Engineers (SAE) classification for Grade 20 motor oil. The two remaining products were in SAE Grade 30.
- (3) Three base oils had flash points less than 400°F (AL-6689, AL-6694, and AL-6702).
- (4) Total acid numbers were all fairly low (0.04—0.22) as were total base numbers (0.00—0.27).
- (5) Sulfur contents were typical of virgin lube oil stocks and ranged from 0.12 to 0.34 percent.
- (6) All stocks exhibited high viscosity index values.
- (7) The following summary table shows the significant trace elemental contaminations which were observed:

<u>Sample AL-</u>	<u>Element</u>	<u>PPM</u>
6692	Cu	111
	Zn	11
	P	65
	Pb	47
	Fe	13
6701	Ca	75
	Mg	11
6697	Ca	44
	P	17
6689	Ca	10
	Zn	15
	P	24
	Mg	12
	Fe	13
6699	Ca	16
6695	Ca	15
	P	33
6691	P	25
6693	P	25
6694	P	30
6702	P	70

The zinc, phosphorus, calcium, and magnesium are probably residual additive components, while the iron and copper are wear-related contaminants. The lead is either left from leaded gasoline or results from engine wear/corrosion.

In comparing the percent of aromatics of Table 2 with the aromaticity values of Table 3, it should be remembered that the D2007 method (Table 2) considers *any molecule* which has an aromatic carbon atom as entirely aromatic. For the UV aromaticity, only those *carbon atoms* which are in aromatic rings are regarded as aromatic. This difference in methods accounts for the difference between values presented in Tables 2 and 3.

B. Feedstocks of Re-Refined Base Oils

As previously mentioned, the participating companies furnished 12 samples of feedstocks. Table 4 shows the data obtained for these stocks. Also indicated is the corresponding re-refined base stock. All samples contained lead, chlorine, and bromine contamination which is indicative of used crankcase drainings obtained from vehicles operated on leaded gasoline. In addition, the feedstock contained phosphorus, barium, calcium, and zinc which is typical of engine oil additive systems. Therefore, it is believed that in all cases the feedstocks came at least in part from vehicle crankcase drainings.

TABLE 4
FEEDSTOCKS ANALYSES

AL-	7028	7029	7030	7031	7032	7033	7034	7035	7036	7037	7038	7039
Related Base oil AL-	6696	6688	6702	6694	6695	6693	6689	6691	6697	None	6690	6698
TAN	2.11	2.58	3.00	3.74	2.53	1.05	2.90	4.11	2.79	2.95	2.11	4.06
TBN	3.95	3.05	3.32	3.80	4.30	0.90	4.28	4.41	4.00	4.14	3.73	3.73
Nitrogen, wt %	0.064	0.045	0.009	0.074	0.030	0.049	0.052	0.078	0.052	0.078	0.056	0.083
KVIS, cSt												
100°F	48.9	55.7	38.3	67.2	62.4	75.0	68.6	56.2	72.4	69.2	52.3	66.2
210°F	7.53	8.47	6.48	10.00	9.71	9.25	10.31	8.77	9.29	10.11	7.91	10.27
Viscosity Index	128	138	134	144	150	110	147	145	115	141	130	154
Water, %	2.7	0.2	0.6	5.3	6.0	0.2	10.1	10.2	0.8	2.8	7.4	3.6
Specific Gravity	0.8953	0.8877	0.8887	0.8960	0.8968	0.8857	0.9067	0.8993	0.8876	0.8956	0.8961	0.8981
Carbon Residue, wt %	1.35	1.40	1.12	1.94	1.35	0.46	1.56	2.07	1.21	1.78	1.20	2.20
Total Ash, wt %	0.70	0.82	0.62	1.07	0.86	0.25	1.14	1.14	0.68	1.13	0.74	0.68
Flash Point, °F	*	350	205	*	*	365	*	*	300	*	*	*
Pour Point, °F	-45	-35	-40	-30	-25	-10	-30	-30	-25	-30	-40	-30
Elements by XRF, wt %												
P	0.083	0.130	0.088	0.120	0.108	0.116	0.35	0.123	0.098	0.138	0.150	0.133
S	0.36	0.54	0.38	0.51	0.42	0.49	0.44	0.52	0.43	0.60	0.49	0.59
Cl	0.135	0.072	0.385	0.20	0.068	0.182	0.08	0.34	0.048	0.064	0.44	0.135
Ca	0.105	0.086	0.058	0.088	0.104	0.06	0.080	0.069	0.135	0.080	0.14	0.07
Ba	<0.010	0.024	0.018	0.023	0.046	<0.010	0.018	0.020	<0.010	0.031	<0.010	<0.010
Fe	0.0135	0.0190	0.0575	0.021	0.0190	0.029	0.0190	0.018	0.0105	0.03	0.0515	0.021
Zn	0.05	0.075	0.026	0.055	0.059	0.035	0.062	0.046	0.068	0.068	0.048	0.068
Pb	0.205	0.185	0.179	>0.33	0.147	0.215	0.22	>0.33	0.11	0.245	0.221	>0.33
Cu	0.003	0.004	0.002	0.0045	0.003	<0.003	0.0035	0.002	0.0045	0.0045	0.0065	0.0025
Other Elements	Br	Br	Br	Br	Br	Ti, Br, Al, Si	Br	Br	Br	Br	Br	Br

