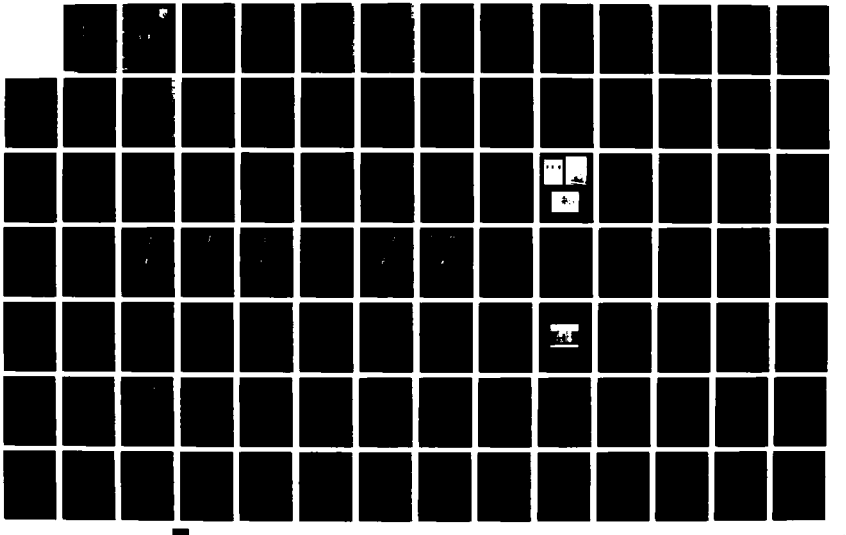
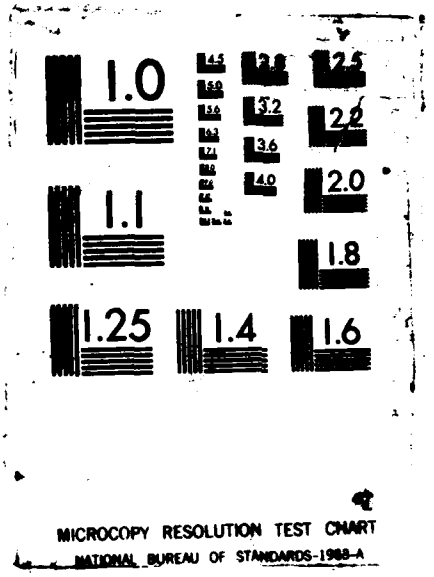
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LUBRICANT PERFORMANCE AND EVALUATION

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

An investigation was conducted in order to develop and improve methods for defining and predicting lubricant performance. This investigation was performed in seven tasks.

In Task I, Arrhenius plots were developed to describe the effective lives of six MIL-L-7808 lubricants as a function of temperature for selected limiting values of changes in total acid numbers, viscosities and weight losses. Relative ranking of lubricants with respect to their stability depends to some degree on defining maximum permissible limiting values. The static coker deposition tester was used to study the coking characteristics of lubricants. Ranking lubricants with respect to their deposit levels using the static coker was in general similar to the MCRT and coking propensity tests. Foaming characteristics of MIL-L-7808 lubricants were investigated using a static foaming test. Parameters studied included sample volume, airflow, ASTM stone and metal spargers. Foaming data shows the 25 ml volume test using the 13/16 inch diameter

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5 micron rated pore size sparger correlates the best with the 200 ml volume Test Method 3213. Chromatographic methods developed for analysis of antioxidants in MIL-L-7808 resulted in isolation and identification of the major intermediates produced during oxidation of PANA and DODPA containing lubricants.

In Task II, significant improvement in AE spectrometer sensitivity was obtained when using air as stabilizing gas, optimizing preburn and exposure times, and profiling refractor plates. The capability of the Wear Particle Analyzer was evaluated for the analysis of metallic iron in lubricants. Even though the instrument suffers from a lack of accuracy in the analysis of metal powders, it responds to changes in concentration of ferromagnetic wear debris. A 5-gallon capacity microfiltration test rig was constructed to simulate fine filtration in turbine engine lubrication systems. A wear test machine was used to generate wear particles in order to study the impact of fine filtration on spectrometric oil analysis.

In Task III, COBRA, conductivity meter and an oil maintenance tester (OMT) were evaluated as lubricant monitoring devices. Even though OMT compared favorably with COBRA it was somewhat less sensitive. Relationships between electrochemical properties and specific chemical changes in stressed lubricants were investigated.

In Tasks IV and V, an assessment of the literature on lubricant load carrying capacity (LCC) test methods was made. The four ball wear test configuration was investigated as a possible technique for determining the LCC of MIL-L-7808 and MIL-L-23699. Effects of load, speed and lubricant formulations containing various concentrations of TCP on wear scar size were determined.

In Task VI, a software system was developed and implemented on the Zenith Z-100 microcomputer for storage, retrieval and correlation of MIL-L-7808 lubricant qualification data.

In Task VII, the remaining useful life of MIL-L-7808 and MIL-L-23699 lubricants evaluation rig (RULLER) was developed based on reductive cyclic voltammetry. A single board voltammograph was developed to reduce the size and cost of the RULLER.

FOREWORD

This report describes the research conducted by personnel of the University of Dayton Research Institute on Contract No. F33615-85-C-2507. The work was conducted at the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories, Wright-Patterson AFB, Ohio.

The work was accomplished under Project 3048, Task, 304806, Work Unit 30480641, Lubricant Evaluation and Performance, with Dr. Phillip W. Centers as the project monitor.

The work reported herein was performed during 17 June 1985 to 17 January 1987.



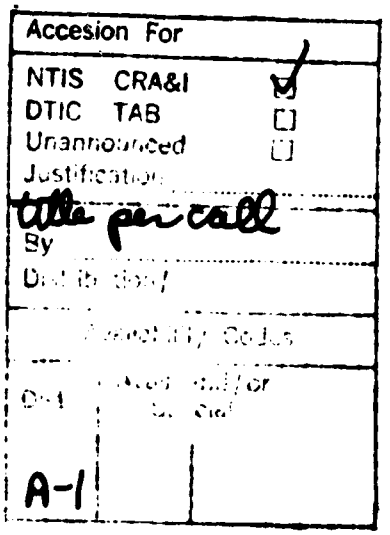


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SECTION I

INTRODUCTION

This work was conducted to develop improved methods for defining and predicting lubrication performance in gas turbine engines. This includes development of lubrication system health monitoring techniques; techniques for lubricant condition monitoring; efficient storage, correlation, and display of current and historical lubricant performance data and development of remaining useful life of a lubricant evaluation rig. Methods development in this program to better define and predict lubricant performance was designed to provide more cost effective methods for lubrication evaluation and the prediction of lubricant performance. The methods are applicable to all types of turbine engine lubricant systems and various lubricating fluids, including the currently used ester based fluids (MIL-L-7808 and MIL-L-23699 oils), mineral oils, polyphenyl ethers, and other high temperature lubricating fluids.

Task I, "The Development of Improved Methods for Measuring Lubricant Performance," was concerned with determining lubricant stability, lubricant coking and lubricant foaming characteristics. The lubricant stability was determined using various high temperature lubricant degradation testing techniques including oxidation, confined heat and corrosion/oxidation. The different testing techniques were used to study the stabilities of all lubricants at temperatures of 215^{o}C or below and at test times between 24 and 768 hours, depending upon test temperatures and other test conditions. Evaluation and correlation of test results were based on changes in composition, acidity, viscosity, volatility and electrochemical

characteristics of the stressed lubricants. The data obtained for each stressed lubricant were used to produce Arrhenius plots. The plots were used to express the stabilities of each lubricant under various thermal and oxidative conditions as maximum temperature capabilities. The prediction capability of each method will then evaluated and refined to develop models which can accurately predict lubricant performance based on required parameters.

The lubricant coking characteristics were investigated with respect to the effects of time, temperature, sample volume, lubricant composition, and test specimen material on the deposition tendencies of various basestock and additive package combinations. The effects of wear debris and lubricant degradation on lubricant deposition characteristics were also studied. The AFAPL Static Coker and Rolls-Royce Coking Propensity Tests were used. The physical properties of the coke deposits produced by the above tests were evaluated and the deposit values were compared with other techniques such as the microcarbon residue tester, bearing deposition rig and tube deposition test.

The third area of Task I was concerned with the study of different size spargers and the development of a lubricant static foaming test which requires 25 ml or less of sample. The results of the developed test were evaluated by comparing them with data obtained from the ASTM static foaming 200-ml sample test.

Task II, "Development of Improved Lubrication System Health Monitoring Techniques," was concerned with determining the morphology of wear debris present in used oil samples taken from turbine engines undergoing normal and severe wear. The particle size detection capabilities of different analytical techniques such as the wear particle analyzer, atomic absorption

spectrophotometry, and atomic emission spectrometry were determined for wear debris of various composition and morphology. The impact of using fine filtration (3-10 micron) in aircraft lubrication systems was evaluated with regard to continued use of present Air Force SOAP wear metal monitors. Requirements for future systems will be defined if current methods prove inadequate.

Task III, "Investigation of Lubricant Monitoring Techniques," involved an investigation into the electrochemical properties of synthetic turbine lubricants. The investigation included a systematic study involving the effects of various basestock and additive package combinations and of different degrees of thermal and oxidative stressing on the electrochemical properties of synthetic turbine lubricants. The Complete Oil Breakdown Rate Analyzer and other applicable instruments for measuring conductivity and dielectric breakdown voltage were used to measure and study the changes in the lubricants' electrochemical properties.

Task IV, "Lubricant Load Carrying Capability Test Assessment," involved an investigation of alternative techniques for measuring load carrying capacity for the Ryder gear test which suffers from a lack of precision. Specifically, the FZG-Ryder (Forschungsstelle fur Zahnrader and Getriebebau) and IAE were assessed as alternatives to replace the Ryder gear test. Factors included in the assessment were test conditions and materials for testing lubricants. The four ball wear test was investigated for determining the load carrying capacity of lubricants. The most promising load carrying assessment procedure will be developed into a test for inclusion into lubricant specifications if appropriate.

Task V, "Development of Specification Wear Test," began with a review of existing techniques and test rigs used for measuring wear. The techniques

and test rigs with the potential of being developed into a specification test for measuring wear prevention characteristics of synthetic turbine lubricants are being ranked according to cost, test time, and repeatability. The ability of each test rig or technique to detect changes in the wear prevention characteristics of different lubricants produced by minor formulation changes is being determined. A recommendation of these techniques will be made for evaluating the wear characteristics of MIL-L-7808 and MIL-L-23699 lubricants. If appropriate, the best technique will be proposed for inclusion in the lubricant specifications.

Task VI, "Development of Lube Data Storage and Retrieval System," will result in the implementation and demonstration of a microcomputer system for lubricant test data storage, retrieval, correlation, and evaluation. The developed system incorporates generated data for MIL-L-7808, MIL-L-23699, and other laboratory formulated lubricants and will incorporate all currently used test data for determining and defining lubricant performance and uses a Zenith Z-100 microcomputer.

Finally, Task VII, "RULLER DEVELOPMENT" was conducted to develop a reductive cyclic voltammetric technique (RCV) for the determination of the remaining useful life of a lubricant. Optimizing experimental parameters of the RCV for determining the remaining useful life of used MIL-L-7808 and MIL-L-23699 was completed. Work is progressing towards miniaturizing the data management system.

SECTION II

DEVELOPMENT OF IMPROVED METHODS FOR MEASURING LUBRICANT PERFORMANCE

1. LUBRICANT OXIDATIVE STABILITY

a. Introduction

The objective of this phase of the program was to investigate the oxidative stability of selected turbine engine lubricants under various temperature conditions, and define stability in terms of their effective lubricant life based upon selected limiting values of various physical and chemical properties. Properties monitored during lubricant stressing included volatility, acidity, viscosity, toluene insolubles, electrochemical characteristics and composition. Arrhenius curves describing effective lubricant life as a function of temperature were developed using only the changes in total acid number (TAN), viscosity and lubricant loss (volatility).

b. Test Apparatus

The oxidation test apparatus is described by DERD Test Method No. 9 prepared by the Ministry of Defence, Directorate of Engine Technology, United Kingdom. This test method is based on Rolls-Royce Ltd. lubricant test development.[1] The test apparatus consists primarily of an oxidation tube shown in Figure 1, a heating bath containing polyphenyl ether as the heat medium and an air flow system including an air filter for removal of oil and large debris and a 5 micron membrane filter for removing fine debris, a pressure regulator, calibrated flow meter and air saturation assemblies. Test assembly is shown in Figures 2 and 3. This test is essentially an oxidation test similar in some respects to many other oxidation tests. To avoid confusion this oxidation test and oxidative test data using this test

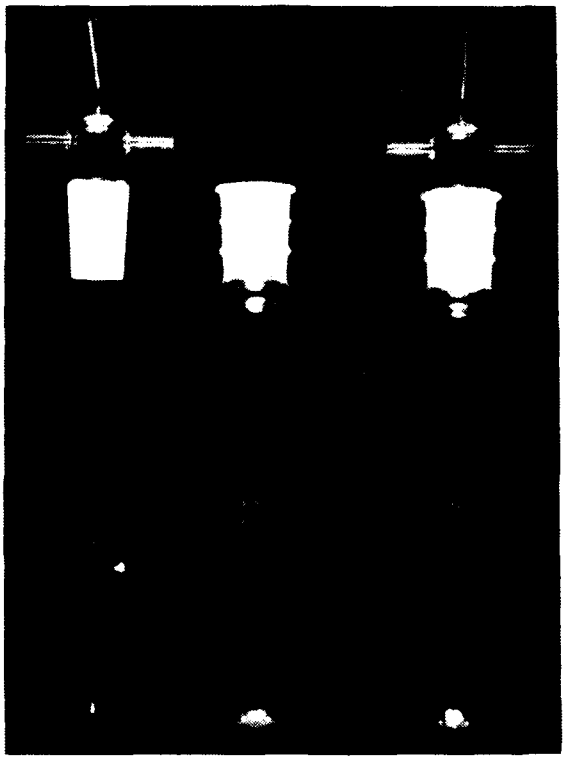


Figure 1. Oxidation Tube for DERD Test Method No. 9

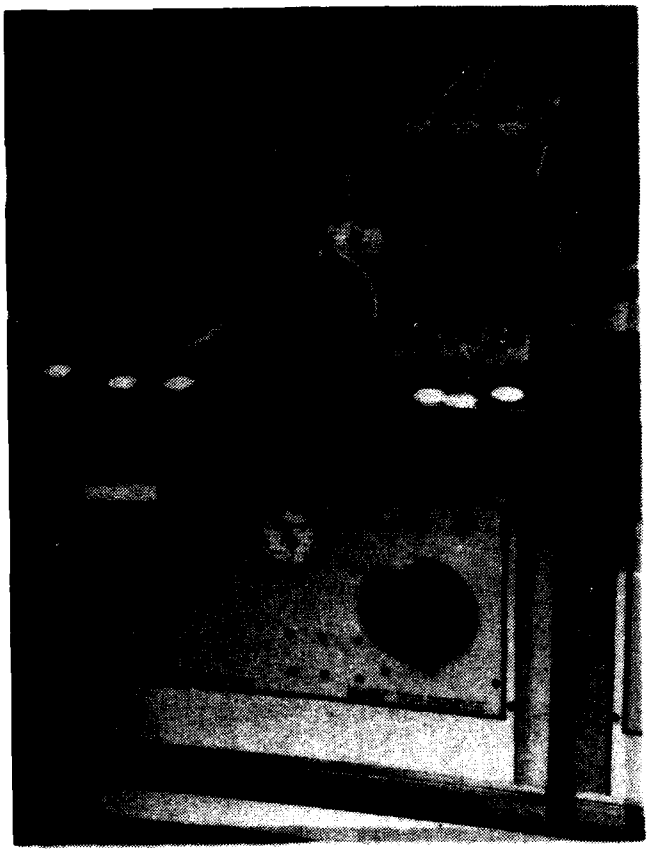


Figure 2. Oxidation Stability Test Unit



Figure 3. Oxidation Stability Test Unit Showing Oxidation Tubes and Confined Heat Assemblies in Place

will be referred to as the Squires oxidation test or Squires oxidative test data as opposed to other corrosion and oxidation testing discussed in this report.

Two variations were made in using the DERD Test Method No. 9 with the first involving the air blowing tubes. Initial tests indicated a small difference in the degree of lubricant stressing between the six air blowing tube assemblies supplied by Rolls-Royce Ltd. having ground glass tapers of 24/29 and those obtained from SGA Inc. having a taper of 29/42. The SGA manufactured tubes gave a slightly lower test severity for all measured properties. Since this difference in severity was small all subsequent testing was to be conducted using SGA manufactured tubes. However, contact with SGA for additional tube assemblies showed they would no longer supply the tubes. Upon request Ace Glass Inc. supplied an air blowing tube of similar dimensions and having a 29/42 taper for evaluation and comparison with the tubes provided by Rolls-Royce Ltd and SGA Inc. Testing of lubricant O-79-16 at 205°C for 168 hours using the different tube assemblies was conducted and a comparison of test data obtained is shown below:

Assembly Manufacturer	R.R.	SGA	Ace Glass Inc.
Weight Loss, % wt	72.2	73.2	71.2
COBRA Reading (Final)	82	84	80
TAN (Final)	1.90	1.84	2.08
Viscosity (Final cSt)	5.66	5.72	5.54

Based upon this data, all subsequent testing was conducted using air blowing assemblies having glass tapers of 29/42 manufactured by either SGA or Ace Glass Inc.

The second variation involved measuring of the TAN and viscosity of the stressed fluids without oil makeup and is discussed in Section II.1.e.

c. Test Procedure

The oxidation tube assemblies were cleaned using the following

stepwise procedure:

Step 1. Washing the assemblies with V.M.P. naphtha and air drying.

Step 2. Soaking the assemblies in carbon remover (Citrikleen, Type HD) for 24 hours or longer if required.

Step 3. Rinsing the assemblies with tap water.

Step 4. Washing the assemblies with Alconox detergent and rinsing with tap water.

Step 5. Rinsing the assemblies with distilled water and drying at about 50°C using a forced air oven.

Steps 1 thru 3 were eliminated for new oxidation tube assemblies since no carbonaceous material existed within the assemblies. The test baths were adjusted to correct temperature and checked with a calibrated Type J thermocouple. Air saturation units (air bubblers) were filled with distilled water and were replenished as necessary during the test to ensure air saturation throughout the duration of the test. Air flow was initiated with the air flow rates being slightly below the required limit of 250 cc/min. Oxidation test assemblies were weighed to the nearest 0.01 gram. Fifty milliliters of the test lubricant was added to the assembly which was then reweighed for providing initial sample weight. The weighed assembly containing the test oil was then placed in the oil bath and the air tube from the saturator was immediately connected to the air inlet tube of the test assembly. Air flow was adjusted to 250 cc/min and was considered starting test time. Testing continued for a specified time period depending upon test temperature and degradation level of previous sample. Test assemblies were then removed from the bath, wiped with a lint free towel, and cooled to room

temperature. Outside of tube assembly was then washed with V.M.P. naphtha to remove any residual bath oil, air dried and then reweighed for determining lubricant loss due to volatility. The lubricants were then poured into an amber vial containing a polyseal cap and used for subsequent testing.

Testing conducted on the stressed lubricant consisted of total acid number (TAN) measurements, viscosity determinations, COBRA measurements, toluene insolubles and for some samples gas chromatography (GC) and high performance liquid chromatography (HPLC). TAN measurements were conducted in accordance with method ASTM D 664 using 1 to 5 gram samples depending upon remaining sample volume.

Kinematic viscosity measurements were conducted in accordance with method ASTM D 445 using semi-micro viscometers.

The COBRA (Complete Oil Breakdown Rate Analyzer) instrument measures changes in the electrochemical characteristics of the lubricant. The COBRA instrument has been previously described[2] and a detailed discussion of its operation and development of calibration standards for this instrument will not be repeated in this report.

Toluene insolubles were determined by heating and sonicating the samples to suspend particulates, weighing 1 to 10 grams of the test lubricant into a glass stoppered flask and then adding 100 ml of pre-filtered (5 micron) reagent grade toluene. The flask was stoppered, swirled for 30 seconds and then placed in dark storage for seven days. The contents of the flask were then filtered through a 5 micron teflon membrane filter. The filter was washed with toluene, dried in an oven at 100°C for 30 minutes and then allowed to equilibrate to room temperature. The filter was weighed and percent toluene insolubles calculated from any increase in filter weight.

d. Test Lubricants and Test Conditions

A total of 11 lubricants, three esters and 38 ester-additive blends containing various percentages and combinations of additives were examined in this study. Oxidative testing of many ester-additive mixtures was conducted for providing stressed samples for deposition studies. Table 1 presents a listing of the lubricants and ester-additive mixtures including a description of each fluid.

Lubricants were stressed at temperatures ranging from 175°C (347°F) to 215°C (419°F). Test durations ranged from 24 hours to 768 hours depending upon the test temperature and the specific fluid being tested. Testing was discontinued for each test fluid after severe degradation or very high lubricant loss had occurred. Lubricant make-up due to volatility oil loss during test was not made prior to analysis of stressed samples.

Total acid numbers and COBRA measurement were conducted on the same day the samples were removed from the test bath since these properties can change with time after being stressed and stored at room temperature.

e. Results and Discussion

The rate of degradation can be expressed by the Arrhenius equation $K = Ae^{-E/RT}$ where

K = rate constant

A = pre-exponential factor (constant)

E = activation energy (cal/mole)

R = gas constant (cal/deg mole)

T = absolute temperature (°K)

Lubricant life is inversely proportional to the rate of degradation and can be expressed as $D = (1/A)e^{-E/RT}$. Representing E/R log e by C, the following logarithmic form to base 10 of this equation is log D = C/T log A. Using this equation and plotting effective lubricant life against reciprocal

TABLE 1

DESCRIPTION OF TEST FLUIDS

TEST FLUID	DESCRIPTION
O-71-6	MIL-L-23699 Lubricant
O-76-5	Trimethylolpropane triheptanoate
O-76-8	Di-2-ethylhexyl adipate
O-77-1	Di-2-ethylhexyl adipate
O-77-1 + I	90.2% wt. O-77-1, Plus 8.3% wt. trimethylolpropane triheptanoate, and 1.5% wt. 2-ethylhexanol (Impurities)
O-77-1 + P	O-77-1, plus 1% wt. phenyl-alpha napthyl amine (PANA)
O-77-1 + IP	O-77-1, plus 9.8% wt. Impurities and 1% wt. PANA
O-77-15	MIL-L-23699 Lubricant
O-79-16	MIL-L-7808 Lubricant
O-79-17	MIL-L-7808 Lubricant
O-79-20	MIL-L-7808 Lubricant
O-82-2	MIL-L-7808 Lubricant
O-82-3	MIL-L-7808 Lubricant
O-82-14	MIL-L-7808 Type Lubricant
O-85-1	4 cSt Candidate Lubricant
TEL 6031	7.5 cSt Fluid
TEL 6032	7.5 cSt Fluid

degrees Kelvin using semilogarithmic graph paper provides a linear relationship between temperature and lubricant life. Appendix A Table A-1 provides all the Squires Oxidative test data developed during the study and from which Arrhenius plots were developed.

Lubricant life must be defined in terms of limiting values for selected properties. For the MIL-L-7808 lubricants, Arrhenius plots were developed using the following criteria as limiting values.

Total Acid Number Changes of 1, 1.5 and 3

Viscosity Increases at 100°C of 15%, 25% and 35%

Volatilization Losses of 15%, 25% and 35%

Figures 4 thru 6 show Arrhenius plots for six MIL-L-7808 type lubricants using TAN increase as the limiting life criteria. For all levels of TAN increase limiting values, two of the six lubricants showed much lower lubricant stability, especially at temperature of 200°C and lower. Differences in effective life of the six lubricants are also shown in Table 2 for test temperatures of 175°C, 200°C, and 225°C for the three limiting TAN values. This table shows lubricants O-82-2 and O-82-14 to have much lower lubricant stability than the other lubricants. The data in Table 2 also shows the ranking of the lubricants with respect to lubricant life depends to some extent on the selected TAN increase limit.

Figures 7 thru 9 show Arrhenius plots for the six MIL-L-7808 type lubricants using viscosity increase as the limiting life criteria. Less difference in effective life is shown for the six oils than the difference in effective life based on TAN increase. This would be expected since most of the viscosity increase is due to volatilization of lighter esters. Table 3 shows the difference in effective life for these lubricants at test temperatures of 175°C, 200°C, and 225°C. The data in this table shows

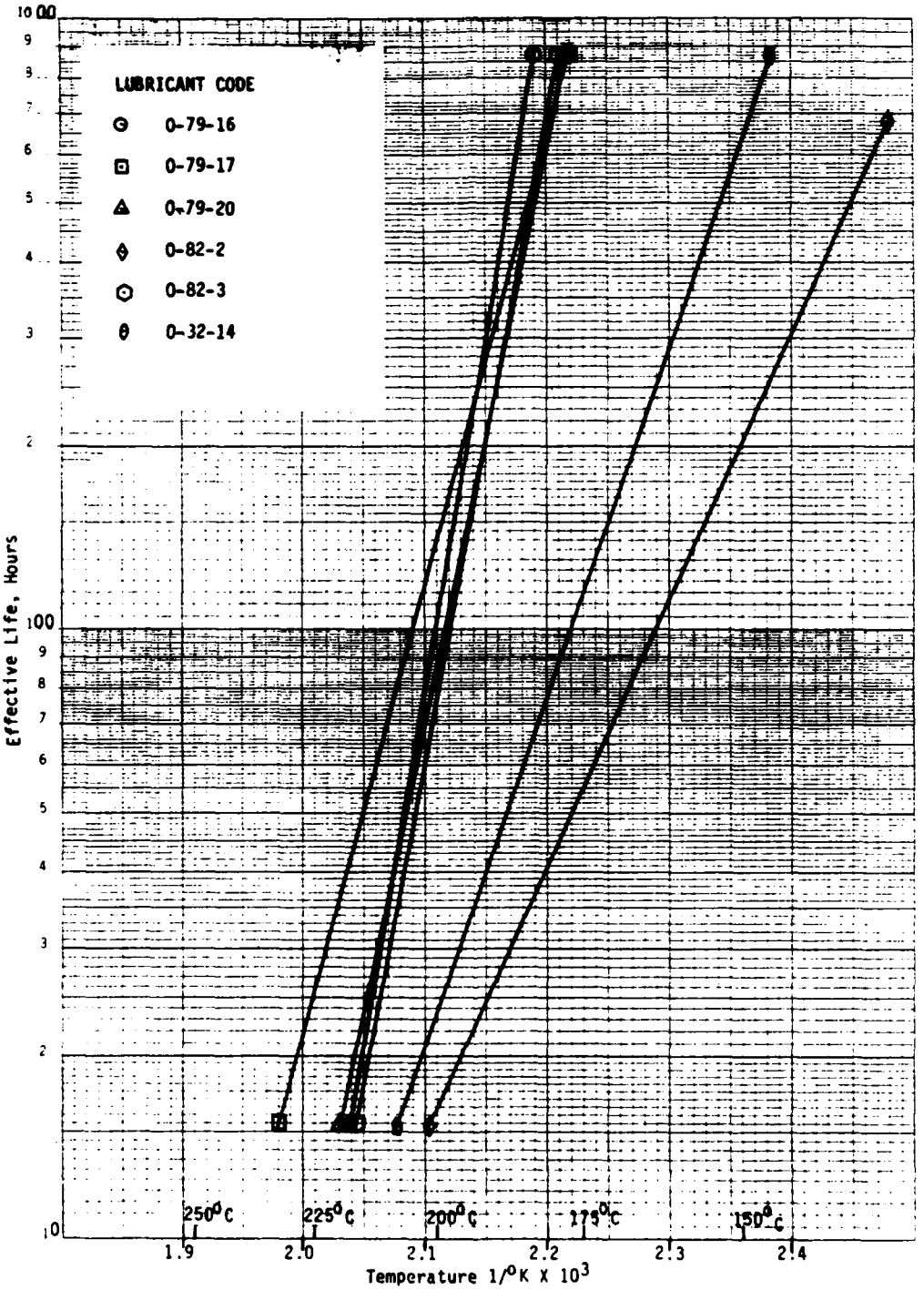


Figure 4. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Total Acid Number Increase Limit of 1.0

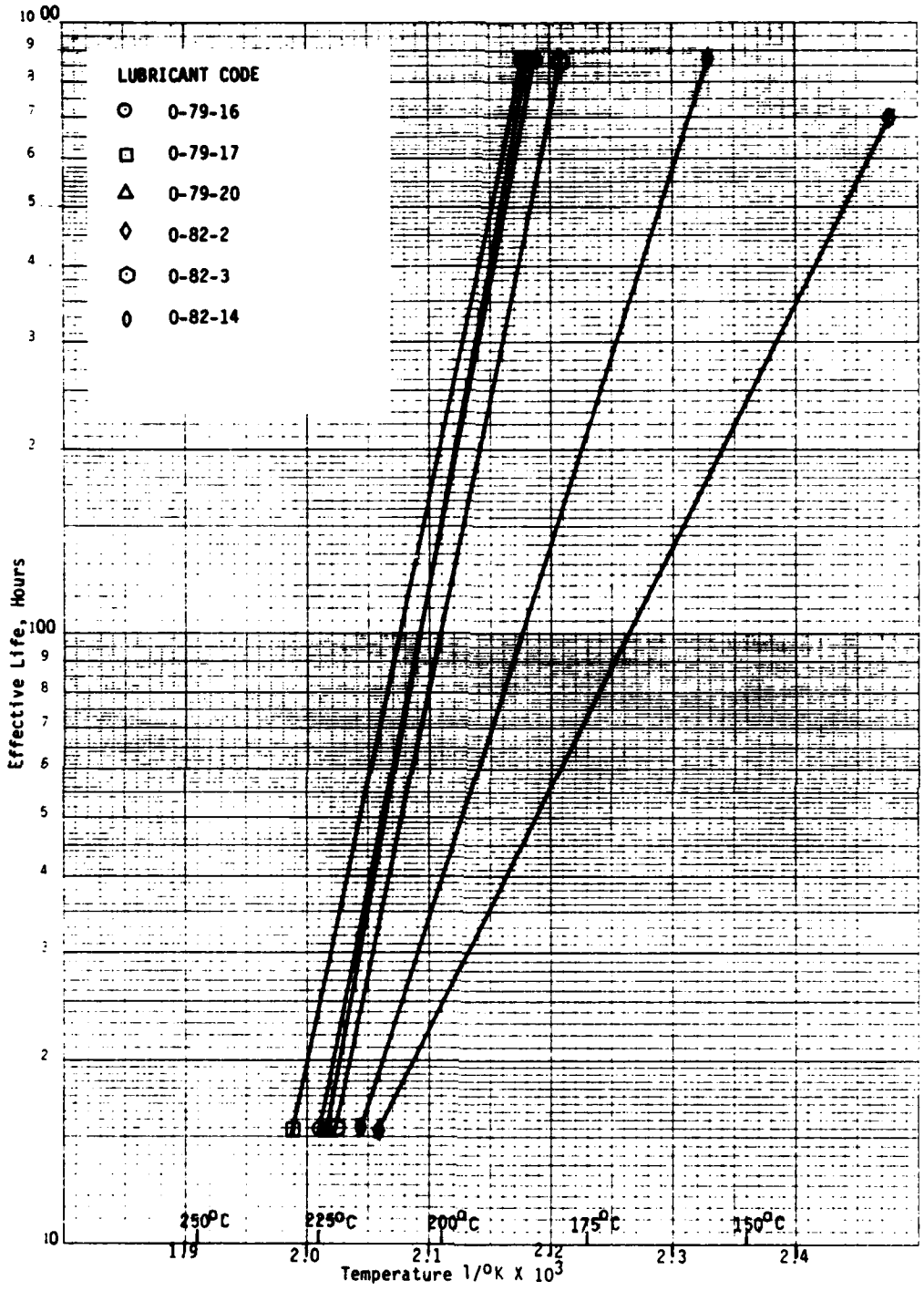


Figure 5. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Total Acid Number Increase Limit of 1.5

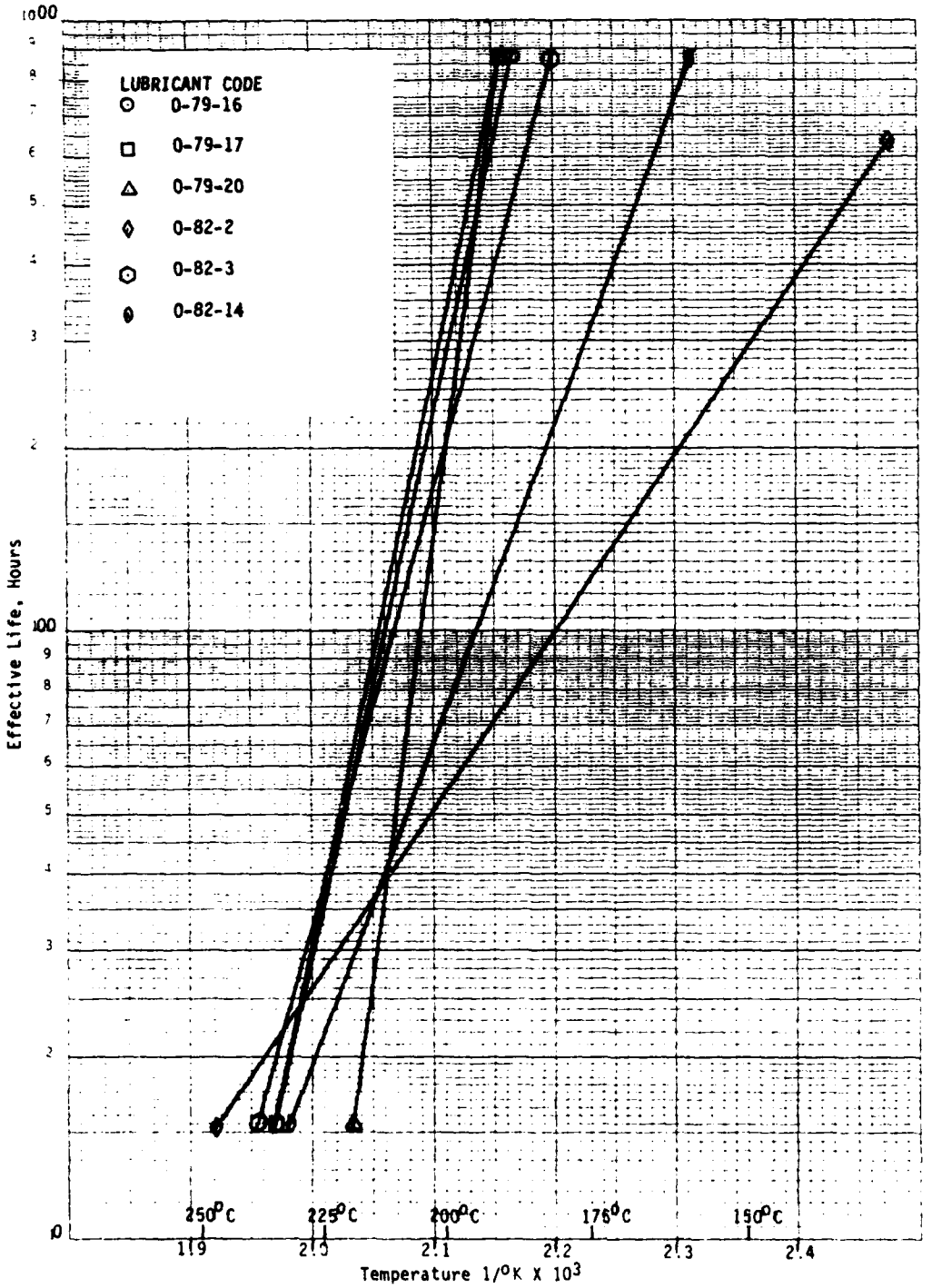


Figure 6. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Total Acid Number Increase of 3.0

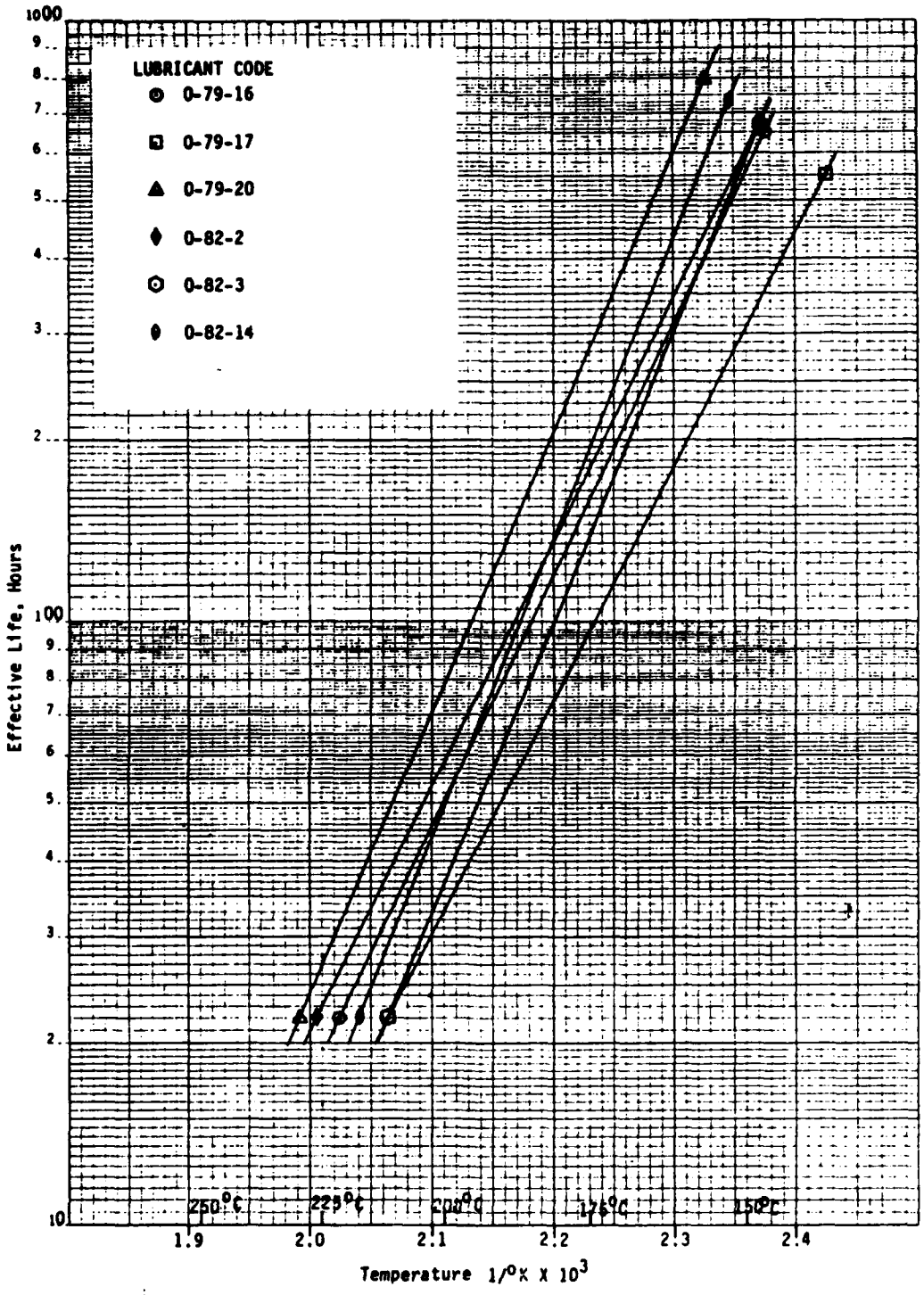


Figure 7. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Viscosity Increase Limit of 15%

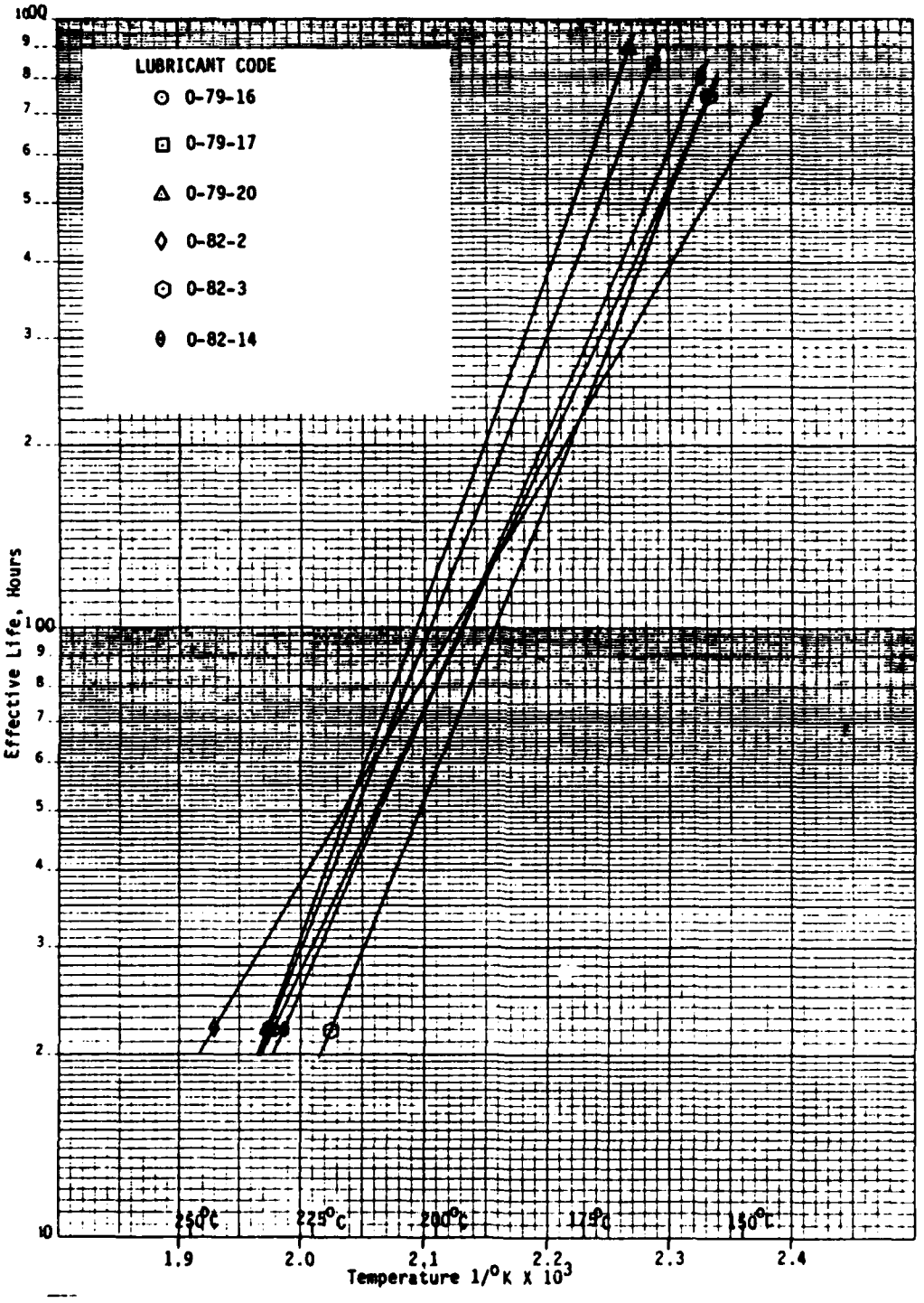


Figure 8. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Viscosity Increase Limit of 25%

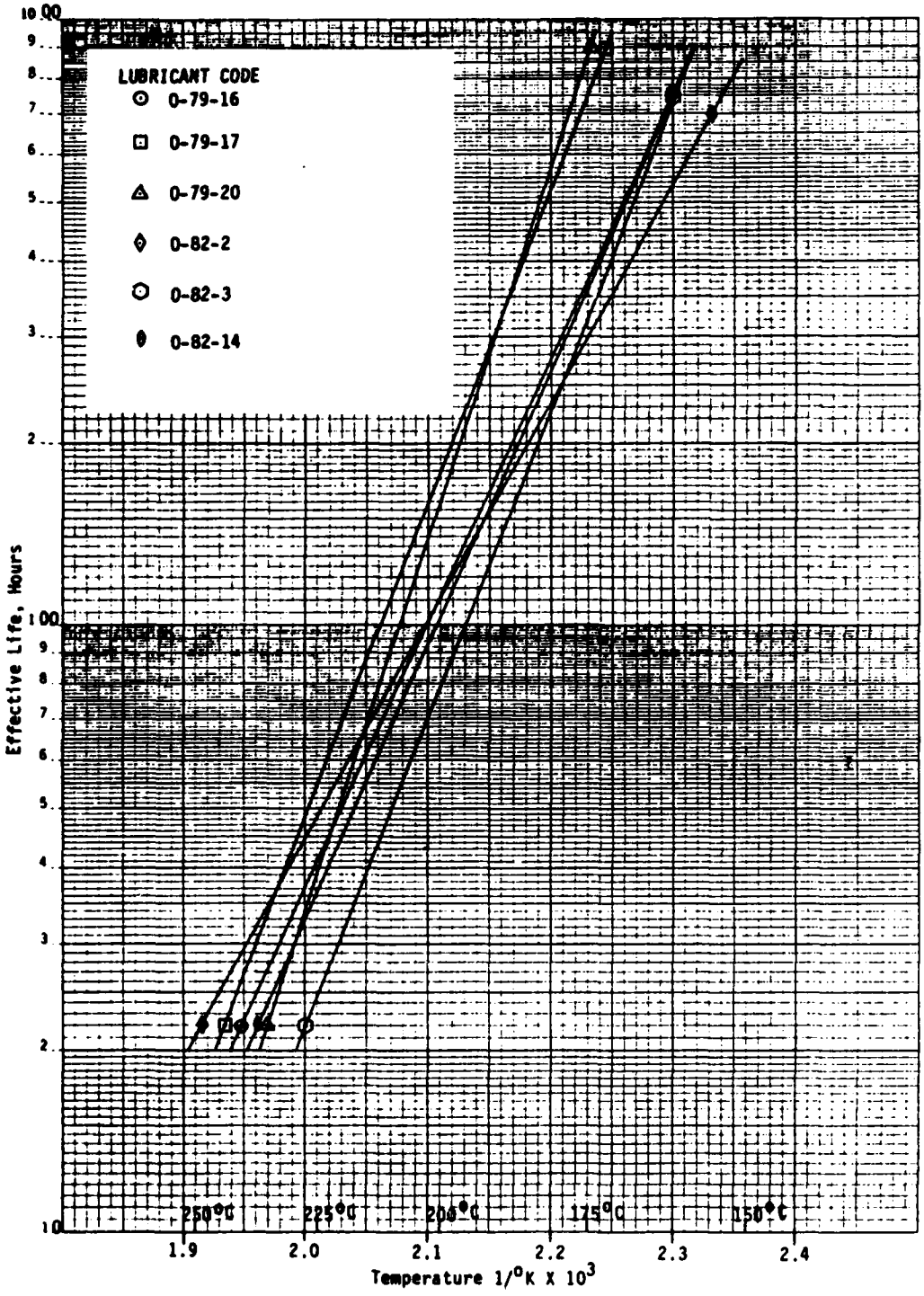


Figure 9. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Viscosity Increase Limit of 35%

TABLE 2

EFFECTIVE LUBRICANT LIFE
DERD METHOD NO. 9 (NO DILUTION)
ACIDITY INCREASE LIMITS

TAN Increase Limit of 1.0
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	2500	1100	1500	55	1400	140
200°C	100	140	72	16	79	24
225°C	7	26	10	6	7	6

TAN Increase Limit of 1.5
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	2800	2600	4000	79	1500	215
200°C	149	211	131	26	102	40
225°C	15	26	8	11	12	10

TAN Increase Limit of 3.0
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	3300	4400	11,000	105	1600	312
200°C	278	334	196	55	202	73
225°C	35	39	7	32	39	22

TABLE 3

EFFECTIVE LUBRICANT LIFE
DERD METHOD NO. 9 (NO DILUTION)
VISCOSITY INCREASE LIMITS

Viscosity Increase Limit of 15%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	162	98	292	181	143	196
200°C	51	34	80	59	38	49
225°C	20	14	27	23	12	16

Viscosity Increase Limit of 25%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	268	436	569	237	236	293
200°C	82	108	125	92	60	83
225°C	31	34	35	42	19	29

Viscosity Increase Limit of 35%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	376	749	885	303	324	364
200°C	112	180	163	112	80	104
225°C	41	55	40	49	25	37

relative ranking of the lubricants with respect to effective life depends upon the selected limiting value of viscosity increase as was shown for different limiting values of TAN.

Figures 10 thru 12 show Arrhenius plots for the six MIL-L-7808 type lubricants using volatilization loss as the limiting life criteria. These curves show less difference in effective life than that based on TAN increase values. Table 4 shows the difference in effective life for these lubricants at the test temperature of 175°C, 200°C, and 225°C. The data shown in Table 4 is similar to the data in Table 3 which is based on viscosity change except in most cases, the volatility loss provides slightly lower effective life values for all temperatures. It must be remembered that the Arrhenius data provided in Figures 4 to 12 are test method dependent and different values would be obtained for other types of oxidation testing. Data obtained using condensate return would tend to decrease volatility loss and increase TAN values. The use of metal corrosion test specimens could also affect lubricant stability.