*Water content too high.

C. Selection of Re-Refined Base Oils for Formulation

The selection of six representative re-refined base oils, to be formulated into finished engine oil, was made with the assistance of the ASTM task force on re-refined oils, and various other interested parties. The group met on February 24, 1977, at EPA headquarters, Washington, D.C. and selected the six base oils. Attendees and their organizations are shown in Table 5. The re-refined base oil analyses presented in Tables 1 and 3 were reviewed and used as the basis for selecting oils to be formulated. The group sought to select base oils which represented a range of potential quality. The selected base oils were grouped in the following three broad categories:

- (1) "Excellent" quality (L-6697, AL-6698)
- (2) "Good" quality (AL-6696, AL-6690, AL-6094)
- (3) "Lesser" quality (AL-6692)

The six stocks were similar in physical properties. From a viscosity standpoint, the stocks would be classified as SAE 20W-20 oils and generally would be comparable with 250-350 neutral high VI virgin base stocks. Two dissimilarities which should be noted are the low flash point for stock AL-6694 and the low pour point value of stock AL-6692. These are probably related to the re-refining level used in their production.

TABLE 5
ATTENDEES AT RE-REFINED ENGINE OIL MEETING
FEBRUARY 24, 1977

Name	Organization
Mr. H.B. Kaufman	EPA
Mr. W.W. Crouse	ASTM (Suntech)
Mr. H.F. Hitchcox	Exxon Research
Mr. C.J. Thompson	BERC/ERDA
Mr. D.M. Stehouwer	GM Research
Mr. H.E. Tiffany	API
Mr. M. Willingham	FEA-State Programs
Mr. M.L. Kerran	Double Eagle Refining
Mr. R.F. Pedall	Motor Oils Refining
Mr. D. Ekedahl	Assoc. of Petroleum Re-refiners
Mr. J. Swain	Consultant (EPA)
Mr. C.F. Schwarz	Consultant
Mr. D.A. Becker	National Bureau of Standards
Mr. T.C. Bowen	MERADCOM
Mr. E.A. Frame	USAFLRL

Although similar in physical properties, the six stocks show significant variation in their chemical makeups. There is a fair range in TAN between stocks, two stocks (AL-6690 and AL-6692) contain high sulfur levels, and a significant difference in trace element content can be observed. The higher level of trace elements in sample 6692 tends to confirm a lesser re-refining level for this stock.

Also, moderate variation was observed in the boiling point distribution and characteristic group analyses. Most significant is the lower 1- and 5-percent point values for sample 6694 and the higher saturate-lower aromatic contents shown for samples 6694 and 6696. The low 1- and 5-percent point values indicate that some of the light ends may not have been removed in the re-refining process and tend to confirm the comment made concerning the low flash point of this sample.

One last point that should be made is that the data for several re-refined stocks reflected values similar to that of the virgin stock. In other words, it would not be possible using these data to distinguish several of the re-refined stocks from a virgin stock.

III. FORMULATION OF RE-REFINED LUBRICANTS

The next phase of the program was the formulation of the selected stocks into finished engine lubricants. The major additive suppliers were contacted and asked for an additive system recommendation to meet MIL-L-46152 requirements. It was desired, but not required, that a single additive package be used for all six stocks. Also, the manufacturers were asked when making a recommendation to consider both the performance and economics of the finished products. A copy of the letter requesting additive company participation is included in Appendix A. Responses from the various additive suppliers are summarized as follows:

- (1) All manufacturers recommended a package currently used in qualified MIL-L-46152 oils.
- (2) With one exception, the additive system was recommended for use in all stocks at the same treatment level. One manufacturer, however, did recommend supplementing the basic system with additional inhibitors for three of the stocks.
- (3) Except when additional inhibitors were recommended, the treating cost for the re-refined oils was the same as for virgin-based products.

In a meeting held on June 17, 1977, at EPA headquarters, the additive recommendations were reviewed with the ASTM task force. Table 6 gives the meeting attendees and their affiliations. One recommendation involving a single additive package for use in all stocks was selected. Drum quantities of the six re-refined stocks were shipped to the additive manufacturer for blending with the selected additive system. Table 7 shows the inspection/properties of the six formulated lubricants which were *all* treated with 7.7 vol % (8.76 wt %) of the recommended additive package. The additive elements show very close agreement among the six lubricants and reflect the excellent blending technique used by the additive manufacturer. In addition, the six oils were tested in accordance with FTMS 791B Method 3470 for compatibility. The test results indicate that the oils are compatible with products qualified under various military engine oil specifications.

TABLE 6
ATTENDEES AT RE-REFINED ENGINE OILS MEETING
JUNE 17, 1977

Name	Affiliation
1. Mr. H.B. Kaufman	EPA
2. Mr. Jack Swain	EPA Consultant
3. Mr. Don Becker	National Bureau of Standards
4. Mr. Bob Pedall	National Bureau of Standards
5. Mr. Duane Ekedahl	Association of Petroleum Re-refiners
6. Mr. Michael L. Kerran	Double Eagle Refining Co.
7. Mr. Michael Willingham	FEA
8. Mr. C.J. Thompson	ERDA/BERC
9. Mr. Harold Tiffany	API
10. Mr. David M. Stehouwer	GM Research
11. Mr. Thomas C. Bowen	MERADCOM
12. Mr. Charles F. Schwarz	Consultant
13. Mr. Leslie Mayot	Exxon
14. Mr. William Crouse	Sun Oil Co. (ASTM)
15. Mr. William Sanjoon	EPA

TABLE 7
FINISHED OIL INSPECTION DATA

Finished Oil	AL-	7067	7068	7069	7070	7071	7072
Re-refined Base	AL-	6692	6698	6690	6694	6696	6697
<u>Properties</u>	<u>Method</u>						
Vis., 210°F, cSt	D445	10.2	8.2	9.3	9.2	9.8	10.6
Vis., 100°F, cSt	D445	86.3	62.7	79.1	72.5	80.8	92.4
Vis., 0°F, cSt (Ext.)	D341	7125	4364	7084	5136	6321	8159
VI	D2270	107	107	103	111	109	106
API Gravity, °	D287	27.2	28.0	26.9	28.4	27.9	27.9
Carbon Residue, wt %	D524	1.17	1.02	1.15	1.11	1.10	1.09
Sulfated Ash, wt %	D874	0.98	0.96	0.95	0.95	0.97	0.96
Pour Point, °F	D97	5	10	10	10	15	10
<u>Elemental:</u>							
Ca, wt %	XRF	0.21	0.21	0.22	0.21	0.22	0.22
Ba, wt %	XRF	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Mg, ppm	AA	9	6	6	7	6	7
Zn, wt %	XRF	0.14	0.14	0.14	0.14	0.15	0.14
P, wt %	XRF	0.12	0.12	0.12	0.12	0.12	0.12
N, wt %	PE	0.019	0.027	0.026	0.025	0.023	0.026
S, wt %	XRF	0.64	0.46	0.59	0.47	0.46	0.46
Base oil S, wt %	XRF	0.34	0.16	0.31	0.14	0.12	0.15

IV. STANDARD ENGINE DYNAMOMETER TESTS

The finished re-refined lubricants were subjected to the standard engine dynamometer tests used to define oil performance under the MIL-L-46152 specification. This specification describes oils intended for use in both gasoline and in low- to medium-output, diesel-powered commercial vehicles which are operated by the military and other federal agencies. Oils that meet MIL-L-46152 specifications satisfy all the requirements of API service SE and CC. Custody of this specification is maintained by USAMERADCOM.