In turbine engine operation periodic oil addition due to consumption would increase the effective life of the lubricant.[1] The portion, O_T of original oil remaining after engine running time, T, is given by

$$O_T = e^{-CT/V}$$

where C = average oil consumption

V = oil system capacity

T = time

Figure 13 shows the oil phase-out rate in turbine engines when the time (T) is expressed in terms of V/C. For example, a turbine engine lubricant system having a consumption of 0.3 pints per hour and a system capacity of 3 gallons would have a V/C value of 80. From Figure 13, 1% of

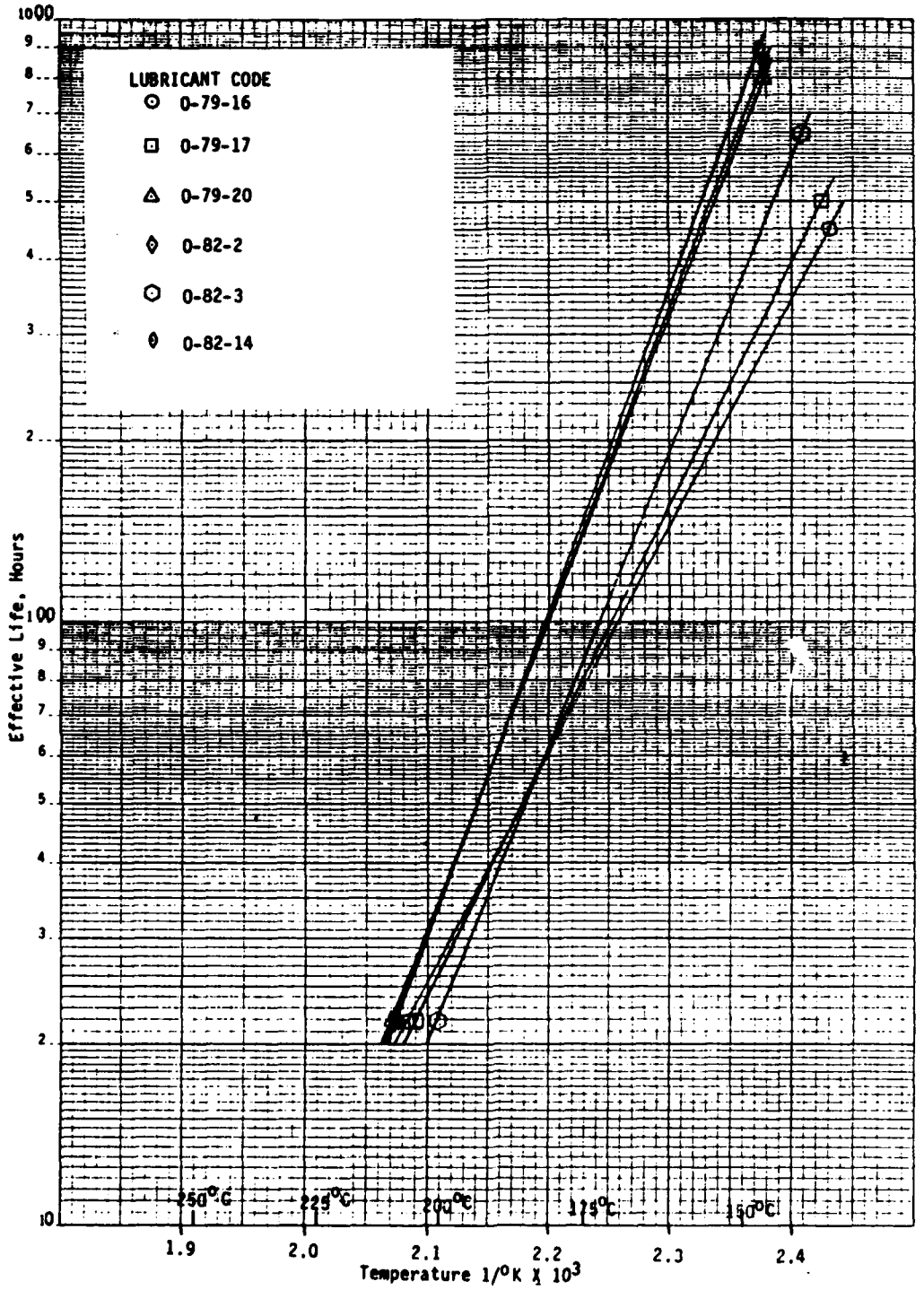


Figure 10. Effect of Temperature on Lubricant Life Using DERD Method N. 9, Volatilization Loss Limit of 15%

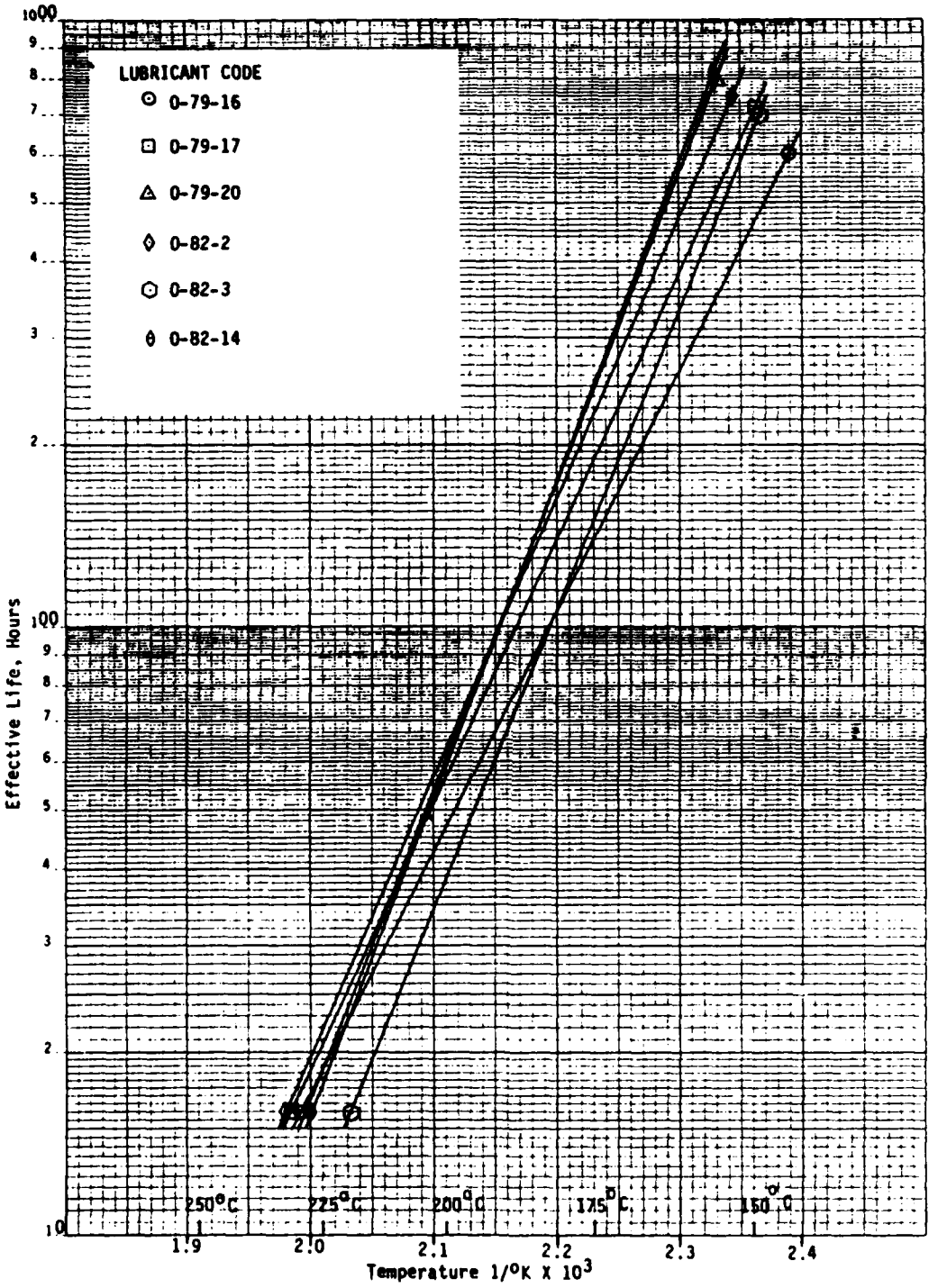


Figure 11. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Volatilization Loss Limit Of 25%

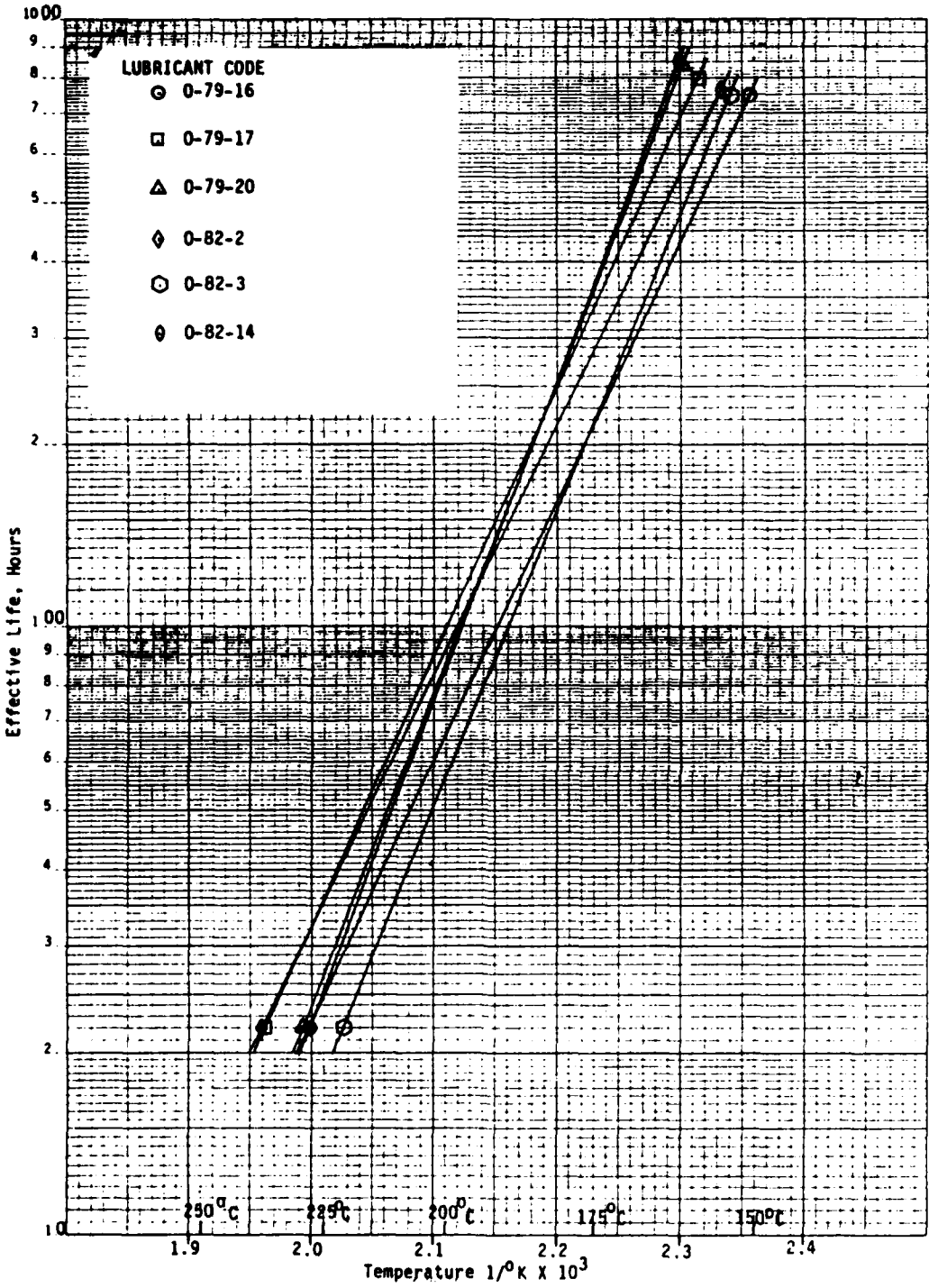


Figure 12. Effect of Temperature on Lubricant Life Using DERD Method No. 9, Volatilization Loss Limit of 35%

TABLE 4

EFFECTIVE LUBRICANT LIFE
DERD METHOD NO. 9 (NO DILUTION)
VOLATILIZATION LOSS LIMITS

Volatilization Loss Limit of 15%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-70-20	0-82-2	0-82-3	0-82-14
175°C	78	81	144	144	87	151
200°C	28	27	36	35	23	34
225°C	12	10	11	11	7	10

Volatilization Loss Limit of 25%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	142	190	250	227	153	253
200°C	47	57	61	64	39	60
225°C	19	21	19	22	13	18

Volatilization Loss Limit of 35%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	219	342	369	290	221	371
200°C	67	100	89	93	57	86
225°C	25	36	27	36	19	26

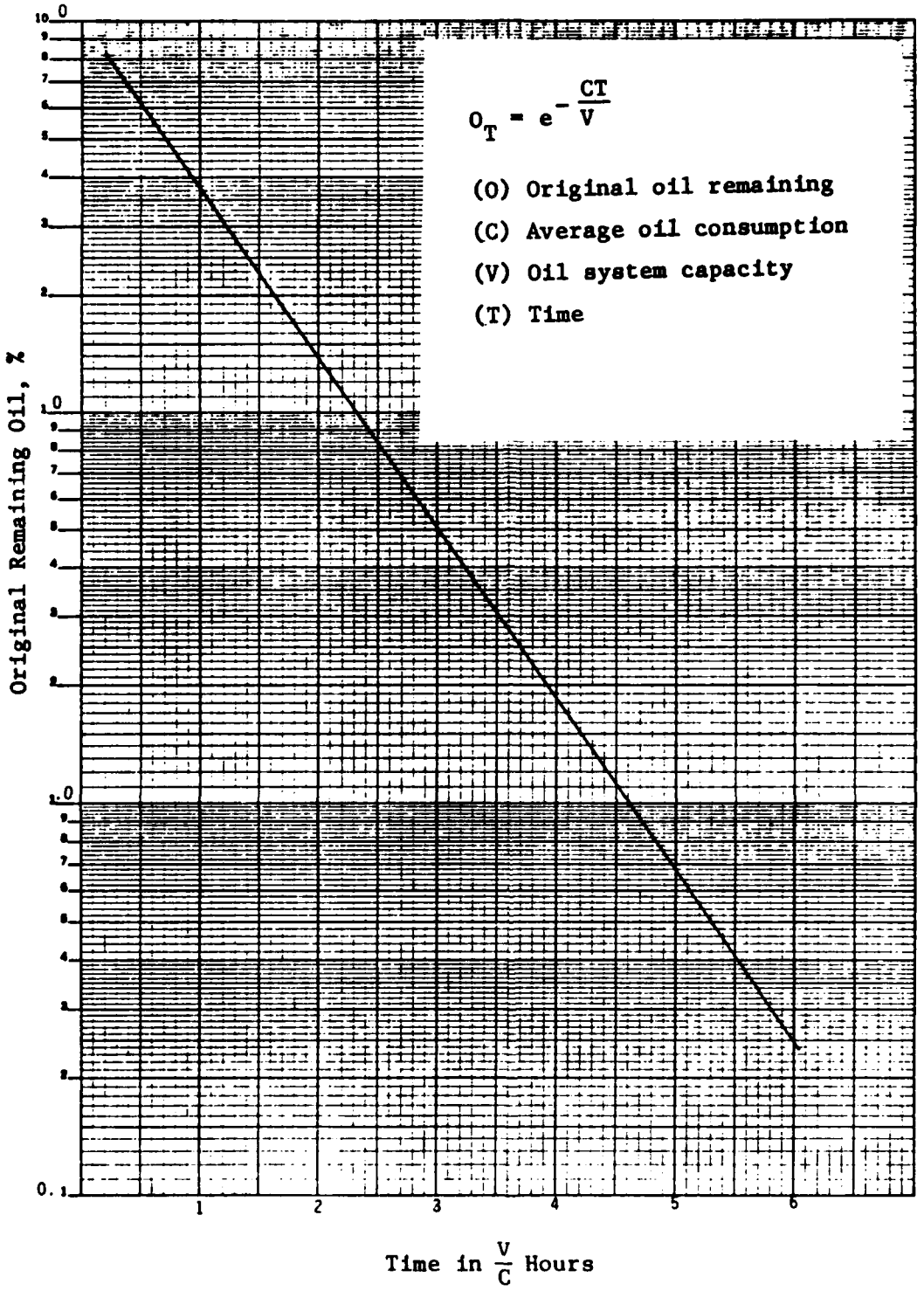


Figure 13. Oil Phase-out Rate in Turbine Engines

the original oil will remain after 4.5 V/C or 360 hours. This shows that oil consumption in turbine engines and volatility loss in oxidation testing can have very large effects on lubricant life.

Arrhenius plots from the Squires oxidative test data given in Appendix A Table A-1 could be developed considering volatility loss. Effect of volatility on TAN changes would be revised by dilution calculations while the viscosity increase values would be revised using ASTM method D 341 using viscosity-temperature charts for petroleum products.

Only small amounts of toluene insolubles were found for any of the six MIL-L-7808 lubricants even after severe lubricant degradation. As shown in Appendix A, Table A-1, 97% of the toluene insoluble measurements were below 0.04% weight and all values were below 0.1% weight.

The effect of additive content on lubricant stability is shown in Table 5 for 0.5, 1.0, 1.5 and 2.0% mixtures of phenyl-alpha-naphthylamine (PANA) and dioctyldiphenylamine (DODPA) and 2.0% mixtures of phenothiazine (PTZ) and dioctylphenothiazine (DOPTZ) in O-76-5 (trimethylolpropane heptanoate) ester and O-76-8 (di-2-ethylhexyl adipate). For the polyol ester O-76-5, PANA provided more improvement in effective lubricant life than any other antioxidant investigated. This additive also provided improved lubricant stability at 0.5 and 1.0% concentrations while 1.5% of DODPA was required before increased stability was obtained. Two factors can influence the effectiveness of these two antioxidants. The first is the 55.7% effective antioxidant property of DODPA relative to PANA based upon the amine radical and the molecular weight of 219 for PANA and 393 for DODPA. This should provide PANA with the better antioxidant capability compared to DODPA. The second factor is the higher volatility of PANA which would reduce its effectiveness compared to DODPA which remains longer in the stressed

TABLE 5

EFFECT OF ADDITIVE CONTENT ON LUBRICANT LIFE AT 205°C

Ester	Additive Content, % Wt									
	0.0		0.5		1.0		1.5		2.0	
	Hours to		Hours to		Hours to		Hours to		Hours to	
	TAN 1.5	Vis 15%	TAN 1.5	Vis 15%	TAN 1.5	Vis 15%	TAN 1.5	Vis 15%	TAN 1.5	Vis 15%
O-76-5										
PANA	2	1	70	60	136	130	220	156	226	130
DODPA	2	1	2	2	3	3	72	78	180	96
PTZ	2	1	-	-	-	-	-	-	48	48
DOPTZ	2	1	-	-	-	-	-	-	36	40
50/50 PANA DODPA	2	1	-	-	-	-	-	-	240	110
O-76-8										
PANA	1	1	2	36	3	36	3	36	9	36
DODPA	1	1	2	26	3	26	3	26	2	24
PTZ	1	1	-	-	-	-	-	-	2	25
DOPTZ	1	1	-	-	-	-	-	-	2	28
50/50 PANA DODPA	1	1	-	-	-	-	-	-	3	24

lubricant. The data in Table 5 does not show that maximum oxidative stability has been achieved with 2.0% concentrations. Table 5 also shows that 1% PANA plus 1% DODPA in O-76-5 provides about the same increase in ester stability as was obtained with either 2.0% of PANA or 2.0% DODPA.

PTZ showed greater improvement in stability compared to DOPTZ which again may be caused by the difference in the effective concentration due to the lower molecular weight of the PTZ which functions as the antioxidant.

The diester O-76-8 shows much less stability with either antioxidant compared to ester O-76-5. However, two differences are noted in the effects of antioxidants for this ester. First, very little increase in stability was obtained using any additive based upon the hours required to reach a TAN value of 1.5. Secondly, both PANA and DODPA increased the stability at 0.5% concentration based upon the hours required to reach a viscosity increase of 15%. Above this concentration, no improvement in stability was obtained and at 2.0% levels, all the various antioxidants gave about the same improvement in ester stability based on viscosity increase.

Evaluation of a second di-2-ethylhexyl adipate ester, O-77-1 showed considerable differences when compared to the di-2-ethylhexyl adipate O-76-8 in stability studies. Analysis of the polyol ester and the two diesters by gas chromatography showed the following compositions:

O-76-5: 89% Trimethylolpropane heptanoate (TMPH) with the remaining 11% being other mixed esters of trimethylolpropane.

O-76-8: 90% Di-2-ethylhexyl adipate, 8.3% TMPH ester, 1.5% 2-ethyl hexanol and other unidentified trace impurities.

O-77-1: 99% Di-2-ethylhexyl adipate, with trace amounts of other esters and 2-ethylhexanol.

Table 6 shows the effect of 1.0% PANA plus 1.0% DODPA on ester stability at

190°C and 205°C for the three esters with each containing 1.0% PANA and 1.0% DODPA. This data shows ester O-76-5 to have much better oxidative stability than either diester at 205°C. At 190°C much less difference is seen between O-76-5 and O-77-1 than between O-76-5 and O-76-8. A large difference is seen in the stability of the two diesters at both temperatures.

TABLE 6

EFFECT OF 1.0% PANA PLUS 1.0 % DODPA
ON ESTER STABILITY AT 190°C AND 205°C

	Temperature			
	190° C		205° C	
	Hours to TAN 1.5	Hours to Vis change 15%	Hours to TAN 1.5	Hours to Vis change 15%
O-76-5	120 +	120 +	240	110
O-76-8	2	48	3	24
O-77-1	96	108	24	40

The effect of the impurities identified in ester O-76-8 was investigated by adding 8.3% wt O-76-5 (TMPH ester), 1.5% 2-ethylhexanol and 1.0% wt PANA to O-77-1 and conducting a 24 hour 205°C oxidation test on the mixture. A comparison of test data obtained on this ester blend with O-76-8 plus 1.0% PANA and O-77-1 with 1.0% PANA is given in Table 7.

TABLE 7

EFFECT OF IDENTIFIED IMPURITIES IN O-76-8 ON ESTER STABILITY
(24 HOUR, 205°C TEST)

	O-76-8 PLUS 1.0% PANA	O-77-1 PLUS 1.0% PANA	O-77-1 Plus Impurities and 1.0% PANA
Weight Loss, %	23.4	12.4	14.4
COBRA Value	78	48	16
TAN Increase	9.55	1.18	1.08
Viscosity at 100°C, % Increase	12.7	-0.8	6.4

This data shows that the identified impurities in O-76-8 are not the cause for its much reduced oxidative stability and that very small quantities of unknown impurities in esters can greatly affect the improvement in ester stability using antioxidants. This is shown by the difference in TAN and viscosity increases between O-76-8 plus 1.0% PANA and O-77-1 plus impurities (identified in O-76-8) and containing 1.0% PANA.

Toluene insoluble content of the esters and ester-additive mixtures was generally slightly higher than that found for the six MIL-L-7808 lubricants with 80% being 0.05% wt or below and 98% being below 0.13%. The highest toluene insoluble value was 0.34% wt for diester O-76-8 containing 2.0% PANA after 48 hours at 205°C test temperature. Tube deposits varied from tacky material to hard coke. These deposits did not appear to be related to test time, test temperature or degree of lubricant degradation. Ester-additive combination appeared to be the main factor in the formation of tube deposits. All esters containing PANA, PTZ and DOPTZ produced various types of tube deposits while the same esters blended with DODPA did not produce tube deposits under any condition including severe lubricant degradation.

f. Summary

Effective lubricant lives of six MIL-L-7808 lubricants were established using different levels of limiting values for changes in TAN, viscosity or weight loss as the maximum permissible degree of degradation. Arrhenius plots were developed describing effective life as a function of temperature for each selected limiting value of degradation. Four of the six lubricants studied were found to have similar lubricant effective lives while the remaining two had much lower effective lives. Relative ranking of the lubricants within each group depended to some degree on the criteria for defining maximum permissible degradation.

The oxidative stability study of the polyol ester trimethylolpropane heptanoate (TMPH) and two diesters (both di-2-ethylhexyl adipate esters) when blended with various antioxidants showed the TMPH ester to have superior oxidative stability when compared to either of the di-2-ethylhexyl adipate esters using the specified test procedures and evaluation criteria. A significant difference was observed between the two diesters which was shown to be due to trace impurities in one diester which could not be identified through various analyses. This shows that a formulated lubricant's oxidative stability can be decreased significantly due to trace amounts of impurities.

g. Future Effort

Oxidative stability studies of selected MIL-L-7808 lubricants, MIL-L-23699 lubricants and 4 cSt candidate lubricants will be continued using air and nitrogen without condensate return. Studies will also include stability testing using air and condensate return. Evaluation of this data will be made and compared with corrosion-oxidation data obtained for these fluids using Federal Test Method Standard 791, Method 5307. Oxidative stability studies will also be made on higher temperature fluids such as polyphenyl ethers and perfluoro polyethers.

2. LUBRICANT CONFINED HEAT STABILITY

a. Introduction

The purpose of this study was to determine the confined heat stability of selected turbine engine lubricants under various temperatures, and to provide a measure of their stability in terms of effective lubricant life. The effective life was determined by selecting limiting values for various physical and chemical properties, and monitoring these properties during lubricant stressing. Properties monitored included weight loss due to volatility, acidity increase, viscosity increase, toluene insolubles, electrochemical properties, and composition. Only the acidity increases and viscosity increases were significant enough to provide data required for the development of Arrhenius plots, which graphically depict the effective life of a lubricant as a function of temperature.

b. Test Apparatus

The thermal or confined heat stability test apparatus consists of a stainless steel vessel shown in Figure 14 and a heating bath containing polyphenyl ether as the heat medium.

Prior to each test the vessels were degreased with V.M.P. naphtha. The vessels were then polished with a cotton pad which had been dampened in heptane and dipped into 3 micron size corundum powder. The condenser tube, aluminum washer and vessel cover were cleaned in the same manner. A small brush with the scouring mixture was used to clean inside the vent tube. The entire vessel was then rinsed and washed with Alconox detergent. Using distilled water and isopropanol as final rinses, the vessels were then blown dry with clean dry air.

c. Test Procedure

The vessel was filled to approximately two-thirds of its volume with

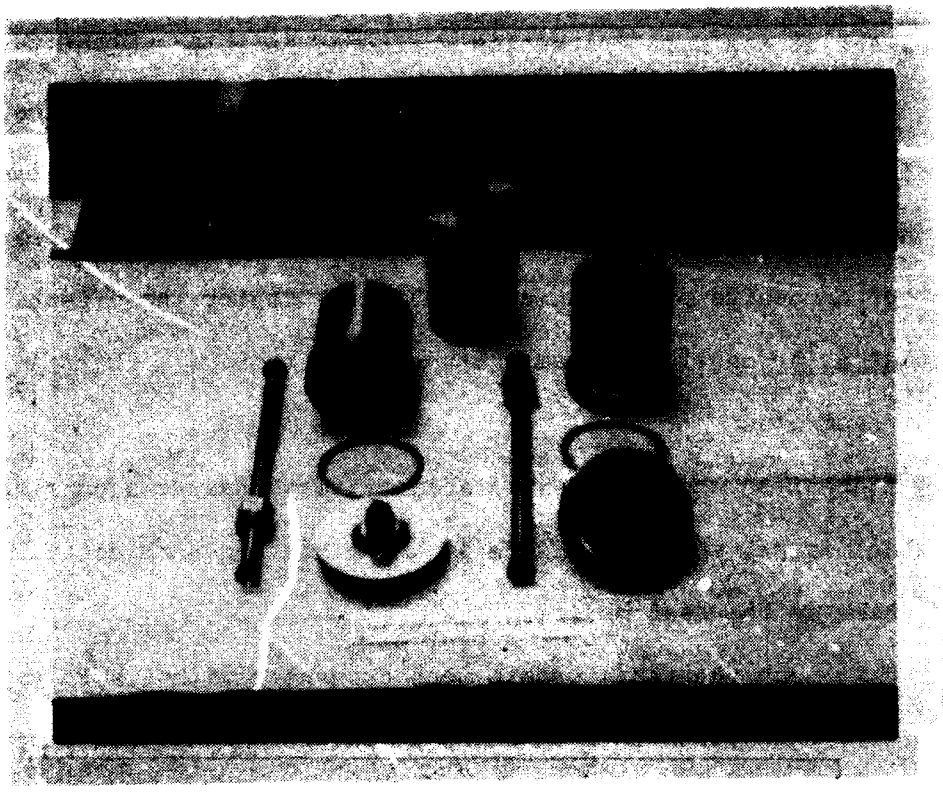


Figure 14. Confined Heat Test Assembly

85 ml of the sample fluid. The lid, aluminum washer, and condenser tube was assembled, screwed firmly into place, and weighed to the nearest 0.1 gram. The apparatus was placed into the heating bath which had been heated to the required test temperature. At the end of the specified test period, the vessels were removed and allowed to cool to room temperature.

The vessels were then wiped with a lint-free towel, washed with V.M.-P. naphtha and reweighed. The percent weight loss was calculated and the test assembly was opened and inspected for deposits. The test fluid was poured into an amber bottle and sealed with a Polyseal cap.

The total acid number, viscosity, toluene insolubles and electrochemical properties were determined as described in Section II.1.c.

d. Test Lubricants and Test Conditions

A total of 12 lubricants, three esters and 32 ester-additive blends containing various percentages and combinations of additives were evaluated in this study. Thermal stressing of some of the ester-additive mixtures was conducted for providing stressed samples for deposition studies. Table 1, Section II.1.d presents a listing of the lubricants including a description of each fluid with the exception of O-79-18 which is a MIL-L-23699 lubricant.

Lubricants were stressed at temperatures ranging from 175°C to 215°C with test durations ranging from 24 hours to 577 hours depending upon the test temperature and the specific fluid being tested. Testing of each fluid was discontinued after severe degradation had occurred. Total acid numbers and COBRA measurements were conducted on the same day the samples were removed from the test bath since these properties can change with time after being stressed.

e. Results and Discussion

Lubricant life of the thermally stressed MIL-L-7808 lubricants were

developed using the following criteria as limiting values.

Total Acid Number increases of 2.0 and 4.0

Viscosity Increases at 100°C of 1%, 3% and 5%

No volatility loss criteria for effective lubricant life of the stressed lubricants were considered since very little lubricant loss occurs. Appendix A, Table A-2 provides all the Squires Confined Heat test data developed during this study and from which the Arrhenius plots shown in this report were developed.

Figures 15 and 16 show Arrhenius plots for six MIL-L-7808 type lubricants using TAN increases as the limiting life criteria. For both levels of TAN increase limiting values, significant differences exist between the lubricants. Figures 17 through 19 show Arrhenius plots of the six lubricants using viscosity increases as the limiting life criteria. For all three levels of viscosity increase limiting values, significant differences exist between the lubricants even to a greater degree than that observed using TAN increase limiting values.

Differences in effective life of the six lubricants are also shown in Table 8 for test temperatures of 175°C, 200°C and 225°C for two limiting TAN increase values. This data shows that lubricants O-82-2 and O-82-14 have much lower thermal stability than the other lubricants. No difference occurred in relative ranking of the lubricants between the two limiting TAN values which occurred with oxidative stressing.

Differences in effective life of the six lubricants based upon viscosity change is shown in Table 9 for test temperatures of 175°C, 200°C and 225°C for three limiting viscosity increase values. This data shows that the thermal stability varies significantly among the six lubricants and relative ranking of the lubricants depends to some extent upon the level of

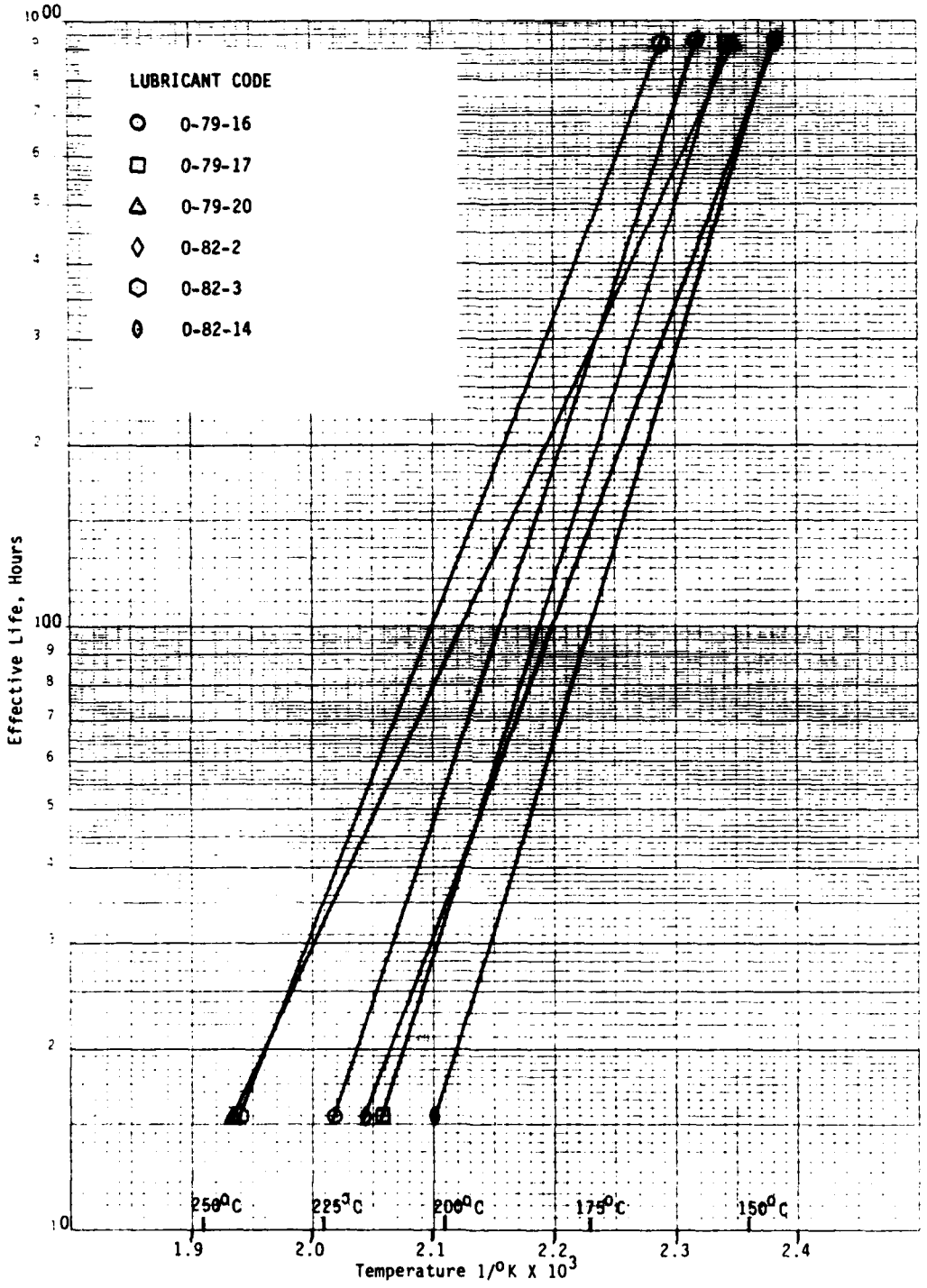


Figure 15. Effect of Temperature on Lubricant Life Using DERD Method No. 1 (Confined Heat Stability), Total Acid Number Increase Limit of 2.0

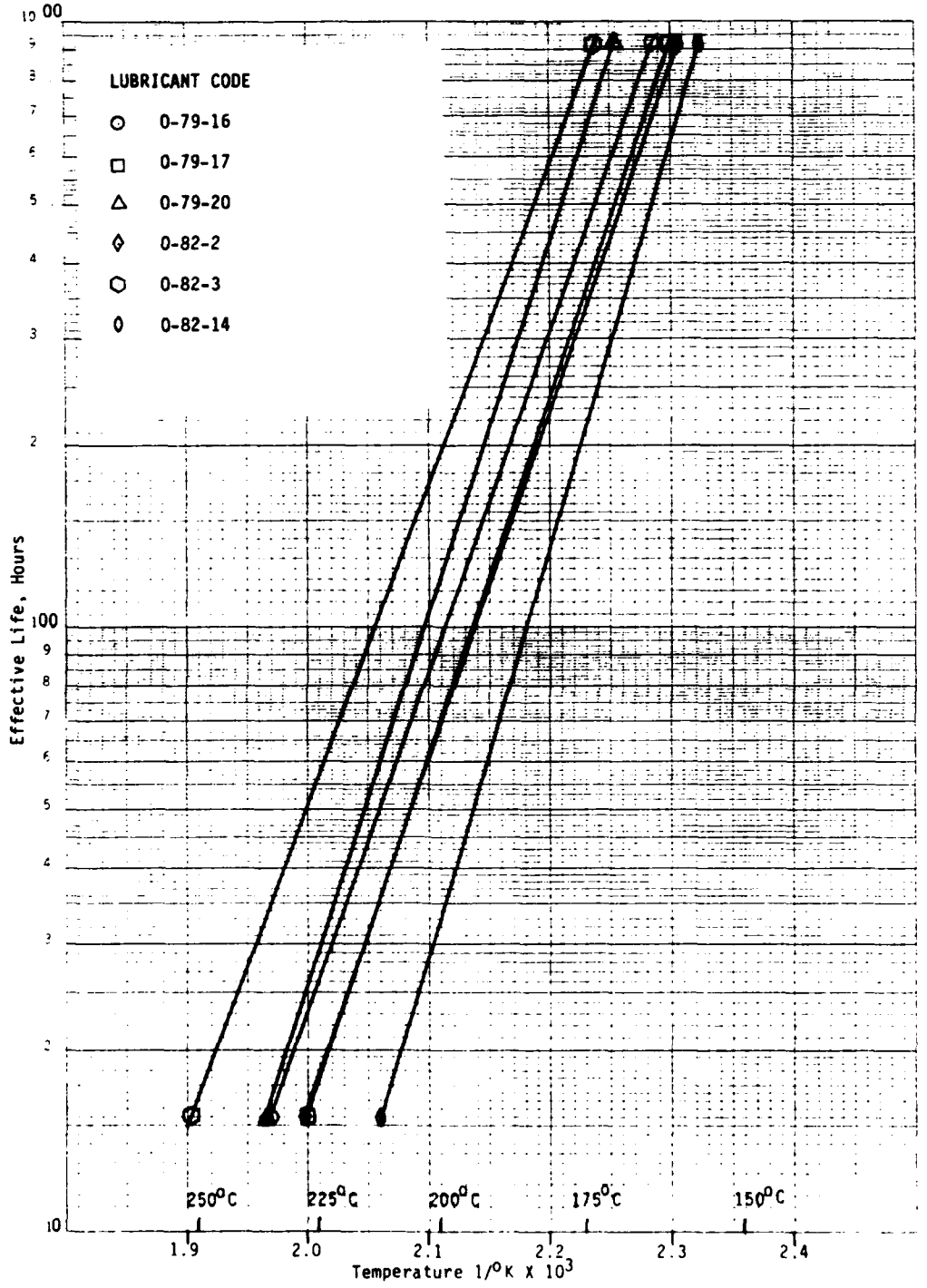


Figure 16. Effect of Temperature on Lubricant Life Using DERD Method No. 1 (Confined Heat Stability), Total Acid Number Increase Limit of 4.0

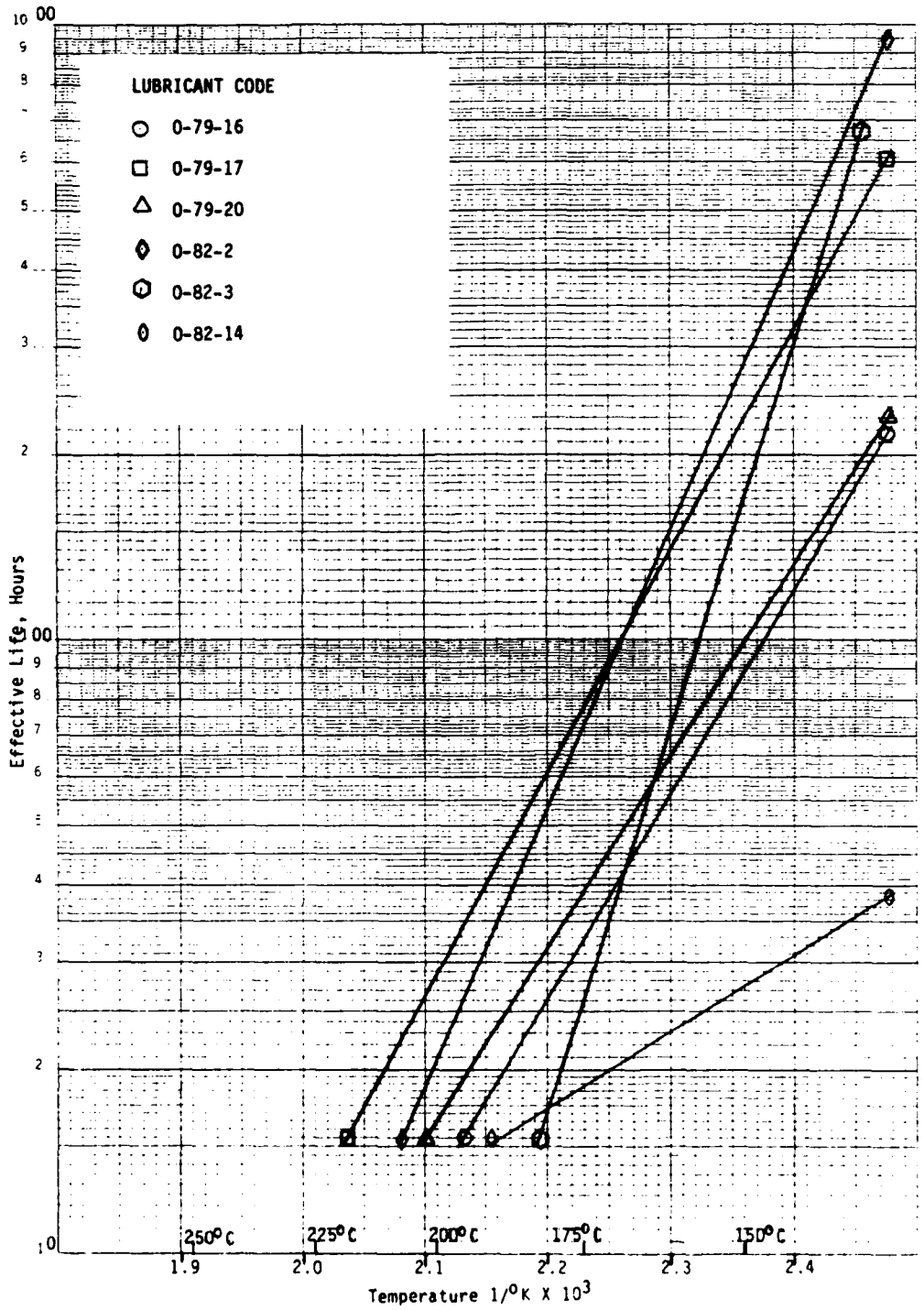


Figure 17. Effect of Temperature on Lubricant Life Using DERD Method No. 1 (Confined Heat Stability), Viscosity Increase Limit of 1%

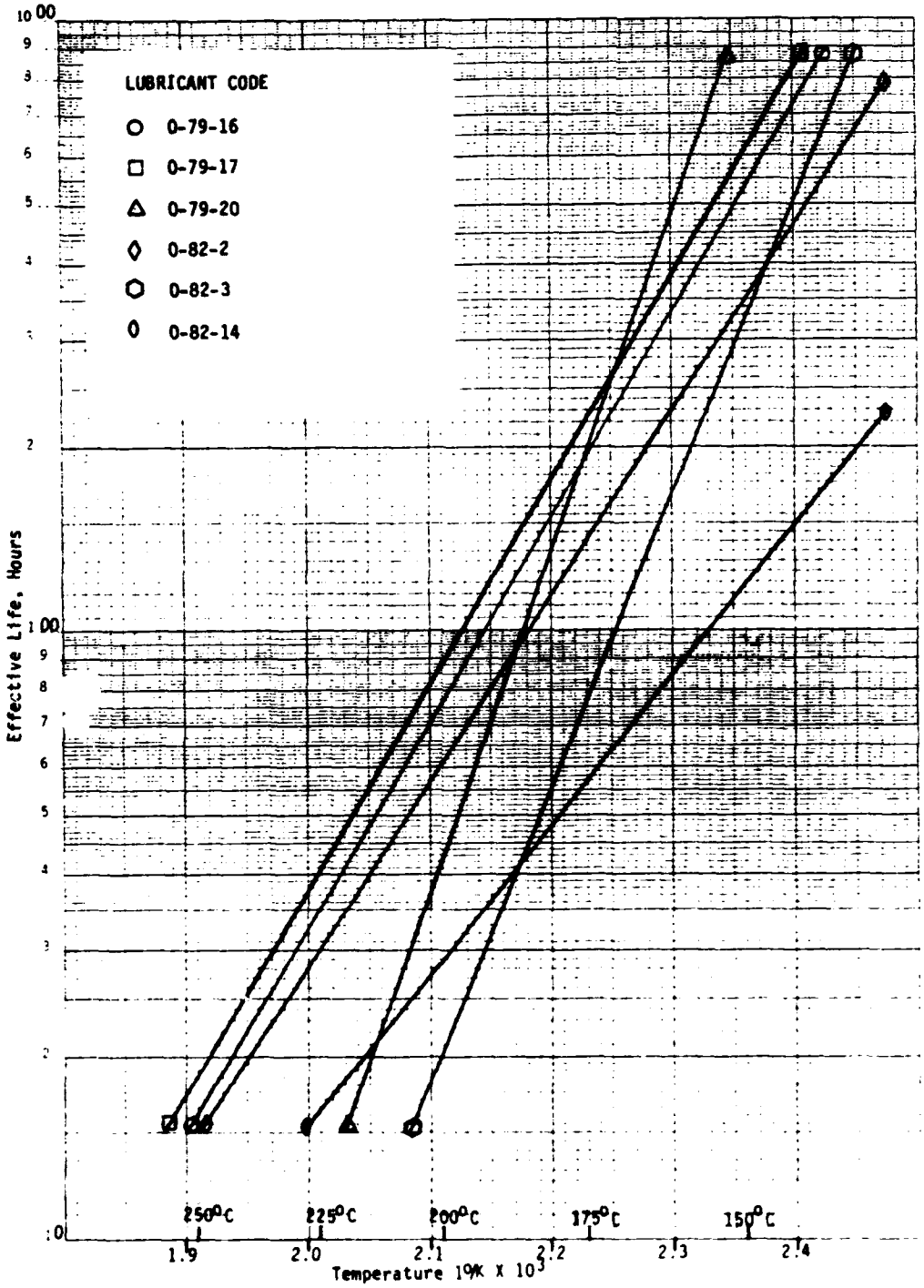


Figure 18. Effect of Temperature on Lubricant Life Using DERD Method No. 1 (Confined Heat Stability), Viscosity Increase Limit of 3%

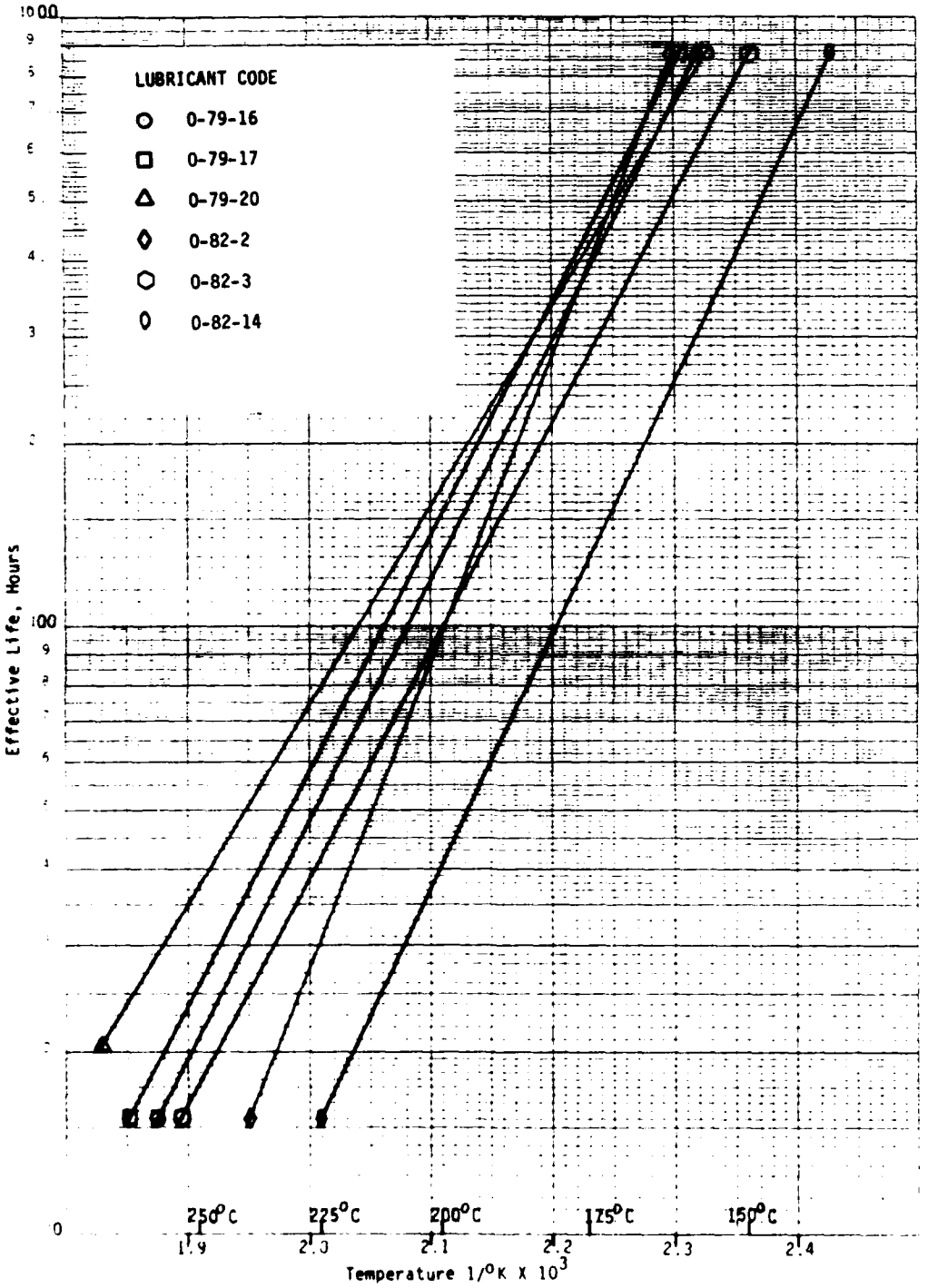


Figure 19. Effect of Temperature on Lubricant Life Using DERD Method No. 1 (Confined Heat Stability), Viscosity Increase Limit of 5%

TABLE 8

EFFECTIVE LUBRICANT LIFE
DERD METHOD NO.1 (NO DILUTION)
ACIDITY INCREASE LIMITS

TAN Increase Limit of 2.0
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	282	186	288	148	460	101
200°C	55	33	89	35	113	18
225°C	14	8	33	11	35	4

TAN Increase Limit 4.0
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	450	363	658	339	844	214
200°C	97	71	123	70	196	33
225°C	27	18	30	19	58	7

TABLE 9

EFFECTIVE LUBRICANT LIFE
DERD METHOD NO. 1 (NO DILUTION)
VISCOSITY INCREASE LIMITS

Viscosity Increase Limit of 1%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	33	79	39	74	27	19
200°C	13	29	17	21	5	13
225°C	1	12	8	7	1	10

Viscosity Increase Limit of 3%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	194	223	198	223	77	57
200°C	76	88	43	63	20	29
225°C	35	40	12	22	7	16

Viscosity Increase Limit of 5%
Lubricant Life, Hours

Lubricant	0-79-16	0-79-17	0-79-20	0-82-2	0-82-3	0-82-14
175°C	390	447	426	413	281	130
200°C	132	154	171	99	100	41
225°C	53	63	80	30	42	16

viscosity increase. The increases in viscosity must be due mostly to degradation since only very little weight loss occurred during testing.

Very small amounts of toluene insolubles were found for any of the six MIL-L-7808 lubricants even after severe degradation. As shown in Appendix A, Table A-2, 98% of the toluene insoluble measurements were below 0.03% weight and the maximum value being 0.05% weight.

The effect of confined heat testing of the esters O-76-5 (TMPH), O-76-8 (D2EHA) and O-77-1 (D2EHA)is shown in Table 10 below.

TABLE 10

EFFECTIVE ESTER LIFE, DERD METHOD NO 1.
(LUBRICANT LIFE, HOURS)

TAN Increase Limit of 4.0

	175°C	190°C	205°C
O-76-5	44	34	32
O-76-8	41	23	19
O-77-1	-	-	>72

Viscosity Increase Limit of 5%

	175°C	190°C	205°C
O-76-5	>72	48	21
O-76-7	>72	57	53
O-77-1	-	-	72

The data in Table 10 shows that the di-2-ethylhexyl adipate ester O-77-1 has much better confined heat stability than either of the other two esters for both effective lubricant life limiting values of changes in TAN and viscosity.

Table 11 shows the effects of additives on the confined heat stability of the three esters when blended with 2% PANA, 2% DODPA and 1% PANA plus 1% DODPA.

TABLE 11

EFFECTS OF ANTIOXIDANTS ON ESTER LUBRICANT LIFE
DERD METHOD NO. 1, 205°C TEST TEMPERATURE

Ester	No Additive	2% PANA	2% DODPA	1% PANA 1% DODPA
	TAN Increase Limit of 4.0 Lubricant Life, Hours			
O-76-5	32	50	55	72
O-76-8	19	26	22	22
O-77-1	>72	-	-	166
	Viscosity Increase Limit of 5%			
O-76-5	21	>96	>72	>96
O-76-8	53	48	60	60
O-77-1	72	-	-	178

The data shown in Table 11 is not what would be expected especially between the polyol ester O-76-5 and the diester O-77-1. This data appears to show that not only ester purity but the breakdown and subsequent reactions of these materials can greatly affect the ester stability.

The toluene insoluble content of some ester-additive mixtures was much higher than the amounts found in the six MIL-L-7808 lubricants, especially with ester O-76-8 and PANA and O-76-5 and PTZ. All mixtures of O-76-8 plus PANA gave toluene insoluble values ranging from 0.05 to 1.25% weight. All mixtures of O-76-5 with PANA gave values below 0.03%. Both esters O-76-5 and O-76-8 gave values below 0.03% with all percentages of

DODPA. Ester O-76-5 with 2% PTZ gave a toluene insoluble content of 0.66% after 48 test hours while O-76-8 gave a value 0.10% after 48 test hours. DOPTZ gave much less toluene insolubles for both O-76-5 (0.06%) and O-76-8 (0.03%). This would be expected due to the increased solubility of the additive breakdown products due to the octyl groups. Test chamber deposits varied from none to black coke and in most cases followed the pattern of toluene insoluble content with the various ester-additive mixtures.

f. Summary

Effective lubricant lives of six MIL-L-7808 lubricants under confined heat (thermal) conditions were determined using different levels of limiting values for changes in TAN and viscosity as the maximum permissible degree of degradation. Arrhenius plots were developed describing effective life as a function of temperature. Both limiting values of TAN increase and viscosity increase showed significant differences between the confined heat stability of the six lubricants. The difference in effective life of the lubricants was greater based upon viscosity change and relative ranking of the lubricants with respect to effective life depended to some extent upon the selected viscosity increase limiting value.

Confined heat stability testing of ester-additive mixtures showed unexpected higher confined heat stability for the diester O-77-1 (di-2-ethylhexyl adipate) than the polyol ester O-76-5 (trimethylolpropane heptanoate) with and without antioxidants.

g. Future Effort

Confined heat study of the MIL-L-23699 lubricants and the 4 cSt candidate lubricant will be continued providing data for developing Arrhenius plots describing lubricant effective life as a function of temperature.

3. CORROSIVENESS AND OXIDATION STABILITY

a. Introduction

Corrosion and oxidation testing of selected lubricants was conducted to establish correlation with other laboratories and for providing stressed lubricants for other studies.

b. Test Apparatus.

Test apparatus is described by Federal Test Method Std. 791, Method 5307 and will not be repeated in this report.

c. Test Procedure

Test Method 5307, basic method, requiring intermediate sampling was used for MIL-L-7808 lubricant testing. Modification of Test Method 5307 was made when stressing the lubricants required for other studies. This modification consisted of using 350 ml samples instead of the normal 200 ml samples and not determining metal test specimen loss although metal test specimens were used during the test. A change in sampling frequency was also made for all tests.

d. Results and Discussion

Corrosion and oxidation testing was conducted on two MIL-L-7808 lubricants O-79-16 and O-79-17 using Method 5307.1 with intermediate sampling. Duplicate tests were conducted for each lubricant with the data obtained from the four tests being given in Tables 12 through 15. A summary of this data is given in Table 16.

Good test repeatability was obtained for each lubricant including both the intermediate and final sampling data. Both lubricants showed good oxidative stability and conform to MIL-L-7808J. Although no corrosion-oxidation test data generated by another laboratory is available for these specific lots of lubricants, comparison of the test data with the manufacturers' data for several other lots of the same lubricant formulations

TABLE 12

RESULTS OF REFLUX CORROSION/OXIDATION TEST
LUBRICANT O-79-16
TEST A

SAMPLE DATA

	Viscosity, cSt/40°C	40°C Visc. Increase, %	Viscosity, cSt/100°C	100°C Visc. Increase, %	Neut. No. mg KOH/g	Neut. No. Increase	COBRA
Initial	12.35	-	3.16	-	0.20	-	3
18 hr.	13.09	6.0	-	-	-	0.13	13
24 hr.	13.20	6.9	-	-	-	0.37	20
42 hr.	13.48	9.2	-	-	-	0.73	42
48 hr.	13.54	9.6	-	-	-	0.77	47
66 hr.	13.70	10.9	-	-	-	0.84	65
72 hr.	13.78	11.6	-	-	-	1.22	70
90 hr.	14.00	13.4	-	-	-	1.41	100
96 hr.	14.24	15.3	3.47	9.8	2.16	1.96	104

METAL SPECIMEN DATA

Metal Type	Wt. Change mg/cm^2	Color and Appearance (visual/20X mag.)
Aluminum	0.0	no change/no change
Silver	-0.1	no change/no change
Bronze	+0.1	tarnish/tarnish
Mild Steel	0.0	blue/blue
M-50 Steel	-0.1	purplish-blue/purplish-blue
Magnesium	-0.1	sl. stain/sl. stain
Titanium	-0.1	sl. stain/sl. stain

TEST CELL DATA

Sludge, Vol. %	0.0
Wt. Loss, %	3.6
Tube deposits	None
Condenser Deposits	None

TEST CONDITIONS

Sample Vol.	200 ml
Air Rate	10 l/hr.
Test Temp.	200°C

TABLE 13

RESULTS OF REFLUX CORROSION/OXIDATION TEST
LUBRICANT O-79-16
TEST B

SAMPLE DATA

	Viscosity, cSt/40°C	40°C Visc. Increase, %	Viscosity, cSt/100°C	100°C Visc. Increase, %	Neut. No. mg KOH/g	Neut. No. Increase	COBRA
Initial	12.35	-	3.16	-	0.20	-	3
18 hr.	13.01	5.3	-	-	-	0.09	12
24 hr.	13.17	6.6	-	-	-	0.30	19
42 hr.	13.40	8.5	-	-	-	0.78	42
48 hr.	13.51	9.4	-	-	-	0.75	47
66 hr.	13.73	11.2	-	-	-	1.12	67
72 hr.	13.77	11.5	-	-	-	1.21	69
90 hr.	14.02	13.5	-	-	-	1.47	101
96 hr.	14.19	14.9	3.47	9.8	1.98	1.78	105

METAL SPECIMEN DATA

Metal Type	Wt. Change mg/cm²	Color and Appearance (visual/20X mag.)
Aluminum	0.0	no change/no change
Silver	-0.1	no change/no change
Bronze	+0.1	tarnish/tarnish
Mild Steel	0.0	blue/blue
M-50 Steel	0.0	bluish-purple/bluish-purple
Magnesium	-0.1	sl. stain/sl. stain
Titanium	-0.1	no change/no change

TEST CELL DATA

Sludge, Vol. %	0.0
Wt. Loss, %	1.6
Tube Deposits	None
Condenser Deposits	None

TEST CONDITIONS

Sample Vol.	200 ml
Air Rate	10 l/hr.
Test Temp.	200°C

TABLE 14

RESULTS OF REFLUX CORROSION/OXIDATION TEST
LUBRICANT O-79-17
TEST A

SAMPLE DATA

	Viscosity, cSt/40°C	40°C Visc. Increase, %	Viscosity, cSt/100°C	100°C Visc. Increase, %	Neut. No. mg KOH/g	Neut. No. Increase	COBRA
Initial	13.40	-	3.35	-	0.08	-	2
18 hr.	14.69	9.6	-	-	-	0.32	31
24 hr.	14.66	9.4	-	-	-	0.42	42
42 hr.	14.96	11.6	-	-	-	0.92	66
48 hr.	15.06	12.4	-	-	-	0.82	74
66 hr.	15.27	14.0	-	-	-	1.05	90
72 hr.	15.30	14.2	-	-	-	1.06	95
90 hr.	15.54	16.0	-	-	-	1.26	114
96 hr.	15.66	16.9	3.66	9.2	1.38	1.30	119

METAL SPECIMEN DATA

Metal Type	Wt. Change mg/cm²	Color and Appearance (visual/20X mag.)
Aluminum	0.0	no change/no change
Silver	0.0	no change/no change
Bronze	0.0	tarnish + purple stain/ tarnish + purple stain
Mild Steel	+0.1	blue green/blue green
M-50 Steel	0.0	purplish blue/purplish-blue
Magnesium	0.0	no change/no change
Titanium	0.0	no change/no change

TEST CELL DATA

Sludge, Vol. %	0.0
Wt. Loss, %	2.9
Tube Deposits	None
Condenser Deposits	None

TEST CONDITIONS

Sample Vol.	200 ml
Air Rate	10 l/hr.
Test Temp.	200°C

TABLE 15

RESULTS OF REFLUX CORROSION/OXIDATION TEST
LUBRICANT O-79-17
TEST B

SAMPLE DATA

	Viscosity, cSt/40°C	40°C Visc. Increase, %	Viscosity, cSt/100°C	100°C Visc. Increase, %	Neut. No. mg KOH/g	Neut. No. Increase	COBRA
Initial	13.40	-	3.35	-	0.08	-	2
18 hr.	14.36	7.2	-	-	-	0.40	33
24 hr.	14.69	9.6	-	-	-	0.53	44
42 hr.	15.01	12.0	-	-	-	0.96	64
48 hr.	15.08	12.5	-	-	-	0.98	73
66 hr.	15.26	13.9	-	-	-	1.09	89
72 hr.	15.33	14.4	-	-	-	1.30	97
90 hr.	15.49	15.6	-	-	-	1.34	121
96 hr.	15.56	16.1	3.67	9.5	1.40	1.32	126

METAL SPECIMEN DATA

Metal Type	Wt. Change mg/cm²	Color and Appearance (visual/20X mag.)
Aluminum	0.0	no change/no change
Silver	0.0	no change/no change
Bronze	0.0	tarnish + purple stain/ tarnish + purple stain
Mild Steel	0.0	blue/blue
M-50 Steel	0.0	purplish blue/purplish blue
Magnesium	0.0	no change/no change
Titanium	0.0	no change/no change

TEST CELL DATA

Sludge, Vol. %	0.0
Wt. Loss, %	1.7
Tube Deposits	None
Condenser Deposits	None

TEST CONDITIONS

Sample Vol.	200 ml
Air Rate	10 l/hr.
Test Temp.	200°C

TABLE 16

SUMMARY OF CORROSION AND OXIDATION TEST DATA

(Fed-STD-791 Method 5307.1, Intermediate Sampling)

	Test A	Test B	Average
	Lubricant O-79-16		
Neut. No. Change	1.96	1.78	1.87
Viscosity, 40°C (Initial) cSt	12.35	12.35	12.35
Viscosity, 40°C, % Change	15.3	14.9	15.1
Viscosity, 100°C, (Initial), cSt	3.16	3.16	3.16
Viscosity, 100°C, % Change	9.8	9.8	9.8
COBRA Reading (Final)	104	105	105
Oil Loss, % Weight	3.6	1.6	2.6
Sludge, % Volume	0.0	0.0	0.0
Tube Deposits	None	None	None
Corrosion of Metals, mg/cm^2	<0.2	<0.2	<0.2
	Lubricant O-79-17		
Neut. No. Change	1.30	1.32	1.31
Viscosity, 40°C (Initial) cSt	13.40	13.40	13.40
Viscosity, 40°C, % Change	16.9	16.1	16.5
Viscosity, 100°C, (Initial), cSt	3.35	3.35	3.35
Viscosity, 100°C, % Change	9.2	9.5	9.3
COBRA Reading (Final)	119	126	123
Oil Loss, % Weight	2.9	1.7	2.3
Sludge, % Volume	0.0	0.0	0.0
Tube Deposits	None	None	None
Corrosion of Metals, mg/cm^2	<0.2	<0.2	< 0.2

showed good test reproducibility. As shown in Tables 12 through 15 intermediate sampling was accomplished at 18, 24, 42, 48, 66, 72, 90 and 96 test hours instead of 16, 24, 40, 48, 64, 72, 88 and 96 test hours. This change of 2 test hours for every other sampling period permits sufficient time to conduct the total acid number (TAN) on the same day the samples are taken during a normal 8 hour work period. This change in the sampling schedule should be considered for Method 5307.1 since the TAN can change with time especially after lubricant stressing. Trace metal analysis was conducted on all intermediate and final samples using rotating disk emission spectroscopy. The concentrations of aluminum, silver, copper (bronze), iron (mild steel, M-50), magnesium and titanium were below 1 ppm in all samples and were equivalent to the new lubricant.

Corrosion and oxidation testing of two MIL-L-23699 fluids (O-71-6 and O-77-15) was completed at a test temperature of 188°C. Testing was continued until the breakpoint was reached for both TAN and viscosity. Two tests for each lubricant were used to provide a total of 13 intermediate samples taken between 24 and 432 test hours. Test data for all the intermediate samples consisting of viscosity and viscosity increase at 40°C, TAN and TAN increase, and COBRA readings are given in Tables 17 and 18. The breakpoint for each lubricant occurred between 408 and 432 test hours for both TAN increase and viscosity change. The COBRA reading did not show the expected increase between 408 and 432 test hours for either oil.

e. Summary

Corrosion and oxidation testing using Federal Test Method Standard No. 791, Method 5307.1 with modified intermediate sampling frequencies has been completed for two MIL-L-7808 lubricants using the normal 96 hour test period and for two MIL-L-23699 lubricants for extended test time of 432

TABLE 17

RESULTS OF REFLUX CORROSION/OXIDATION TEST
LUBRICANT O-71-6

Sample	Viscosity cSt/40°C	40°C Viscosity Increase, %	Neut. No. mg KOH/g	Neut. No. Increase	COBRA
Initial	25.71	-	0.06	-	2
24	27.06	5.2	0.22	0.16	6
48	28.07	9.2	0.28	0.22	18
96	28.91	12.4	0.49	0.43	30
144	29.54	14.9	0.54	0.48	48
192	30.00	16.7	0.48	0.42	49
264	30.94	20.3	0.68	0.62	60
288	31.27	21.6	0.66	0.60	61
312	31.66	23.1	0.64	0.58	64
336	32.04	24.6	1.19	1.13	68
360	32.54	26.6	0.97	0.91	58
384	33.28	29.4	1.42	1.36	65
408	34.49	34.2	1.71	1.65	51
432	47.99	86.6	10.26	10.20	59

METAL SPECIMEN DATA

Metal Type	Visual Appearance
Aluminum	no change
Silver	no change
Bronze	sl. tarnish
Mild Steel	bluing
M-50 Steel	bluing
Magnesium	no change
Titanium	sl. tarnish

TEST CELL DATA

Tube Deposits	slight streaks of varnish above oil level
Condenser Deposits	none

TEST CONDITIONS

Sample Vol.	250 ml per 2 tubes
Air Rate	10 l/hr.
Test Temp.	188°C (370°F)

TABLE 18

RESULTS OF REFLUX CORROSION/OXIDATION TEST
LUBRICANT O-77-15

Sample	Viscosity cSt/40°C	40°C Viscosity Increase, %	Neut. No. mg KOH/g	Neut. No. Increase	COBRA
Initial	24.04	-	0.43	-	2
24	25.50	6.1	0.30	0.13	10
48	26.28	9.3	0.49	0.06	20
96	27.16	13.0	0.70	0.27	40
144	27.79	15.6	1.01	0.58	63
192	28.40	18.1	0.81	0.38	71
264	29.36	22.1	1.23	0.80	78
288	29.77	23.8	1.15	0.72	78
312	30.08	25.1	1.12	0.69	76
336	30.56	27.1	1.56	1.13	71
360	31.37	30.5	1.45	1.02	66
384	31.84	32.4	2.01	1.58	64
408	33.82	40.7	2.65	2.22	55
432	41.56	72.9	8.65	8.22	61

METAL SPECIMEN DATA

Metal Type	Visual Appearance
Aluminum	no change
Silver	sl. discoloration
Bronze	sl. tarnish
Mild Steel	bluing
M-50 Steel	bluing
Magnesium	no change
Titanium	sl. tarnish

TEST CELL DATA

Tube Deposits	slight varnish streaks above oil level
Condenser Deposits	none

TEST CONDITIONS

Sample Vol.	250 ml per 2 tubes
Air Rate	10 l/hr.
Test Temp.	188°C (370°F)

hours. Good test repeatability was obtained between duplicate testing and with the lubricant manufacturers' data.

f. Future Effort

Corrosion oxidation testing using Federal Test Method 791, Method 5307.1 will be conducted to the extent necessary to provide stressed lubricants required for study by other test areas such as deposition and development of lubricant monitoring techniques.

4. CORROSION INHIBITING PROPERTIES OF LUBRICANTS

a. Introduction

DERD Test Method No. 18, "Corrosion-Inhibiting Properties" developed by the Ministry of Defence, United Kingdom was investigated for determining differences in the corrosion inhibiting properties of ester base gas turbine lubricants under conditions of high humidity and temperature cycling from 40°C to 5°C and using a high and a low corrosion-inhibiting reference oil.

b. Test Apparatus and Materials

The corrosion test specimens consisted of 0.5 inch bearing balls made of SKF Grade 1 steel normally used for IP239 and IP300 Four Ball Wear Testing.

The humidity chamber consisted of a glass tank having general dimensions of 300 mm in length, 150 mm in width and 230 mm high and being similar to a thin layer chromatography developing tank. The glass tank is fitted with a ground glass lid and has a high ledge to support the drip control paper. The glass tank lid has an opening for inserting a thermocouple into the glass chamber for measuring and recording cycling temperatures. A pyrex tray having general dimensions of 225 mm in length, 65 mm in width and two inches high was used for supporting the test balls above the water in the tank. The top part of the tray was drilled to provide

counter sunk ball bearing seats and oil drainage from the seats. The tray contained 18 seats with each being numbered for identification of test specimens. An underneath drip tray prevents the drippings from covering the water layer and preventing vaporization of the water to provide 100% relative humidity. The tray was fitted with legs to provide a standard elevation of the test specimens in the humidity chamber.

A laboratory oven was used to elevate the internal air temperature to 40°C from 5°C within 3 hours and maintaining a constant temperature of 40 ± 1°C. Cooling the chamber to 5°C from 40°C within 12 hours and maintaining a constant internal air temperature of 5 ± 1°C was accomplished using a refrigerator. Temperature recording throughout the complete test was made using a calibrated type J thermocouple and strip chart recorder. Whatman Number 1 filter paper cut to fit the top of the humidity chamber was used as the condensate drip control paper. Handling of the test specimens prior to cleaning was done using polyethylene gloves. A test specimen handling device (wire loop) was used to handle the test specimens after cleaning and soaking in the test lubricant, and for placing them on the support tray. Test specimen inspection was accomplished using a dynazoom binocular microscope with 10X magnification.

c. Test Procedures

The humidity chamber, lid and sample handling device were cleaned with acetone followed by petroleum ether and air dried. The specimen tray was cleaned with petroleum ether and air dried. Test specimens were inspected prior to cleaning for signs of rust, staining or surface damage. Specimens showing imperfections were rejected. The test specimens were first degreased by placing them in boiling acetone for 10 minutes and then washed with boiling petroleum ether for ten minutes. Residual solvent was then

removed by air drying.

Three test specimens were used for each fluid to be tested and for each of the low and high reference oils. The test specimens were soaked in the appropriate test fluid for 30 minutes at a temperature of 40°C. During the 30 minute soaking period, distilled water was added to the humidity chamber to a depth of 2 cm (one test was conducted using de-ionized water). The test specimens were removed from the test fluid using the wire loop and removing excess oil from the test specimens by touching the specimen on the rim of the beaker used for soaking and then onto a clean flat tissue. The prepared specimen was placed in a seat of the support tray and its position recorded. A clean specimen handling device was used for the three test specimens for each test fluid.

The condensation drip control paper was placed above the support tray supported by the chamber ledge and by glass rods being placed across the width of the tank. Silicone stop cock grease was used to seal the chamber lid. A thermocouple was inserted into the lid opening with its junction being below the drip paper and above the specimen tray.

The humidity test chamber containing the specimens was placed in the oven preheated to 40°C with a temperature recorder being attached to the thermocouple. After 3 hours in the oven, the chamber was transferred to the refrigerator pre-set at 5°C for a period of 12 hours with the temperature being monitored throughout the test. The chamber was removed from the refrigerator after the 12 hour test period and returned to the 40°C oven for a 3 hour test period. Cycling from 40°C to 5°C is repeated for a total of seven cycles. At the end of the seventh cycle, the chamber was removed from the refrigerator and allowed to obtain room temperature. Since one week-end was involved during the testing, the test was initiated on a Tuesday with the

week-end occurring during the 4th and 5th cycle. The test chamber was kept in the refrigerator at 5°C over the week-end.

After the 7th cycle the test specimens were removed from the support tray after reaching room temperature and washed with acetone followed by boiling petroleum ether. The test specimens were then air dried on lint free towels.

Test specimens were examined under 10X magnification with each specimen below assessed according to the following criteria:

Number of Corrosion Centers	Corrosion Category
0	Zero
1 to 5	Low
Greater than 5	High

d. Description of Test Fluids

A total of 6 test fluids and uncoated test specimens were examined in this study. These test fluids are described below:

Test Fluid	Description
O-79-16	MIL-L-7808 Lubricant
O-79-20	MIL-L-7808 Lubricant
O-82-2	MIL-L-7808 Lubricant
TEL-7002	MIL-L-23699 Lubricant
H	High Reference Oil
L	Low Reference Oil

e. Results and Discussion

The first test was conducted using distilled water in the humidity chamber. For this first test continuous recording of temperature was not made although periodic checks of temperature were made. Data obtained from this test is as follows.

Lubricant O-79-16

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Lubricant O-82-2

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: Two small corrosion centers.

Lubricant O-79-20

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Lubricant H (High Corrosion Reference Oil)

Test Ball #1: One small corrosion center.

" " #2: Three small corrosion centers.

" " #3: No corrosion centers.

Lubricant L (Low Corrosion Reference Oil)

Test Ball # 1: No corrosion centers.

" " # 2: One corrosion center.

" " # 3: No corrosion centers.

Uncoated Test Balls

Test Ball #1: Over 15 corrosion test centers.

" " #2: " " " " "

" " #3: " " " " "

Although this test showed some corrosion, no significant difference was seen between the low and high reference oils. These two oils were examined by gas chromatography and were identified as two completely different ester base lubricants.

The test was repeated with distilled water but with the cooling cycle temperatures being recorded and the heating cycle temperature being monitored with a Doric Temperature Indicator. Data obtained from this second test is

as follows:

Lubricant O-79-16

Test Ball #1: One corrosion center.

" " #2: Two corrosion centers.

" " #3: One corrosion center.

Lubricant O-79-20

Test Ball #1: No corrosion centers.

" Ball #2: " " "

" Ball #3: " " "

Lubricant TEL-7002

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Lubricant H (High Reference Oil)

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Lubricant L (Low Reference Oil)

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Uncoated Test Balls

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: Two corrosion centers.

Again, this test showed no corrosion for the high corrosion reference oil and very little corrosion for the uncoated balls in comparison with high

corrosion for the uncoated balls in the first test.

A third test was conducted using de-ionized water having a pH value of 3.8. The following test data was obtained:

Lubricant O-79-16

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Lubricant O-79-20

Test Ball #1: No corrosion centers.

" " #2: One corrosion center.

" " #3: Eight corrosion centers.

Lubricant TEL-7002

Test Ball #1: Over five corrosion centers.

" " #2: " " " "

" " #3: " " " "

Lubricant H (High Reference Oil)

Test Ball #1: No corrosion centers.

" " #2: " " "

" " #3: " " "

Lubricant L (Low Reference Oil)

Test Ball #1: No corrosion centers.

" " #2: One corrosion center.

" " #3: " " "

Uncoated Test Balls

Test Ball #1: Very much corrosion.

" " #2: " " "

" " #3: " " "

The use of de-ionized water gave much more corrosion for lubricants O-79-20 and TEL-7001 but gave no corrosion for lubricant O-79-16 or lubricant H (high corrosion reference oil). A summary of the three tests is given below using the previously described rating procedure. The rating shown is the highest rating assigned to any one of the three balls for each lubricant.

	Test 1	Test 2	Test 3
Test Lubricant	Distilled Water	Distilled Water	De-ionized Water
O-79-16	Zero	Low	Zero
O-79-20	Zero	Zero	High
TEL-7002	-	Zero	High
O-82-2	Low	-	-
H Reference Oil	Low	Zero	Zero
L Reference Oil	Low	Zero	Low
Uncoated Balls	High	Low	High

f. Summary

The following conclusions can be made based upon the data obtained from the three tests:

a. Poor repeatability was obtained between tests.

b. No difference in the corrosion inhibiting propensity of the low and high reference oils was obtained.

c. Use of de-ionized water appears to make a big difference for some lubricants and no difference for other lubricants.

5. ADDITIVE ANALYSIS

a. Introduction

In order to characterize lubricants stressed in the Squires oxidative test, chromatographic methods were used for analysis of additives in MIL-L-7808 and laboratory formulated lubricants. In addition, identification of some of the intermediate antioxidant products was made in order to gain insight into the mechanism of oxidative depletion of the aromatic amine antioxidants.

b. Experimental

A procedure was developed for analysis of volatile nitrogen and phosphorous containing additives by gas chromatography with a thermionic specific detector (GC-TSD). The procedure was specifically developed for the analysis of PANA, Octyl-PANA, DODPA and TCP. Details of this method are listed in Appendix B. The selectivity of the TSD allowed this analytical procedure to be relatively free of interferences and was shown to be linear over a range of 0.05 to 2.5 weight percent with good accuracy and precision.

The reverse phase liquid chromatography (RPLC) procedure used was adapted from a method by Klenke.[4] The conditions of this method are listed in Table 19.

TABLE 19

LIQUID CHROMATOGRAPHY CONDITIONS FOR ANTIOXIDANT ANALYSIS

Instrument:	Hewlett Packard 1084B	
Detector:	Ultraviolet Variable wavelength at 254 nm	
Column:	Hi-Chrom 4.6 mm X 25 cm 5 micron ODS (Regis)	
Solvent:	Acetonitrile : water (A:B)	
Flow Rate:	1.0 ml/min	
Solvent Program:	Time (min)	% B
	0.0	10
	5.0	0
	22.0	0

Intermediate antioxidant species produced in O-77-1 with 2% PANA, 2% DODPA or 1% PANA and 1% DODPA after 24 hours of the Squires oxidative test at 190°C were collected preparatively by this RPLC procedure. An ultraviolet (UV) spectrometric scan over the range of 225 to 540 nm was done on these species by using the stopped flow scan accessory of the ultraviolet detector of the liquid chromatograph, although extinction coefficients could not be measured with this equipment. The isolated samples were submitted to AFWAL/MLSA for mass spectrometric (MS) analysis. These samples were also analyzed by gel permeation chromatography using 50 and 100 angstrom pore size 30 cm X 7.5 mm ID, 5 micron PL-GEL columns (Polymer Laboratories LTD) and using THF at a flow rate of 1.0 ml/minute with UV detection at 254 nm. The isolated species that were of sufficient volatility were analyzed by GC-TSD by modifying the method in Appendix B to allow a final column temperature of 325°C with a 5 minute final hold. Quantitative analysis of these species was accomplished by calculating response factors relative to PANA assuming the same per

nitrogen response. However, since a compounds' response is also somewhat dependent on structure these concentrations must be considered to be semiquantitative although the similarity in structures of all these compounds should make the results approximately correct.

c. Results and Discussion

(1) Antioxidant Concentrations in Stressed MIL-L-7808 Lubricants

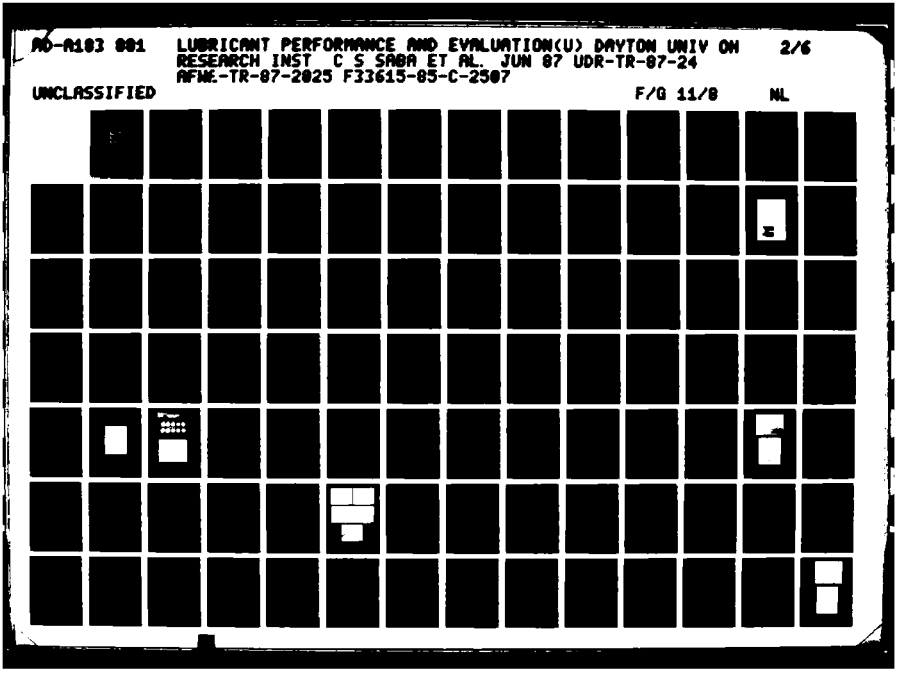
The concentrations of antioxidants (PANA, Octyl-PANA and DODPA) during the Squires oxidative test at 190°C for six MIL-L-7808 lubricants were measured by GC-TSD and are plotted in Figures 20 to 25. The lubricant O-82-3 contained small amounts of other nitrogen or phosphorous containing compounds which may have antioxidant properties. In general the plots show that the more active antioxidant (PANA or Octyl-PANA) depletes very rapidly, usually by 48 hours, though the PANA is at least partially lost by evaporation due to its relatively high volatility. The other antioxidant (DODPA) depletes more slowly. The physical properties of the lubricants seem to degrade rapidly at the point where the DODPA depletes to a concentration between 0.05 to 0.10 weight percent.

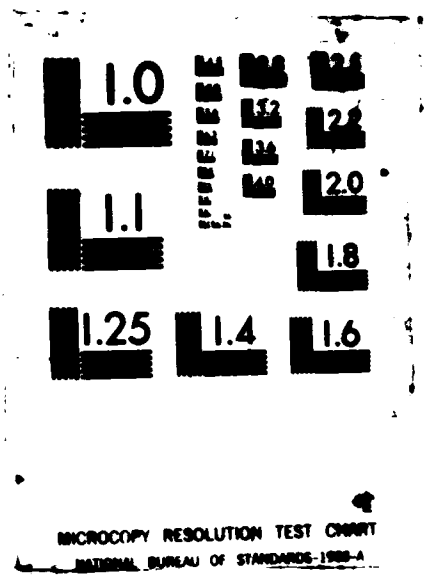
(2) Intermediate Antioxidant Species of PANA and DODPA

In assessing the ability of secondary aromatic amine antioxidants to inhibit the autoxidation of ester basestocks it is necessary to consider the intermediate species that form from the oxidation of the primary antioxidants. Previous research[5-10] has identified some of these species and has demonstrated their autoxidation inhibiting properties. An investigation into some of these species produced during the oxidation of PANA and DODPA containing MIL-L-7808 lubricants was made. Individual intermediate antioxidant species were preparatively isolated by RPLC and identified and their concentration during an oxidative test was measured.

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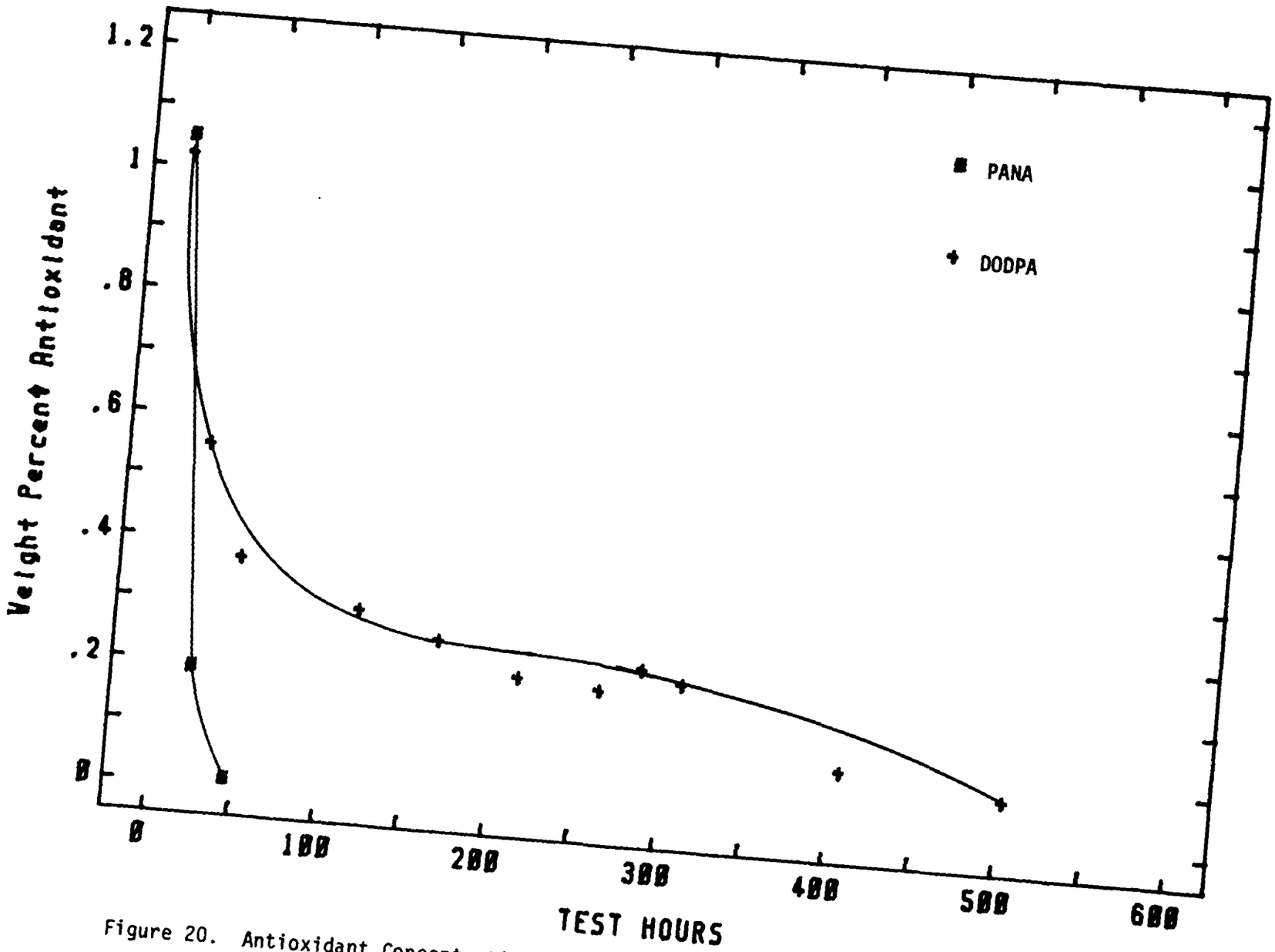


Figure 20. Antioxidant Concentration in O-79-16 during the Squires Oxidative Test at 190°C

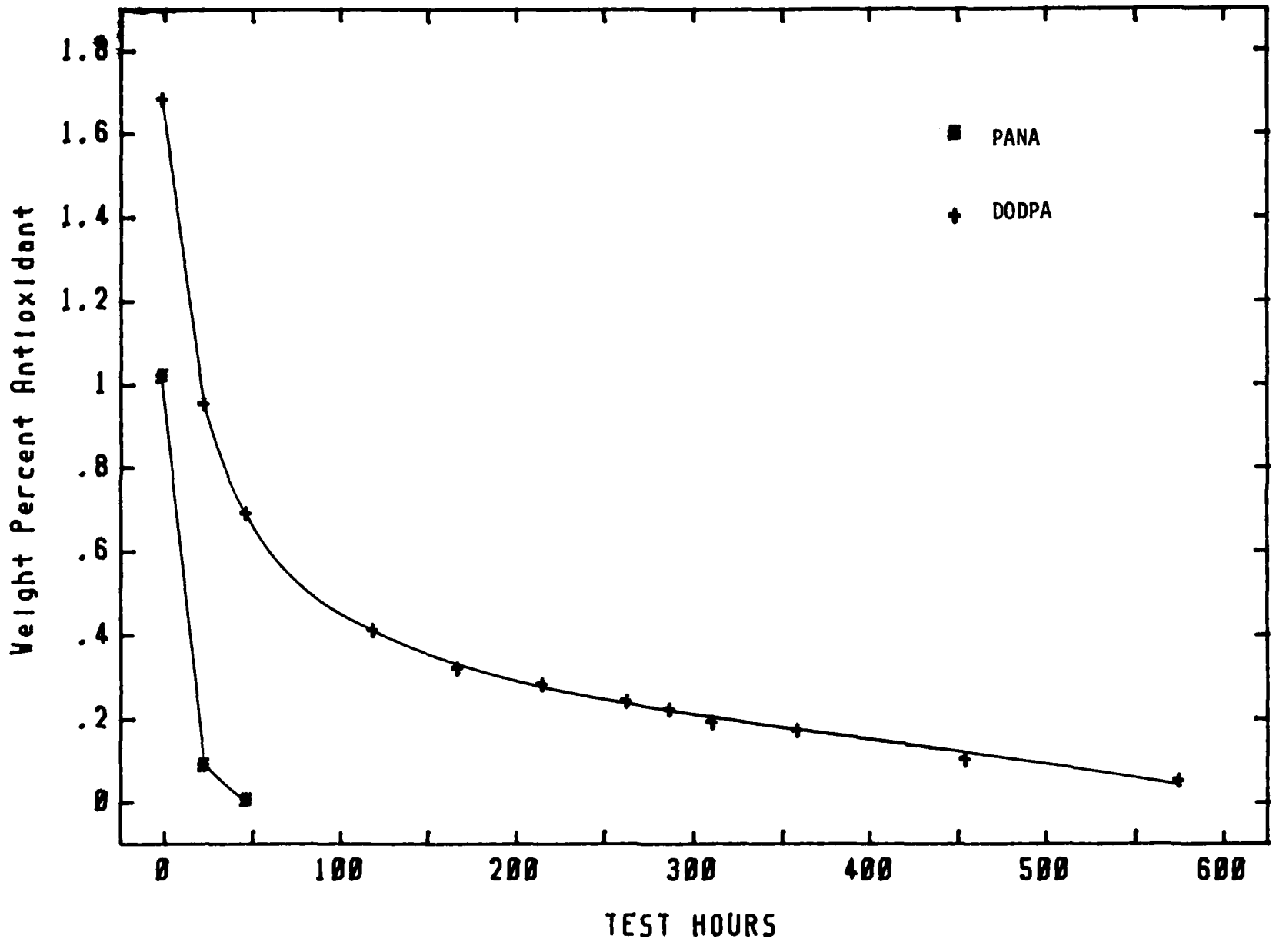


Figure 21. Antioxidant Concentration in O-79-17 during the Squires Oxidative Test at 190°C

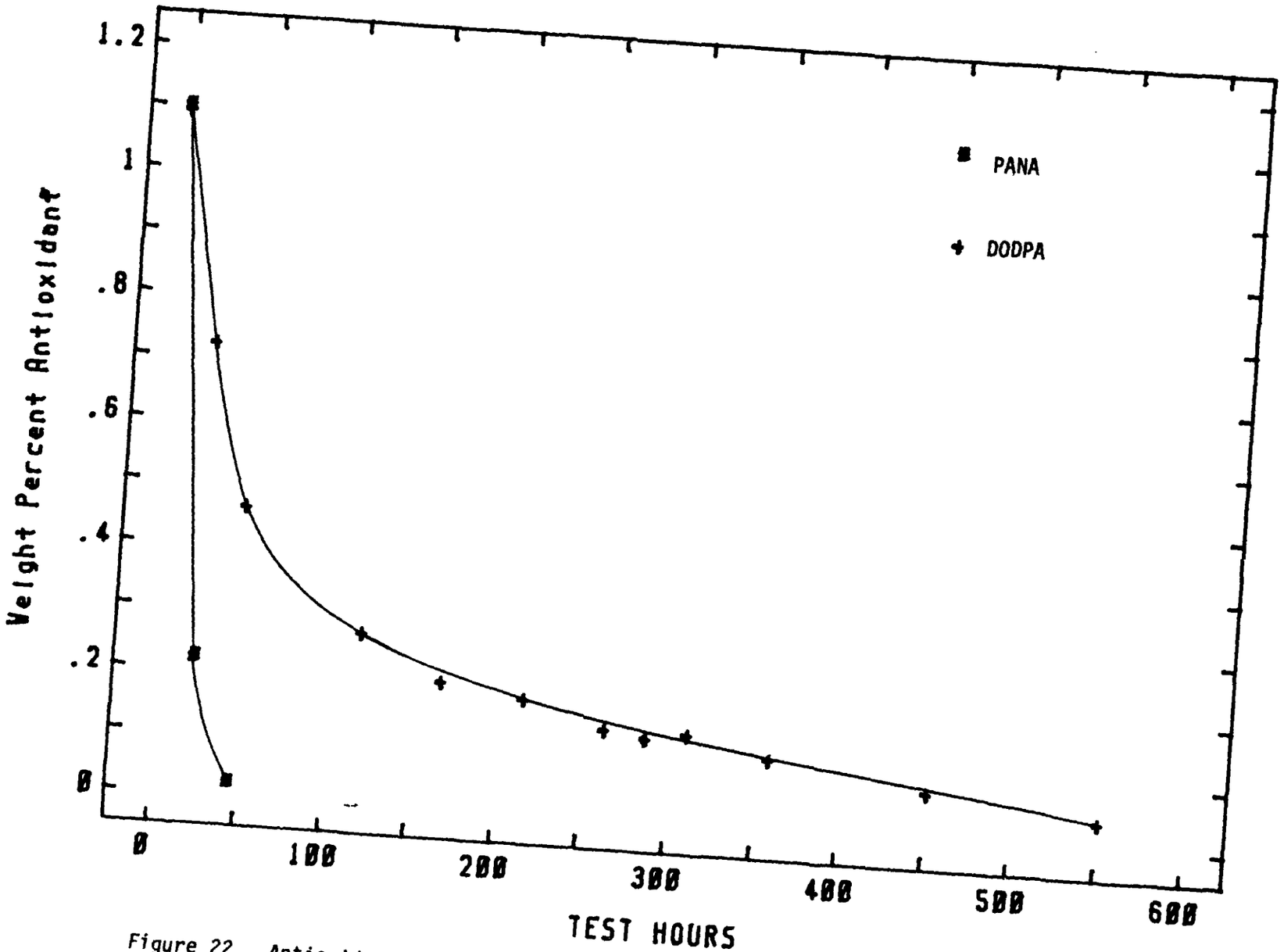


Figure 22. Antioxidant Concentration in O-79-20 during the Squires Oxidative Test at 190°C

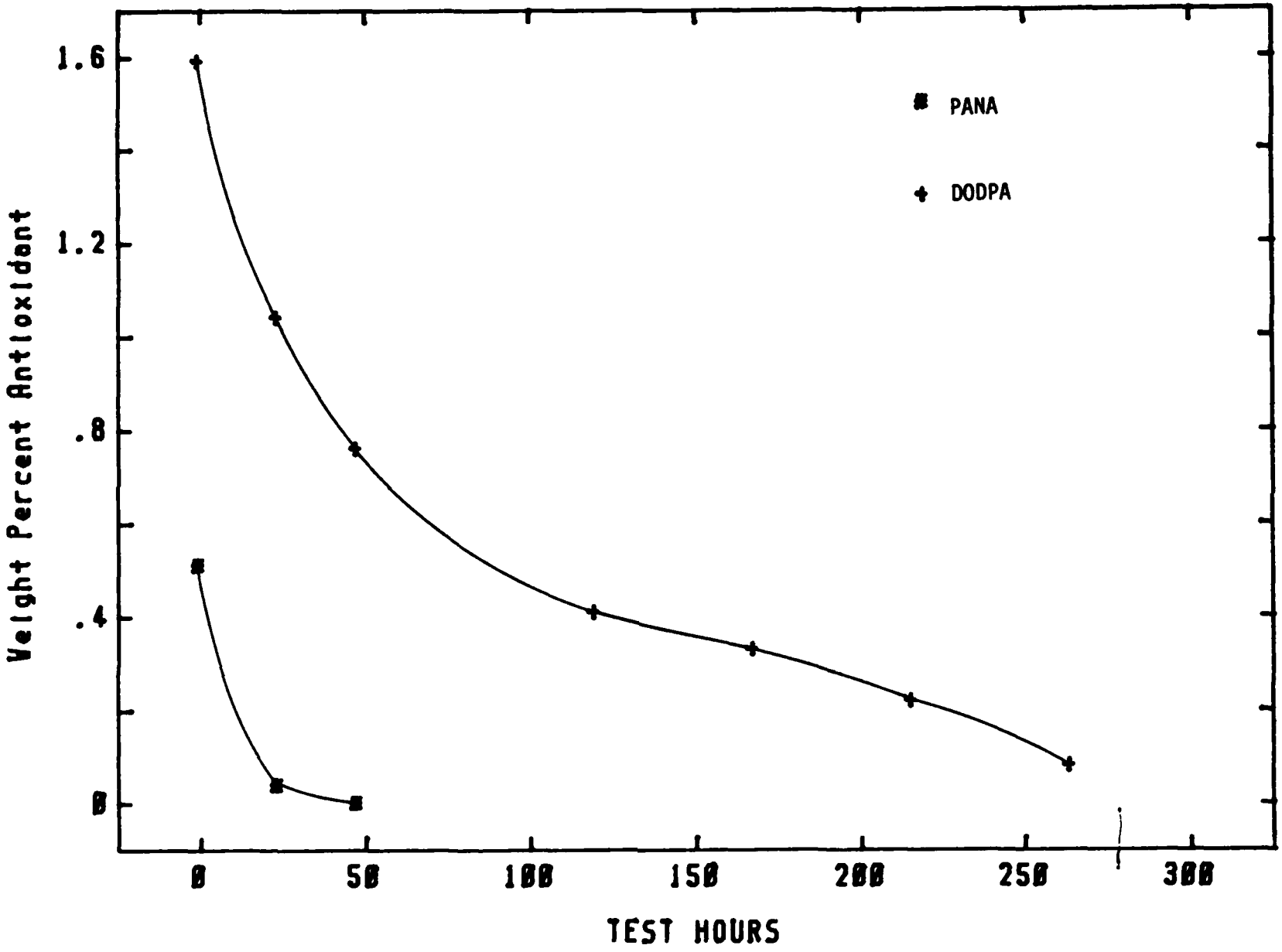


Figure 23. Antioxidant Concentration in O-82-2 during the Squires Oxidative Test at 190°C

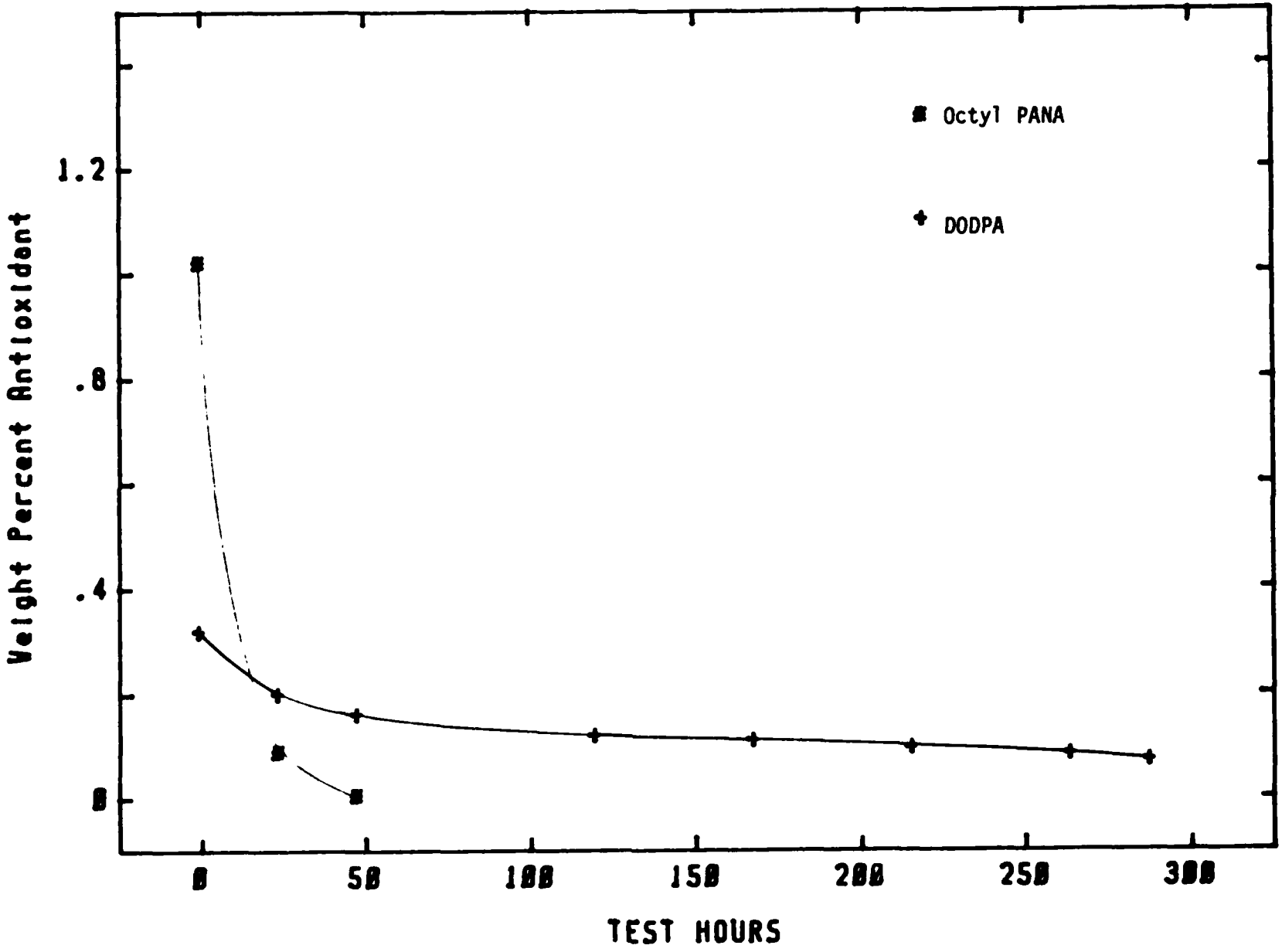


Figure 24. Antioxidant Concentration in O-82-3 during the Squires Oxidative Test at 190°C

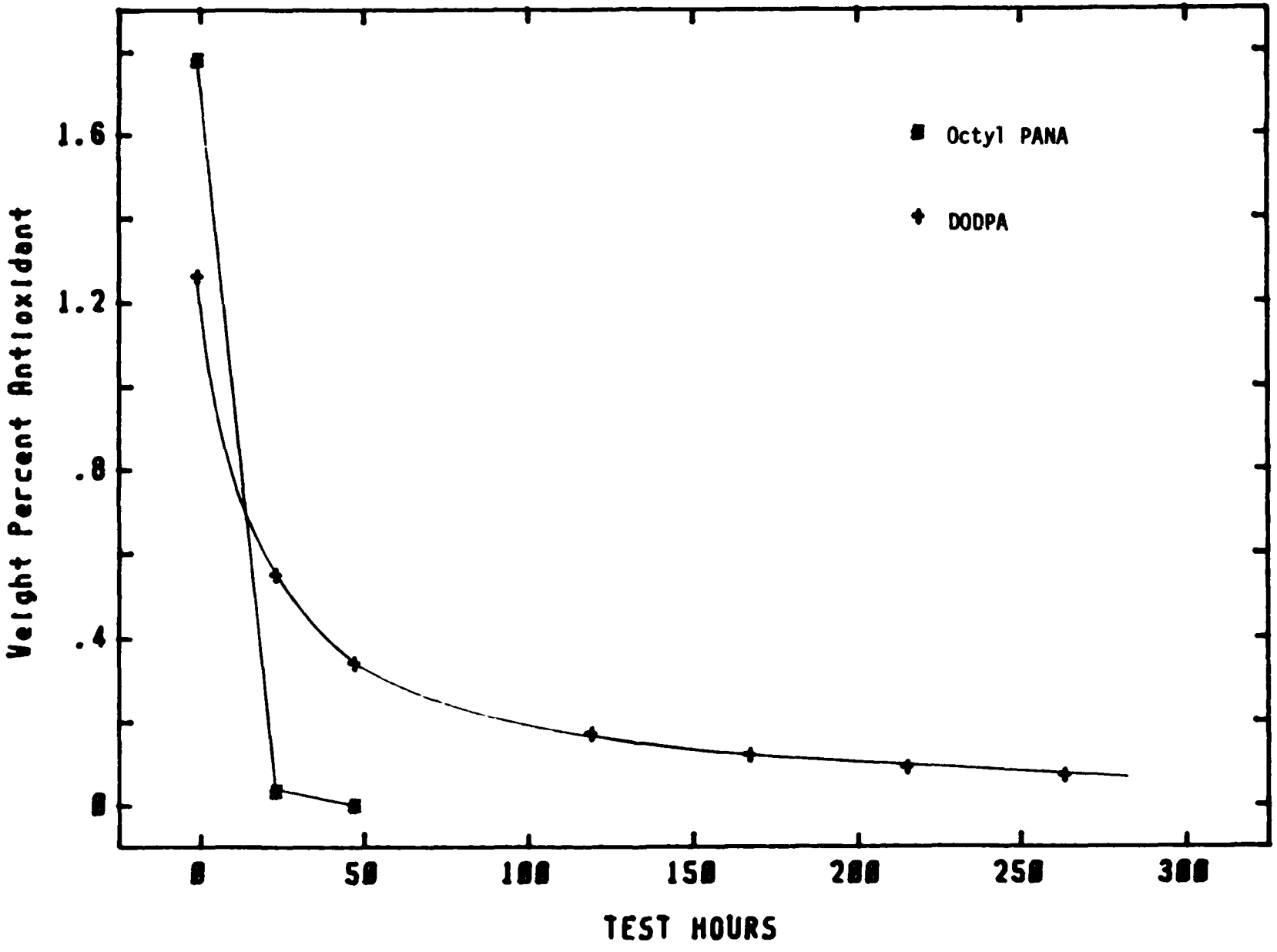


Figure 25. Antioxidant Concentration in O-82-14 during the Squires Oxidative Test at 190°C

The RPLC chromatograms of the 24 hour Squires oxidative tested lubricants with 2% PANA, 2% DODPA or 1% PANA and 1% DODPA are shown in Figures 26, 27 and 28 respectively. Compounds I through V were isolated by RPLC. Compounds I, IV and V were submitted for MS analysis. In addition, compounds I, II and III were analyzed by GPC. The MS and UV data for these compounds and their identification from this data is shown in Table 20. Compounds II and III were identified as the PANA trimer and PANA tetramer respectively based on the similarity of their UV structure to the PANA dimer (I) and the linearity of the plot (Figure 29) of the log of the molecular weight of PANA, I, II and III vs. their respective GPC retention times (corr. coeff. = 0.99996). Identification of compound IV could not be made and is assigned the structure $C_{28}H_{39}NO_2$ based on its MS molecular ion peak.