Table 8 summarizes the standard engine dynamometer tests required by MIL-L-46152 and shows the primary performance areas determined by each test. All engine tests were

TABLE 8
ENGINE TESTS OF MIL-L-46152

<u>Engine Test</u>	<u>Primary Performance Areas</u>
L-38	Bearing corrosion Piston varnish Multiviscosity oil-shear stability
Sequence IIC	Engine rust
Sequence IIIC	Oil Oxidation/Thickening Engine varnish Engine wear
Sequence VC	Engine sludge Engine varnish
Caterpillar 1-H2	Diesel engine piston deposits

performed by Southwest Research Institute (Division of Engines, Fuels and Lubricants), San Antonio, Texas.

Table 9 compares engine test results to the requirements of MIL-L-46152. The data are single test results which have been adjusted for laboratory severity effects observed during the testing period. The results have also been adjusted for accuracy as reviewed at the initial meeting of the SAE Interim Engine Oil Review Committee in Chicago, Illinois, October 31-November 2, 1977. It should be noted that data adjustments did not affect the results in regard to meeting specifications. All oils performed satisfactorily in the single-cylinder CLR L-38 and the Sequence IIC tests. In the Sequence IIC test, all oils passed except

TABLE 9
SUMMARY OF PERFORMANCE TESTS ON REFINED ENGINE OILS

Test	MIL-L-46152	Lubricant Formulation					
		Finished Lube					
		AL-7069	AL-7067	AL-7070	AL-7071	AL-7072	AL-7068
		Base					
		AL-6690	AL-6692	AL-6694	AL-6696	AL-6697	AL-6698
<u>L-38</u>							
BWL, mg	40 max	28.4	22.0	14.9	4.8	8.2	20.9
Piston Varnish	-	9.7	9.7	9.7	9.7	9.7	9.7
<u>Sequence IIC</u>							
Avg. Rust	8.4 min	8.4	8.7	8.7	8.9	8.5	8.7
Lifter Sticking	None	None	None	None	None	None	None
<u>Sequence IIIC</u>							
% Vis. Increase @ 40 hr	400 max	105	111	126	88	69	62
@ 64 hr	-	166	173	4774	117	114	103
Sludge	9.2 min	9.8	9.8	9.6	9.7	9.7	9.7
Piston Varnish	9.3 min	9.3	9.2	9.5	9.4	9.5	9.4
Ring Land Deposit	6.0 min	7.95	8.1	7.6	8.3	8.0	8.1
Ring Sticking	None	None	None	None	None	None	None
Wear (Cam & Lifter)							
Scuffing	None	None	None	None	None	None	None
Average, in $\times 10^{-4}$	10	2	2	3	4	3	3
Maximum, in $\times 10^{-4}$	20	6	6	6	7	6	5
<u>Sequence VC</u>							
Piston Varnish	7.9 min	7.9	8.0	7.9	8.2	8.1	7.9
Avg. Engine Varnish	8.0 min	7.9	8.3	8.2	8.3	8.6	8.4
Avg. Sludge	8.7 (8.5) min	9.6	9.6	9.6	9.6	9.6	9.6
Screen Clogging, %	5 max	0	0	0	0	0	0
Oil Ring Clogging, %	5 max	0	0	0	0	0	0
Compression Ring Sticking	None	None	None	None	None	None	None
<u>1-H2</u>							
Top Groove Filling, %	45 max	9	1	24	2	12	10
Weighted Total Demerit	140 max	80.4	73.6	176.0	67.8	188.3	155

Value in parentheses indicates Manual No. 10 rating, other values by Manual No. 12 rating.

AL-7067 which was borderline fail in piston varnish. In the Sequence VC test, a potential problem was revealed in varnish deposits. Many average piston varnish results are at or just above the minimum MIL-L-46152 requirement level. Oil AL-7069 was a borderline fail, missing the required average engine varnish by 0.1 merits. In other VC areas such as sludging, clogging, and ring sticking, all oils performed satisfactorily. In the 1-H2 test, all oils showed acceptable performance in controlling top groove deposits. Piston cleanliness (lacker deposits) resulted in three of the oils (AL-7070, AL-7072, AL-7068) failing to meet the specifications.

Oil AL-7071 passed *all* the engine performance tests of MIL-L-46152 (API service SE-CC). Oils AL-7070, AL-7071, AL-7068, and AL-7072 met the requirements for API service SE, while oils AL-7069, AL-7067 and AL-7071 met the API CC service requirements. These findings are summarized in Table 10.