The formation and transitory nature of these intermediate antioxidant species can be explained given the present stage of knowledge of the mechanism of oxidation inhibition by aromatic amine antioxidants. These antioxidants are presumed to be effective inhibitors of oxidation by interrupting the peroxy radical chain transfer process by offering an easily extractable hydrogen. The resulting antioxidant radicals have too low an activity to continue the chain transfer process and will form inactive species by radical combination or disproportionation. Compound I is probably formed by dimerization of two hydrogen abstracted PANA radicals. Similarly, compounds II and III (trimer and tetramer respectively) are formed by continuation of this same process and doubtlessly smaller amounts of higher oligomeric compounds are formed. A likely structure for these three compounds would involve a nitrogen to carbon bond repeating link through the 4 position of the naphthyl ring although this is not certain. A similar type compound was found to be produced by the oxidation of phenyl-2-naphthylamine

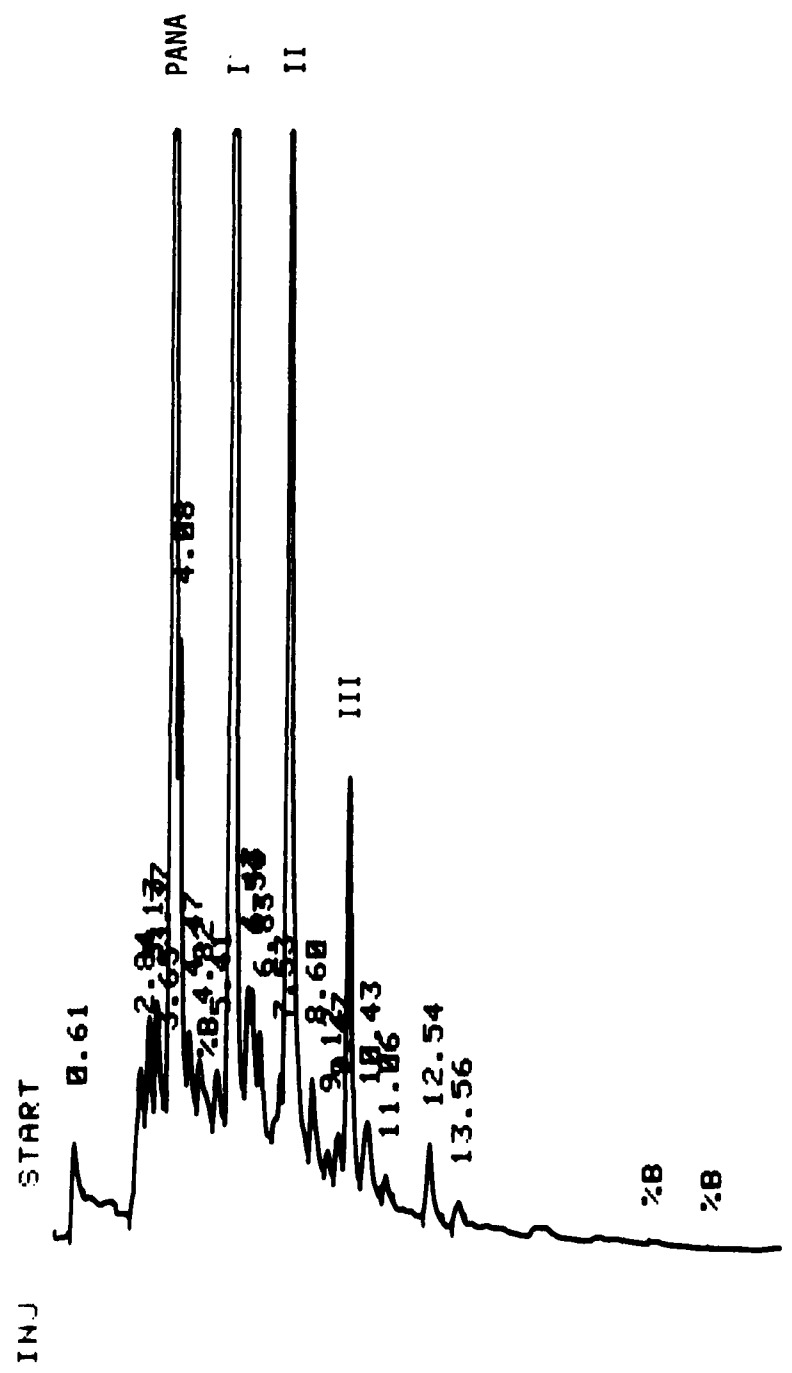


Figure 26. RPLC Chromatogram of O-76-5A + 2% PANA From Squires Oxidative Test at 205°C After 24 Hours.

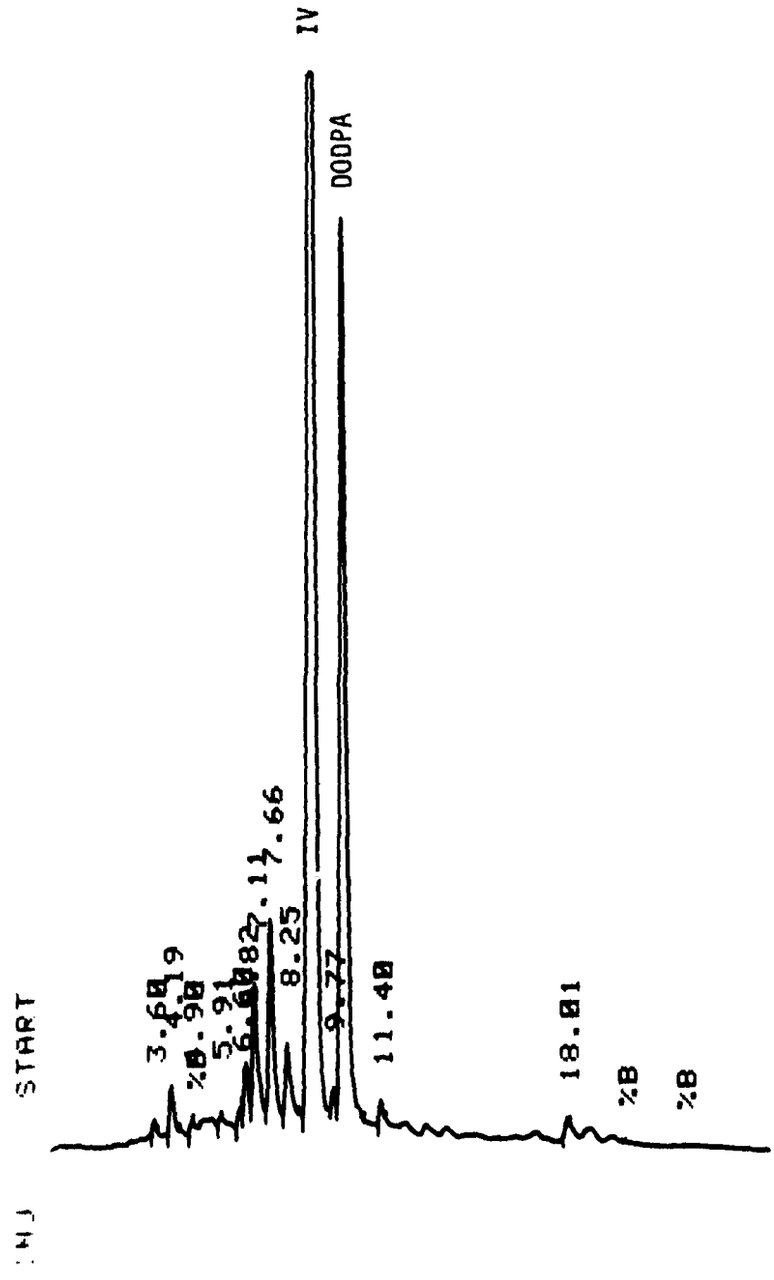


Figure 27. RPLC Chromatogram of O-76-5A + 2% DODPA from Squires Oxidative Test at 205°C After 24 Hours.



Figure 28. RPLC Chromatogram of O-79-17E From Squires Oxidative Test at 205°C After 24 Hours.

TABLE 20

ULTRAVIOLET SPECTROSCOPY AND MASS SPECTROMETRY DATA FOR INTERMEDIATE ANTIOXIDANT SPECIES

COMPOUND	IDENTIFICATION	ULTRAVIOLET λ_{Max} (nm)	MASS SPECTRAL IONS (m/z)
-	PANA	256,341	219(100),218(46),217(32),55(11),43(11)
-	DODPA	295	-
I	PANA Dimer	267,365	436(100),359(18),344(19),217(14),216(15),77(10)
II	PANA Trimer	268,375	-
III	PANA Tetramer	271,382	-
IV	$C_{28}H_{39}NO_2$	251,286(sh)	421(9),350(100),250(10),210(3),135(14),57(8)
V	PANA/DODPA Dimer	303,378	610(100),539(98),467(50),270(26),234(28),217(19),57(93),43(70)

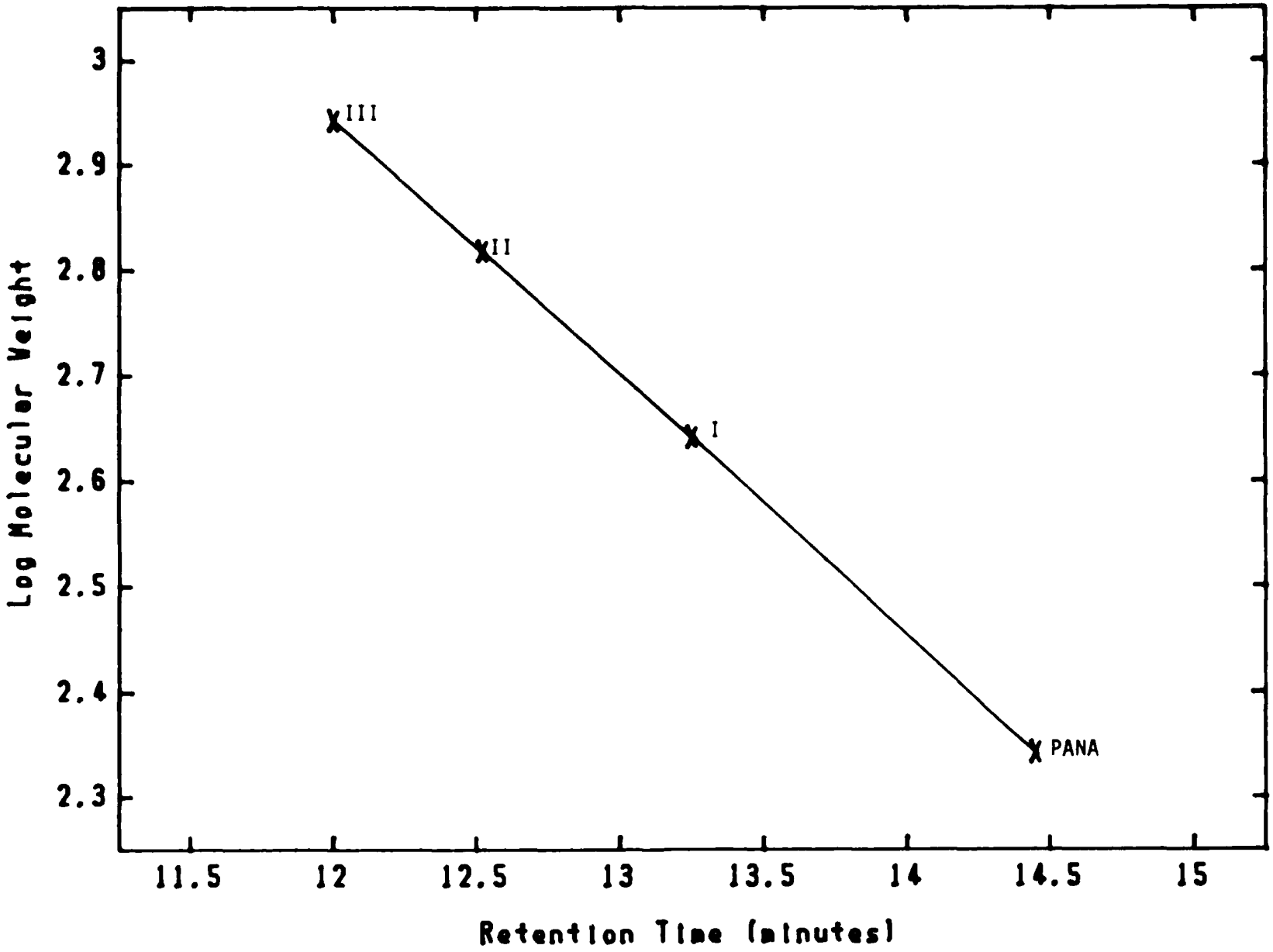
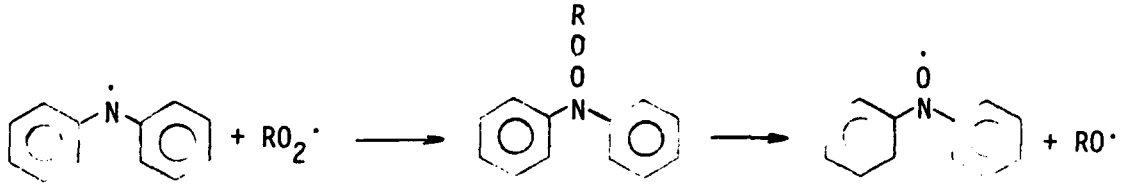


Figure 29. Log Molecular Weight of Compounds I, II, III and PANA vs Gel Premeation Chromatography Retention Times

(PBNA) by alkylperoxy radicals[8] and was shown to be an effective inhibitor of the AIBN initiated oxidation of styrene. Compound V is likely formed in a similar way by dimerization of a PANA and DODPA radical with a probable nitrogen to carbon link. The identity of compound IV is not known and if the MS derived formula of $C_{28}H_{39}NO_2$ (a bi-oxidation product of DODPA) is correct it is difficult to assign a structure or conceive of this compound as a primary intermediate DODPA species. Other research has indicated that diphenylamine and substituted diphenylamines tend to form nitroxide radicals during oxidation[6,7] which are products of the reaction of peroxy radicals with the aminyl radicals:



This nitroxide radical has not been observed in any system analyzed and is probably not stable under the conditions of chromatographic analysis. The presence of such species are generally detected by electron spin resonance. It is possible that compound IV is an oxidation product of a nitroxide radical.

Since compounds I, II, III and V all contain an amine hydrogen they can function as a radical trap much as the primary antioxidants that formed them. The transitory nature of compounds I and V as well as IV can be seen in their concentrations in O-77-1 with 1% PANA and 1% DODPA during the Squires oxidative test at 190°C (Figure 30). While PANA and DODPA undergo depletion typically observed in MIL-L-7808 lubricants (Figures 20-25), compounds I and V reach a maximum near the point of PANA depletion and then are rapidly depleted. Compound IV may possess some radical scavenging

ability.

The lubricant in Figure 30 reaches its breakpoint fairly close to the point of depletion of the intermediate antioxidant species. However, this is generally not true of the MIL-L-7808 lubricants analyzed. Figure 31 shows the concentration of intermediate antioxidant species in O-79-20 during the Squires oxidative test of 190°C. Despite the fact that PANA and compounds I and V are depleted by 120 hours test time, the lubricant does not show its breakpoint until a point between 456 and 552 hours. The 0.23% DODPA level at 120 hours could not sufficiently protect the lubricant from oxidation over this length of time. Therefore, there must be other intermediate antioxidants species present in the lubricant that are not detected by the present chromatographic methods due to lack of volatility, low polarity, or lack of stability under the conditions of analysis. Also, it would be expected that since the intermediate antioxidant species formed initially are the result of radical processes they would eventually lead in time to a large number and variety of compounds. Such a complexity of species would make chromatographic analysis difficult. Nevertheless, it can be seen that the major pathway for the production of intermediate antioxidant species that possess considerable oxidation inhibition properties lies, at least initially, through the coupling of antioxidant radicals that are produced by the initial radical hydrogen abstraction.

The concentration of intermediate antioxidant species in O-77-1 with 1% PANA and 1% DODPA stressed in the Squires confined heat test at 205°C is shown in Figure 32. Though this is an oxygen limited test compounds I and V are produced and seem to exhibit behavior similar to that seen in the oxidative test. PANA depletes more slowly in this test probably due to much smaller volatility losses. Because of the limited oxygen availability in

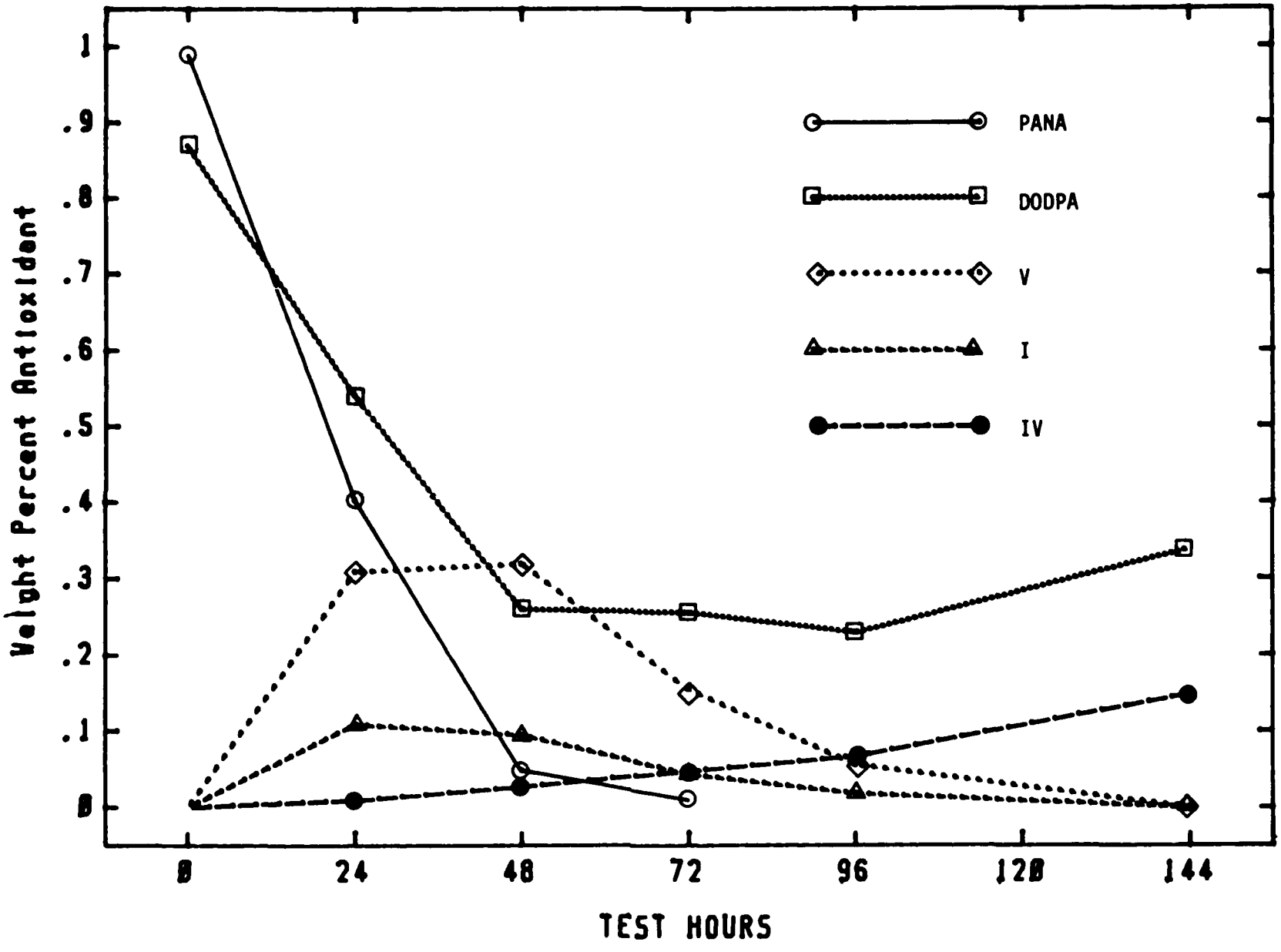


Figure 30. Concentration of Intermediate Antioxidant Species in O-77-1 with 1% PANA and 1% DODPA from the Squires Oxidative Test at 190°C

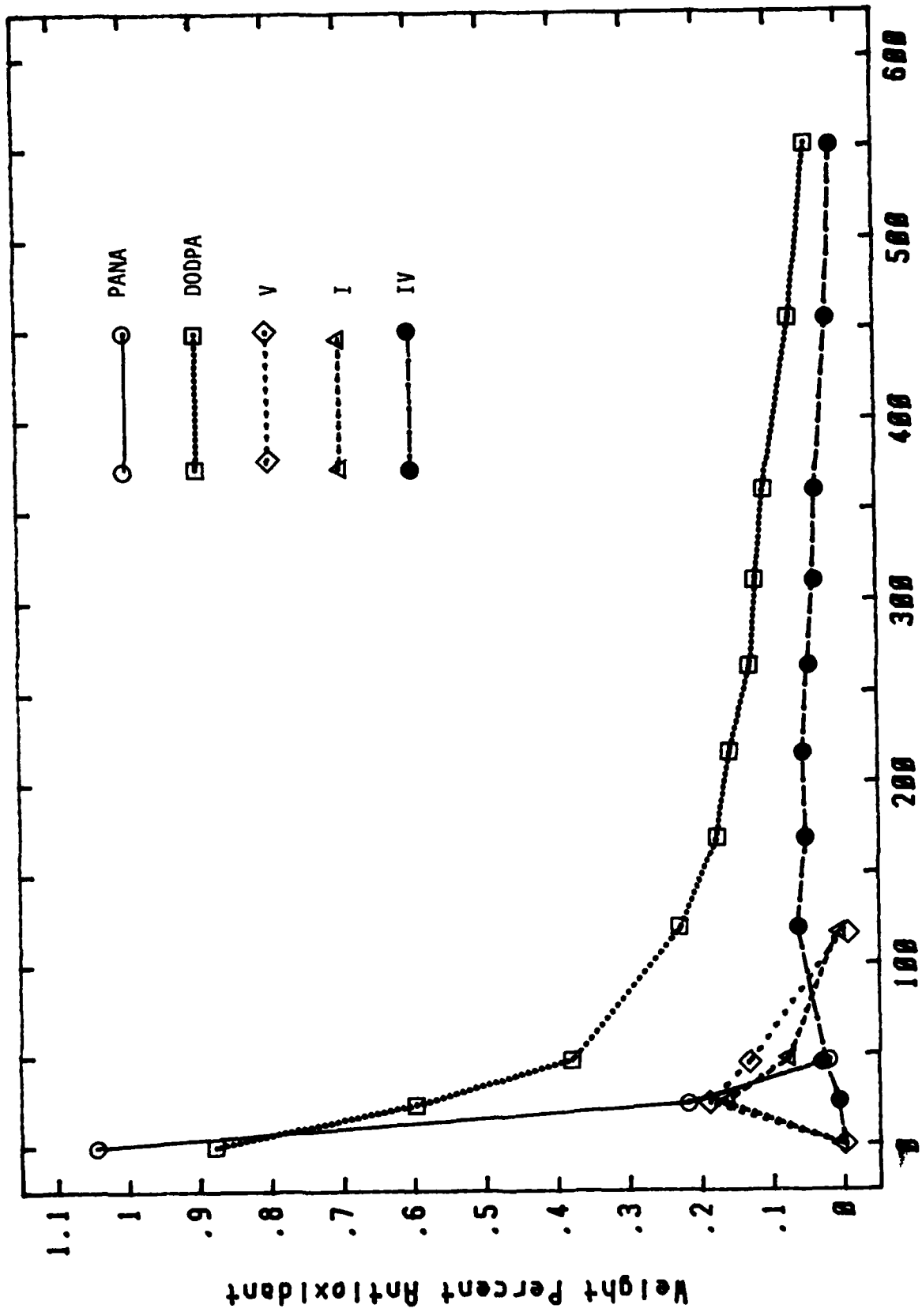


Figure 31. Concentration of Intermediate Antioxidant Species in 0-79-20 from the Squires Oxidative Test at 190°C

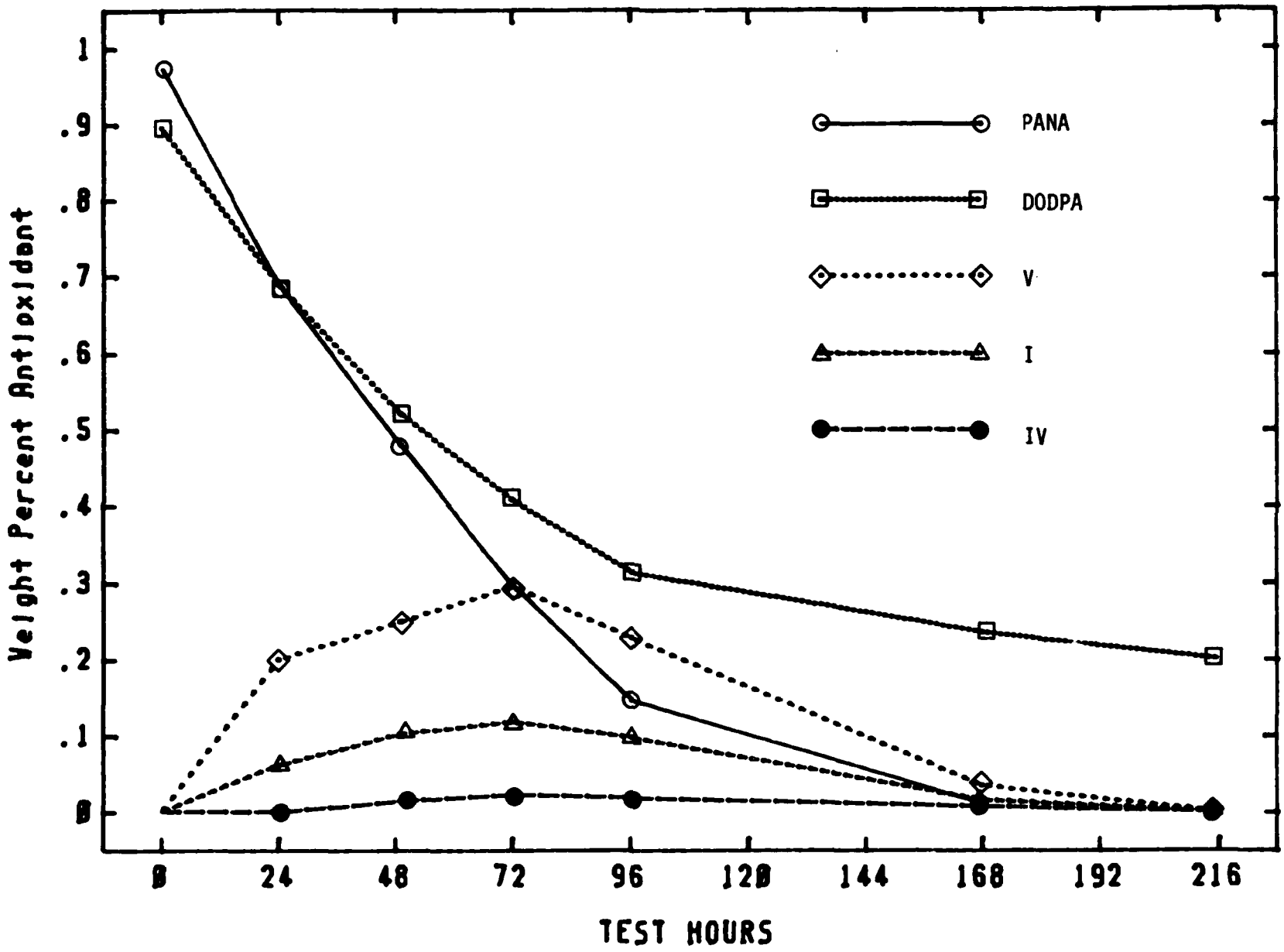


Figure 32. Concentration of Intermediate Antioxidant Species in O-77-1 with 1% PANA and 1% DODPA from the Squires Confined Heat Test at 205°C

this test Compound IV is formed in much smaller concentrations due to its being an oxygenated compound.

d. Summary

Chromatographic methods have been developed for analysis of antioxidants in MIL-L-7808 lubricants. Analysis of these lubricants stressed in the Squires oxidative test reveal a rapid depletion of the primary antioxidant (PANA or Octyl-PANA) and a much slower loss of the secondary antioxidant (DODPA). The physical properties of these lubricants in general degraded rapidly when the DODPA concentration depleted to a level of between 0.05 and 0.10%. Preparative isolation by RPLC and subsequent chromatographic and mass spectroscopic analysis of various compounds from these stressed lubricants has resulted in the identification of the major intermediate antioxidant species produced during oxidation of PANA and DODPA containing lubricants. Semiquantitative analyses of these species from lubricants stressed in the Squires oxidative test indicate that while they are of importance early in the test the lack of a breakpoint at the point of their depletion means that there are other species present to provide oxidation inhibition. Similar behavior was observed in Squires confined heat tested lubricants.

e. Future Effort

Method development for analysis of new additives will be completed as required. Investigation into intermediate antioxidant species will continue.

6. INVESTIGATION OF REFRACTIVE INDICES OF LUBRICANTS

a. Introduction

Refractive indices of various new and stressed oils were measured for identifying changes due to lubricant formulations and degradation produced by oxidative and thermal stressing.

b. Test Apparatus

An Abbe Model Fisher Refractometer, capable of measuring refractive indices with an accuracy of 0.0001 refractive unit, was used for the refractive index measurements.

c. Test Procedure

The instrument was allowed to warm up for 30 minutes prior to use and calibration was conducted according to the manufacturer's instructions. A drop of sample was applied to the prism, the refractive index reading was taken and the temperature recorded.

d. Results and Discussion

The samples were tested at measured room temperature (20-22^{o}C) and values adjusted to a standard temperature of 25^{o}C. The adjustments were made using an experimentally determined value of 0.00035 as the increment of change in refractive index per degree Celcius.

Table 21 and Figure 33 illustrate that a change in refractive index occurs when a lubricant experiences volatility loss. Samples which showed significant degradation but negligible volatility loss showed no change in refractive index.

A relationship exists between the molecular weight of an ester and its refractive index, which is shown in Table 22.

TABLE 21

REFRACTIVE INDICES at 25°C OF STRESSED LUBRICANTS

Oxidatively Stressed

Test Hours	O-79-16J	O-79-17E	O-79-20	O-82-2	O-82-3	O-82-14
0	1.4515	1.4520	1.4517	1.4525	1.4521	1.4545
24	1.4527	1.4537	1.4527	1.4534	1.4538	1.4563
48	1.4537	1.4542	1.4527	1.4542	1.4554	1.4575
120	1.4583	1.4554	1.4561	1.4579	1.4633	1.4632
168	1.4599	-	1.4585	1.4643	1.4678	1.4709
216	1.4618	1.4607	1.4614	1.4709	-	1.4741
264	1.4643	-	1.4672	-	-	-

Thermally Stressed

Test Hours	O-79-16J	O-79-17E	O-79-20	O-82-2	O-82-3	O-82-14
0	1.4515	1.4520	1.4517	1.4525	1.4521	1.4545
24	1.4519	1.4517	1.4520	1.4527	1.4522	1.4545
48	1.4519	1.4521	1.4523	1.4527	1.4522	1.4548
72	1.4519	1.4521	1.4523	1.4527	1.4522	1.4548
96	1.4519	1.4521	1.4523	1.4527	1.4523	1.4548
144	1.4519	1.4521	1.4523	1.4531	1.4523	1.4548
192	1.4519	1.4521	1.4523	1.4531	1.4523	1.4548

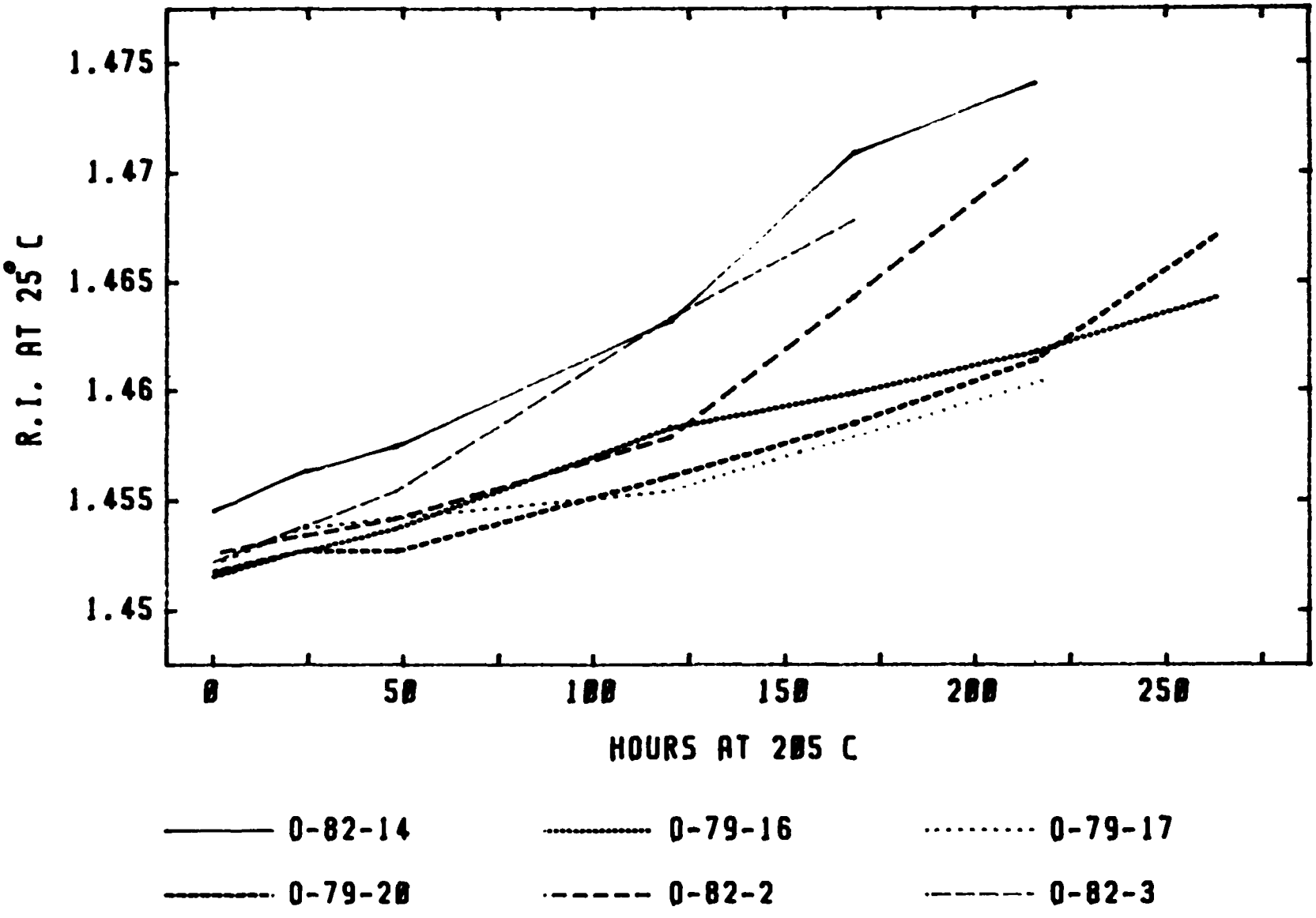


Figure 33. Refractive Indices of Oxidatively Stressed Oils.

TABLE 22

REFRACTIVE INDICES FOR VARIOUS ESTERS

	Ester	M.W.	n_D^{25}
E-105	(di-2-ethylhexyladipate)	371	1.4460
O-77-1	(di-2-ethylhexyladipate)	371	1.4456
O-76-8	(di-2-ethylhexyladipate)	371	1.4461
E-120	(2,2-dimethyltrimethylenenonanoate)	384	1.4434
E-129	(trimethylolpropane triheptanoate)	470	1.4485
O-76-5	(trimethylolpropane triheptanoate)	470	1.4490
E-149	(pentaerythritol tetraheptanoate)	584	1.4513

This data shows the refractive index increases as the molecular weights of the esters increase.

e. Summary

Based on the results of this study, the increase in refractive index of oxidatively stressed lubricants is attributed to the volatilization of lower molecular weight esters, rather than degradation of the lubricant. This conclusion is supported by the fact that the same oils, which were thermally stressed and did not experience weight loss, showed no change in refractive index even after considerable degradation had occurred.

f. Future Effort

Investigation will be made using refractive index measurements for identifying changes in oxidatively and thermally stressed high temperature lubricants such as polyphenyl ethers and perflouroalkylethers.

7. LUBRICANT DEPOSITION STUDIES

a. AFAPL Static Coker Study

(1) Introduction

Many requirements must be met by a lubricant for satisfactory performance in turbine engines. This performance has generally been obtained through upgrading of specifications requirements which resulted in lubricant formulations with improved performance characteristics and through engine designs which have reduced the severity of lubricant stressing. However, with increased emphasis on higher engine performance resulting in smaller oil capacities and higher operating temperatures, the need for defining and measuring lubricant properties continues. Lubricant deposition has been and is continuing to be an extremely important lubricant property. A variety of tests have been developed for measuring and describing lubricant deposition. These include bearing deposition tests (11-13), a tube deposition test (14), a panel coker test (15), a hot wall deposition test (16), a rotating cylinder deposition test (17) and an engine simulator test (18). These tests have provided information relative to deposit formation and have been used for measuring deposition characteristics of ester type lubricants. Since test parameters are quite varied, correlation among tests and with actual engine performance is marginal. Most of these tests require large sample volumes and are time consuming and expensive.

The initial effort involving the static coker (19) was directed toward developing a small volume, short time test which would permit good control over test variables and provide for determining weight of deposits along with deposit description. The effort herein describes further development of the static coker and its use for investigating the effects of test specimen material, test temperatures, lubricant prestressing, ester

volatility, antioxidants, and wear metals on lubricant deposition.

(2) Apparatus and Procedure

The configuration of the static coker is illustrated in Figures 34 and 35. The base section is heated by a controlled hot plate and is fabricated from brass for efficient heat transfer. A thermocouple (Type J) is positioned dead center just below the surface and is used for recording and adjusting the test temperature. The base top is machined to provide a vacuum plenum for holding down the metal test specimen. A second thermocouple (Type J) is positioned near the bottom of the base and used for controlling temperature by replacing the hot plate temperature controls with fully adjustable electronic controllers.

A polytetrafluoroethylene (PTFE) seal is used to confine the lubricant to the test specimen and reduce oil creep up the walls of the coker. Small vent holes in the seal serve to maintain a small steady updraft of air over the surface of the test lubricant. The PTFE seal is supported with a stainless steel sleeve which aids in sealing. The test specimen, PTFE seal and seal sleeve are surrounded with a metal convection shield. A brass sealing ring (retainer ring) is placed around and on the ferruled convection shield to prevent leakage of oil between the metal test specimen and the PTFE seal.

The coker base and the hot plate are positioned on a leveling table to provide more uniform layers of deposits. The coking unit is assembled on the hot plate and is then surrounded by two sections of machined calcium silicate insulation. The complete unit including leveling table is surrounded with a metal shield to provide better temperature control.

Initially a series of surface temperature measurements were made at five positions using shim stock test specimens on four coking units for



Figure 34. Static Coker Deposition Tester; (A) Base Section (B) Test Coking Surface (C) PTFE Seal (D) Steel Sleeve Retainer (E) Convection Shield (F) Brass Sealing Ring (G) Insulation

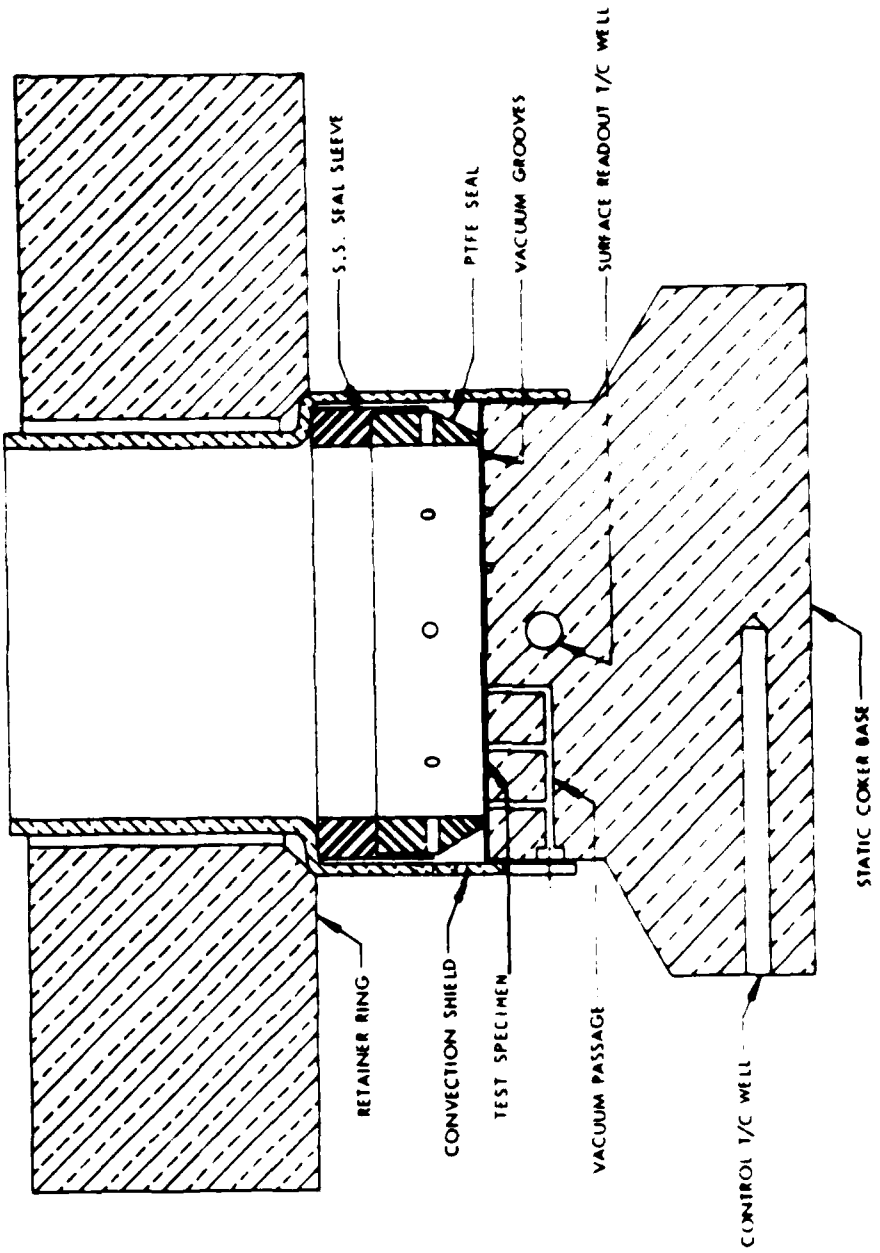


Figure 35. Schematic of the Static Coker Unit

determining temperature uniformity of the test specimens and for identifying any temperature differences between the four test units. Positions measured were the center and at 12, 3, 6, and 9 o'clock (18 mm from the center). Those measurements were made at six temperature settings ranging from 245°C to 330°C. The average of each five measurements obtained on the four cokers at six temperatures were within 2°C of each individual measurement which shows a good uniformity of surface temperature. The average surface temperature measured with a thermocouple surface probe was 15±2°C lower than the recorded base temperature of 330°C obtained with the thermocouple just below the top of the base surface. Between recorder readings of 245°C to 315°C, the surface temperature was 19±2°C below the recorder temperature. Surface temperature measurements made on stainless steel and aluminum surfaces showed the same temperature uniformity and difference between recorder temperature and surface temperature. Test temperatures in this study are test specimen surface temperatures and not recorded coker base temperatures.

The metal test specimens (shim stock, stainless steel and aluminum) were cleaned with petroleum ether and cotton swabs, rinsed with petroleum ether, dried with lint free towels, heated at 50°C and then cooled before weighing. The weighed test specimens were placed on the coker base using forceps and vacuum was applied. The PTFE seal and seal sleeve were centered on the test specimen and the convection shield was placed over the test specimen, seal and seal sleeve. The insulation blocks were put around the shield and the sealing ring was placed on the ferruled shield. After the test temperature was obtained, a one-gram sample was added to to the test specimen using a preweighed syringe containing the sample. This was accomplished by holding the tip of the syringe needle approximately 5 mm

above the center of the test specimen. The syringe was then reweighed for determining exact sample weight. At the conclusion of each test, the coker was disassembled and the test specimen was removed, cooled, and reweighed. Deposits were reported in milligrams per gram of oil along with a description of the deposits.

The purity of the esters used in this study was determined using gas chromatography. These analyses utilized a flame ionization detector, a 25-meter capillary column coated with SE-52 (phenyl methyl silicone) with a temperature program of 100°C to 325°C and a heating rate of 8°C/minute with a final five minute hold. Purity was calculated from peak area using a unity response factor.

The effects of lubricant thermal and oxidative stressing on coking were investigated using the Ministry of Defence (United Kingdom) Test Method DERD NO. 9, "Volatility and Oxidative Stability, High Temperature" (without oil make-up) and DERD NO. 1 "Confined Heating Stability" for stressing the lubricant prior to coking studies. Test Method DERD NO. 9 is essentially an oxidation test utilizing 50 ml test samples with no intermediate sampling, and 250 cc/minute flow of saturated air. This method does not require metal test specimens and water condenser for condensate return. Test Method DERD NO. 1 is a thermal stressing test utilizing a 100 ml stainless steel test chamber fitted with a 190 mm long, 6 mm inside diameter air condenser and a test sample size of 85 ml. Wear metal generation in the oil was accomplished using a multi-specimen friction and wear test machine (20). The pin-on-disk wear tests utilized AISI 1018 mild steel discs and AISI 440C stainless steel pins having a hardness of R_c 55 to 60, 1078 N (110 kgf) and a speed of 0.63 m/s. The gear simulation wear configuration consisted of two AISI 8620 steel pins (R_c 56) and two annular

rings of AISI 8620 steel having the same hardness. The pins roll and slide simultaneously on the two rings. A drive velocity of 0.51 m/s and a load of 1078 N (110 kgf) were used for these studies.

The iron content of the lubricant used for investigating the effect of wear on coking was accomplished using the acid dissolution method (21) and flame atomic absorption analysis. Particle size distribution was determined by filtering the lubricant containing the wear debris through various pore size membrane filters and subsequent analysis for iron concentration.

(3) Test Lubricants

Lubricants, esters and ester-additive mixtures used in the AFAPL Static Coker Study are described in Table 1, Section II.1.d

(4) Results and Discussion

(a) Test Repeatability

An ester base MIL-L-7808 lubricant was selected for determining test repeatability using four static coker units. Four coking tests were conducted on each unit and the results are given in Table 23. This data along with all static coking test data is given in Table A-3. A test temperature of 315^{o}C and a standard test time of 180 minutes using shim stock test surface were used for this test repeatability study. A mean deposit value of 14.1 mg/g oil was obtained with the standard deviation being 1.1. This standard deviation is typical for lubricant deposition testing and was considered satisfactory for these studies. The evaluation of this lubricant nine months later using the same four coking units gave a mean deposit value of 14.3 mg/g and a standard deviation of 1.7. Data generated during the course of this study has shown that test repeatability can vary for different lubricant formulations. All test values reported are the mean

TABLE 23

REPEATABILITY OF STATIC COKER DEPOSITS USING MIL-L-7808 LUBRICANT, 315°C TEST TEMPERATURE AND SHIM STOCK SURFACE

	Coking Deposit, mg/g (Static Coker Number)			
Test No	1	2	3	4
1	13.6	14.9	15.4	15.0
2	12.2	13.3	12.8	15.0
3	13.5	15.9	13.0	13.2
4	13.0	14.7	14.0	15.3
Mean	13.1	14.7	13.8	14.6
Std. Dev.	0.6	1.1	1.2	1.0

Mean Value, All Data - 14.1 mg/g oil

Std. Dev. - 1.1

of two or more tests. The number of tests conducted for obtaining each test value depended on the deviation obtained for the initial two tests.

(b) Effects of Test Surface Material on Lubricant Coking

Table 24 gives the coking values obtained using aluminum, stainless steel, quartz and shim stock coking surfaces for two classes of ester base fluids. Shim stock gave the highest deposit levels for all six MIL-L-7808 (specification for 3 cSt ester base lubricants) fluids and two of the MIL-L-23699 (specification for 5 cSt ester base lubricants) fluids. Quartz gave the second highest values for four of the MIL-L-7808 fluids. The two MIL-L-7808 fluids (O-82-3 and O-79-17) not giving the second highest deposit with quartz showed very small differences in the deposit levels between the aluminum, stainless steel and quartz test surfaces. The effect of test surface was less for the MIL-L-23699 fluids than the MIL-L-7808 fluids with the MIL-L-23699 differences being marginal when considering test repeatability. Shim stock was selected as the coking surface for most of this study due to its superior test repeatability, low cost, uniformity of material, and ease in cutting 50.8 mm diameter test surfaces. Aluminum provided the worst coking surface due to poor wettability of the oil on aluminum at coking temperatures which can contribute to poor test repeatability.

(c) Coking Deposits Versus Molecular Weight of Ester

Lubricant volatility is an important factor in lubricant deposition testing, especially for those tests involving very low to no lubricant flow during testing. This condition is typical of many engines having areas of low oil flow and trapped pockets of lubricant due to fog or mist lubrication in high temperature regions. Temperature rise due to heat soak-back on engine shut-down also contributes to coking under these

TABLE 24

EFFECT OF TEST SURFACE MATERIAL ON LUBRICANT COKING DEPOSITS AT 315°C

Lubricant	Aluminum AMS-4037F (0.032 in)	Stainless Steel AISI 302 (0.019 in)	Quartz - (0.052 in)	Shim Stock AMS 5045 (0.005 in)
MIL-L-7808 Type				
	mg/g	mg/g	mg/g	mg/g
O-79-16	9.6	11.2	12.3	14.1
O-79-17	11.8	9.8	11.2	13.2
O-79-20	9.3	11.8	12.2	14.7
O-82-2	13.9	11.1	13.9	14.6
O-82-3	9.8	8.6	8.6	14.7
O-82-14	11.9	13.8	16.1	16.9
MIL-L-23699 Type				
O-71-6	28.9	29.2	-	32.0
TEL-6021	35.6	32.0	-	33.4
TEL-6022	36.3	37.7	-	39.9

conditions.

Two competing mechanisms involving the rate of volatility and the rate of degradation with subsequent deposition determine the coking characteristic of the lubricant under these conditions. If the volatility is sufficiently high, very low deposits will be formed. Volatility is a function of vapor pressure and for esters used in turbine lubricants, the vapor pressure varies with molecular weight according to Log P=2.48318-0.00539M (22). Figure 36 shows the relationship between coking deposits for six technical grade basestock esters and their respective theoretical molecular weights. The purity of these esters determined by GC is also shown. As expected, coking deposits increased as the molecular weight increased. The impurities could increase or decrease the deposit level depending upon the type of contaminants.

(d) Effect of Antioxidants on Coking Deposits

The effect of antioxidants on lubricant coking was investigated using two diesters, one polyol ester and commonly used antioxidants. The esters consisted of commercial grade trimethylolpropane triheptanoate (TMPH) having a 93 percent purity and two di-2-ethylhexyl adipate esters (D2EHA) with one having a relatively low purity of 89 percent and one having a high purity of 99 percent. Each ester was blended with 0.5, 1.0, 1.5 and 2.0 percent weight of phenyl-alpha-naphthylamine (PANA) and dioctyldiphenylamine (DODPA). The antioxidants were commercial grade and were not purified prior to use.

Table 25 shows the coking deposits obtained for the various esters and ester-additive mixtures. The addition of 0.5 percent of both antioxidants reduced the quantity of deposits for the polyol ester TMPH. Increasing the concentration of both antioxidants from 0.5 to 2.0 percent in

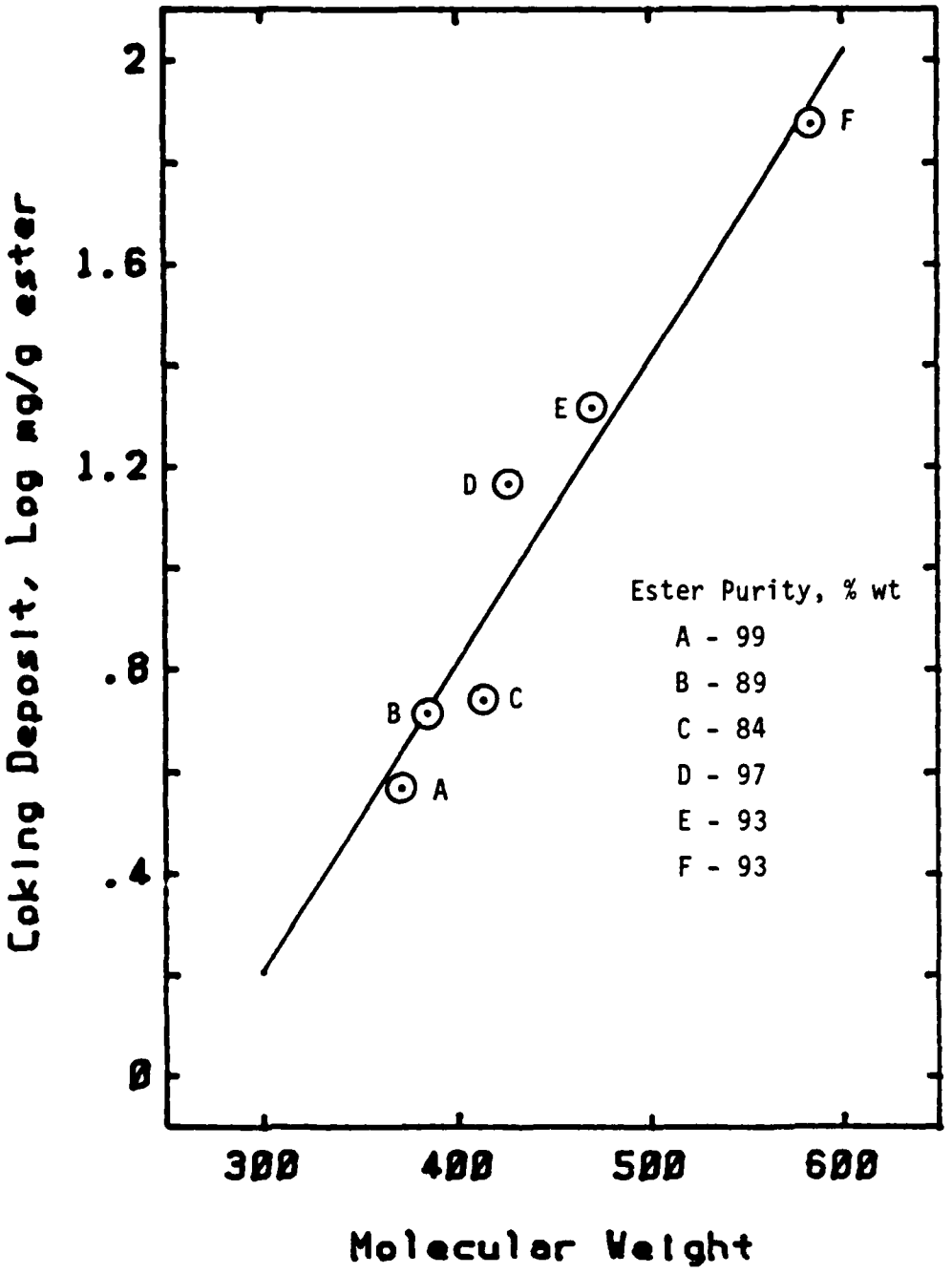


Figure 36. Coking Deposit Versus Molecular Weight of Ester; (A) di-2-ethylhexyl adipate (B), 2, 2-dimethyltrimethylene nonanoate (C) di-2-ethylhexyl azelate, (D) di-2-ethylhexyl sebacate, (E) trimethylolpropane triheptanoate, (F) pentaerythritol tetraheptanoate

TABLE 25

EFFECT OF ANTIOXIDANTS ON COKING DEPOSITS AT 315°C
TEST TEMPERATURE AND SHIM STOCK SURFACE

	% Wt Phenyl-alpha-naphthylamine				
	0.0	0.5	1.0	1.5	2.0
	mg/g	mg/g	mg/g	mg/g	mg/g
O-76-5*	21.2	17.0	16.8	17.3	16.3
O-76-8#	5.9	6.2	8.8	12.0	12.0
O-77-1+	3.7	6.5	6.4	7.8	7.6

	% Wt Dioctyldiphenylamine				
	0.0	0.5	1.0	1.5	2.0
	mg/g	mg/g	mg/g	mg/g	mg/g
O-76-5	21.2	16.0	14.8	13.6	14.2
O-76-8	5.9	6.4	7.6	11.2	12.8
O-77-1	3.7	3.8	4.2	4.3	5.1

* Trimethylolpropane triheptanoate, 93% purity
\# Di-2-ethylhexyl adipate, 89% purity
\+ Di-2-ethylhexyl adipate, 99% purity

this ester did not significantly increase deposits. GC analysis of the TMPH ester showed the 7 percent impurities to be primarily trimethylolpropane esters other than TMPH. The lowering of deposits using 0.5 percent antioxidant in the TMPH could be due to reactions between the antioxidant and some undetected impurity giving rise to a more volatile material and/or reducing the rate of degradation of the ester.

The addition of the two antioxidants to both of the two diesters increased the quantity of deposits as the additive concentration increased, with the lower purity ester deposits increasing to twice the deposit level of the purer ester at 2 percent additive concentration. GC analysis of the lower purity ester showed the impurities to be 8.3 percent TMPH, 1.5 percent 2-ethylhexanol and 1.5 percent unidentified components. The TMPH impurity would increase the deposit level slightly due to its lower volatility and as shown by Table 25, the lower purity ester does have the highest deposit level between the two diesters without additives. The data indicates the increase in deposits for the diesters with antioxidants is due mostly to reactions between the antioxidants and ester impurities. No significant difference was found between the two antioxidants with respect to their effect on lubricant coking.

No differences were seen in the physical appearance of deposits formed by the three esters or the ester/additive mixtures. All tests gave hard, brown to black, glossy smooth deposits.

(e) Effect of Temperature on Lubricant Coking

Coking characteristics of four classes of ester base lubricants representing 13 different formulations and three esters were studied at 245, 260, 300 and 315°C using shim stock test surfaces. Table 26 shows the deposit values of each fluid at the various test temperatures with

TABLE 26

EFFECT OF TEMPERATURE ON LUBRICANT COKING DEPOSITS
USING SHIM STOCK TEST SURFACE

Lubricant	Test Temperature 245°C	260°C	300°C	315°C
MIL-L-7808 Type	mg/g	mg/g	mg/g	mg/g
O-79-16	16.0	16.4	13.4	14.1
O-79-17	13.9	13.4	12.8	13.2
O-79-20	22.2	22.6	15.3	14.7
O-82-2	22.6	24.0	15.6	14.6
O-82-3	23.8	17.7	14.7	14.7
O-82-14	12.9	15.0	16.2	16.9
TEL-6034	5.3	5.6	8.4	8.2
MIL-L-23699 Type				
O-71-6	82.2	58.2	34.5	32.0
TEL-6021	98.2	68.1	35.7	33.4
TEL-6022	116.6	64.1	36.1	39.9
4 Centistoke Candidate Fluid				
O-85-1	33.5		27.6	27.7
7.5 Centistoke Fluids				
TEL-6031	57.8	41.4	30.1	30.5
TEL-6032	24.2	20.5	12.8	10.4
Basestock Esters				
O-76-5	28.2	23.4	19.9	21.2
O-76-8	12.5	12.5	13.0	5.9
O-77-1	9.7	6.9	7.9	3.7

each value being the mean of two or more determinations.

Each of the seven MIL-L-7808 lubricants showed no coking differences between 300°C and 315°C. Below 300°C, three of the lubricants showed a significant increase in deposits, one showed no difference and two lubricants showed a slight decrease. The mean of the deposit values for the MIL-L-7808 fluids was 13.8 mg/g oil at 315°C and 17.4 mg/g oil at 245°C. All of the oils produced hard brown to black deposits at 260°C and above. Three of the lubricants (O-79-17, O-82-3 and TEL-6034) had tacky deposits at 245°C.

The three MIL-L-23699 lubricants showed a decrease in deposits with increasing temperature throughout the range of 245°C to 315°C. Their deposit values were much greater than the deposit values for the MIL-L-7808 fluids with the mean deposit value being 34.4 mg/g oil at 315°C and 99.0 at 245°C. All three lubricants produced hard brown to black deposits at 300°C and 315°C. Lubricant O-79-18 produced a tacky deposit at 260°C and at 245°C. Lubricant O-77-15 produced a tacky deposit at 245°C.

The 4 cSt candidate fluid produced a deposit value at 315°C close to the values of MIL-L-23699 fluids. Only a very small increase in deposit occurred with a decrease in temperature. At 245°C, the deposit value was near the values for the MIL-L-7808 fluids. The deposits of this fluid were hard and brown to black for all test temperatures except 245°C. At this temperature the deposit was tacky.

The two 7.5 cSt lubricants did not give the amount of deposits expected for higher viscosity fluids. This is partially due to the narrow molecular weight range of the esters used for the basestocks. However, this does not explain the vast difference between the two 7.5 cSt fluids since TEL-6031 has a lower apparent molecular weight than TEL-6032 based on GC retention times. The 7.5 cSt fluid TEL-6032 very closely

resembles MIL-L-7808 fluids with respect to coking. The deposits formed from these lubricants were hard and brownish black at all test temperatures.

The basestock esters, identified in Table 25, resembled MIL-L-7808 fluids producing about the same level of deposits and with all deposits being hard and brownish black. One difference found between the deposits of formulated lubricants and basestock esters was the tendency of the ester deposits to be flaky, especially if the test specimen cooled too fast. No flaky deposits were observed for any of the formulated lubricants.

(f) Effect of Lubricant Thermal and Oxidative Stressing on Coking Deposits

Table 27 shows the effect of 205°C, 48 hour confined heat stressing on lubricant coking at 315°C using shim stock surface. The degree of stressing is shown by the increase in total acid number (TAN) and viscosity from new fluid. The MIL-L-7808 lubricants showed moderate to high increases in TAN (1 to 6) and small increases in viscosity (2 to 6 percent). All of these lubricants had significant increases in coking deposits which cannot be attributed to volatility. The MIL-L-23699 fluids showed moderate increases in TAN (1 to 3) and small increases in viscosity (1 to 4 percent). The coking values increased only slightly for two of the fluids while the third fluid showed a slightly lower (within test repeatability) coking value. No correlation exists between the increase in coking deposits and changes in TAN or viscosity.

Table 28 shows the effect of 205°C, 24 hour and 48 hour oxidative stressing using the same coking test conditions. The 24 hour stressed MIL-L-7808 lubricants had low to moderate TAN increases, moderate viscosity increases and moderate deposit increases which were slightly higher than that obtained after 48 hours thermal (confined heat) stressing. The

TABLE 27
EFFECT OF 205°C CONFINED HEAT STRESSING ON LUBRICANT COKING
AT 315°C ON SHIM STOCK SURFACE

Ester Base Lubricants	NEW LUBRICANT Total Acid No	NEW LUBRICANT Viscosity at 100°C cSt	NEW LUBRICANT Deposit mg/g oil	48 HOUR STRESSED LUBRICANT Total Acid No Increase	48 HOUR STRESSED LUBRICANT Viscosity Increase %	48 HOUR STRESSED LUBRICANT Coking Deposits mg/g oil	48 HOUR STRESSED LUBRICANT Coking Deposits Increase %
MIL-L-7808 Type Fluid							
O-79-16	0.20	3.16	14.1	2.49	2.2	18.2	29.1
O-79-17	0.08	3.35	13.2	2.66	2.4	19.2	45.5
O-79-20	0.20	3.47	14.7	1.38	4.0	24.6	67.3
O-82-2	0.08	3.33	14.6	3.45	2.7	18.4	26.0
O-82-3	0.12	3.45	14.7	1.11	3.5	17.9	21.8
O-82-14	0.15	3.40	16.9	6.05	6.5	26.4	56.2
MIL-L-23699 Type Fluid							
O-71-6	0.06	4.95	32.0	2.67	4.4	35.7	11.6
O-77-15	0.43	4.95	33.4	2.99	4.0	39.0	16.8
O-79-18	0.07	5.29	39.9	0.76	0.9	39.0	-2.3

TABLE 28

EFFECT OF 205°C OXIDATIVE STRESSING ON LUBRICANT COKING
AT 315°C ON SHIM STOCK SURFACE

Ester Base Lubricants	24 HOUR STRESSED LUBRICANT				48 HOUR STRESSED LUBRICANT			
	Total Acid No Increase	Viscosity Increase %	Coking Deposits mg/g oil	Coking Deposits Increase %	Total Acid No Increase	Viscosity Increase %	Coking Deposits mg/g oil	Coking Deposits Increase %
MIL-L-7808 Type Fluid								
0-79-16	0.31	8.5	22.1	56.7	0.58	17.7	25.0	77.3
0-79-17	0.44	13.1	21.2	60.6	0.69	20.3	23.4	77.3
0-79-20	0.35	7.5	26.6	81.0	0.55	14.4	29.0	97.3
0-82-2	1.82	7.8	20.2	38.3	3.12	14.7	26.2	79.5
0-82-3	0.56	13.3	26.5	80.2	1.18	26.1	33.4	127.2
0-82-14	1.43	11.5	30.6	81.1	2.62	17.9	35.3	108.9
MIL-L-23699 Type Fluid								
0-71-6	0.29	10.3	35.2	10.0	0.68	14.1	45.6	42.5
0-77-15	0.31	7.5	37.6	12.6	0.09	11.1	47.8	43.1
0-79-18	0.30	2.8	40.7	2.0	0.83	5.3	44.3	11.0

higher viscosity change caused by the oxidation test would contribute to an increase in deposits. Very little change occurred for the MIL-L-23699 lubricants for either TAN, viscosity or deposits. The 48 hour stressed MIL-L-7808 lubricants showed low to moderate TAN increases not too different from the 24 hour stressed samples and a large viscosity change. Deposit levels increased for all the lubricants but not proportional to changes in TAN or viscosity. The MIL-L-23699 fluids showed only slight changes in TAN or viscosity after 48 hour oxidative stressing but deposits increased significantly and showed a much greater percentage increase between the 24 hour and 48 hour stressed samples than the MIL-L-7808 lubricants.

(g) Effect of Wear Debris on Lubricant Coking

The particle size distribution of the nascent wear debris (wear particles generated within the lubricant) and the effect of the wear debris on lubricant coking are given in Figure 37 and Table 29. The data in Figure 37 shows the wear debris from the pin-on-disk wear test generates particles of which 94 percent weight have sizes below 8 microns. As expected the gear simulation test produces larger size particles with approximately 90 percent weight of the particles being above 3 microns. The pin-on-disk test produces more of the smaller particles with approximately 67 percent being below 3 microns. These particles sizes are typical of normal wear particles generated in aircraft turbine engines.

The effect of wear debris on lubricant coking is shown in Table 29. Coking deposits for two MIL-L-7808 lubricants and trimethylolpropane triheptanoate, with and without wear debris are given using both quartz and shim stock coking surfaces. All fluids show the same effect in coking deposits. No difference was seen between the coking values conducted on fluids containing wear debris using quartz and shim stock test

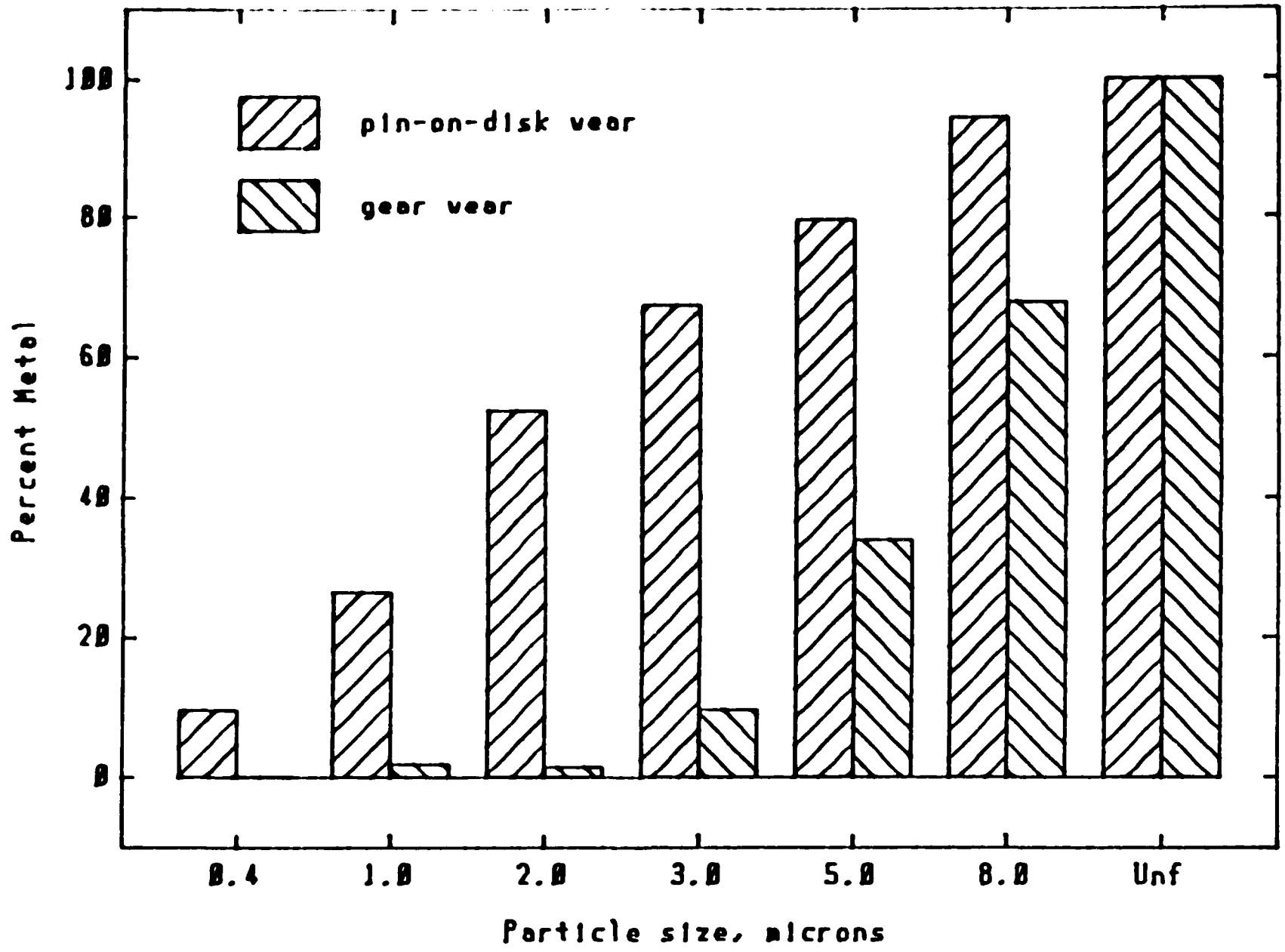


Figure 37. Particle Size Distribution of Wear Metal Generated from the Pin-on-Disk and Gear Wear Tests

TABLE 29

EFFECTS OF WEAR DEBRIS ON LUBRICANT COKING AT 315°C
ON SHIM STOCK SURFACE

Lubricant	Type of Wear Test	Iron Content ppm	Coking Deposits, mg/g	
			Quartz Surface	Shim Stock Surface
MIL-L-7808 Type				
O-79-16	None	0	12.3	14.1
O-79-16	Pin-on-Disk	136	14.5	14.8
O-79-16	Gear Simulation	510	14.8	14.0
O-82-3	None	0	8.6	14.7
O-82-3	Gear Simulation	271	14.2	14.8
Ester*				
O-76-5	None	0	16.9	21.2
O-76-5	Gear Simulation	532	20.7	21.3

*Trimethylolpropane triheptanoate, 93% purity

surfaces. A difference is seen between the new fluid and corresponding wear containing fluid when coking is done with quartz specimen. This difference is of the same order of magnitude as that seen between new lubricant using quartz and shim stock test surfaces. This data indicates that in this test, iron has a limited effect on lubricant coking and this effect occurs with wear debris or iron shim stock to the same degree.

(h) Effect of Lubricant Filtering on Coking Deposits

The effect of filtration on the coking characteristics of thermally and oxidatively stressed ester-additive mixtures was investigated using three esters and two antioxidants with each being blended at 2.0% concentrations. The three esters were O-76-5 (TMPH) O-76-8 and O-77-1 (both D2EHA esters) and the two antioxidants were phenothiazine (PTZ) and dioctylphenothiazine (DOPTZ). These additives were selected for determining the effect of the two octyl groups on solubilizing lubricant additive breakdown products. Table 30 shows the properties of the stressed esters after 48 hours confined heat testing and after 24 hours and 48 hours oxidative stressing at 205°C. The 48 hour confined heat stressing caused a significant increase in TAN for all ester-additive mixtures. COBRA values increased for the ester-additive mixtures but not in the same order of magnitude as the TAN values. Only moderate increases occurred for changes in viscosity. The esters containing PTZ gave more visual deposits in the test oil and higher toluene insoluble contents, especially for O-76-5 plus 2% PTZ. Each ester blended with 2% DOPTZ showed the equivalent degree of degradation to that of 2% PTZ in each ester. However, the level of visual deposits and toluene insolubles were much lower with the DOPTZ additive.

The 24 hour oxidative stressed lubricant test data given in Table 30 shows moderate degradation for esters O-76-5 and O-77-1 containing

TABLE 30

PROPERTIES OF STRESSED ESTERS CONTAINING ANTIOXIDANTS
USED FOR COKING STUDIES AT 315°C AND SHIM STOCK SURFACE

48 Hour Confined Heat Stressing at 205°C

Lubricant	Weight Loss, %	TAN Increase	COBRA	Viscosity at 100°C % Increase	Visual Deposits in Oil	Toluene Insolubles, % wt
O-76-5/2% PTZ(1)	0.0	3.89	7	2.8	large amount	0.66
O-76-8/2% PTZ	0.3	5.71	120	7.5	large amount	0.10
O-77-1/2% PTZ	0.0	1.78	100	9.7	moderate amount	0.02
O-76-5/2% DOPTZ(2)	0.0	4.80	28	2.8	None	0.06
O-76-8/2% DOPTZ	0.4	6.12	200 +	4.5	slight amount	0.03
O-77-1/2% DOPTZ	0.6	2.81	46	0.8	slight amount	0.03

(1) Phenothiazine

(2) Dioctylphenothiazine

TABLE 30 (CONCLUDED)

PROPERTIES OF STRESSED ESTERS CONTAINING ANTIOXIDANTS
USED FOR COKING STUDIES AT 315°C AND SHIM STOCK SURFACE

24 Hr Oxidative Stressing at 205°C

Lubricant	Weight Loss, %	Increase	COBRA	Viscosity @ 100°C % Increase	Visual Deposits In Tube	Toluene Insolubles, % Wt
O-76-5/2% PTZ	7.0	0.34	5	4.3	Heavy Coke	0.06
O-76-8/2% PTZ	21.1	11.88	25	14.2	" "	0.04
O-77-1/2% PTZ	18.4	0.74	4	2.1	" "	0.02
O-76-5/2% DOPTZ	7.2	0.28	4	2.2	" "	0.05
O-76-8/2% DOPTZ	22.3	9.90	24	12.2	" "	0.05
O-77-1/2% DOPTZ	14.9	0.82	7	0.0	" "	0.02

48 Hr Oxidative Stressing at 205°C

O-76-5/2% PTZ	13.8	0.40	6	5.7	Heavy Coke	0.01
O-76-8/2% PTZ	-	-	-	-	-	-
O-77-1/2% PTZ	34.1	0.90	6	3.0	Heavy Coke	0.02
O-76-5/2% DOPTZ	18.2	5.33	110	32.3	Slight Varnish	0.00
O-76-8/2% DOPTZ	-	-	-	-	-	-
O-77-1/2% DOPTZ	33.7	9.10	200 +	9.9	Slight Varnish	0.01

either antioxidants but severe degradation for diester O-76-8. Heavy coke deposits existed on all tubes usually just above the oil level. Toluene insoluble content was low for all 24 hour samples. After 48 hours of oxidative stressing, neither O-76-5 nor O-77-1 ester showed significant degradation with the antioxidant PTZ. Both esters showed much greater degradation when containing the same concentration of DOPTZ. Although the weight percent of each antioxidant blend was 2.0%, the amount of active antioxidant would be less due to the octyl groups which may explain the difference seen between PTZ and DOPTZ. The two esters with PTZ gave heavy coke deposits on the oxidation tubes while the DOPTZ/ester mixtures had only slight varnish deposits. The toluene insoluble content was very low for both antioxidants in each ester.

The data given in Table 31 shows the effect of 0.45 micron filtering on coking deposits for both the thermally and oxidatively stressed esters (O-76-5, O-76-8 and O-77-1) containing the antioxidants PTZ and DOPTZ. The addition of PTZ and DOPTZ changed the coking level of the three esters as shown below:

Ester	No Additive	2.0% PTZ	2.0% DOPTZ
	(Deposit values, mg/g oil)		
O-76-5	21.2	21.0	21.8
O-76-8	5.9	10.7	14.8
O-77-1	3.7	6.6	6.8

The polyol ester O-76-5 showed no change in coking values using either PTZ or DOPTZ. This is different with respect to the addition of both PANA and DODPA to O-76-5 which decreased the deposit level. The increase in deposit levels for O-76-8 and O-77-1 containing either PTZ or DOPTZ was the same as that obtained by the addition of PANA or DODPA to the

TABLE 31

EFFECT OF FILTERING PRESTRESSED ESTERS CONTAINING ANTIOXIDANTS ON LUBRICANT COKING AT 315°C AND SHIM STOCK SURFACE

	New Oil Deposits mg/g oil	Oxidative Stressing at 205°C mg Deposits/g oil				Thermal Stressing at 205°C mg Deposits/g oil	
		24 hr Unfiltered	24 hr Filtered(1)	48 hr Unfiltered	48 hr Filtered	48 hr Unfiltered	48 hr Filtered
0-76-5/2% PTZ(2)	21.0	26.8	21.3	28.6	26.7	29.8	26.2
0-76-8/2% PTZ	10.7	18.8	14.6	-	-	14.2	15.3
0-77-1/2% PTZ	6.6	8.0	7.2	12.2	9.3	8.3	8.3
0-76-5/2% DOPTZ	21.8	25.2	20.4	69.0	68.6	25.8	21.3
0-76-8/2% DOPTZ	14.8	18.2	15.6	-	-	13.6	15.0
0-77-1/2% DOPTZ	6.8	9.3	9.2	17.2	16.8	16.4	13.8

(1) 0.45 Micron Filter

(2) Phenothiazine

(3) Dioctylphenothiazine

esters with the increase being twice as great as for O-76-8 as for O-77-1. As expected the unfiltered thermally and oxidatively stressed samples had increased deposit levels ranging from 16 to 76% for 24 hour oxidatively stressing, 36 to 216% for 48 hour oxidatively stressing and 0 to 141 % for the 48 hour thermally stressed ester additive mixtures. Ester O-76-5 gave a lower deposit increase with PTZ than O-77-1 with PTZ. With DOPTZ, O-76-5 showed the greatest increase (216%) in deposits compared to O-77-1 (153%). For thermal stressing, ester O-77-1 with 2.0% DOPTZ gave a very high deposit increase of 141%. This high increase was not obtained with any other ester-additive mixture as shown in Table 31. For the esters containing PTZ, filtering decreased the deposit values with the decrease ranging from 10 to 26% for 24 hour oxidative stressing, 7 and 24% for 48 hour oxidative stressing and 0 to 12% for 48 hour thermal stressing. For the esters containing DOPTZ, filtering also decreased the deposit values with the decrease ranging from 1 to 19% for 24 hour oxidative stressing, 1 and 2 % for 48 hour oxidative stressing and 16 to 17% for 48 hour thermal stressing. Fluid O-76-8 plus 2% DOPTZ gave a 10% increase after filtering which is probably within test repeatability.

(i) Comparison of the Static Coker with Other Deposition Tests

Data in Table 32 shows the static coker, the coking propensity test and the microcarbon residue tester (MCRT) which has previously been described (23) differentiate between the MIL-L-7808 and MIL-L-23699 lubricants in a similar manner relative to deposits. This is probably due to the similarity of tests.

Repeatability of each test must be considered in the ranking of the lubricants within each class of fluids. The repeatability of the tube deposition test deposit rating (weight of sludge plus 10 times the

TABLE 32

LUBRICANT COKING TENDENCIES WITH VARIOUS DEPOSITION TESTS

Ester Base Lubricants	Static Coker* 315°C Residue mg/g	Coking Propensity# 260°C Residue mg/20 cycles	MCRT+ 300°C/15h Residue % wt	TUBE DEP Rating	BEARING DEP Rating
MIL-L-7808 Type					
O-79-16	14.1	9.6	12.57	0.7	49
O-79-17	13.2	7.8	12.56	0.1	30
O-79-20	14.7	14.0	-	0.8	27
O-82-2	14.6	10.8	13.01	0.4	35
O-82-3	14.7	8.8	8.49	0.2	40
O-82-14	16.9	9.0	13.70	0.2	38
MIL-L-23699 Type					
O-71-6	32.0	113.4	24.15	-	37
TEL-6021	33.4	126.6	24.17	-	20
TEL-6022	39.9	136.2	-	-	
4 Centistoke Candidate Fluid					
O-85-1	27.7	-	-	0.1	33
7.5 Centistoke Fluids					
TEL-6031	30.5	97.0	10.04	-	-
TEL-6032	10.4	50.4	7.38	-	-

* Shim Stock Surface
\# 316 SS Cups
\+ Microcarbon Residue Tester

weight of coke) is plus or minus 0.75 of the mean of two or more values (15). Therefore, the merit of ranking the six MIL-L-7808 fluids having deposition ratings ranging from 0.1 to 0.8 is questionable. The mean of the overall deposit demerit rating using the bearing deposition test for the six MIL-L-7808 fluids is 37 with a standard deviation of 8. The test repeatability of the bearing deposition test, assuming each value is the mean of four tests, is plus or minus 8 with a 90 percent confidence limit (24). Thus considering test repeatability, only one MIL-L-7808 lubricant (O-82-3) showed a difference in coking characteristics using the five different tests (Table 32). With this lubricant, the coking propensity test and the MCRT gave lower deposit levels.

The bearing deposition test did not show the increased deposit levels for the MIL-L-23699 fluids as did the static coker, MCRT or coking propensity test. This is probably due to these tests being more affected by lubricant volatility.

The two 7.5 cSt lubricants were ranked in the same order with the static coker, MCRT, and coking propensity tests, with each test giving approximately twice the deposits for one oil compared to the other. However, levels of deposits are not what would be expected when comparing the data obtained from the MIL-L-7808 and MIL-L-23699 lubricants using the same three tests. The coking propensity values would be expected to be lower when compared to the deposit levels shown by the other two tests.

(5) Summary

This study has shown the static coker deposition tester to be a good low cost, short time laboratory test for studying the coking characteristics of lubricants under high temperature static oil conditions. The test has shown good repeatability and permits the measurement of the true

coking surface temperature.

Different deposit values were obtained for some of the lubricants when using different coking surfaces such as aluminum, stainless steel, quartz and shim stock. The shim stock gave the highest deposit values for all MIL-L-7808 and MIL-L-23699 lubricants and provided the best test repeatability.

The static coker was used to examine the effects of two antioxidants on deposit quantity when used with various esters. Phenyl-alpha-naphthylamine and dioctyldiphenylamine (antioxidants) produced the same effects on deposits for three different esters. Deposits decreased for one ester and increased for the other two esters with the addition of either additive. This study has shown the type and quantity of ester impurity have a very large effect on deposits when antioxidants are present. This emphasizes the importance of knowing ester purity and composition in any lubricant degradation study.

Thermal and oxidative stressing were found to also influence the quantity of coking deposits produced in the static coker. Four classes of ester base lubricants displayed very little change in coking deposits between temperatures of 300°C and 315°C. Between 245°C and 300°C major increases in the quantity and type of deposits occurred for some lubricants while the deposit level for other lubricants remained the same. The effects of oxidative and thermal stressing of the lubricant prior to deposit testing varied from 0 to 100 percent increase in deposits. These increases are partially due to lowering of volatility during the oxidation stressing but the deposit increase for the thermally stressed lubricants cannot be attributed to volatility loss.

The influence of wear debris on coking deposits was investigated

by generating metallic wear debris in the test oil and observing changes in coking deposits produced in the static coker. The wear debris study showed small increases in deposits between the new and wear debris containing lubricant if quartz coking surfaces were used. No difference was observed when using shim stock test surface. This indicates that the effect of iron is limited with respect to coking using the static coker and the effect occurs to the same degree with either wear debris or the shim stock test surface.

The effect of 0.45 micron filtration decreases the deposit values for the esters and antioxidants studied. The degree of decrease depended upon the specific ester-additive mixture, stressing time and type (thermal or oxidative) stressing.

Ranking of the MIL-L-7808 and MIL-L-23699 lubricants with respect to deposit levels was similar for the static coking test, MCRT and the coking propensity test with exception of one lubricant. This MIL-L-7808 lubricant (0-82-3) gave much lower deposits for the MCRT and coking propensity test. Considering test repeatability, the tube deposition test and the bearing deposition test show no significant difference between the lubricants studied. The 7.5 cSt fluids had lower deposits than would be expected for a heavier fluid when compared to the MIL-L-7808 or MIL-L-23699 fluids. This is apparently due in part to the relatively low and narrow range of molecular weight esters used for the lubricant basestocks. Although the static coker has demonstrated good capability in studying lubricant deposition, it must be remembered that other conditions exist (lubricant flow, varying oil residence times and environmental conditions) for lubricant coking within turbine engines' lubrication systems. Under these conditions, the static coker along with all other deposition tests which are influenced

by lubricant volatility should not be expected to provide correlative data with other dynamic deposition tests.

(6) Future Effort

Investigation of coking characteristics using the AFAPL Static Coker will be continued for other test conditions, test times and advanced high temperature ester base lubricants. Evaluation of the AFAPL Static Coker will be made with respect to its value as a specification test.

b. Coking Propensity Test

(1) Introduction

The coking characteristics of lubricants were investigated using the Coking Propensity test developed by Rolls-Royce Ltd.[1] This test measures the deposits formed by a lubricant under static conditions where lubricant volatility is an important factor. These conditions exist in areas of turbine engines where there is low or no oil flow and trapped pockets of lubricants are present due to fog or mist lubrication. Temperature rise due to heat soak-back on engine shut-down also contributes to coking under these conditions.

(2) Test Apparatus

The test apparatus consists of cylindrical metal dishes with the procedure requiring sandblasted aluminum dishes. Stainless steel (302) and polished aluminum test dishes were also used in this study for investigating the effect of metal and surface type on deposit levels. Coking of the test lubricants was conducted using a Blue M, Class A, Batch oven, Model No. PDM-106A-GHP having horizontal forced air circulation. A baffle was used to prevent the air flowing directly over the test dishes. Figure 38 shows the high temperature oven used for these studies. Coking propensity dishes are shown in Figure 39.

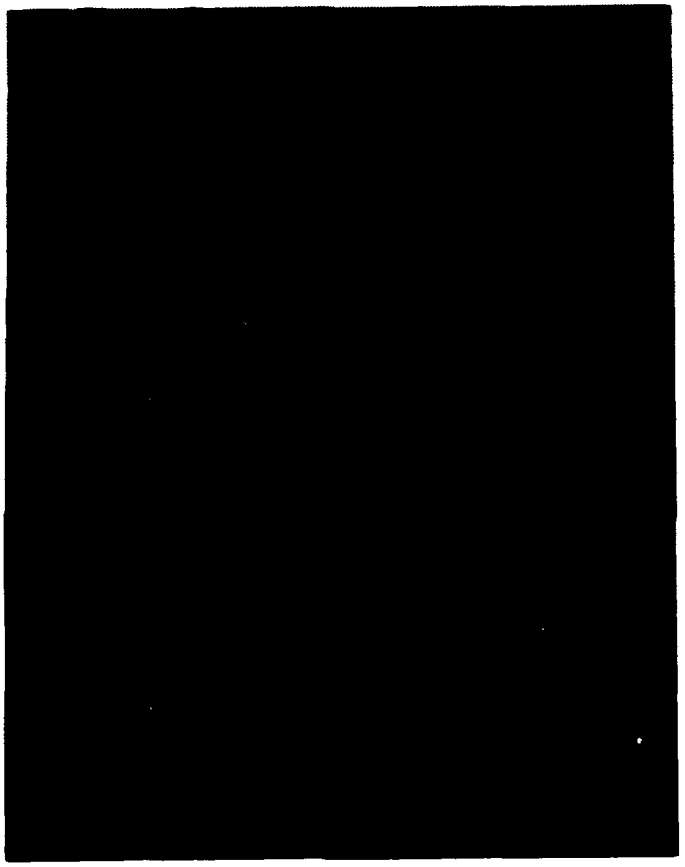


Figure 38. High Temperature Oven Used for Coking Propensity Test

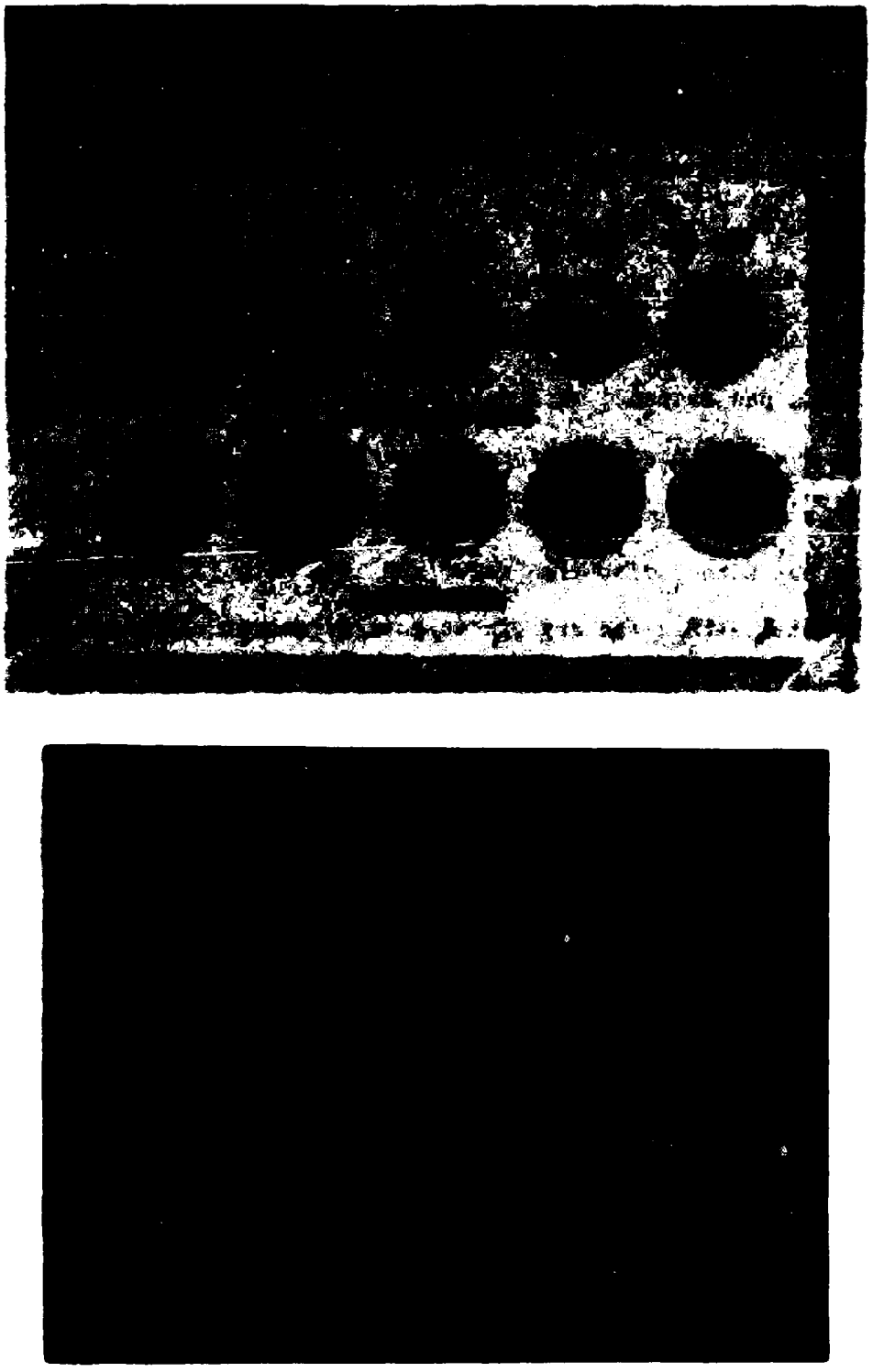


Figure 39. Photographs of Coking Propensity Test Dishes with Deposits

(3) Test Procedure

The stainless steel and aluminum dishes were polished using 400 grit silicon carbide paper to remove surface roughness and 600 grit silicon carbide paper for finer polishing. The test area of the aluminum sandblasted dishes was sandblasted by the base machine shop using 80 grit sandblasting material. Prior to each test using polished dishes, each dish was polished using a cotton pad dampened with reagent grade heptane, and dipped into 3 micron size corundum powder to form a scouring paste. The test surface was rubbed with a circular motion including the test walls. The dishes were rinsed with hot water, distilled water and then with isopropanol. Each dish was then treated with clean dry air to ensure removal of the scouring particles and for drying the dish. Sandblasted dishes were treated with clean dry air prior to use.

All dishes were heated to several degrees higher than the test temperature for a minimum of two hours and allowed to cool to room temperature prior to initiation of test.

The test dish was weighed to the nearest 0.1 mg and approximately 0.4 ml of test fluid was added to the dish. The dish was rotated and tilted to ensure coverage of the test surface. The dish containing the sample was re-weighed and then arranged on an aluminum tray for placing into the preheated oven set at the selected test temperature. Normally 10 to 12 test dishes were used during a test run. The dish (or dishes) was heated at the test temperature for 22 hours. The dish was then removed and placed in a large metal desiccator and allowed to equilibrate to room temperature which occurred within 1 to 1 1/2 hours. The test dish was then weighed for determining the amount of deposits. The test was repeated for 9 additional cycles.

After completion of the 10 test cycles, the total weight of the residue was divided by the weight of test fluid for providing a value expressing the residue in terms of milligrams of residue per gram of test fluid. This value was determined in two ways; one using the average milligrams per cycle and the second using the difference in dish weight between the final and initial dish weight. The expectant residue for 20 test cycles was determined statistically from the 10 test cycles.

(4) Test Lubricants and Test Conditions

A total of 12 lubricants consisting of 6 MIL-L-7808 type fluids, 3 MIL-L-23699 type fluids, a 4 cSt candidate lubricant and two 7.5 cSt lubricants were investigated. A description of these lubricants is given in Table 1, Section II.1.d with exception of O-79-18 which is a MIL-L-23699 type lubricant.

Testing was conducted at temperatures of 210, 235, 260 and 300°C.

(5) Results and Discussion

All coking propensity test data is given in Appendix A, Table A-4 with a summary of this data being given in Table 33. The data in Table 33 shows that the amount of deposits varies with the type of test dish. Stainless steel gives the highest coking values while sandblasted dishes produce the lowest coking values. This effect occurs for all the lubricants but is more pronounced for the higher viscosity fluids such as the MIL-L-23699 fluids, the 4 cSt candidate lubricant and the two 7.5 cSt lubricants. The effect of test dish material on coking propensity at 300°C test temperature is shown in Table 34 for these higher viscosity fluids.

TABLE 33

SUMMARY OF COKING PROPENSITY TEST DATA

DEPOSITS, mg/g Oil

Type Fluid	Stainless Steel Dishes				Polished Aluminum Dishes				Sandblasted Dishes		
	210°C	235°C	260°C	300°C	210°C	235°C	260°C	300°C	235°C	260°C	300°C
MIL-L-7808											
0-79-16	0.9	1.7	1.2	-	0.6	1.4	1.7	-	0.6	0.9	-
0-79-17	-	1.8	0.9	-	-	1.4	0.9	-	0.8	0.2	-
0-79-20	2.1	1.4	1.7	-	2.1	1.0	1.6	-	-	0.1	-
0-82-2	-	3.2	1.4	-	-	2.4	1.6	-	-	0.4	-
0-82-3	-	2.0	1.0	-	-	2.1	0.6	-	-	-	-
0-82-14	3.7	1.7	1.0	-	3.1	1.4	0.7	-	0.7	0.4	-
MIL-L-23699											
0-71-6	-	15.6	14.0	11.1	-	-	12.4	9.6	-	-	0.9
0-77-15	-	12.2	15.2	9.1	-	-	10.3	7.5	-	-	1.0
0-79-18	-	16.7	15.8	10.1	-	-	14.9	9.7	-	-	3.1
4 cSt											
0-85-1	-	12.6	8.6	6.2	-	-	8.6	5.9	-	-	1.8
7.5 cSt											
TEL-6031	-	9.4	10.2	6.3	-	-	10.5	6.2	-	-	2.0
TEL-6032	-	3.5	5.9	3.2	-	-	4.4	2.9	-	-	0.3

TABLE 34

EFFECT OF TEST DISH MATERIAL ON COKING PROPENSITY AT 300°C TEST TEMPERATURE

Test Lubricant	Deposit, mg/g oil: Sandblasted Aluminum Dish	Polished Aluminum Dish	Polished Stainless Steel Dish
O-71-6	0.9	9.6	11.1
O-77-15	1.0	7.5	9.1
O-79-18	3.1	9.7	10.1
O-85-1	1.8	5.9	6.2
TEL-6031	2.0	6.2	6.3
TEL-6032	0.8	2.9	3.2

The data in Table 33 also shows that the level of deposits varies more with the class of lubricants than between different lubricants of the same class. Table 35 more clearly illustrates this difference.

TABLE 35

COKING PROPENSITY VERSUS LUBRICANT TYPE
(Stainless Steel Dishes, 260°C Test Temperature)

Type Fluid	Number Tested	Mean Deposit Value mg/gm	Deposit Value Range Lowest mg/gm	Highest mg/gm
Mil-L-7808	6	1.2	0.9	1.7
4 cSt Candidate	1	8.6	8.6	8.6
MIL-L-23699	3	15.0	14.0	15.8
7.5 cSt Lubricants	2	8.0	5.9	10.2

In general, the higher the viscosity the higher the deposit level. This would be expected since the higher viscosity oils usually have lower volatility. However, the two 7.5 cSt fluids do not give the amount of deposits expected for the higher viscosity. This is partially due to the narrow molecular weight range of the esters used for the basestocks of these lubricants. However, this does not explain the vast difference between the two 7.5 cSt fluids since lubricant TEL-6031 has lower apparent molecular weight than TEL-6032 based on gas chromatography retention times, but gives about twice the amount of deposits as TEL-6032.

As shown by the data in Tables A-4 and A-5 some of the lubricants had a tendency to creep up the sides of the test dishes which appeared to be slightly worse with the sandblasted dishes.

Good correlation was usually obtained between the deposit values calculated from the mean value for 10 cycles and the value calculated from the final and initial weight of the test dishes and is shown in Table A-4.

Ranking of the MIL-L-7808 and MIL-L-23699 relative to deposit levels varied to a small degree depending upon the test dish being stainless steel or polished aluminum but the overall ranking of the 12 lubricant was the same as for the AFAPL Static Coker shown in Table 32. Varying the test temperature also changed the relative ranking of the lubricants (within each class) with respect to deposit levels. Part of these differences in ranking of the lubricants could be due to test repeatability and very low deposit values. Tables 36 and 37 show test repeatability using polished aluminum and stainless steel dishes at a test temperature of 235^{o}C and aluminum test dishes at 260^{o}C. Repeatability was determined for both deposit values calculated from the mean and from the final and initial weight of the test dish. All lubricants used in the repeatability study were MIL-L-7808 type

TABLE 36

REPEATABILITY OF LUBRICANT COKING PROPENSITY TEST DATA
235° TEST TEMPERATURE

ALUMINUM TEST DISHES

	RESIDUE, mg/gm oil[a]				RESIDUE, mg/gm oil[b]			
	Test 1	Test 2	$\bar{X}$	σ	Test 1	Test 2	$\bar{X}$	σ
0-79-16	1.9	0.9	1.4	0.7	1.7	0.8	1.2	0.6
0-79-17	1.6	1.2	1.4	0.3	1.4	1.4	1.4	0.0
0-79-20	1.1	0.9	1.0	0.1	0.9	0.7	0.8	0.1
0-82-2	2.4	2.5	2.4	0.1	2.4	2.4	2.4	0.0
0-82-3	2.2	2.0	2.1	0.1	2.2	2.0	2.1	0.1
0-82-14	1.6	1.3	1.4	0.2	1.50	1.3	1.4	0.1

STAINLESS STEEL TEST DISHES

	RESIDUE, mg/gm oil[a]				RESIDUE, mg/gm oil[b]			
	Test 1	Test 2	$\bar{X}$	σ	Test 1	Test 2	$\bar{X}$	σ
0-79-16	2.2	1.2	1.7	0.7	2.0	0.3	1.1	1.2
0-79-17	2.0	1.7	1.8	0.2	2.0	1.2	1.6	0.6
0-79-20	1.8	0.9	1.4	0.6	1.6	0.6	1.1	0.7
0-82-2	3.5	2.8	3.2	0.5	3.5	2.9	3.2	0.4
0-82-3	2.0	1.9	2.0	0.1	2.1	2.1	2.1	0.0
0-82-14	1.8	1.5	1.6	0.2	1.9	1.6	1.8	0.2

a - Calculated from the mean for 10 cycles

b - Calculated from final and initial weight for 10 cycles

fluids. This data showed fair to poor repeatability which is probably due to the very low amounts of deposits formed. Test repeatability was about the same for both stainless steel and polished aluminum dishes.

TABLE 37
REPEATABILITY OF LUBRICANT COKING PROPENSITY TEST DATA
260°C TEST TEMPERATURE

Aluminum Test Dishes

	Residue, mg/gm oil[a]				Residue, mg/gm oil[b]			
	Test 1	Test 2	$\bar{X}$	σ	Test 1	Test 2	$\bar{X}$	σ
O-79-16	1.7	1.6	1.6	0.1	1.7	1.2	1.4	0.4
O-79-17	0.8	1.1	0.9	0.2	0.7	0.5	0.6	0.1
O-79-20	1.5	1.6	1.6	0.1	1.4	1.4	1.4	0.0
O-82-2	1.7	1.5	1.6	0.1	1.7	1.2	1.4	0.4
O-82-3	0.5	0.7	0.6	0.1	0.4	0.0	0.2	0.3
O-82-14	0.6	0.8	0.7	0.1	0.4	0.2	0.3	0.1

a - Calculated from the mean for 10 cycles

b - Calculated from the final and initial weight for 10 cycles

The MIL-L-7808 lubricants showed very little differences in deposits when tested at 210, 235 or 260°C. Testing of the MIL-L-7808 lubricants at 300°C was initiated but was discontinued after four test cycles due to the lack of deposits. The MIL-L-23699 fluids showed a general increase in deposits with decreasing temperatures.

A description of coking propensity test deposits is given in Appendix A, Table A-5. Coking propensity test deposits for lubricants O-71-6, O-77-15, O-79-18, TEL-6031 and TEL-6032 and O-85-1 are shown in Figure 39. For these fluids, deposits varied from hard brown varnish to hard black deposits depending upon the temperature and type of test dish. The

texture of the deposits also varied and consisted of smooth, flaky or wrinkled deposits, again depending upon the temperature and type of test dish. The MIL-L-7808 lubricants varied from sticky brown varnish to hard dark brown to black deposits. These deposits were usually smooth for all three types of test dishes.

(6) Summary

Coking propensity testing conducted on the various lubricants gave very low levels of deposits for the MIL-L-7808 fluids and much higher deposit levels for the MIL-L-23699 fluids. The two 7.5 cSt fluids and the 4 cSt fluid had intermediate deposit levels. Rating of the 12 fluids with respect to deposit formation was the same as for the AFAPL Static Coker for all test temperatures investigated, although the deposit levels for all fluids were much lower with the coking propensity test. This is probably due to each test having a different effect on lubricant volatility.

(7) Future Effort

Coking propensity testing will be conducted on the higher viscosity lubricants at lower test temperatures along with coking studies of new ester base lubricants at high test temperatures.

8. LUBRICANT FOAMING STUDY

a. Introduction

The foaming characteristics of turbine lubricants are normally determined using both static and dynamic testing described in AFAPL-TR-75-91, ASTM Method D 892 and Federal Test Method Standards 3213 and 3214. A limitation of these foam tests is the large quantity of sample required which prevents determining the foaming characteristics of small samples of used lubricating fluids.

The objective of this study was the development of a static foaming

test requiring 25 ml or less sample and which correlates with Federal Test Method 3213.

b. Test Apparatus

The test apparatus for conducting the standard foam test requiring 200 ml of sample is described in Federal Test Method 3213(25). The test consists of passing air through a 25.4 mm diameter spherical air diffusion stone which is immersed in 200 ml of the test fluid being contained in a 500 cc graduated cylinder immersed in a constant temperature bath of 80°C.