TABLE 10
SUMMARY OF PERFORMANCE

Lubricant	MIL-L-46152	API Service	
		SE	CC
AL-7069	F	F	M
AL-7067	F	F	M
AL-7070	F	M	F
AL-7071	M	M	M
AL-7072	F	M	F
AL-7068	F	M	F

M = Met Requirements
F = Failed to meet requirements.

V. FIELD EVALUATION OF RE-REFINED ENGINE OIL

A portion of the joint EPA/DoD program involved a limited field evaluation of a re-refined oil. Specifically, the evaluation involved the teardown inspection of engines from six police cruisers operated by the city of San Diego, CA where re-refined oil had been used since February 1974. The complete results of this evaluation were reported in AFLRL Report No. 92, "Inspection of Police Cruiser Engines Operated Using Re-Refined and Virgin-Based Crankcase Lubricants" which is available through the Defense Documentation Center, Cameron Station, Alexandria, VA under accession number AD A045330.

Table 11 shows a summary of the vehicles inspected and the observed deposit ratings. To briefly summarize these data, the San Diego (SD) vehicles had moderate engine varnish, light sludge, and very light rust. It is believed these data indicate the re-refined product provided satisfactory performance under the operating conditions encountered by the city of San Diego police cruisers.

TABLE 11
1974 FORD TORINOS
SAN DIEGO, CA POLICE CRUISERS
OPERATED ON RE-REFINED ENGINE OILS^a

SDPD Unit No.	Veh. ID No.	Eng. Type	Manuf. Date	Odometer Miles	Avg. Deposit Ratings, Merits				Int. Valves
					Varnish		Sludge	Rust	
					Pistons	Engine	Engine	Lifters	
701	4H27H178785	351C	3-74	96,590	5.7	7.0	9.3	9.8	7.8
712	4H27H178853	351C	3-74	91,328	6.3	6.8	9.5	9.8	8.0
723	4H27H178864	351C	3-74	109,703	6.4	5.7	9.2	9.8	9.4 ^b
730	4H27H178851	351C	3-74	104,514	6.3	6.3	9.3	9.8	8.1
764	4H27H178820	351C	3-74	88,267	6.3	6.8	9.3	9.8	9.2
742	4H27H178798	351C	3-74	91,395	6.0	6.2	9.3	9.8	8.1

^aOil and filter changed at 4,000-mile intervals.

^bRecond. heads@ 84,664, 5/76.

Deposit ratings made in accordance with standard CRC techniques where 10 = clean.

VI. SUMMARY/CONCLUSIONS

Overall, the test work produced encouraging results for the future use of re-refined engine oils. Based on the single test evaluations of the six formulated oils, four passed the performance requirements of API service SE, three met the API CC classification, and one oil passed all the performance tests of specification MIL-L-46152 (API service SE and CC). Although variations in specific pass/fail results could be expected with further testing or reformulation, the data as reported demonstrate the technical feasibility of producing SE, CC, and MIL-L-46152 quality level lubricants from re-refined base stocks.

Characterization of the base stocks using standard laboratory analytical techniques showed significant variation between physical and chemical makeup of these samples. Although these differences were noted and could be related to re-refining treatment, they could not be effectively correlated with the performance demonstrated by the finished engine oils. Therefore, further research needs to be conducted to establish characterization techniques and quality conformance tests for both virgin and re-refined base stocks.

VII. RECOMMENDATIONS

Considering the encouraging results of this program, the following recommendations are made:

- (1) Develop baseline data concerning the feasibility of formulating MIL-L-2104C lubricants from re-refined components.
- (2) Conduct a *well-controlled* fleet test of administrative service vehicles at a government installation using commercially available re-refined engine oil and/or

a re-refined oil of the same type as was engine tested in the current program. The test should include a qualified MIL-L-46152 virgin-base lube which uses the same additive treatment as the re-refined oils. This fleet test will provide a *direct performance comparison* between re-refined engine oil and a qualified MIL-L-46152 oil in administrative-type service.

- (3) Perform additional research to better define base stock characterization techniques and quality conformance tests which correlate with engine performance for both virgin and re-refined base stocks.