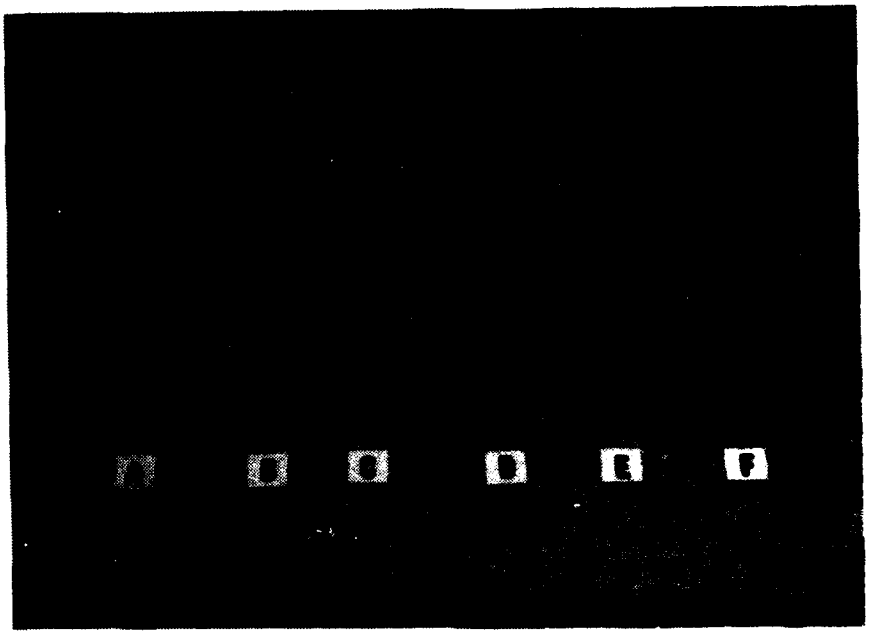
The test apparatus used for developing the small volume foam test consisted of various pore size and various diameter metal spargers and a sealed frit foaming tube, both of which are shown in Figure 40. The 25 ml volume foam testing using the air spargers and standard ASTM Stones was conducted in a 250 cc graduated cylinder immersed in an 80°C constant temperature bath. The sealed frit foaming tube was placed directly in the constant temperature bath.

c. Test Procedure

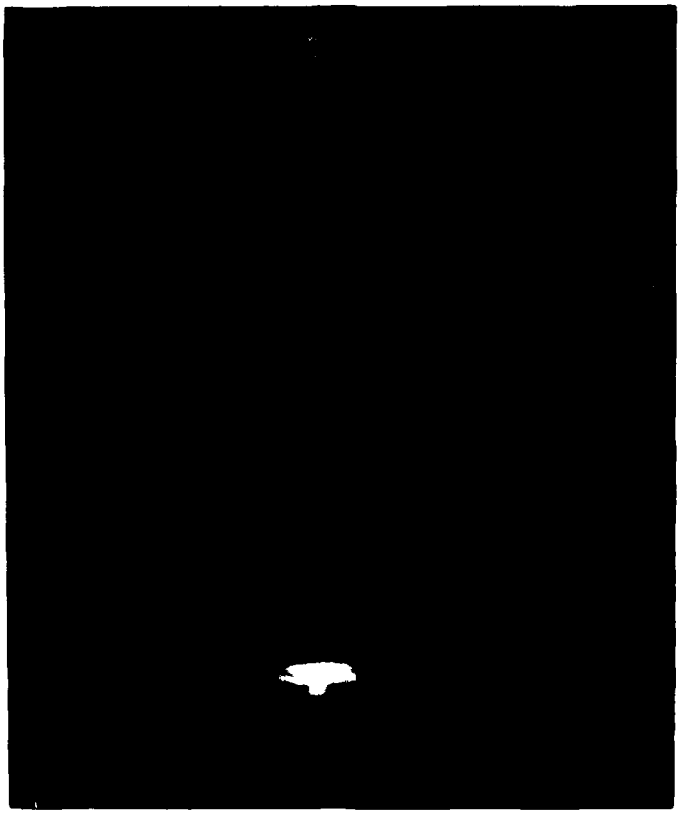
Aeration of the test lubricant was continued at a rate of 1000 cc/min for 30 minutes for both the standard and small volume testing. The maximum volume of foam occurring during the 30 minute test period was reported as the foaming value of the test fluid at that airflow rate. Aeration was then stopped and the time required for the foam to collapse was recorded. The test fluid was then aerated at 500 cc/min and the foam volume recorded at the end of 5 minutes. Foam collapse time was also recorded after the 5 minute aeration period.

Pore size and permeability of the ASTM air diffuser and the metal spargers were determined in accordance with ASTM Methods D 892 and E 128.

d. Test Lubricants



A



B

Figure 40. Small Volume Foam Test Equipment. A. Metal Spargers, (B) Sealed Frit Foamer Tube

Nine fluids having varied foaming characteristics were examined in this study and are described in Table 38.

TABLE 38

LUBRICANTS AND FLUIDS USED FOR FOAMING STUDY

Lubricant or Fluid	Description
O-76-1 plus 3 ppm DC-200-20 cSt*	MIL-L-7808 Lubricant with Silicone
O-76-1 plus 6 ppm DC-200-20 cSt	MIL-L-7808 Lubricant with Silicone
O-76-5	Trimethylolpropane Heptanoate Ester
O-76-8 plus 3 ppm DC-200-20 cSt	Di-2-Ethylhexyl Adipate with Silicone
O-76-8 plus 6 ppm DC-200-20 cSt	Di-2-Ethylhexyl Adipate with Silicone
O-79-17	MIL-L-7808 Lubricant
O-79-17 plus 3 ppm DC-200-500 cSt**	MIL-L-7808 Lubricant with Silicone
O-76-5 plus 2.0% TCP and 2.0% PANA	As Described
Fluid 5K3L6	MIL-L-7808 Lubricant

* Dow Corning Methyl Silicone DC-200 with 20 cSt Viscosity at 25°C
**Dow Corning Methyl Silicone DC-200 with 500 cSt Viscosity at 25°C

The fluids containing the DC-200 methyl silicone were prepared by blending the required amount of a 1.00% silicone concentration in toluene with the base fluid using mechanical agitation.

e. Results and Discussion

(1) Physical Properties of the Air Diffusers and Spargers Used for the Standard and Small Volume Foam Testing

Initial efforts involved determining the physical characteristics of the various physical size and pore size rating of the diffusers and spargers used in the foaming study. Table 39 gives a

TABLE 39

COMPARISON OF LUBRICANT DIFFUSER STONES AND AIR SPARGERS

Type of Diffuser	Shape	Size cm	Surface Area cm^2	Maximum Porosity microns(a)	Perme-ability cm^3/min(b)	Flow Rate cm^3/min/ cm^2 Surface(c)	Equiv Flow Rate/cm^2 Area to Diffuser 1&2
ASTM Stone 1A	Sphere	2.54 dia	14.74	60	3188	68	1.0
ASTM Stone 2A	Sphere	2.54 dia	14.74	48	3249	68	1.0
Glass Frit # 1	Cylinder	1.20 dia 2.20 lgth	9.42	41	4242	106	1.6
Glass Frit # 2	Cylinder	1.20 dia 2.20 lgth	9.42	40	3442	106	1.6
1/2" Dia. Sparger # 1 (40 Micron) (Rating)	Cylinder	1.25 dia 2.54 lgth	11.40	94	2489	88	1.3
1/2" Dia. Sparger # 2 (40 Micron) (Rating)	Cylinder	1.25 dia 2.54 lgth	11.40	84	2550	88	1.3
3/8" Dia. Sparger # 1 (10 Micron) (Rating)	Cylinder	0.95 dia 2.54 lgth	8.23	109	1947	121	1.8
3/8" Dia. Sparger # 2 (10 Micron) (Rating)	Cylinder	0.95 2.54 lgth	8.23	67	1918	121	1.8

TABLE 39 (Cont'd)

COMPARISON OF LUBRICANT DIFFUSER STONES AND AIR SPARGERS

Type of Diffuser	Shape	Size cm	Surface Area, cm^2	Porosity microns(1)	Permeability cm^3/min(2)	Flow Rate cm^3/min/ cm^2 Surface(3)	Equiv Flow Rate/cm^2 Area to Diffuser 1&2
1/4" Dia Sparger # 1 (2 Micron) (Rating)	Cylinder	0.64 dia 2.54 lgth	5.43	59	684	184	2.7
ASTM Stone 6A	Sphere	2.54 dia	14.74	152	3621	68	1.0
Glass Stone	Sphere	2.54 dia	14.74	88	3422	68	1.0
13/16" Dia Sparger # 3 5 Micron Rating	Cylinder	2.06 dia 2.54 lgth	16.43	24	2713	61	0.9

a. As Determined by ASTM D861

b. At 250 mm H_2O pressure

c. Total flow rate of 1000 cm^3/min

d. Top value for 200 cm^3 sample Lower value for 25 cm^3 sample

comparison of diffuser stones and spargers showing dimensions, surface area, maximum pore size, permeability and relative flow rates.

Initial foam testing using both 200 ml and 25 ml sample sizes and various pore size air diffusers and spargers showed no correlation between foam volumes and pore size as determined by ASTM D 892. An additional study of porosity and permeability was made with the data obtained being shown in Table 39. In addition to determining maximum pore size, the "mean" pore size was determined from the pressure required for giving visual uniform airflow (bubbles) all around the diffuser. Duplicate measurements, each by a different technician were made. As shown by Table 40, much less difference exists between the "mean" porosities than the maximum porosities for the various diffusers. The metal spargers show a characteristic decrease in pore size based upon airflow when compared to their micron ratings based upon spherical particle retention. This relationship is shown in Figure 41. Table 40 also shows good agreement between determining the maximum pore size of the 1/2" 2 micron sparger calculated using water (ASTM D 892) and a MIL-L-7808 lubricant (ASTM E 128). Pore size shown by microphotographs of various metal spargers obtained from an SEM (Scanning Electron Microscope) correlates closely with the calculated mean pore size as shown in Figure 42.

This study also showed changes in the porosity and permeability of the air diffusers occurring during the last six months. These changes are shown in Table 41. Four of the five air diffusers showed an increase in maximum pore size while one diffuser showed a decrease in maximum pore size. Four of the diffusers also showed an increase in permeability. These changes may be due to handling and continuous heating and cooling of the diffusers.

Pressure versus flow rate of the air diffusers was determined for characterizing the air diffusers and spargers. This data is shown in

TABLE 40

POROSITY AND PERMEABILITY OF FOAM TEST AIR DIFFUSERS

(Measurements made with 100 mm H_2O head pressure)

Diffuser	Pressure For First Bubbles (mm H_2O)	Maximum[1] Porosity (microns)		Pressure For Uniform Bubbles Around Diffuser (mm H_2O)	Equivalent[1] (Mean) Porosity (microns)		Permeability At 250 mm H_2O Pressure (ml/min air)
ASTM-1A	491	75	(82)[2]	648	53	(55)[2]	3546
ASTM-2A	368	109	(114)	630	55	(56)	4352
ASTM-6A	423	90	(95)	622	56	(56)	4571
1/2" Sparger # 1 40 Micron	321	132	(127)	525	69	(72)	2094
1/2" Sparger # 2 40 Micron	325	130	-	500	73	(85)	1089
1/2" Sparger # 12 20 Micron	432	88	(92)	686	50	(55)	1357
1/2" Sparger # 13 10 Micron	618	56	(57)	940	35	(38)	1276
1/2" Sparger # 14 5 Micron	940	35	(46)	1575	20	(25)	1112
1/2" Sparger # 15 2 Micron	1151	28	(27)	2324	13	(19)	915

(Measurements made with 100 mm MIL-L-7808 (0-82-3) head pressure)

1/2" Sparger # 14 5 Micron	610 mm oil	26[3]		1194	12		

(1) Calculated according to ASTM 892

(2) Repeat test by second operator

(3) Calculated according to ASTM E 128

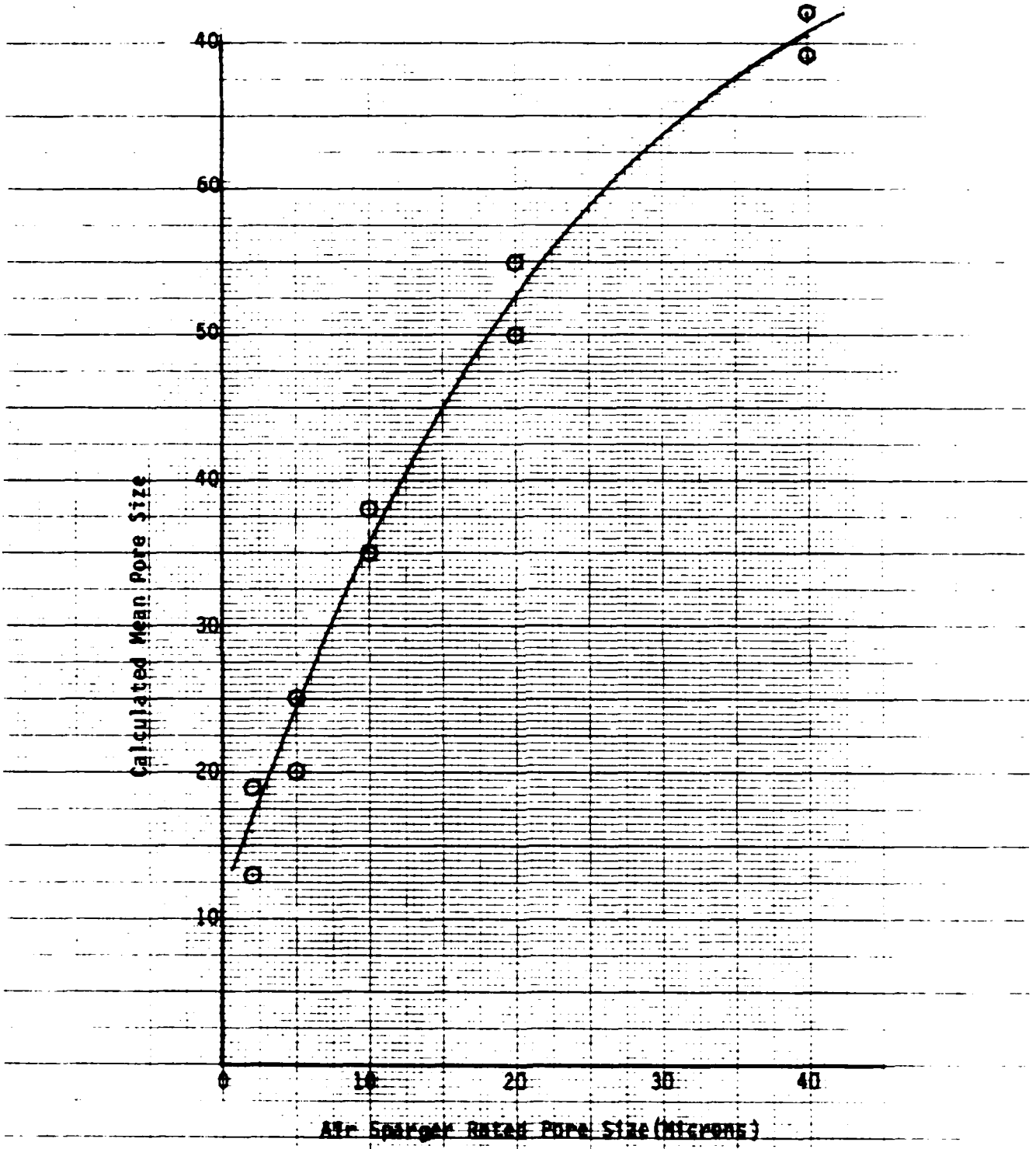


Figure 41. Air Sparger Rated Pore Size Versus Calculated Porer Size.

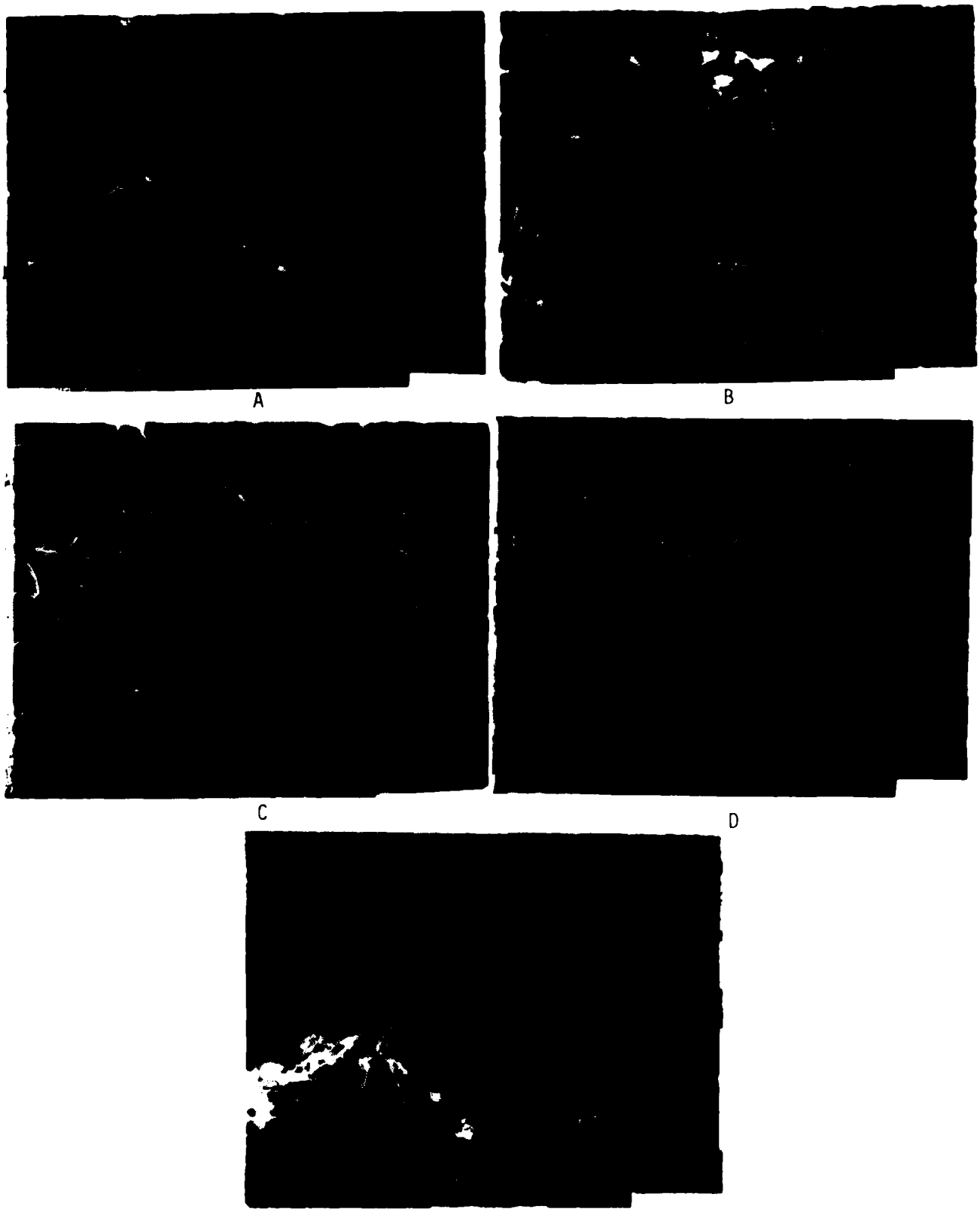


Figure 42. Microphotographs of Various Pore Size Metal Sparger Units at 4.5X Stone: (A) 2 Micron Sparger, (B) 5 Micron Sparger, (C) 10 Micron Sparger, (D) 40 Micron Sparger and (E) 4.5X Stone

TABLE 41

CHANGES IN POROSITY AND PERMEABILITY
OF FOAM TEST AIR DIFFUSERS

Type Diffuser	Maximum Pore Size (microns) Date of Test			Permeability (cc/min at 250 mm H_2O) Date of Test	
	30/9/85	27/3/86	31/3/86	10/10/85	27/3/86
ASTM-1A	60	80	78	3188	3909
ASTM-2A	48	114	116	3249	4466
ASTM-6A	152	95	92	3621	4475
1/2" Sparger # 1 (40 micron)	94	127	132	2489	2332
1/2" Sparger # 2 (40 micron)	84	115	130	2250	2366

Table 42. The pressures ranged from 406 mm H_2O (0.575 psi) at an airflow of 500 cc/min to 2108 mm H_2O (2.988 psi) at an airflow of 1000 cc/min. As expected this data shows that less pressure is required to provide a given air flow rate when reaching the required flowrate from a higher pressure due to reduced surface tension effects. This data also shows a much reduced pressure required for a given flowrate when using a head pressure of 100 mm of oil instead of 100 mm of water. The difference in densities (1.000 for H_2O versus 0.925 for the lubricant) is insufficient to account for the differences in pressure. However, the great difference in surface tensions (72 for water and 30 for the lubricant) would account for the large differences in pressures.

(2) Effect of Diffuser Pore Size and Sample Volume on Foaming

Initial foam testing involved various sample sizes of lubricants 5K3L6 and O-79-17 containing 3 ppm DC-200 500 cSt fluid using Test Method 3213 and a ASTM diffuser stone. Figures 43 and 44 show the effects of sample volume on foam volume for airflows of 1000 cc/min and 500 cc/min. For 1000 cc/min the change in foam volume with respect to change in sample volume is linear for both fluids. At an airflow of 500 cc/min using Federal Test Method 3213, a reversal in the foaming characteristics occurred between the two lubricants with a maximum foam volume occurring at approximately 40 ml sample volume for lubricant 5K3L6. The test data in Table A-5 (which includes all foaming test data), tests 3 through 8 shows that the maximum pore size determined in accordance with ASTM method D 892 is not related to foam volumes. For example, ASTM Stone 1A having a maximum porosity of 60 microns gave a foam volume of 450 ml while ASTM Stone 6A had a maximum porosity of 152 microns and gave a foam volume of 470 ml.

As shown by the data in Table A-6, tests 15 through 22, doubling

TABLE 42

PRESSURE VERSUS FLOWRATE OF FOAM TEST AIR DIFFUSERS

(Pressures in mm H_2O. Includes 100 mm H_2O head pressure)

Diffuser	Pressure Required for Flowrates of: 500 cc/min airflow Increasing Pressure	500 cc/min airflow Decreasing pressure	700 cc/min airflow Increasing Pressure	700 cc/min airflow Decreasing Pressure	1000 cc/min airflow Increasing Pressure	1000 cc/min airflow Decreasing Pressure
ASTM-1A	572	463	584	484	610	533
ASTM-2A	457	432	483	483	521	521
ASTM-6A	635	533	686	559	737	622
1/2" Sparger # 1 40 Microns	457	457	483	-	533	-
1/2" Sparger # 2 40 Microns	406	406	432	-	470	-
1/2" Sparger # 12 20 Microns	572	648	686	-	749	-
1/2" Sparger # 13 10 Microns	1067	1067	1118	914	1219	1118
1/2" Sparger # 14 5 Microns	1410	1410	1499	1168	1626	1397
1/2" Sparger # 15 2 Microns	1905	1905	1984	1613	2108	1880
(Measurements made using 100 mm MIL-L-7808 (0-82-3) head pressure)						
1/2" Sparger # 14 5 Microns	876	762	940	805	1041	940

Minimum pressure: 406 mm H_2O = 0.575 psi; Maximum pressure: 2108 mm H_2O = 2.988 psi

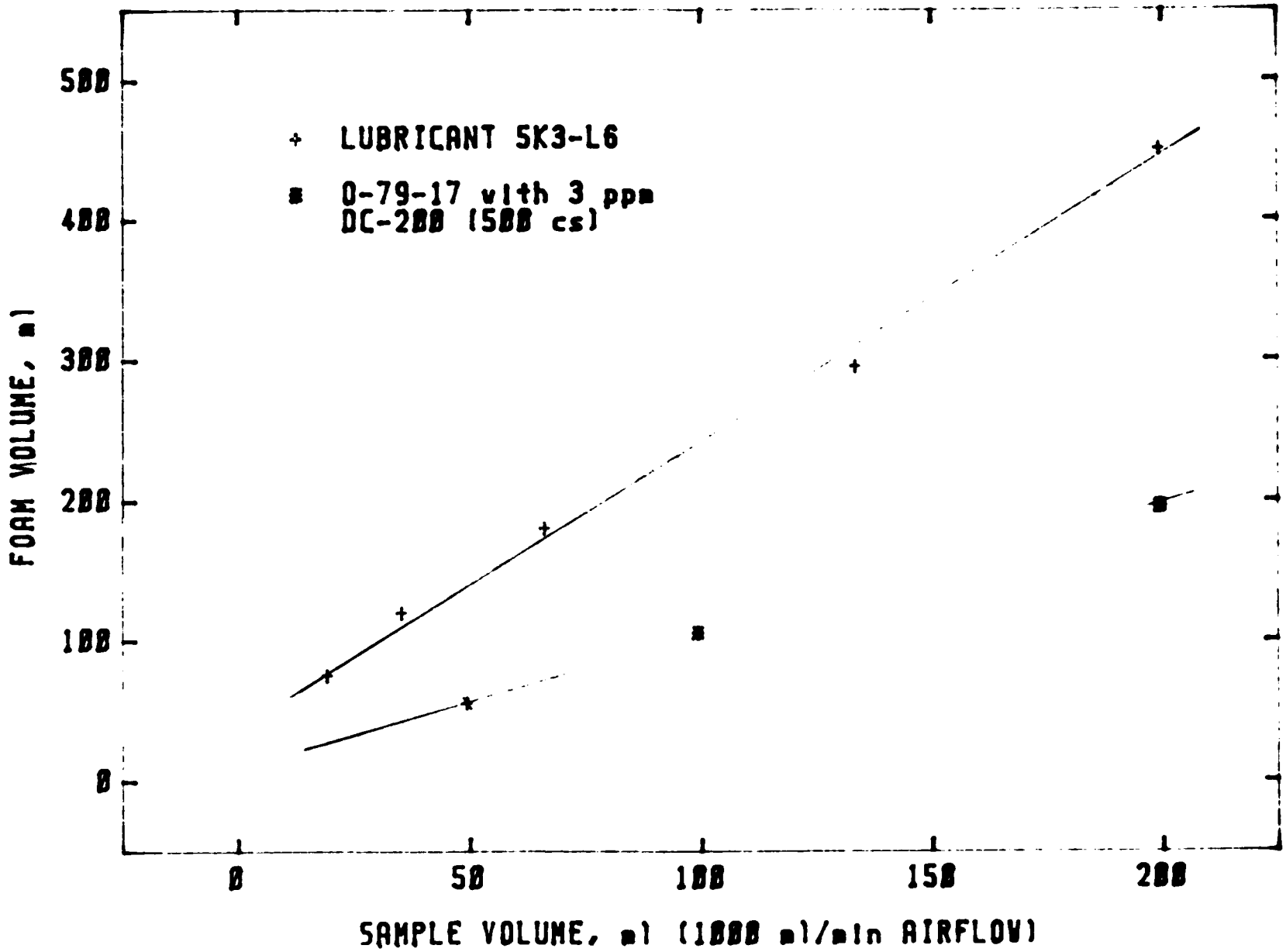


Figure 43. Effects of Sample Volume on Foam Volume. (Fed. Test Method 3213)

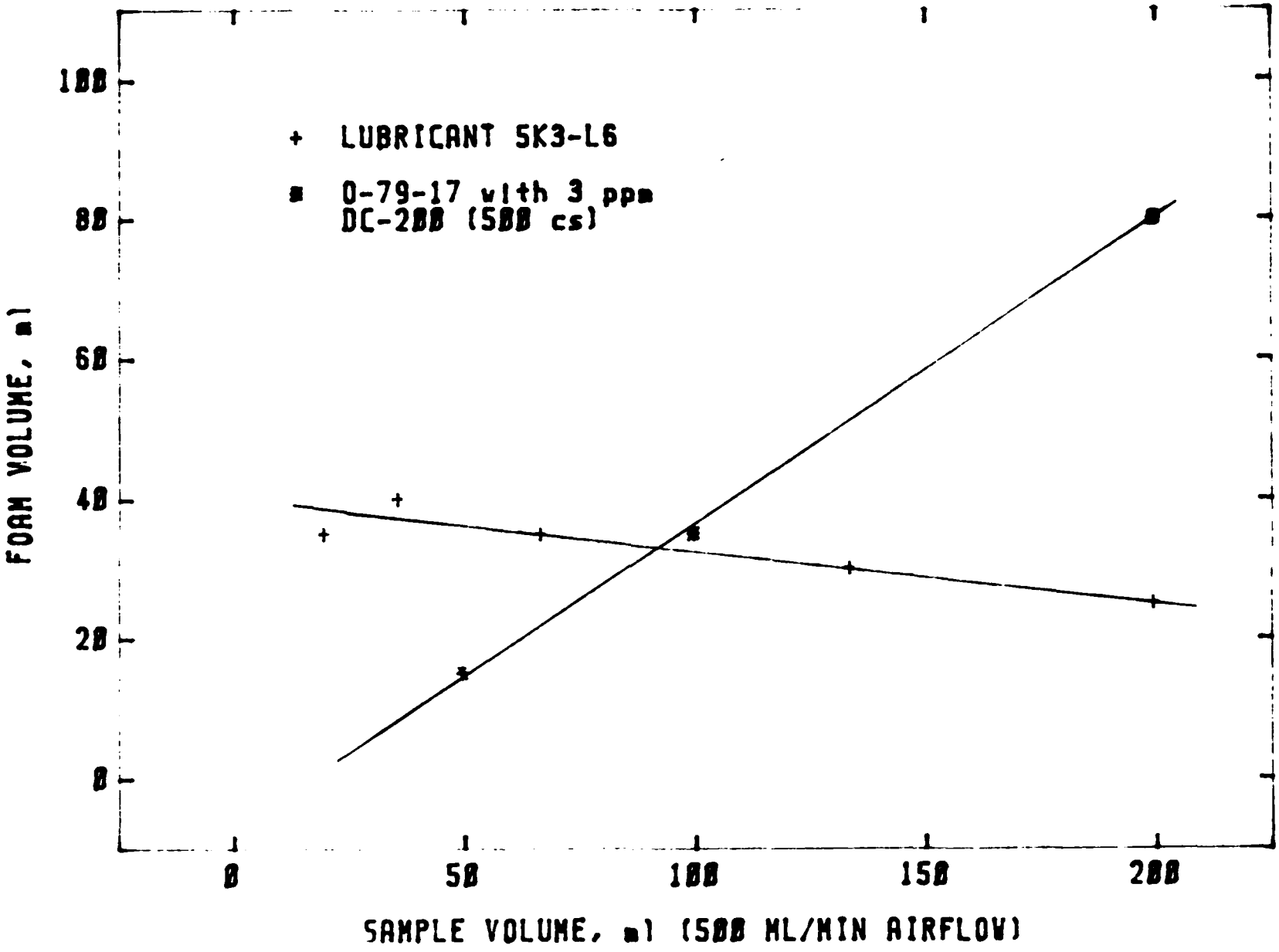


Figure 44. Effects of Sample Volume on Foam Volume.
(Fed. Test Method 3213)

the surface area of the ASTM diffuser stones and metal spargers using the same total airflow, sample size and test cylinders increases the foam volume only slightly. However, doubling the surface area of the glass frits increases the foam volume fourfold and approaches the foam volume obtained using a standard ASTM diffuser stone.

The effect of air diffuser pore size on lubricant foaming was investigated using 1/2" metal spargers having porosity ratings of [illegible], 5, [illegible], 20 and 40 microns. These ratings are based upon particle retention and not upon bubble point determinations. Three lubricants were studied which had various foaming levels and which foam for different reasons.

(a) Lubricant 5K3L6 - Previously qualified MIL-L-7808 lubricant

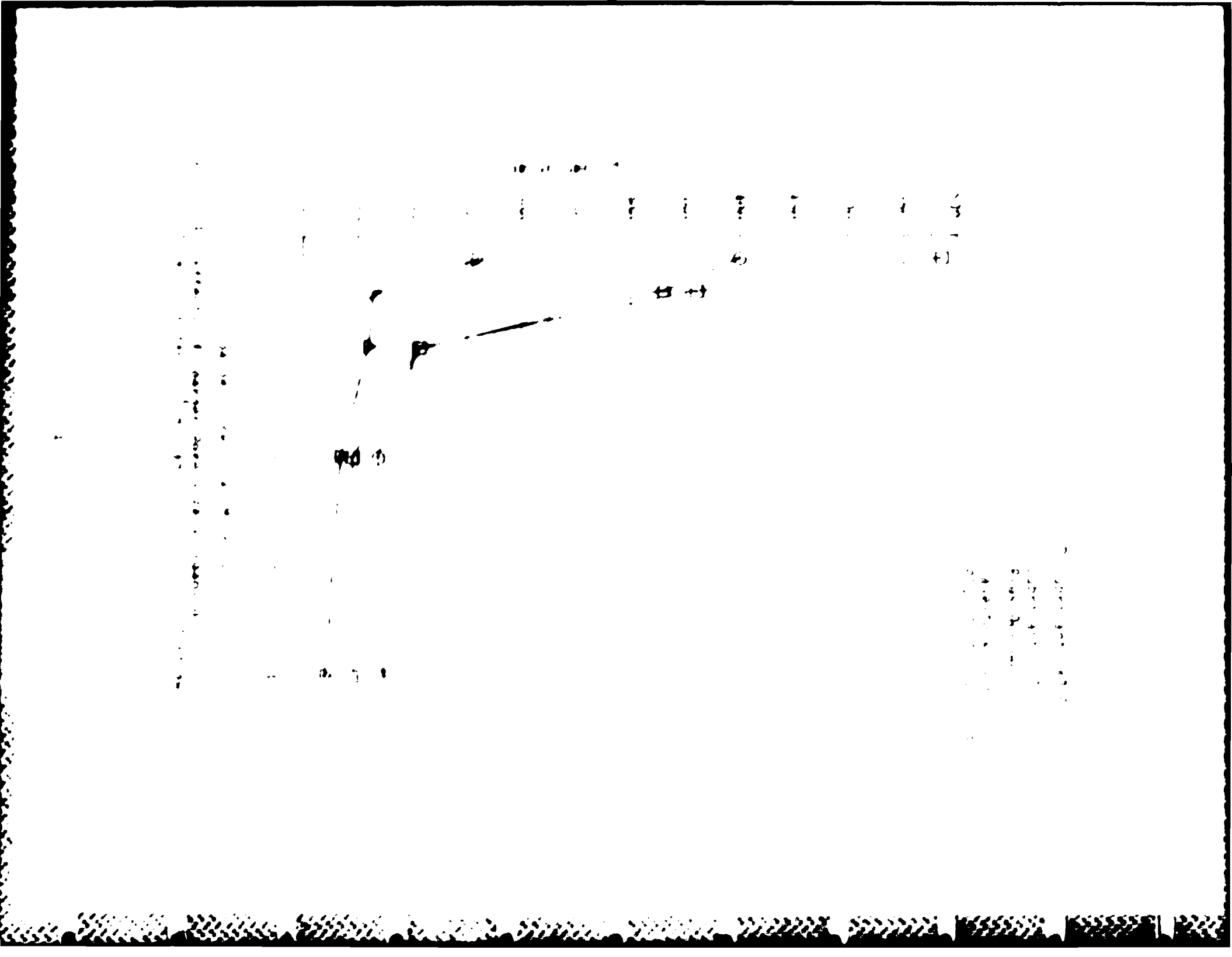
(b) O-79-17 plus DC-200 - Foaming due to soluble silicone.

(c) Basestock O-76-5 plus 2% TCP and 2% PANA - Synergistic effect of additives.

Data obtained from those studies are shown graphically by Figures 45 through 48. The effects on foaming due to air sparger pore size were the same for tests using 200 ml of sample and for tests using [illegible] sample. For the O-79-17 plus DC-200 lubricant and the basestock plus 2% TCP and 2% PANA, foaming increased greatly for air sparger sizes below [illegible] and 25 ml sample sizes. Lubricant 5K3L6 had the highest foaming level when using a 10 micron sparger and a sample size of 25 ml. For 200 ml size samples, lubricants O-79-17 plus DC-200 and O-76-5 plus the additives showed increased foaming for air spargers of pore sizes below [illegible] microns while the foaming characteristics changed very little for lubricant 5K3L6 for pore sizes below 10 microns.

(3) Lubricant Foaming Due to Silicones

Foaming tests conducted on lubricant 5K3L6 showed maximum



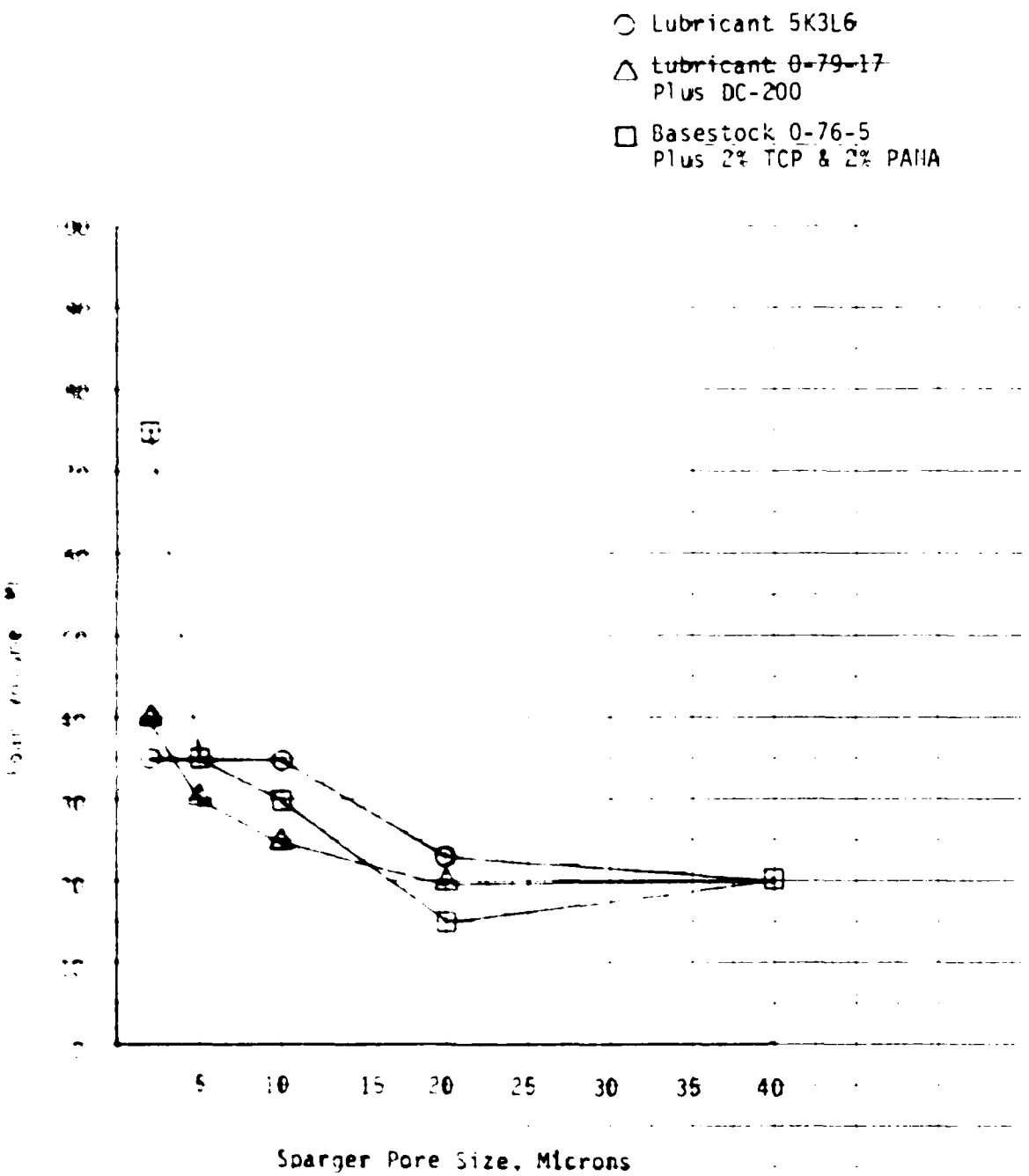
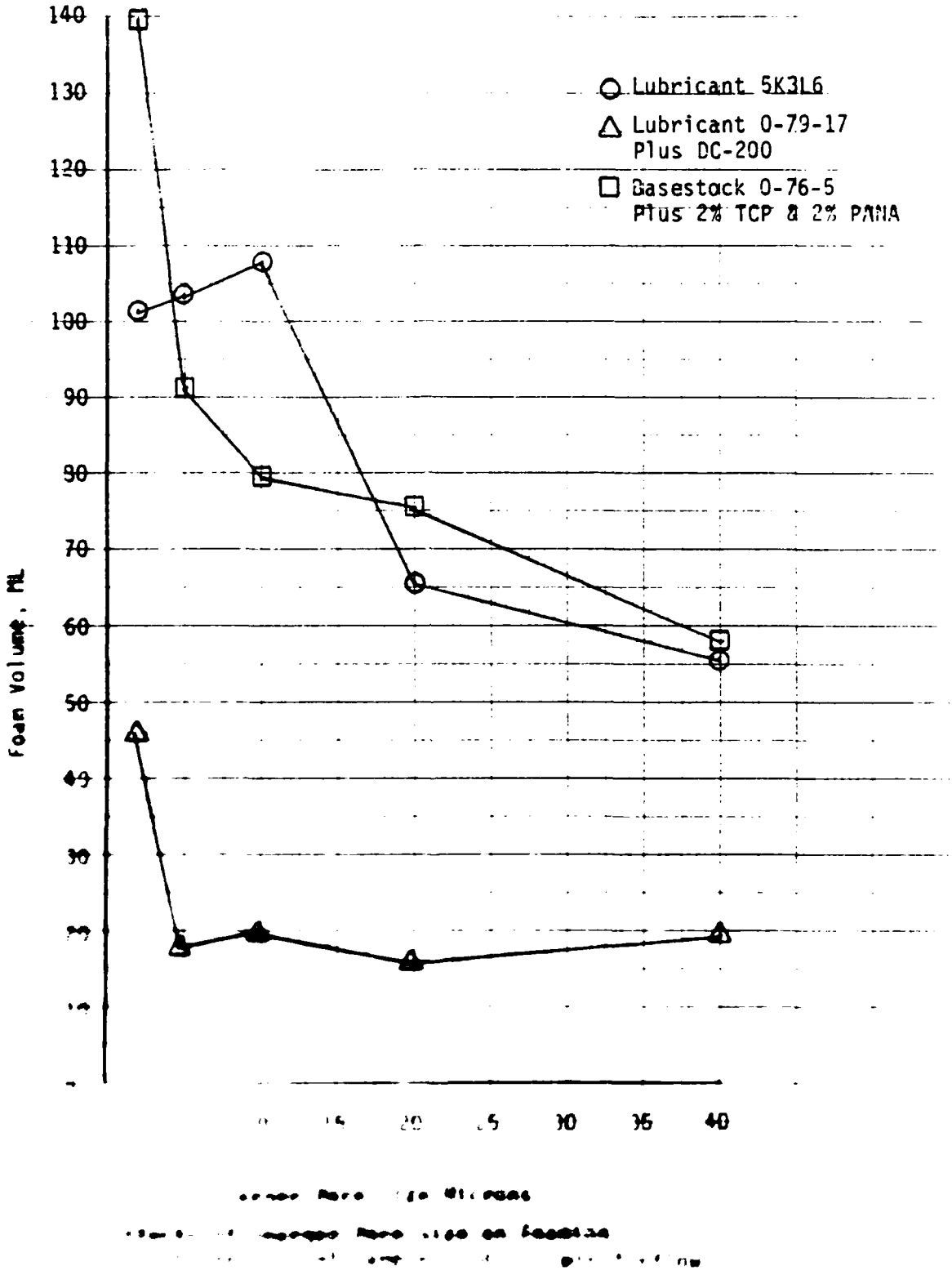


Figure 4[illegible]. Effects of Sparger Pore Size on Foaming
(500 ml Cylinder, 200 ml Sample, 500 cc/min Airflow)


Lubricant 5K3L6
Lubricant 0-79-17
Plus DC-200
Basestock 0-76-5
Plus 2% TCP & 2% PANA
Foam Volume, ML
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
5
10
15
40

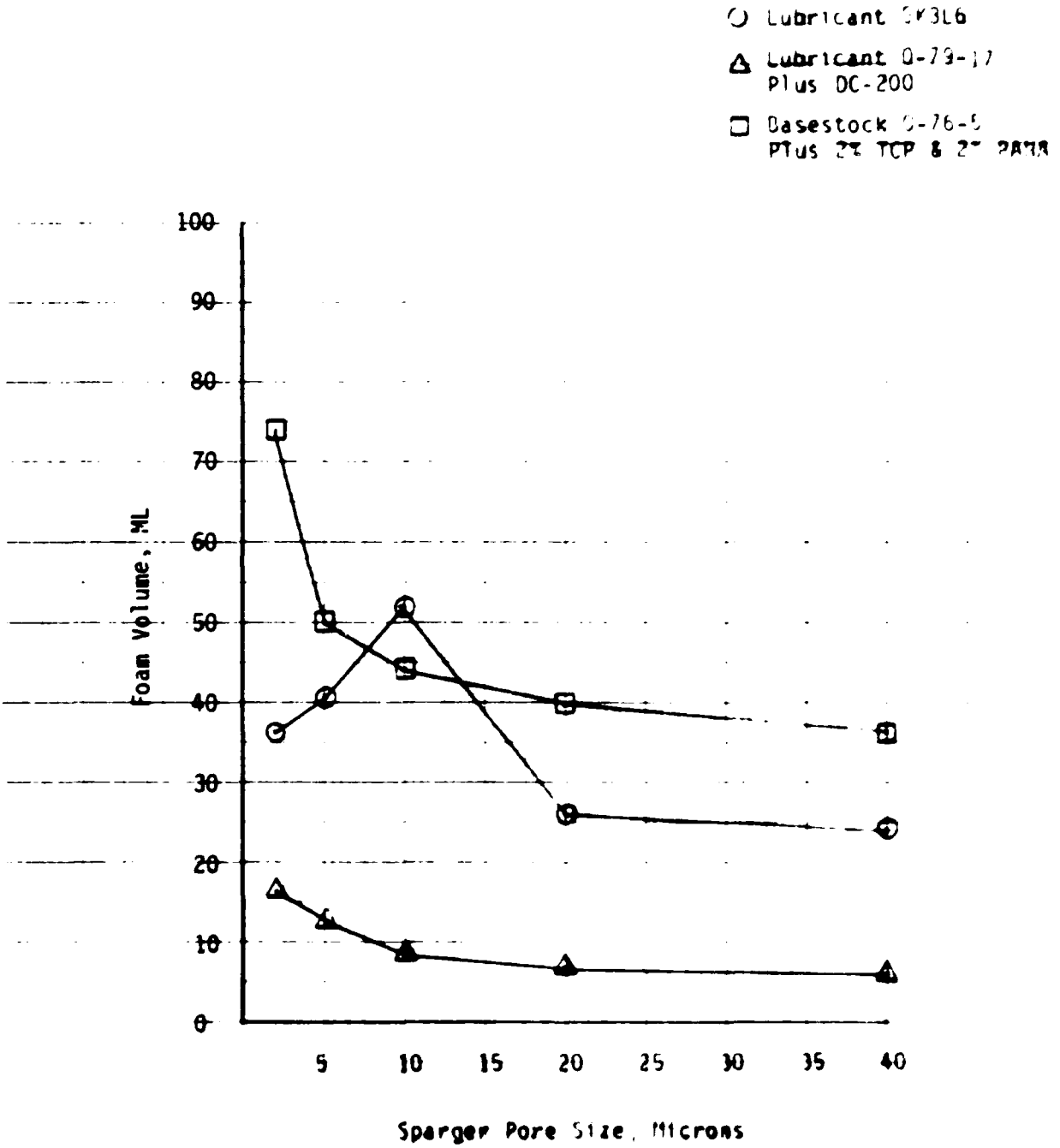


Figure 48. Effects of Sparger Pore Size on Foaming (250 ml Cylinder, 25 ml Sample, 500 cc/min Airflow)

foaming to occur during the first few minutes of testing while 20 to 25 minutes of testing was required for lubricant O-79-17 containing 3 ppm DC-200-500 cSt to approach maximum foaming level. Also, during the testing of O-79-17 containing the silicone the air bubbles changed from large to small bubbles prior to lubricant foaming.

Lubricant O-79-17 plus the 3 ppm DC-200-500 cSt was examined using phase contrast microscopy. A very small quantity (less than 1 ppm) of dispersed silicone two microns or less in size was present in this lubricant. This may explain the 20 to 25 minute delay in foaming of this lubricant.

Lubricant O-79-17 containing 3 ppm DC-200-500 cSt fluid was tested without shaking the container using the standard test method (500 ml cylinder and 200 ml of sample). Foaming did not reach its maximum foam value of 225 ml until 25 minutes of aeration. Aeration was stopped and the foam collapsed in 49 seconds. After waiting 5 minutes, the test was repeated with the lubricant reaching maximum foam volume of 230 ml well before 5 minutes of aeration. This is probably due to the loss of the trace amounts of insoluble silicone. After completing the second test, the ASTM "A" diffuser stone was removed and cleaned according to Method 3215. The test was again repeated using the original sample and the cleaned diffuser stone. A foaming value of 230 ml was obtained almost immediately. Lubricant O-79-17 containing the 3 ppm DC-200-500 cSt fluid was then mechanically shaken for 1 hour. The lubricant was then tested using the standard test method with the test oil being instrumented with a thermocouple to ensure 80°C oil temperature during aeration. Maximum foaming did not occur until 25 to 30 minutes of aeration had been achieved. A maximum foaming value of 195 ml of foam was about the same as previous testing. Test lubricant temperature remained 80°C throughout the test. After the test was completed, the lubricant, diffuser

and test cylinder remained in the bath for 15 minutes. The standard test was repeated and again, maximum foaming occurred during the first few minutes of testing.

Due to this problem of trace quantities of insoluble silicones when blending with the 500 cSt DC-200 fluid with other lubricants, a formulated lubricant O-76-1 and a di-2-ethylhexyl adipate ester O-76-8 were blended with 3 ppm DC-200-20 cSt to improve the silicone solubility. Lubricant O-76-1 plus the 3 ppm silicone gave a foaming value of about 150 using test method 3213 which was expected based on prior testing. However, contrary to past experience with the DC-200-20 cSt fluid causing lubricant foaming in formulated oils, the diester (O-76-8) plus the 3 ppm of DC-200-20 cSt fluid did not foam when using either the ASTM stones or the metal spargers. This is most likely due to an increase in the solubility of the DC-200 fluids when other additives are not present. The diester did not foam with concentrations of 6 ppm DC-200-20 cSt fluid.

Foaming values obtained on lubricant O-76-1 containing the 3 ppm DC-200 (cSt) fluid using metal spargers were much lower than expected because of equivalent foaming values being obtained with all other foaming lubricants when using the ASTM stones and the 5 and 2 micron metal spargers.

(4) Sealed Frit Foaming Tube Studies

During foaming testing with the ASTM stones and the metal spargers using either 200 or 25 ml samples, only part of the sample was being aerated for either 25 or 200 ml volume samples. The sealed frit foaming tube (Figure 40) was made to permit aeration of the complete 25 ml volume samples. Foaming test data obtained using the sealed frit tube is compared with foaming data using Test Method 3213 in Table 45. The sealed frit apparatus shows a reversal in the ranking of some of the lubricants compared to Test

TABLE 43

COMPARISON OF TEST METHOD 3213 FOAMING DATA
WITH THE SEALED FRIT 25 ml SAMPLE FOAMING DATA

LUBRICANT	TEST METHOD 3213 ml foam	SEALED FRIT TEST ml foam
O-79-16	45[a] (20)[b]	50 (29)
O-79-17	10 (5)	50 (12)
O-79-20	15 (10)	70 (14)
O-82-2	10 (10)	50 (14)
O-82-3	10 (10)	15 (10)
O-82-14	75 (20)	188 (64)
O-79-17 plus 3 ppm DC-200-500cs	200 (80)	105 (34)
O-76-5 plus 2% TCP + 2% PANA	480 (35)	134 (79)
O-76-8 plus 3 ppm DC-200-20 cs	10 (10)	10 (6)
O-76-8 plus 6 ppm DC-200-20 cs	10 (10)	16 (10)
O-76-1 plus 3 ppm DC-200-20 cs	148 (50)	22 (14)
O-76-1 plus 6 ppm DC-200-20 cs	290 (128)	88 (52)
5K3L6	15 (33)	148 (76)

a - Airflow of 1000 cc/min.

b - Airflow of 500 cc/min.