VIII. REFERENCES

- (1) Kimball, V.S., "Waste Oil Recovery and Disposal," Noyes Data Corporation, 1975.
- (2) U.S. Military Specification MIL-L-46152, "Lubricating Oil, Internal Combustion Engine, Administration Service," November 1970.
- (3) API Publication 1509 (sixth edition).
- (4) ASTM Research Report D2:1002, January 1971.
- (5) SAE J183a, Engine Oil Performance and Engine Classification.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the assistance provided by Mr. S.J. Lestz of AFLRL, SwRI Division of Engines, Fuels and Lubricants, to all additive suppliers who participated in the program, and especially to the supplier whose package was used. Special recognition is made of the Association of Petroleum Re-Refiners and companies who submitted samples for the program. The review and guidance of the ASTM Re-Refined Oils Task Force was greatly appreciated.



133 Pennsylvania Ave. N.W.
Washington, D.C. 20004

July 7, 1970

APPENDIX A CORRESPONDENCES

Mr. Larry Bowen
Environmental Protection
401 M Street S.W.
AM 403
Washington DC 20460

Dear Larry:

Enclosed for your file are copies of the mailing label to the
members of the Association of Petroleum Refiners and all other
parties on your list regarding full cooperation with your
test program. A copy has also been sent to Tom Bowen at Fort
Belvoir.

We should have a good idea of the number of companies participating
in the test soon as received, and we will keep you posted on
this.

Let us know if we can help in any other way.

Attached is the list of location to-refiners requested.

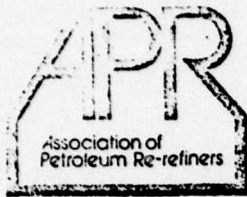
With best regards,

Ernest H. Bradish
Executive Director

ENB:jj
Enclosures

cc: Mr. Tom Bowen, Fort Belvoir, Virginia

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1730 Pennsylvania Avenue, N.W.
Washington, D. C. 20006

202 / 785-0500

July 7, 1976

Mr. Larry McEwen
Environmental Protection Agency
401 M Street S.W.
AW 463
Washington DC 20460

Dear Larry:


Enclosed for your files are copies of the mailing sent to the members of the Association of Petroleum Re-refiners and all other re-refiners on your list encouraging full cooperation with your test program. A copy has also been sent to Tom Bowen at Fort Belvoir.

We should have a good idea of the number of companies participating as the return forms are received, and we will keep you posted on this.

Let us know if we can help in any other way.

Attached is the list of Canadian re-refiners requested.

With best regards,



Duane H. Ekedahl
Executive Director

DHE/jl
Enclosures

cc: Mr. Tom Bowen, Fort Belvoir, Virginia

THIS PAGE IS BEST QUALITY PRACTICABLE
FROM COPY FURNISHED TO DDG



1730 Pennsylvania Avenue, N.W.
Washington, D. C. 20006

200 100

June 30, 1976

Subject: Test Program By
Environmental Protection
Agency

Gentlemen:

This letter, by request of EPA, is being addressed to the members of APR, including Canadian members, and other re-refiners from a list submitted by EPA.

The purpose of the EPA test program as described in the enclosed materials is to reevaluate federal procurement policies which discriminate against re-refined products.

"The purpose of this effort is to examine variations in re-refined oil base stock and to estimate the effect of these variations on the ability of the blended product to qualify under existing military specifications. The results of this contract will be used in re-evaluating Federal lubricating oil procurement specifications which currently prohibit the purchase of re-refined products."

At the meeting of the Board of Directors of APR in Washington in February, and at our October annual meeting in Las Vegas, the test plan was reviewed and it was decided that the Association would give its full support to this effort.

Although changing the federal specifications may not seem to directly affect your business, it is clear that this prohibition has hurt the industry in many ways which go far beyond the mere selling of re-refined oil to government agencies. It has cast into question the quality of re-refined products for all purposes. We see this as an opportunity for the industry to bury this issue once and for all. Positive results will not only lead to greater acceptance of re-refined oil broadly by industry and the public, but will enhance our chances of gaining a national program for waste oil collection and re-refining and help our efforts toward the acceptance of federal and state regulations to encourage the growth of the industry.

The results of the EPA tests will be incorporated in the Bureau of Standards testing program. As you know, the Bureau has now begun to develop tests to demonstrate the equivalency of re-refined oil with new oil as mandated in the Energy Conservation and Oil Policy Act of 1975, which APR is following closely.

The enclosed materials describe the EPA test program. You will see the testing is in two stages. First is a laboratory analysis on all samples and second the engine sequence test on selected samples. In total, you are being asked to prepare three samples:

1. Ten gallons of base stock for the laboratory analysis
2. A 55 gallon drum of base stock for possible engine sequence testing.
3. Five gallons of feed stock from which the base stock is produced.