Method 3213. This reversal in ranking is less using a 500 cc/min airflow rate. With the 1000 cc/min airflow rate, the sealed frit tube is more sensitive to oil aeration and contributes to higher foaming values for some very low foaming lubricants

(5) Effects of Various Diameter 5 Micron Rated Spargers and ASTM Stone on Foam Values

The effects of various diameter 5 micron rated spargers and an ASTM stone on foam values were investigated using four lubricants having varying foaming characteristics with the data being shown in Figures 49 through 52. This data shows that no correlation exists between foaming values of the lubricants and various diameter spargers or the ASTM stone for either the 200 ml or 25 ml sample volume tests. The data does show that the 13/16 inch diameter sparger provides the best correlation with the ASTM air disperser for both sample volumes.

Lubricant foaming test data has been studied relative to the effects of airflow using different micron rated air spargers, different diameter air spargers, and ASTM stone and four different "foaming" lubricants. Figure 53 shows the effect of lubricant formulation on the ratio of foam volume at 1000 cc/min airflow to the foam volume at 500 cc/min using 2, 5 and 10 micron rated spargers having sizes of 1/4, 3/8, 1/2, 11/16 and 13/16 inch diameter and an ASTM stone for two lubricants. These lubricants (5K3L6 and O-76-5 TMPH containing 2% TCP and 2% PANA) foam due to synergistic effects of the lubricant components other than silicone material. This figure shows that for these fluids, this ratio increases as the level of foaming increases at a constant rate regardless of the type, size or micron rating of the air diffuser. In comparison, Figure 54 shows the effect of lubricant formulation on the foam ratio for two lubricants which foam due to

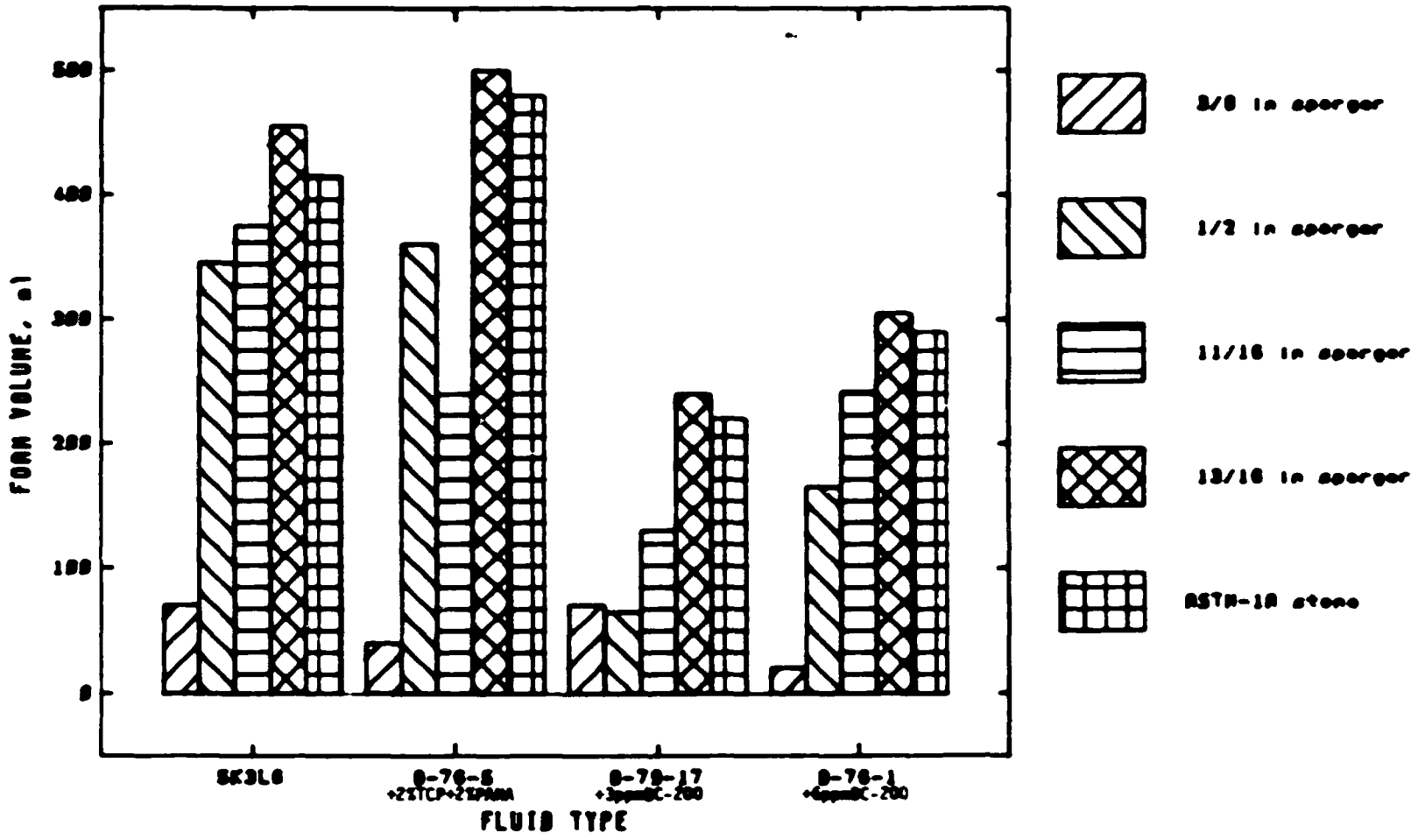


Figure 49. Effects on Various Diameter 5 Micron Spargers and ASTM Stone on Foaming Using 200 ml Sample Size. Data Grouped for Each Lubricant

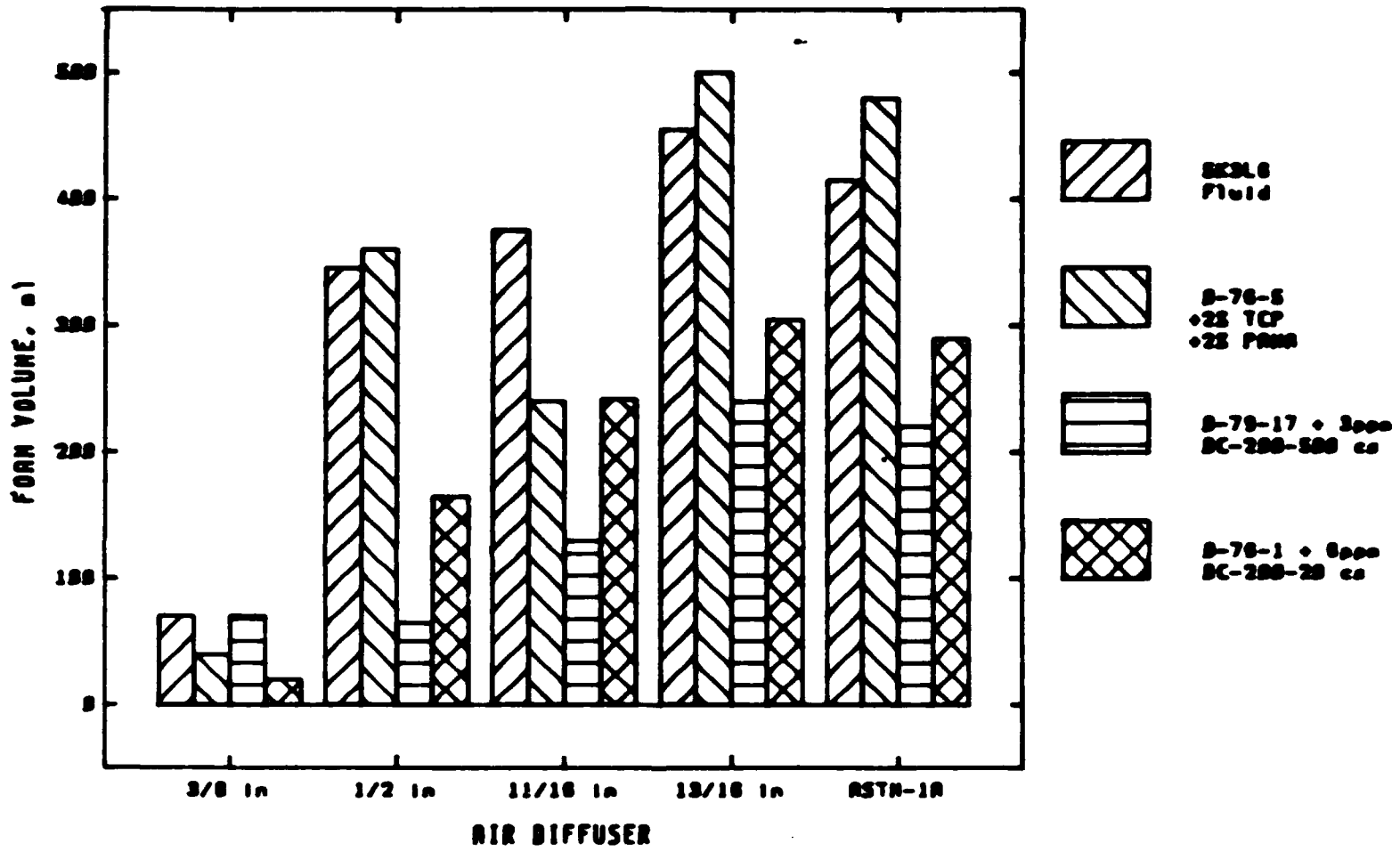


Figure 50. Effects of Various Diameter 5 Micron Spargers and ASTM Stone on Foaming Using 200 ml Sample Size. Data Grouped for Each Size Sparger

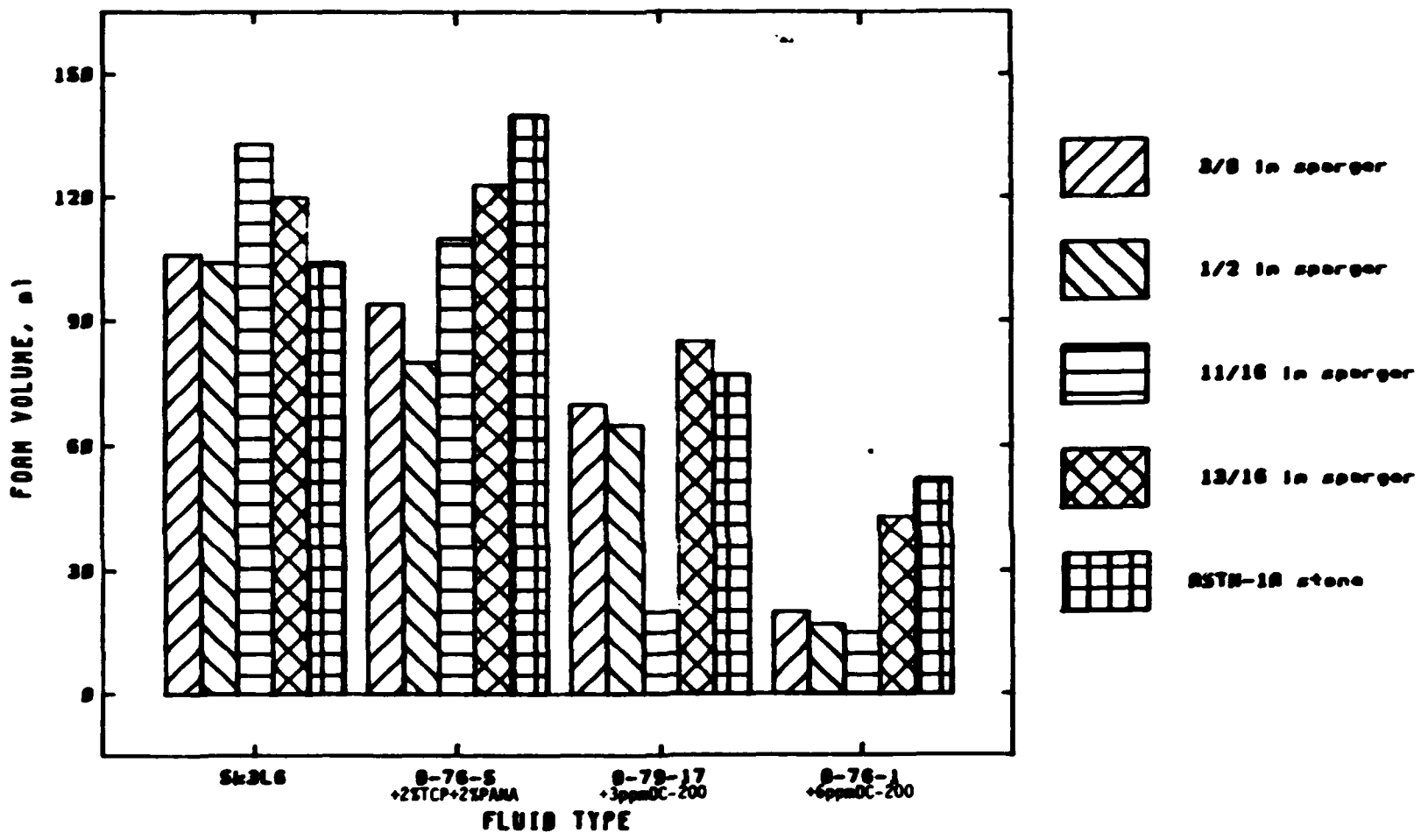


Figure 51. Effects of Various Diameter 5 Micron Spargers and ASTM Stone on Foaming Using 25 ml Sample Size. Data Grouped for Each Lubricant

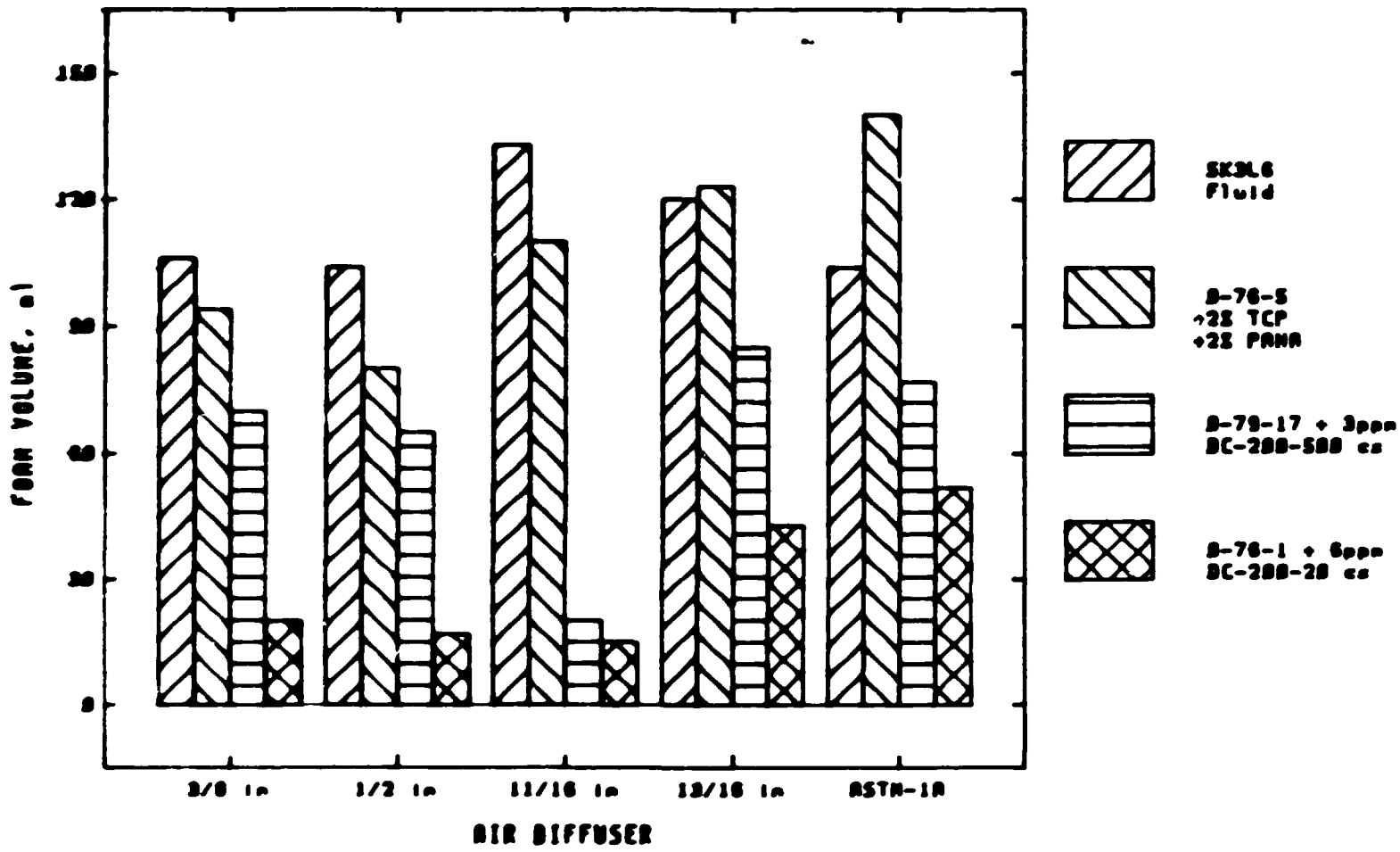


Figure 52. Effects of Various Diameter 5 Micron Spargers and ASTM Stone on Foaming Using 25 ml Sample Size. Data Grouped for Each Size Sparger

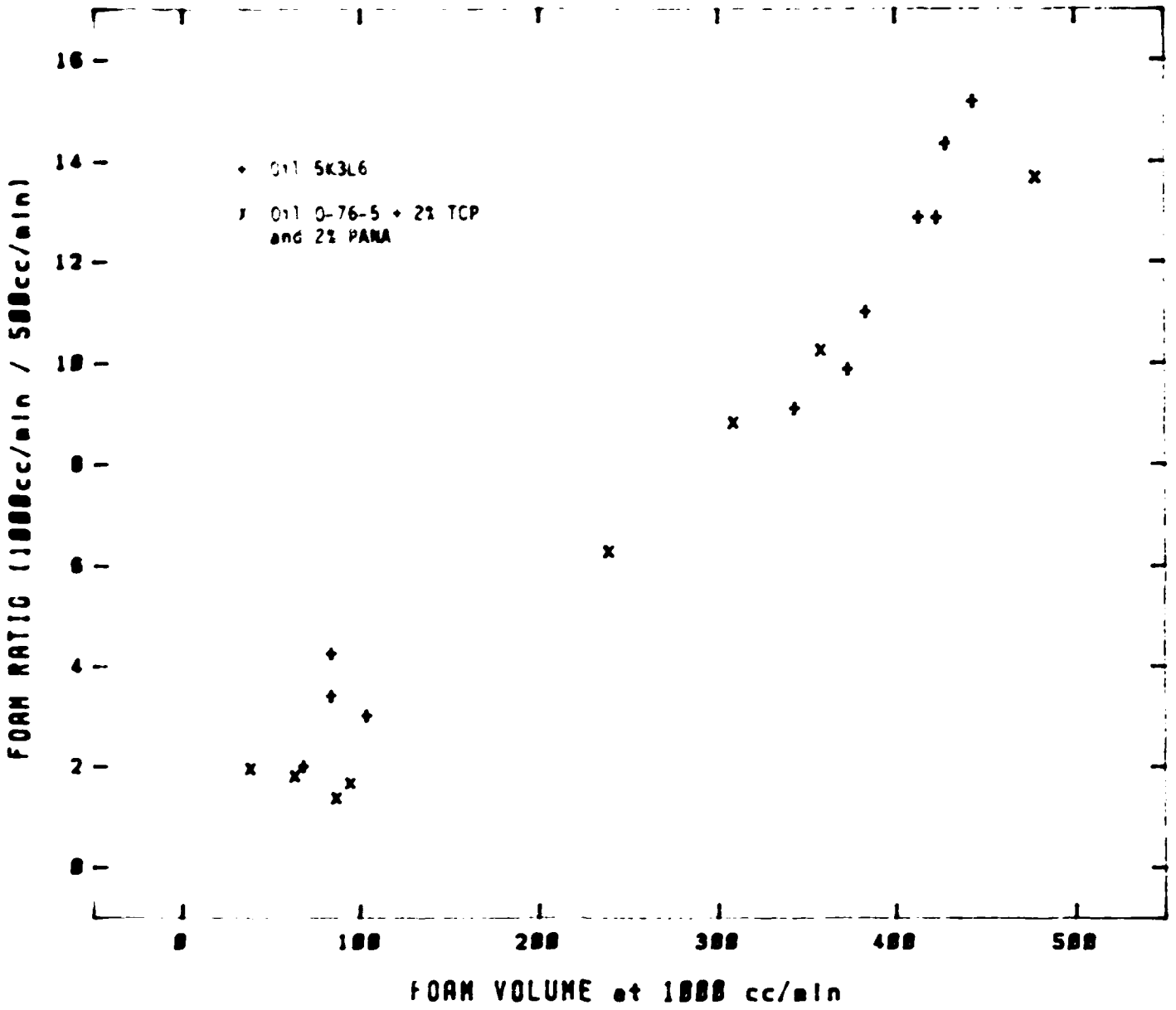


Figure 53. Effect of Lubricant Formulation on the Ratio of Foam Volume at 1000 cc/min Airflow to Foam Volume at 500 cc/min Airflow Using Various Diameter and Micron Rated Air Dispersers, 200 ml Sample Size, Non-Silicone Containing Fluids.

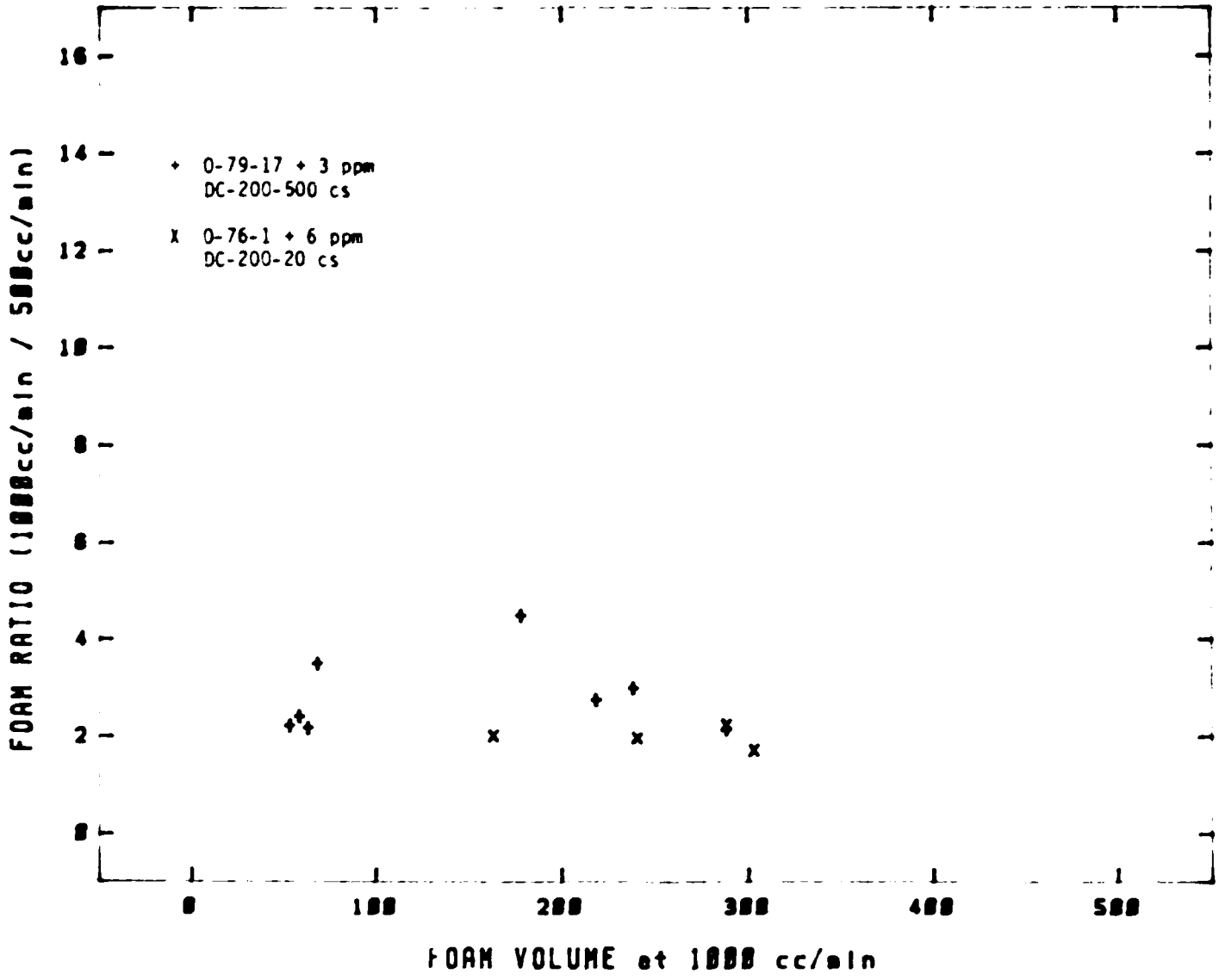
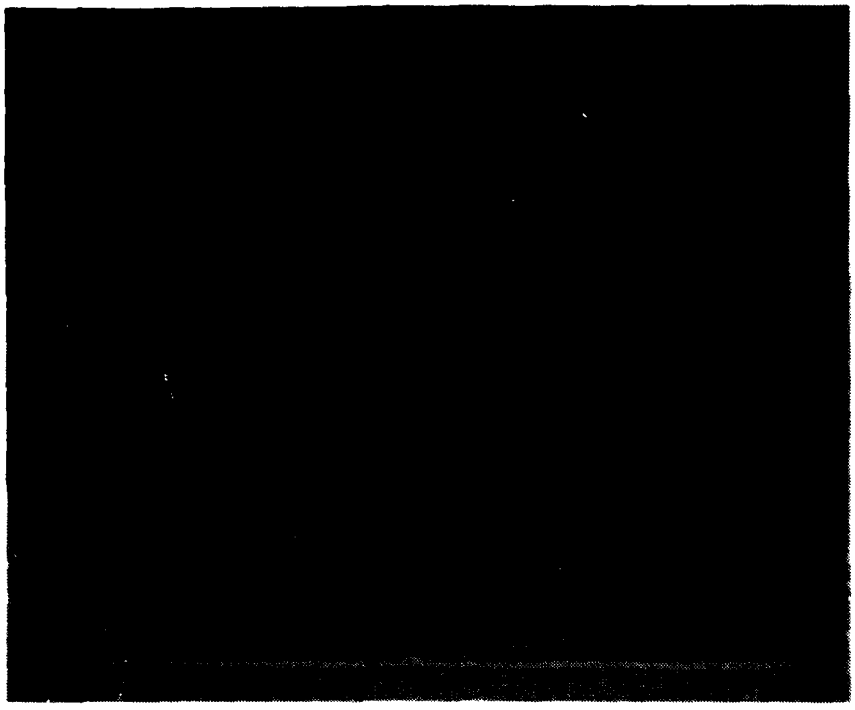


Figure 54. Effect of Lubricant Formulation on the Ratio of Foam Volume at 1000 cc/min Airflow to Foam Volume at 500 cc/min Airflow Using Various Diameter and Micron Rated Air Dispersers, 200 ml Sample Size, Silicone Containing Fluids.

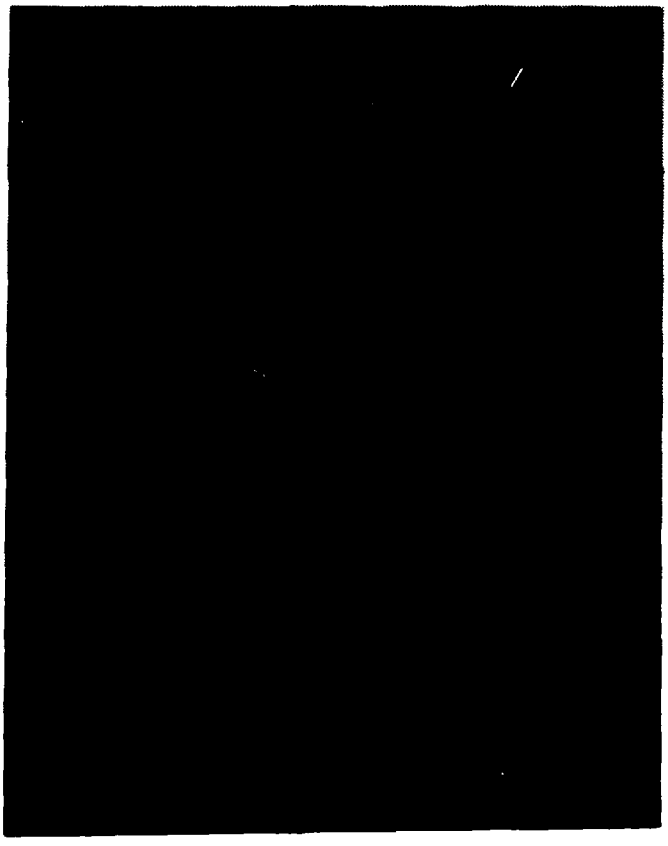
silicone using various type, size and micron rated air diffusers. This figure shows that no correlation exists between the foam volumes at 1000 cc/min and 500 cc/min airflow for these fluids. For tests involving 25 ml sample sizes, no correlation existed between the foam volume ratios at the two airflow rates for any of the four lubricants. The above data shows that changing the airflow in the small sample volume test being developed would not provide correlation between the ASTM test and a small volume test for all "types" of foaming lubricants.

(6) Effect of Pore Size on Bubble Size

The effects of various pore size (micron rated) 1/2 inch diameter spargers and an ASTM stone on air bubble size are shown in Figures 55 through 61. Figure 55 displays photographs of an air sparger and an ASTM stone with no airflow and shows a magnification of 4X which applies to all the figures showing foaming at 1000 cc/min airflow. This magnification means that a bubble measuring one millimeter is 250 microns in size and is about the smallest bubble distinguishable. Figures 56 through 58 show the pore size effects for a non-foaming lubricant. For this lubricant only a very small decrease is seen in bubble size as the rated pore size decreases. The bubble size of the ASTM diffuser stone is the same as for the 40, 20, 10, 5 and 2 micron pore size spargers. In all cases the spargers show almost complete coverage of bubbles (airflow through most of the sparger surface). Figures 59 through 61 show the pore size effects for a high foaming lubricant. These figures show that as the pore size decreases, the bubble size decreases, except that the 5 micron sparger shows more small bubbles than the 2 micron sparger. However, the measured micron rating (Method ASTM D 892) and also the pressure required for developing uniform flow of bubbles around the diffusers and calculating apparent bubble size (ASTM E 128) show



ASTM Diffuser Stone

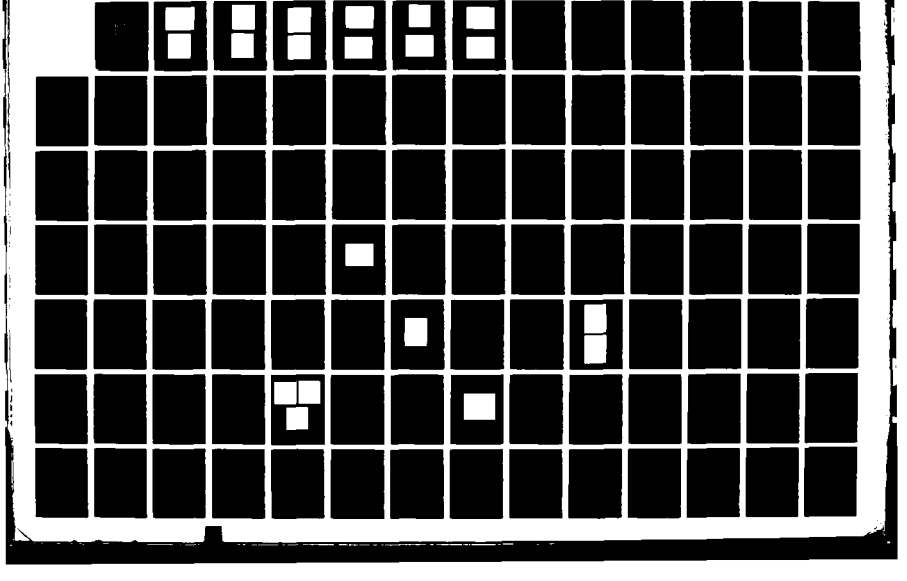


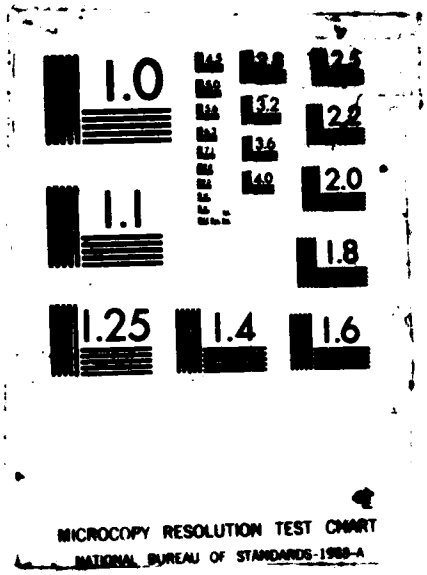
1/2 Inch Diameter Carbide

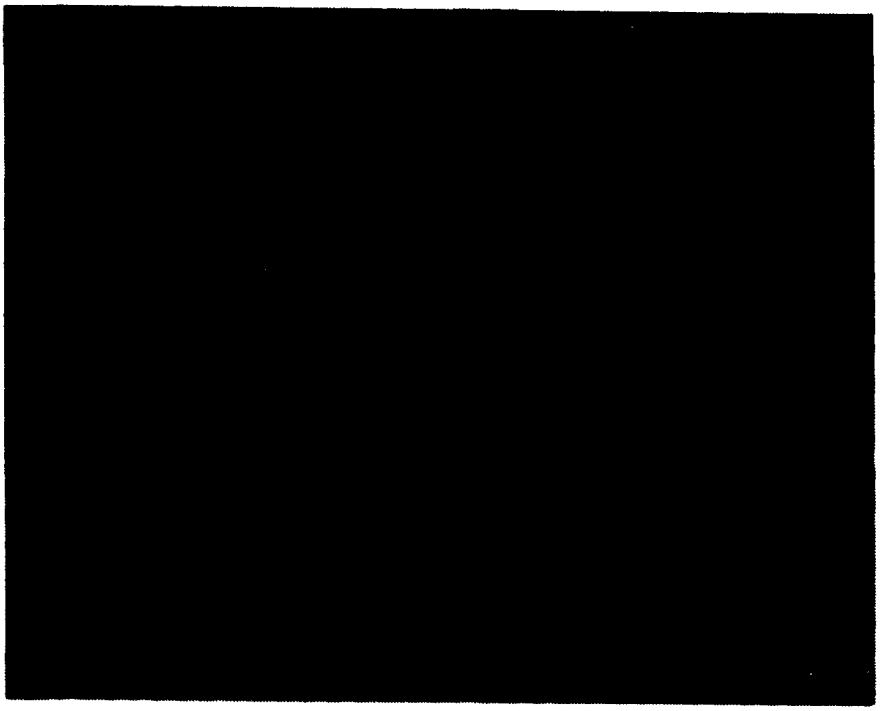
Figure 55. Photograph [illegible] ASTM [illegible] Diameter [illegible]

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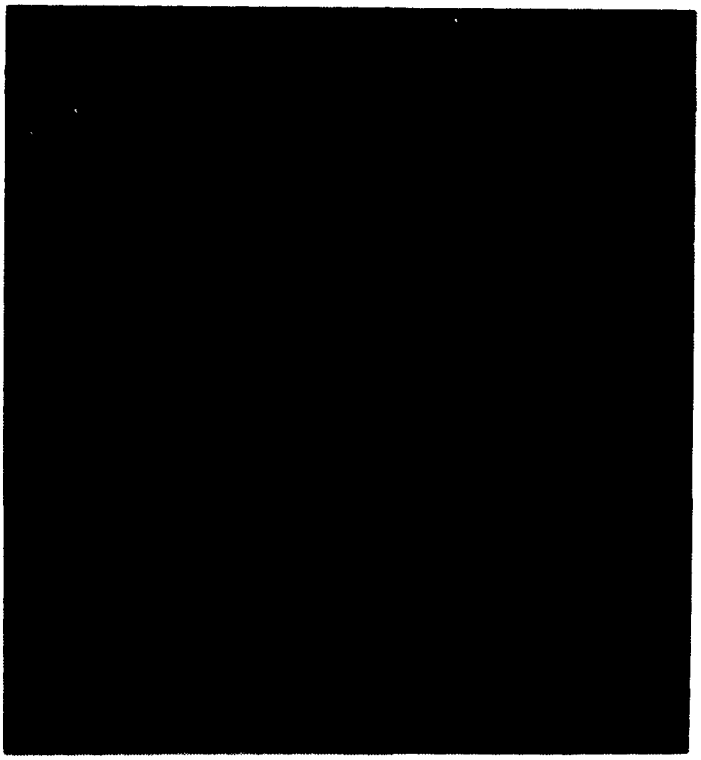
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1.0
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MICROCOPY RESOLUTION TEST CHART
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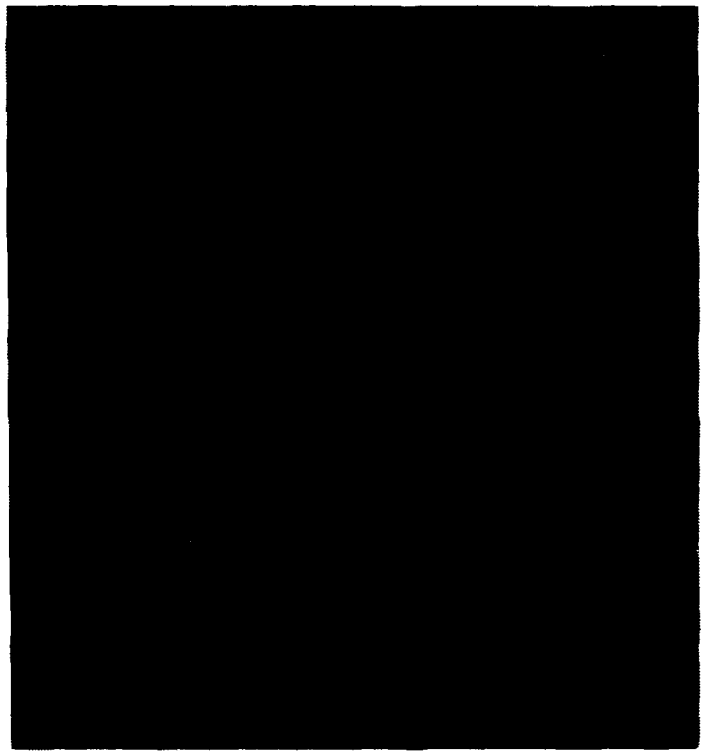


ASTM Diffuser Stone

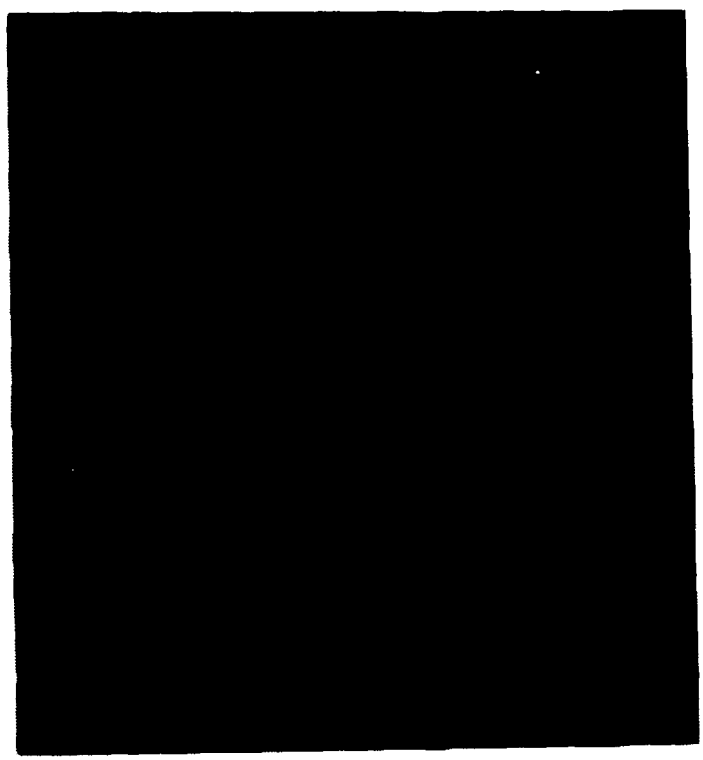


40 Micron Pore Size Sparger

Figure 56. Photographs of Air Bubbles Using Non-foaming Fluid 0-76-8 and 1000 cc/min Airflow with ASTM Diffuser Stone and 40 Micron 1/2 Inch Diameter Sparger (4X Magnification)

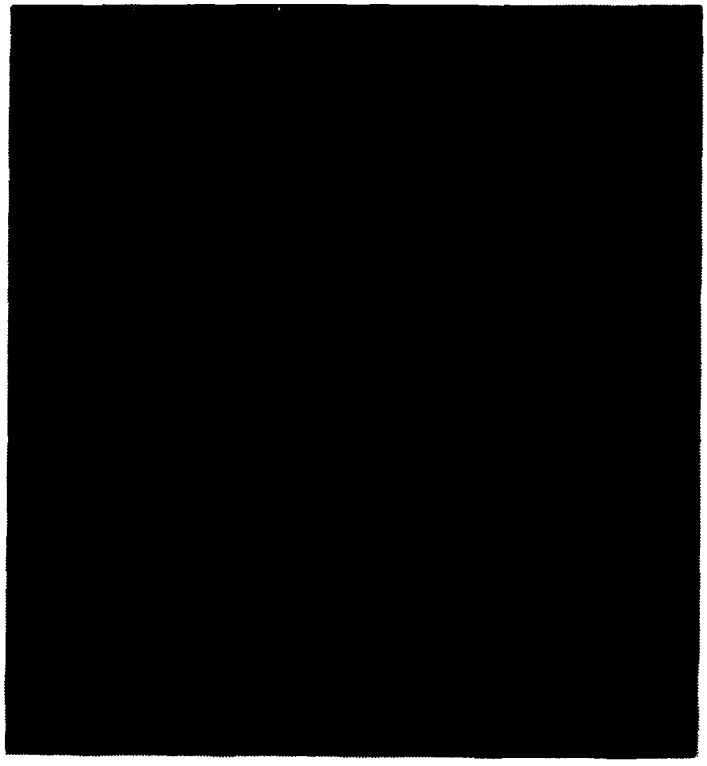


20 Micron Pore Size Sparger

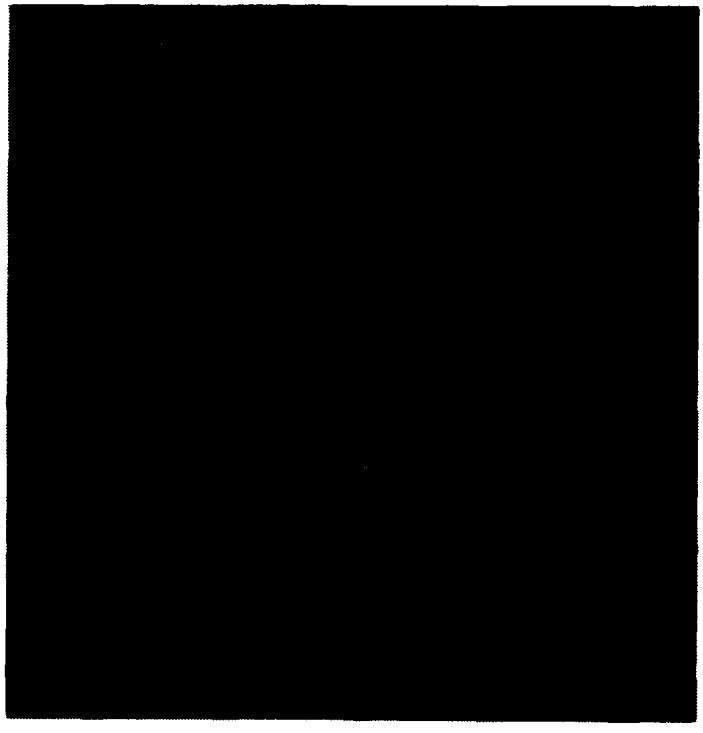


10 Micron Pore Size Sparger

Figure 57. Photographs of Air Bubbles Using Non-foaming Fluid O-76-8 and 1000 cc/min Airflow with 1/2 Inch Diameter 20 Micron and 10 Micron Pore Size Spargers (4X Magnification)

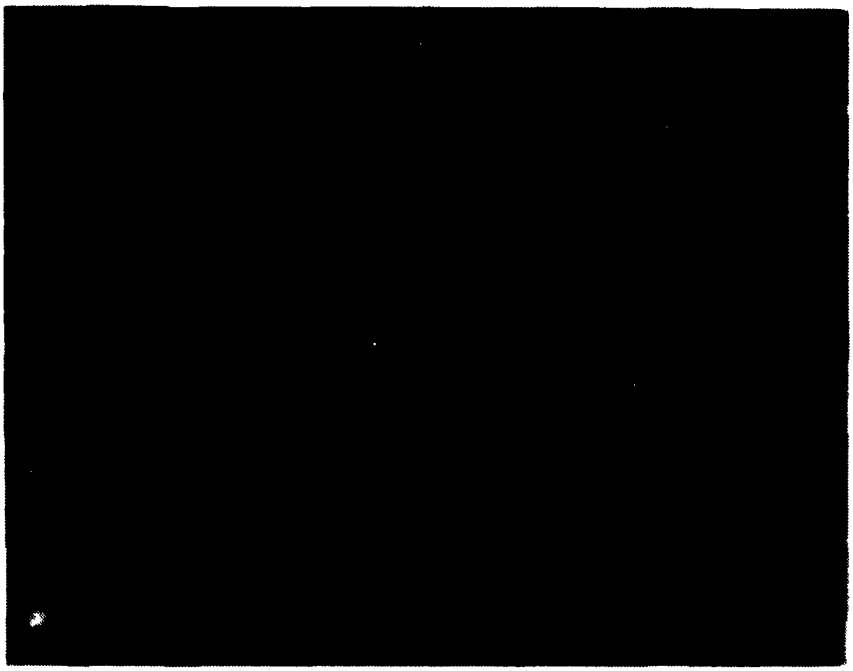


5 Micron Pore Size Sparger

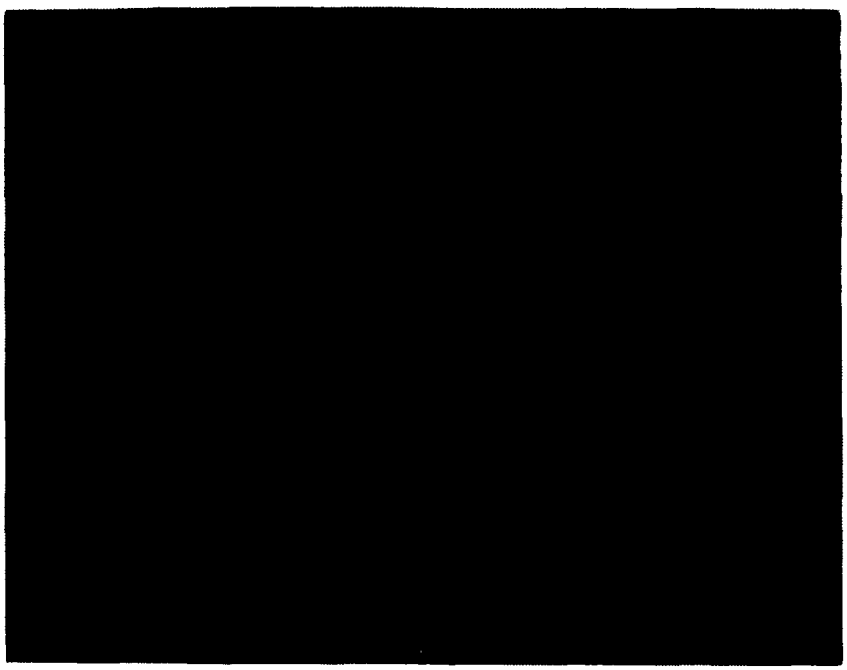


2 Micron Pore Size Sparger

Figure 58. Photographs of Air Bubbles Using Non-foaming Fluid O-76-8 and 1000 cc/min Airflow with 1/2 Inch Diameter 5 Micron and 2 Micron Pore Size Spargers (4X Magnification)

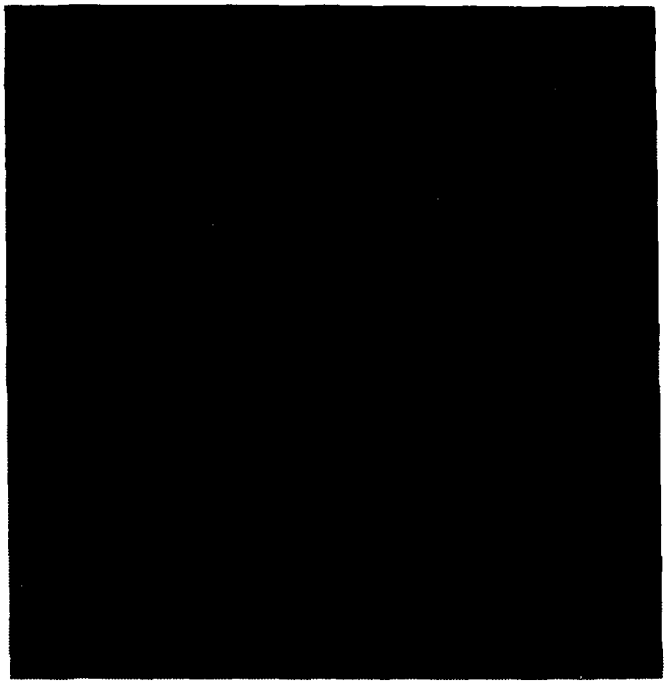


ASTM Diffuser Stone

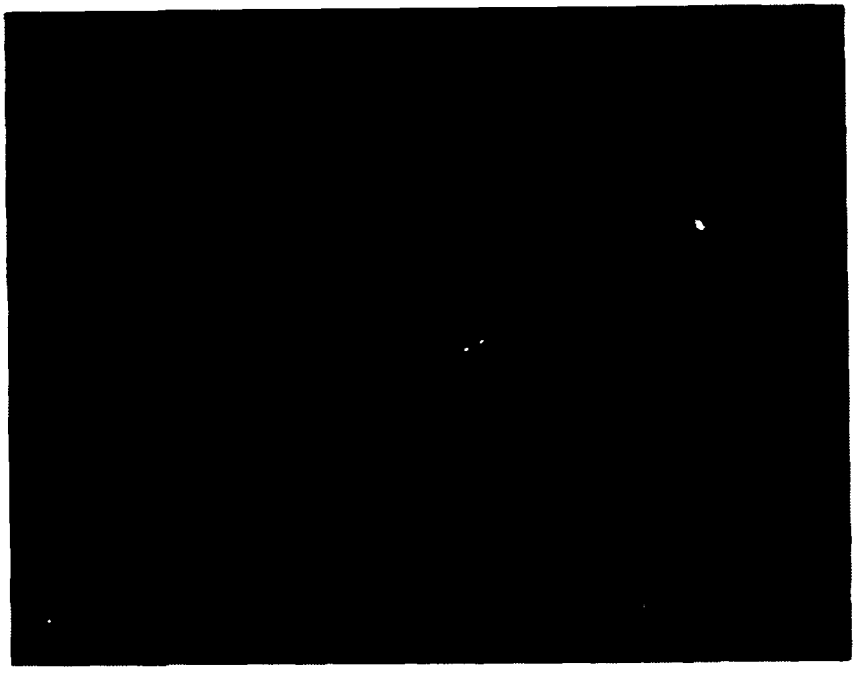


40 Micron Pore Size Sparger

Figure 59. Photographs of Air Bubbles Using Foaming Fluid 5K3L6 and 1000 cc/min Airflow with ASTM Diffuser Stone and 40 Micron 1/2 Inch Diameter Sparger (4X Magnification)

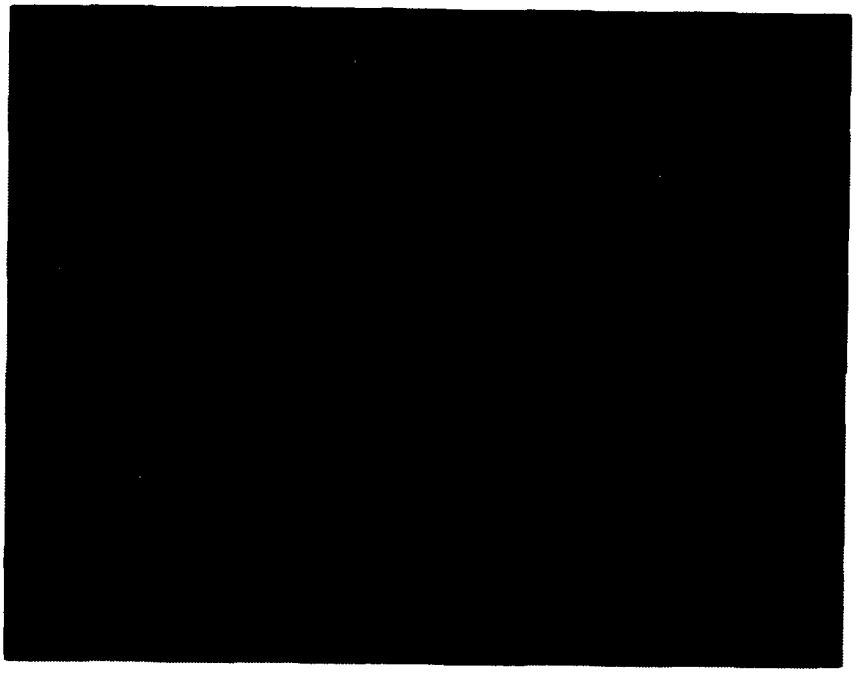


20 Micron Pore Size Sparger

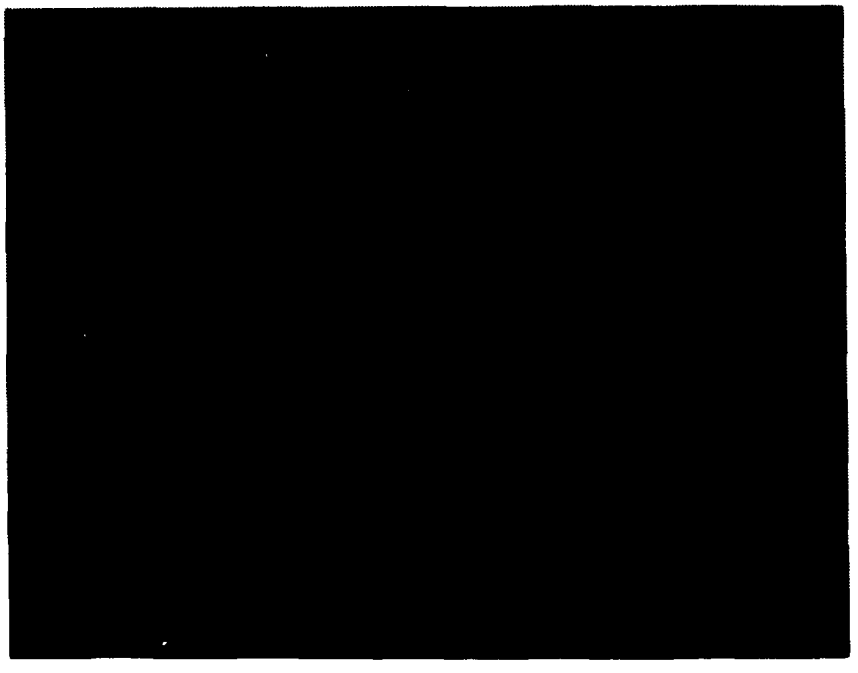


10 Micron Pore Size Sparger

Figure 60. Photographs of Air Bubbles Using Foaming Fluid 5K3L6 and 1000 cc/min Airflow with 1/2 Inch Diameter 20 Micron and 10 Micron Pore Size Spargers (4X Magnification)



5 Micron Pore Size Sparger



2 Micron Pore Size Sparger

Figure 61. Photographs of Air Bubbles Using Foaming Fluid 5K3L6 and 1000 cc/min Airflow with 1/2 Inch Diameter 5 Micron and 2 Micron Pore Size Spargers (4X Magnification)

the 2 micron sparger to be of smaller pore size than the 5 micron sparger. Figures 59 through 61 also show that for the foaming fluid decreasing the pore size increases the area of the sparger having airflow which was not the effect for the non-foaming fluid. The 5 micron rated pore size 11/16 inch and 13/16 inch diameter spargers used in the foaming study have complete bubble coverages (1000 cc/min airflow) using both the non-foaming and foaming fluids although photographs have not been taken for these diffusers.