Upon completion of the laboratory analysis, we are told that each re-refiner will be advised of the findings on his product. At that time he will have the opportunity of declining to have his material subjected to the engine sequence test if he wishes.

We ask that you now do the following:

- A. Return the enclosed form to this office, indicating you have received this letter and whether or not you intend to participate.
- B. Prepare the 5 gallon feedstock sample and 10 gallon base stock sample to be sent by July 30, if possible, to:

Mr. Tom Bowen
U.S. Army Mobility Equipment Research &
Development Command
Building 335
Fort Belvoir VA 22060
Attention: DRXFB-GL

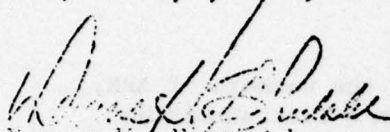
- C. Send these samples to Mr. Bowen with a letter giving information as to the intended use of the base stock, where the feed stock originated if available, and any information you wish to submit regarding blending operations, including bright stock and additive packages which would be helpful in the evaluation.
- D. Also, set aside and hold 55 gallons of the same base stock for possible engine sequence test at a future date.

PLEASE RETURN THE ENCLOSED FORM TODAY.

PLEASE SEND THE TWO SAMPLES TO MR. BOWEN BY JULY 30, IF POSSIBLE.

Thank you for giving this your attention. Call us if you have any questions.

Very truly yours,


Duane H. Ekedahl
Executive Director

DHE/jj1
Enclosures

**THIS PAGE IS BEST QUALITY PRACTICABLE
FROM COPY FURNISHED TO DDC**

LETTER RETYPED FOR REPRODUCTION PURPOSES

June 30, 1976

Subject: Test Program By The
Environmental Protection
Agency

Gentlemen:

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The purpose of the EPA test program as described in the enclosed materials is to reevaluate federal procurement policies which discriminate against re-refined products.

"The purpose of this effort is to examine variations in re-refined oil base stock and to estimate the effect of these variations on the ability of the blended product to qualify under existing military specifications. The results of this contract will be used in re-evaluating Federal lubricating oil procurement specifications which currently prohibit the purchase of re-refined products."

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Upon completion of the laboratory analysis, we are told that each re-refiner will be advised of the findings on his product. At that time he will have the opportunity of declining to have his material subjected to the engine sequence test if he wishes.

We ask that you now do the following:

- A. Return the enclosed form to this office, indicating you have received this letter and whether or not you intend to participate.
- B. Prepare the 5 gallon feedstock sample and 10 gallon base stock sample to be sent by July 30, if possible to:

Mr. Tom Bowen
U.S. Army Mobility Equipment Research &
Development Command
Building 335
Fort Belvoir VA 22060
Attention: DRXFB-GL

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- D. Also, set aside and hold 55 gallons of the same base stock for possible engine sequence test at a future date.

PLEASE RETURN THE ENCLOSED FORM TODAY.

PLEASE SEND THE TWO SAMPLES TO MR. BOWEN BY JULY 30, IF POSSIBLE.

Thank you for giving this your attention. Call us if you have any questions.

Very truly yours,

Duane H. Ekedahl
Executive Director

DHE/jl
Enclosures



DEPARTMENT OF THE ARMY Bowen/lt/43576
US ARMY MOBILITY EQUIPMENT RESEARCH & DEVELOPMENT COMMAND
FORT BELVOIR, VIRGINIA 22060

EAF
4-12-77

JRDME-GL

7 April 1977

SEE DISTRIBUTION

Gentlemen:

The purpose of this letter is to solicit your assistance in the EPA-DoD Re-refined Oil Program. To date, fifteen (15) samples of re-refined base stocks have been collected and their physical and chemical characteristics determined using standard laboratory techniques. Through cooperative effort with the ASTM Re-refined Oil Task Force, the six (6) stocks described in the inclosed table have been selected for formulation with additives and performance testing of the finished products. We would appreciate your recommendations for an optimum additive system (s) to be used with the selected stocks and your assistance in blending the finished products.

It is desired to formulate the finished oils to meet the performance requirements of Military Specification MIL-L-46152. Since only re-refined stocks will be used, the viscosity and flash point requirements of the specification need not be considered. Although a single additive system for use in all stocks is preferred, recommendations for several systems are acceptable. It is noted that consideration should be given to both the performance characteristics and economics of the finished products. Samples of the re-refined stocks and additional information as necessary to provide a recommendation may be obtained by contacting Mr. Bowen of this office, (703-664-4595).

7 April 1977

It is envisioned that all products will be formulated with additive systems obtained from a single supplier. Selection of a recommendation will be made in cooperation with the ASTM Task Force. To assist in this selection, we would appreciate being provided as much of the background information as possible on which you based your recommendation. Also, it is desired that data be provided relating your recommendation to systems used with virgin stocks from an economical and general compositional basis. Since there are time constraints on completing the program, it is requested that all recommendations be received by this office no later than 30 April 1977.

In October 1978, Public Law 94-580 will require Federal Agencies to "Procure items composed of the highest percent of recovered materials practicable". With the guidelines provided, it is clear this legislation is intended to include re-refined engine oil. Therefore, your participation in this program will provide valuable assistance in addressing this complex technological problem.

Thank you for your consideration in this matter.

Sincerely yours,

Maurice E. Lepera

MAURICE E. LEPERA
Chief, Fuels & Lubricants Division

CF: EPA, (Mr. Kaufman)
AFLRL, (Mr. Lestz)
AFLRL, (Mr. Frame)
Sun Oil (Mr. Crouse)

DISTRIBUTION LIST

Amoco Chemicals Company, Mr. G.D. Barth
Chevron Chemicals Company, Mr. W.F. Wagner
Edwin Cooper Incorporated, Mr. D.W. Dinsmore
The Lubrizol Corporation, Mr. James Creedon
Paramins Laboratories, Mr. A.A. Schetelich
Texaco, Incorporated, Mr. R.E. Paggi

APPENDIX B

PROCEDURE FOR THE AROMATICITY DETERMINATION (wt % ring carbon) BY UV SPECTROSCOPY

This is a procedure developed at USAFLRL (See AFLRL Report No. 103, 2/78)

- (1) Weigh to the nearest 0.1 mg, approximately 50 mg of sample and dilute to 10 ml with spectroscopic grade cyclohexane.
- (2) Scan in a variable path length cell* from 350-190 nm.
- (3) The mono-, di-, and tri-aromatics have absorbance maxima at 195, 225, and 255 nm respectively.
- (4) Using Beer's Law: $A = abc$, where
 - A = absorbance
 - a = absorption coefficient, ml/mg-cm
 - b = path length
 - c = concentration
- (5) Standards were scanned to calculate average absorption coefficients.

*The variable cells are used so that path length may be varied, thus eliminating the error which accompanied dilution methods. Also, off-the-shelf spectrograde solvents may be used without further purification because of the short paths possible in the deep UV region.

APPENDIX C

ANALYSES OF LATE ARRIVING OILS

Sample I.D.	Method	7040	7041	7042
AL-				
Inspections				
KVis @ 40°C	D445	85.06	85.26	56.13
KVis @ 100°C	D445	10.31	10.38	7.77
VI	D2270	103	104	102
API°	D287	29.1	29.4	29.9
ASTM Color	D1500	4.5	4.5	4.5
Carbon Residue, wt %	D524	0.16	0.13	0.12
Pour Point, °C	D97	-13	-24	-12
Flash Point, °C	D92	240	246	240
TAN	D664	0.01	0.01	0.09
TBN	D2896	0.00	0.00	0.00
Saponification No.	D94	0.15	0.15	0.35
Elemental Analyses				
Barium, ppm	AA	<5	<5	<5
Calcium, ppm	AA	<5	<5	<5
Chlorine, ppm	XRF	<100	<100	<100
Lead, ppm	AA	2	2	2
Copper, ppm	AA	<1	<1	<1
Iron, ppm	AA	6	<2	<2
Sodium, ppm	AA	515	508	511
Silicon, ppm	AA	<1	<1	<1
Magnesium, ppm	AA	<1	<1	<1
Nitrogen, ppm	PE	26	33	45
Sulfur, wt %	XRF	0.14	0.14	0.36
Phosphorus, ppm	XRF	<100	<100	<100
GC BP Distribution				
wt % off @ °C				
1		358	358	325
5		395	395	371
10		415	415	391
50		498	502	476
90		-	-	555
Residue, wt % >600°C		11	12	0 (598 EP)

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