(7) Correlation of Test Method 3213 Foam Test Data with 25 ml Volume Foam Test Data

Test data used for determining the correlation between the Test Method 3213 foam data and the 25 ml volume foam test data is in most cases the average of 2 to 4 individual foam tests being conducted on the same fluid with the same ASTM Stone or air sparger. Repeat testing was not conducted immediately after first testing and as much as one year could have occurred between some repeat tests. The number of repeat tests depended upon the variation on the foam test data, time between repeat testing and level of expected foaming.

The effects of air diffuser type, diameter and pore size on different lubricants are shown graphically in Figures 62 through 66. Figure 62 shows the comparison of foaming data obtained from Test Method 3213 and 200 ml samples using various diameter 5 micron pore size spargers. This data shows good correlation between the 13/16" sparger and the ASTM diffuser stone with very little bias (16) for the ASTM stone and a correlation slope of 0.903. The 11/16" sparger gave a much larger bias (128) for the ASTM stone and a much lower correlation slope of 0.754. The 1/2" sparger gave a larger bias(148) than either of the other two diffusers but a slope more equal to the 13/16" sparger. Figure 63 shows the comparison of foaming data obtained

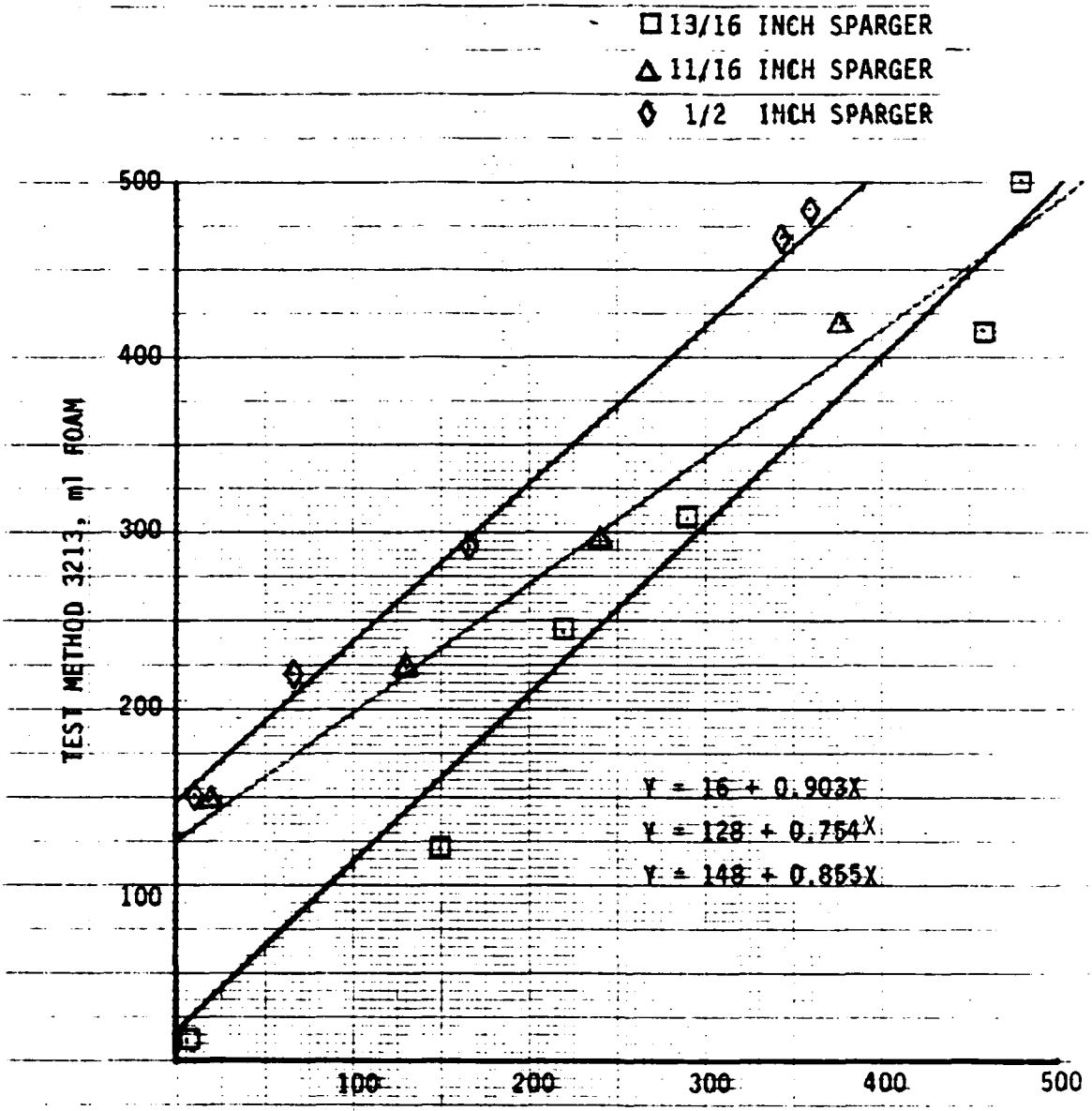


Figure 62. Comparison of Foaming Data Obtained from Test Method 3213 and 200 ml Samples Using Various Diameters 5 Micron Spargers

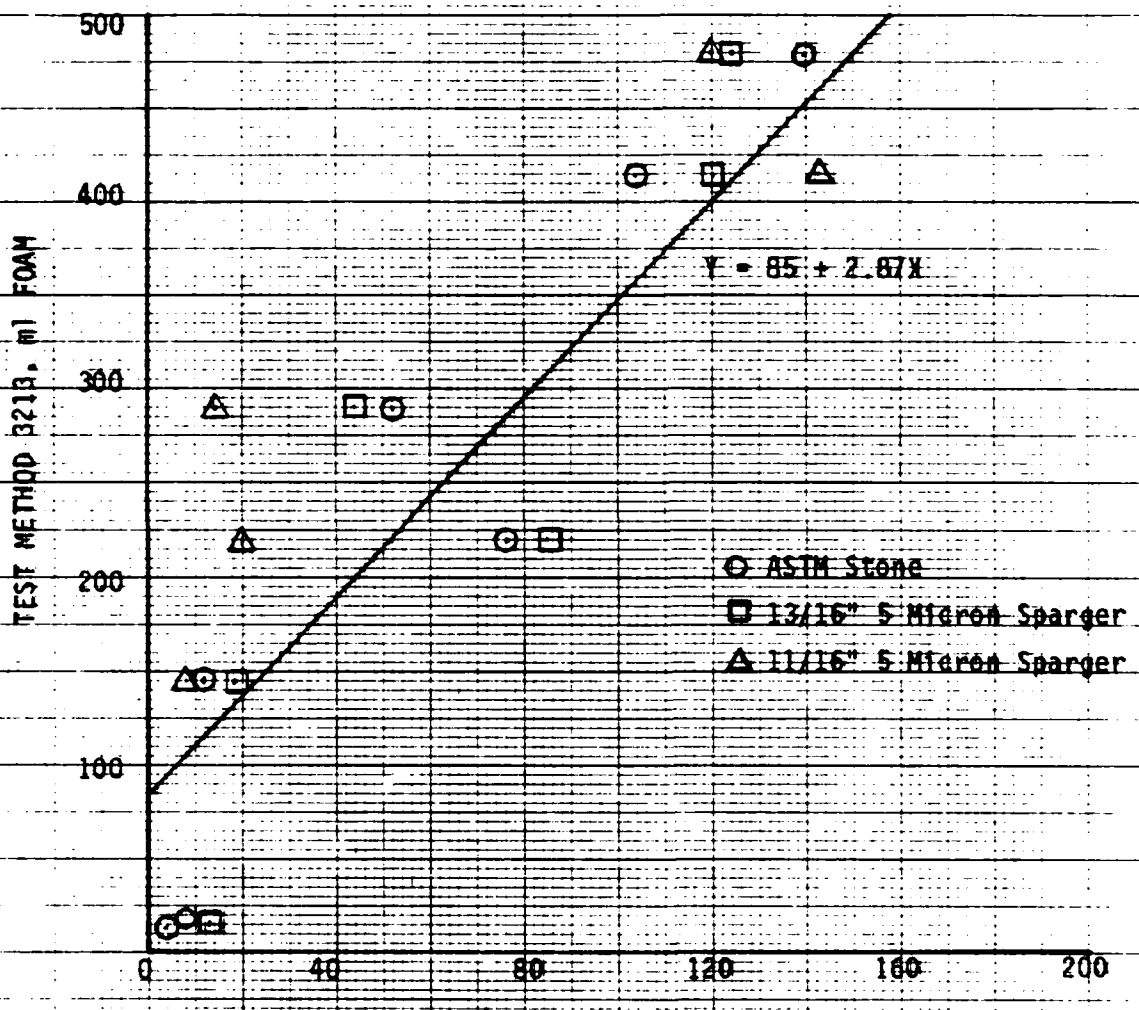


Figure 63. Comparison of Foaming Data Obtained from Test Method 3213 and 25 ml Volume Samples Using ASTM Stone and Various 5 Micron Spargers

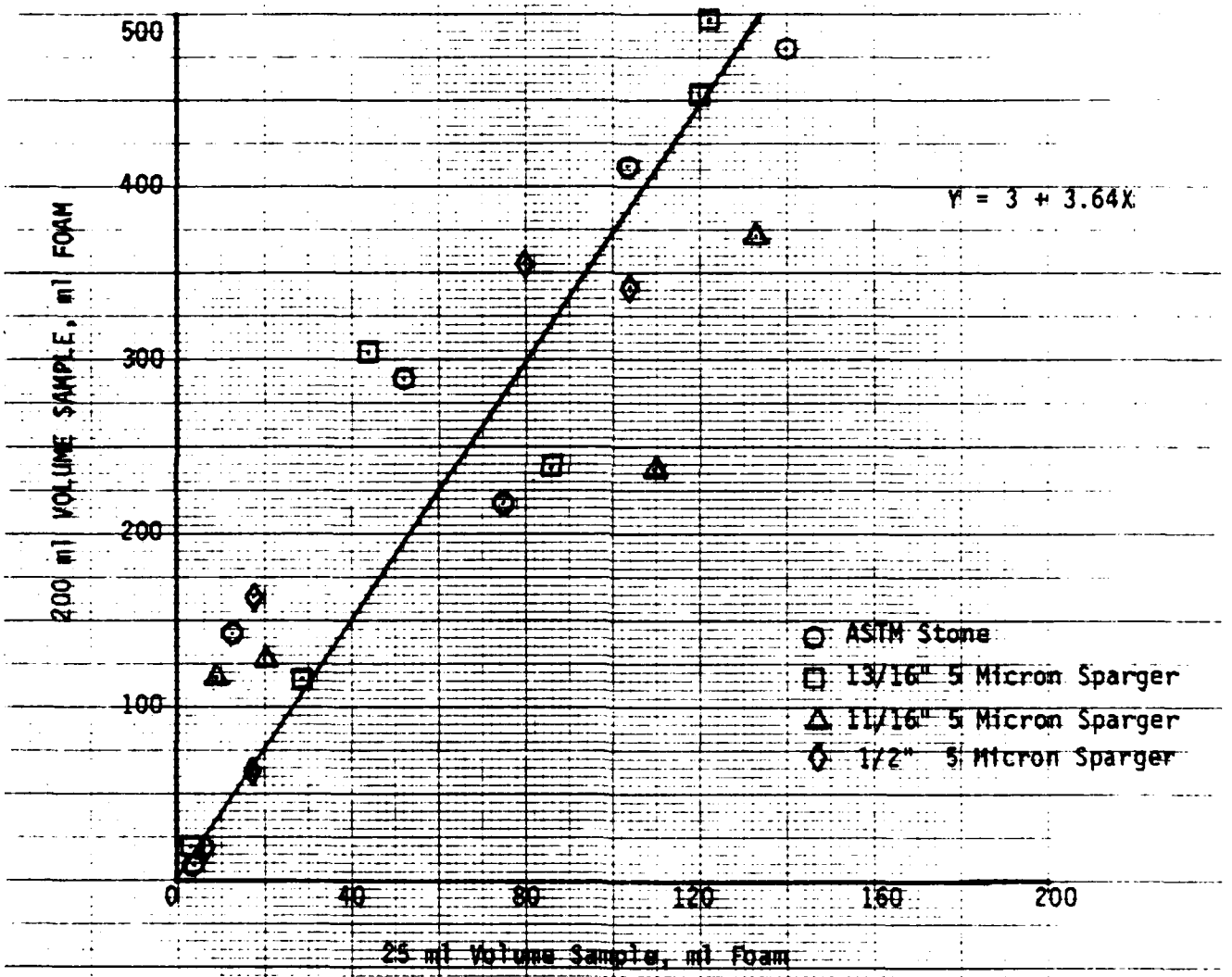


Figure 64. Comparison of Foaming Data Obtained from 200 ml and 25 ml Volume Samples Using the Same Air Diffuser

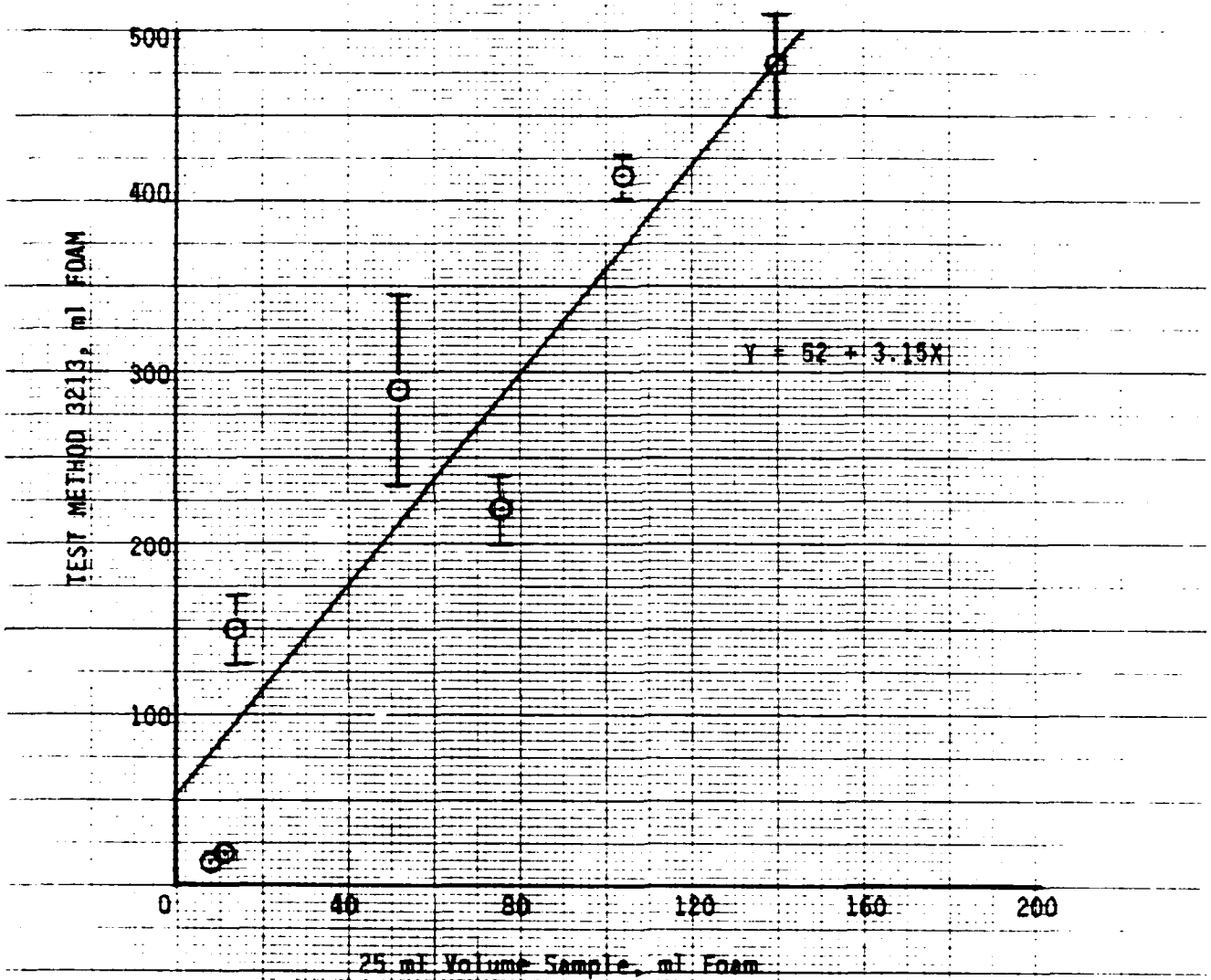


Figure 65. Comparison of Foaming Data Obtained from Test Method 3213 and 25 ml Volume Samples Using ASTM Stone and Showing Standard Deviation for Test Method 3213 Foaming Data

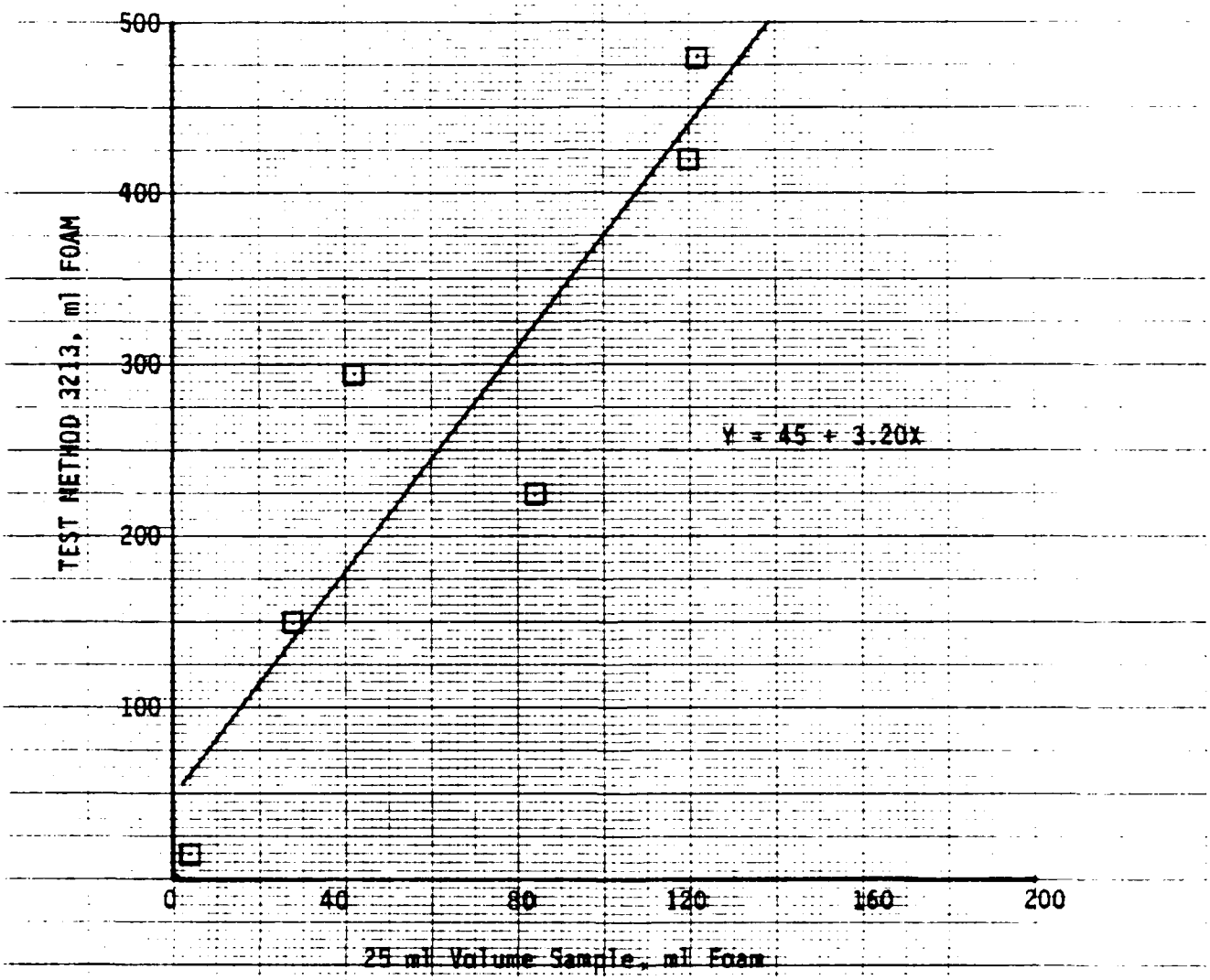


Figure 66. Comparison of Foaming Data Obtained from Test Method 3213 and 25 ml Volume Samples Using 13/16" Diameter 5 Micron Sparger

from Test Method 3213 and 25 ml volume samples using the ASTM stone and various diameter 5 micron spargers. This data shows a poorer correlation than the data in Figure 62, especially for the 11/16" diameter sparger data. Figure 64 shows the comparison of foaming data obtained from 200 ml and 25 ml samples using the same air diffuser. Here the correlation was much better than the data given in Figure 63 showing that the type of air diffuser and nominal porosity (not maximum porosity) has a great effect on foaming. Figure 65 gives the comparison of foaming data obtaining from Test Method 3213 and 25 ml volume samples using the ASTM stone along with the standard deviation obtained with Test Method 3213. Although foam testing normally gives poor repeatability[26], two factors are believed to have contributed to the large variation shown by Figures 62 through 66. First, lubricant foaming can change with time. These foaming studies have been conducted over a one year period. Secondly, preparing duplicate blends of lubricants using DC-200 silicone fluids having identical foaming values offers another variable affecting test repeatability. Figure 66 shows the comparison of foaming data obtained from Test Method 3213 and 25 ml samples using a 13/16" diameter sparger. This data shows about the same variation as that in Figure 65 and the same degree of correlation. This equivalent correlation is shown by the two linear regression equations of $Y = 52 + 3.15X$ and $Y = 45 + 3.20X$.

f. Summary

This study has shown that many physical properties of foam test equipment, test conditions and synergistic effects of some lubricants and test equipment affect foaming values obtained during testing. Specifically, this study has shown the following:

(1) The maximum pore size of ASTM diffuser stones and metal spargers does not correlate with foaming values and shows little value in controlling

the quality or "overall" pore size of the stones or spargers. Determining the "mean" pore size based on uniform flow of air bubbles all around the stone or sparger appears to be a better measurement, although having the disadvantage of being somewhat subjective.

(2) The effect of sample volume on foaming varies for different foaming lubricants when airflow rates are changed.

(3) Foaming increased greatly for air sparger pore sizes below 10 microns for both 200 ml samples and 25 ml samples for all fluids except the lubricant 5K3L6. This lubricant showed increased foaming for air spargers below 20 microns.

(4) Trace quantities of silicones which settle during storage or plate out on the test cylinder wall during foam testing can affect foaming values and test repeatability.

(5) The effect of soluble silicone on lubricant foaming depends on the lubricant's formulation.

(6) No correlation existed between foaming values of the lubricants and various diameter spargers or the ASTM stones for either the 200 ml or 25 ml sample volume tests.

(7) No correlation existed between foam volume ratios of two airflow rates for any of the fluids studied using 25 ml samples. When testing 200 ml volume samples, this ratio increased for the two non-silicone containing fluids but not for silicone containing fluids.

(8) Air bubble sizes of ASTM stones or metal spargers depends upon the foaming characteristics of the fluid as well as pore size of the stones or diffusers.

(9) The best correlation between the Test Method 3213 foaming data and the 25 ml volume foaming data was obtained using the 13/16 inch diameter

5 micron rated pore size metal sparger and a 250 cc graduated cylinder.

g. Future Effort

Future effort in the area of development of a small test volume foam test will be directed towards improving correlation between the two tests. This will involve foam testing of 4 specific fluids using the ASTM diffuser, 11/16" and 13/16" 5 micron spargers for both the standard test and small volume test. Testing will be scheduled such that each oil will be tested with all three tests being conducted on the same day. This will eliminate changes in the foaming characteristics of silicone containing fluids due to time.

SECTION III

DEVELOPMENT OF IMPROVED LUBRICATION SYSTEM HEALTH MONITORING TECHNIQUES

1. INTRODUCTION

Diagnostic methods for determining the health of gas turbine engines include the use of oil contamination monitors as important indicators of the condition of lubricant-wetted components. The primary engine health monitoring technique used extensively by the United States Air Force is the Spectrometric Oil Analysis Program (SOAP). Health monitoring devices used by SOAP are specifically designed to monitor the change in specific wear metal concentration with time. Atomic emission (AE) and atomic absorption (AA) spectrometric analysis involves the measuring and trending of contamination levels while in-line magnetic plugs and chip detectors are used to detect a rapidly progressing component failure before it becomes catastrophic.

Emission and absorption spectrometers have been shown to be effective in detecting wear particles smaller than 3-10 micron.[27] Chip detectors generally monitor particles larger than 100 micron. Wear particles with sizes above the SOAP spectrometers' and below the chip detectors' detection limits can be determined using Ferrographic techniques.[28] The Ferrograph has been developed to magnetically precipitate wear particles according to their sizes onto a glass slide. Individual particles may be observed and studied by using a bichromatic microscope or a scanning electron microscope.

Since all currently used monitoring techniques are particle size dependent, costly, and not specific for all wear mechanisms, the particle detection capability of engine health monitoring techniques is being investigated. During the evaluation period, techniques that can improve

particle detection and improve the diagnostic capabilities of wear metal analysis methods are being considered. The impact of using of fine filters of pore sizes 3-10 micron in the engine lubrication system is being evaluated with regard to continued use of present Air Force engine health monitoring techniques. A microfiltration test rig was built to simulate fine filtration in lubrication systems, to determine the effect of fine filtration on engine wear debris and determine the impact of fine filtration on SOAP. Requirements for future health monitoring systems will be defined if current SOAP methods prove inadequate.

2. BACKGROUND

During the past 15 years, increasing costs of materials and labor have focused attention on the problems generated from the wear of oil-wetted components. The detection and identification of wear particles is of critical importance since their presence is indicative of component wear. Another critical factor is that these particles can often accelerate more complex wear mechanisms which further degrade component surfaces. Several cases have been documented where large metallic wear particles produced by severe wear mechanisms are not being quantitatively analyzed. The inability to detect large wear particles has led to engine component failure without prior indication of wear as determined by spectrometric analysis techniques. The failure of SOAP to predict impending engine failure is directly related to the particle detection limitations of currently used SOAP wear monitors.

Corrosion, fluid breakdown, fatigue, adhesion, and abrasion are primary wear mechanisms which produce wear metal particles of different types and morphology. Normal rubbing wear produces small particles which are readily detected by SOAP spectrometers while the large particles produced by abrasion and erosion are not readily detected and their presence can rapidly degrade

component surfaces in the oil-wetted system.

Filtration constantly removes the large particles of wear and other debris suspended in the fluid. This process should maintain a relatively clean lubricant within the system. If wear particles are not removed, they can generate secondary wear modes which may further degrade component surfaces. However, if wear debris is removed by filtration, valuable information about the health of the engine can be lost. Because wear particles carry within them important information about the mechanisms of wear, and because their size is directly proportional to severity of wear, it may be important that these particles be captured and analyzed.

The analysis of wear particles is an important tool for diagnosing wear regimes and predicting life expectancy of oil-wetted components. The study of particle morphology can reveal a wealth of detailed and important information about the health of an engine. Information which can be determined by analyzing wear debris includes particle shapes, composition, size distribution, concentration, and rate of wear. If obtained, this information can be used to identify engine components experiencing wear so that appropriate maintenance action can be taken to replace abnormally wearing engine components.

The effectiveness of two rotating disk plasma emission spectrometers and one atomic absorption spectrometer was previously evaluated for analyzing metallic particles. None of these instruments quantitatively analyzed particles larger than 3-10 microns. The rotating disk electrode (RDE) emission spectrometer utilized by SOAP can detect 10 m particles but cannot quantitatively analyze particles larger than 1-3 μm.[28,29]

The poor particle detection capability of the various spectrometric methods is primarily the result of the limitations inherent in their sample

introduction systems and source energy. Although the present sample introduction systems are designed to transport homogeneous solutions, these systems can be improved to transport nonhomogeneous samples and thereby improve the particle detection capabilities of the spectrometer.[28]

Complete characterization of wear particles as to their size, shape, and chemical nature is not possible by SOAP. Clearly, supplementary methods of independently measuring the size, shape, and chemistry of particles are required.

3. ATOMIC EMISSION SPECTROMETER

The operational performance of the Jarrell-Ash Model 750 AtomComp was examined by analyzing several oil standards in the concentration range of 0-100 ppm. It became immediately evident that most of the analytical channels were not sensitive enough for the analysis of trace metals in oils.

a. Profiling and Optical Alignment

In order to solve the sensitivity problems of the Jarrell-Ash, Model 97-750, atomic emission spectrometer, it was examined first for profiling and alignment. Profiling is usually performed by measuring the peak response of the Mercury Monitor Lamp or the profile of an analytical line. Using the mercury lamp as a stable source, the micrometer reading was measured at 60-80% of the peak meter reading on one side of the maximum and a second reading was taken at the same meter reading on the other side. The average of the two readings is the best position of centration of the mercury line in the slit. However, to profile all the exit slits as accurately as possible, it was necessary to do the following technique:

With a D13-500 Conostan Standard containing 500 ppm of 13 elements, measurements were taken at different micrometer settings. The measurements were taken at -25, -20, -15, -10, -5, 0, 5, 10, 15, 20 and 25 μ profile

setting with 0 being the standard setting. Plotting the integrated reading vs the profile setting for each spectral line will determine the contours of the lines. The maximum reading of the peak is the profile reading and is expected to be within 2-5 microns. Several refractor plate positions were found incorrect and fine adjustments of the refractor plates were made to obtain exact centration.

Intensity measurements were taken for the D20-100 standard with and without the splash protection window. Since the readings with the window in position were higher than the readings taken without the window, it was suspected that the spectrometer might be out of optical alignment. After checking the optical alignment with a laser, it was discovered that the arc stand was out of alignment because a large crack had developed close to its base. A new stand was installed. As a result, the sensitivity of ten elements out of fourteen was substantially improved. The remaining four elements still suffered from a low signal to noise ratio. In spite of these problems, the spectrometer was calibrated using 0, 3, 10, 30, 50 and 100 ppm D14 standards obtained from the Joint Oil Analysis Program. The spectrometer was also standardized with a high (D14-100) and a low (D14-0) standard in order to examine the accuracy and repeatability of the analytical data. Unfortunately the accuracy and precision were best for most of the elements only at concentration levels greater than 30 ppm. Even though the original problem of the spectrometer's sensitivity was greatly minimized, still a solution had to be found before the spectrometer could produce reliable analyses.

b. Refractor Plates

During profiling, it was discovered that most of the refractor plates were frosted and some were pitted. Cleaning the surface with a cotton swab

thoroughly examined for any malfunction which could be in the rotation of the disk electrode, elevation setting of the sample stand, setting of the analytical gap, or bad discharge. The sample stand was found to be free from any malfunction. The flow of CO_2 which stabilizes the discharge and improves the line-to-background ratio was also checked. In addition to the sample stand and gas flow of CO_2 the effect of other gases (Ar, air), the preburn and exposure times on the accuracy and precision of the analyses was determined.

c. Optimizing Gas Flow

(1) Background Signal for Ester and Hydrocarbon Oils

Signals from blank MIL-L-7808 and 245 basestock (heavy hydrocarbon oil) were also examined for the three gases under different flow rates. Except for Mg and Zn, the background signals of MIL-L-7808 were lower than those of 245 basestock for corresponding elements, gases and flows. Figures 67-72 graphically show the effects of flow rates of Ar, air, and CO_2 on the background signal, for Fe, Cr, Cu, Mg, Mo and Ti. Based on the above findings air flowing at 5 LPM should produce optimum signals and lowest backgrounds for all elements, except for Mg. Since the Mg signal is approximately an order of magnitude higher than all elements considered at the 90 ppm level, using air for Mg instead of Ar should be as effective.

(2) Optimizing Signal-to-Background Ratio for Conostan Standard

One parameter that might influence the signal-to-background ratio of the Jarrell-Ash atomic emission spectrometer was optimized. The gas jet flowing through the boron nitride cone that surrounds the counter-electrode (top electrode) stabilizes the discharge and should improve the signal-to-background ratio. Carbon dioxide has been used as the gas and its flow was optimized earlier. However, analytical results for some

was in vain. Jarrell-Ash engineers explained that fungus activity could have damaged the surfaces of these Cortex plates. This could happen in a high humidity environment. The refractor plates permit fine adjustment of the spectral line with respect to its exit slit. Damaged surfaces will not refract the light efficiently and therefore reduce the intensities of the desired spectral lines. A replacement of the damaged plate of the iron channel with a new one increased the sensitivity of the iron line threefold. As a result all the damaged refractor plates were replaced with new ones and their respective channels were profiled. The spectrometer was recalibrated and standardized using Conostan standards. Concentrations of 900, 600, 300, 100, 50, 30, 10, and 0 ppm were used to establish calibration curves for fourteen elements. Calibration curves were obtained by plotting the intensity ratios versus concentrations. These values were also entered in the Data Base Manager software under group 5. Entries in this group must be made for every element that is to be read out in concentrations. Two point standardization was accomplishment restoring the intensity ratio for each element in a "high" and "low" standard. The two point standardization introduces a "gain" change and "offset" which correct for any change in the background not compensated for by the background corrections. The 100 and 0 ppm were chosen to be the high and low standards, respectively. In the normal operation of the spectrometer, these standards will be used to standardize the instrument periodically prior to analyzing samples. The instrument was then tested for accuracy and precision. Accuracy was dramatically improved. Ten out of fourteen elements gave accuracies within five percent. However, precision was not as good as expected and it ranged from less than one percent for Mg to ten percent for Si at the 100 ppm level. Since achieving reproducible results was difficult, the sample stand was

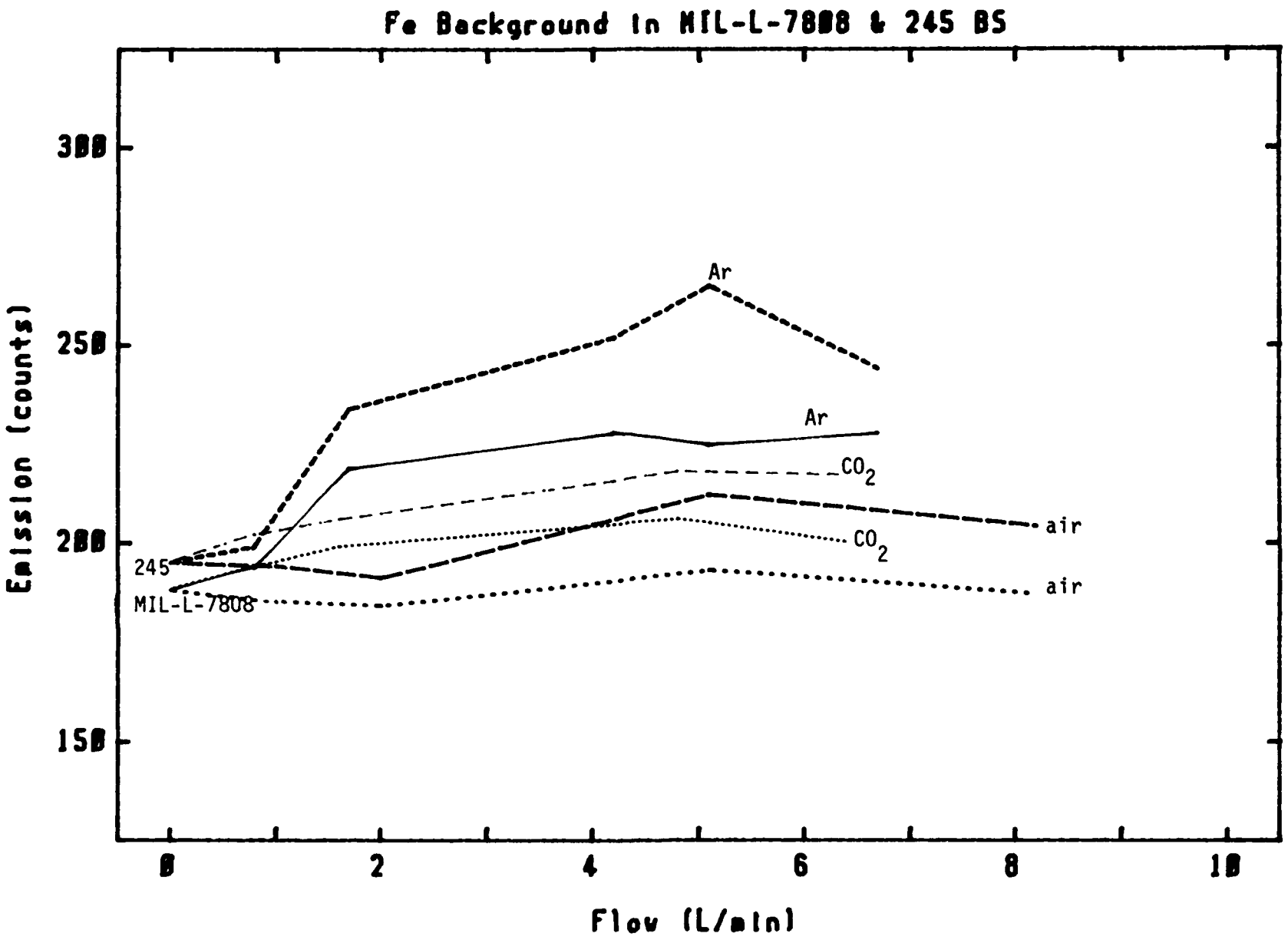


Figure 67. Gas Flow Rate Effect on Background Emission Signal for Fe in MIL-L-7808 and 245 Basestock

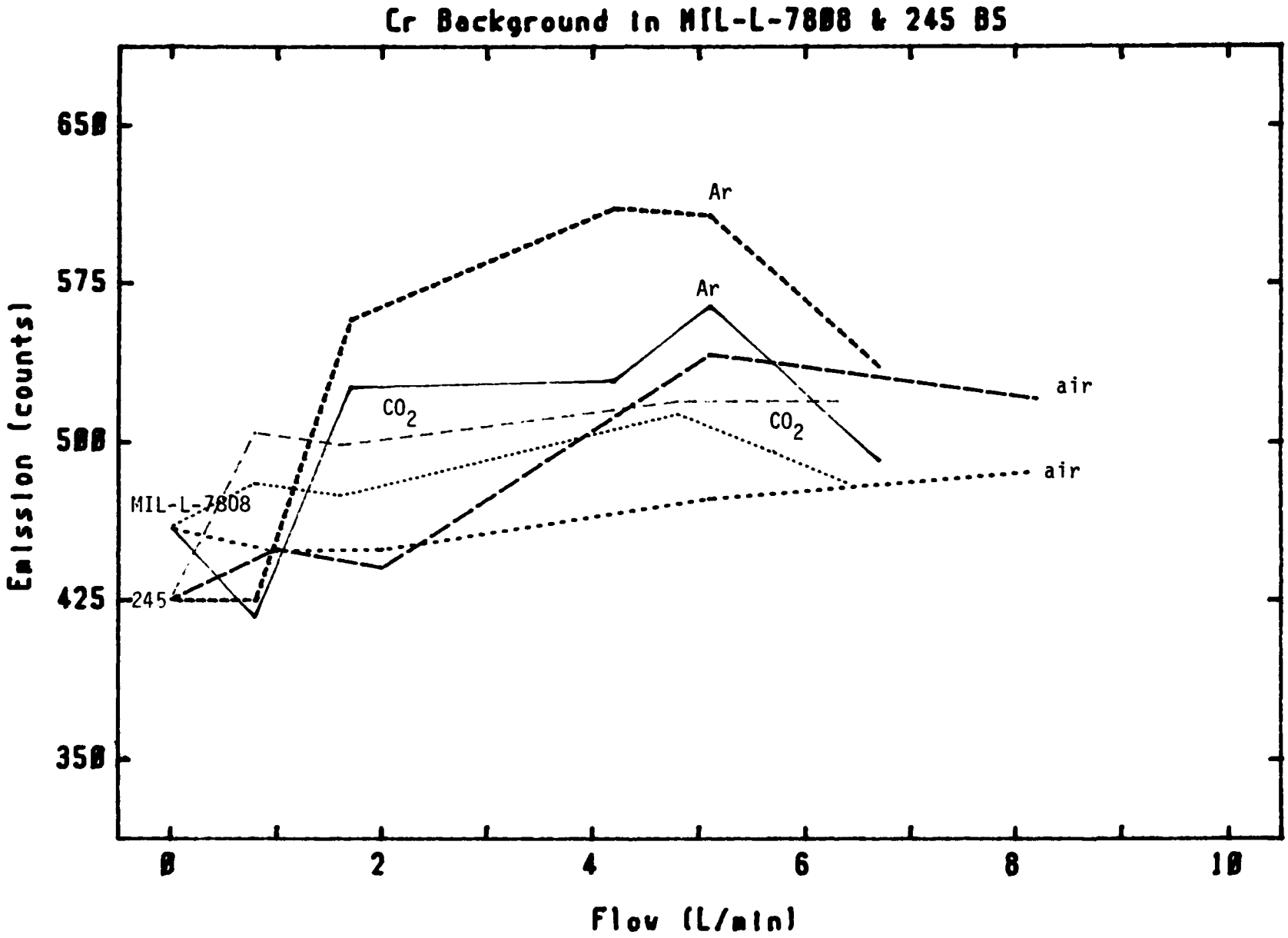


Figure 68. Gas Flow Rate Effect on Background Emission Signal for Cr in MIL-L-7808 and 245 Basestock

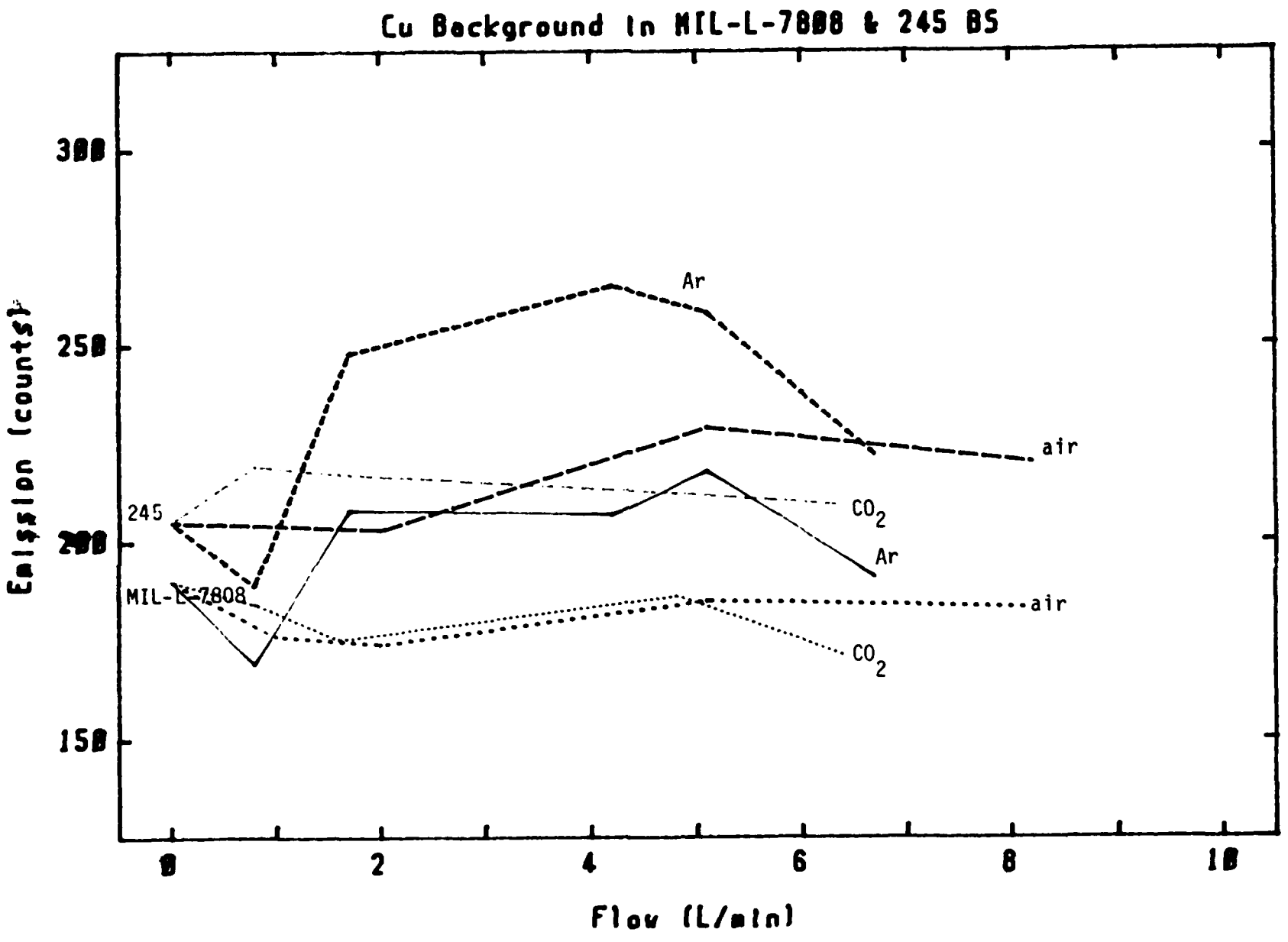


Figure 69. Gas Flow Rate Effect on Background Emission Signal for Cu in MIL-L-7808 and 245 Basestock

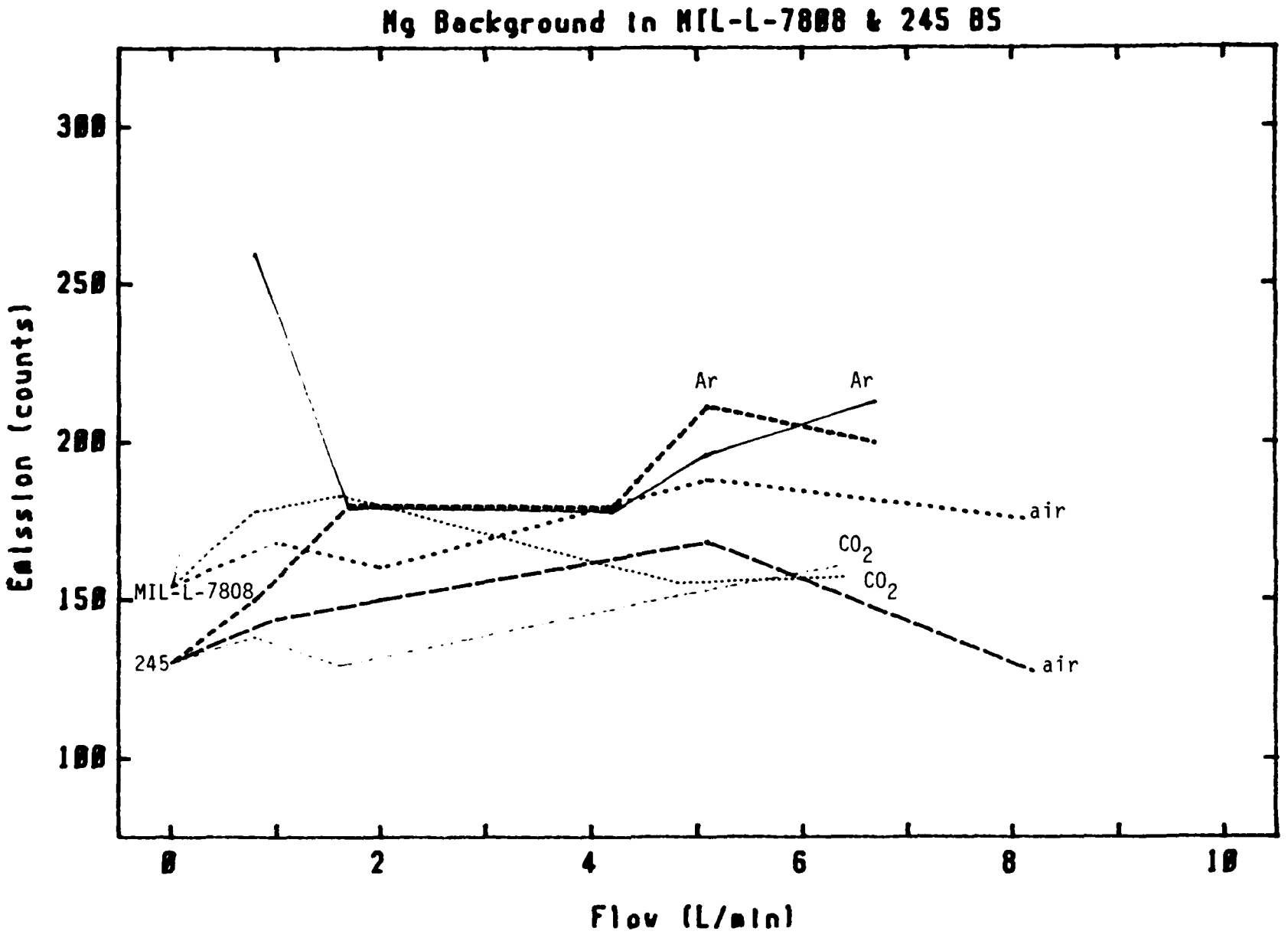


Figure 70. Gas Flow Rate Effect on Background Emission Signal for Mg in MIL-L-7808 and 245 Basestock

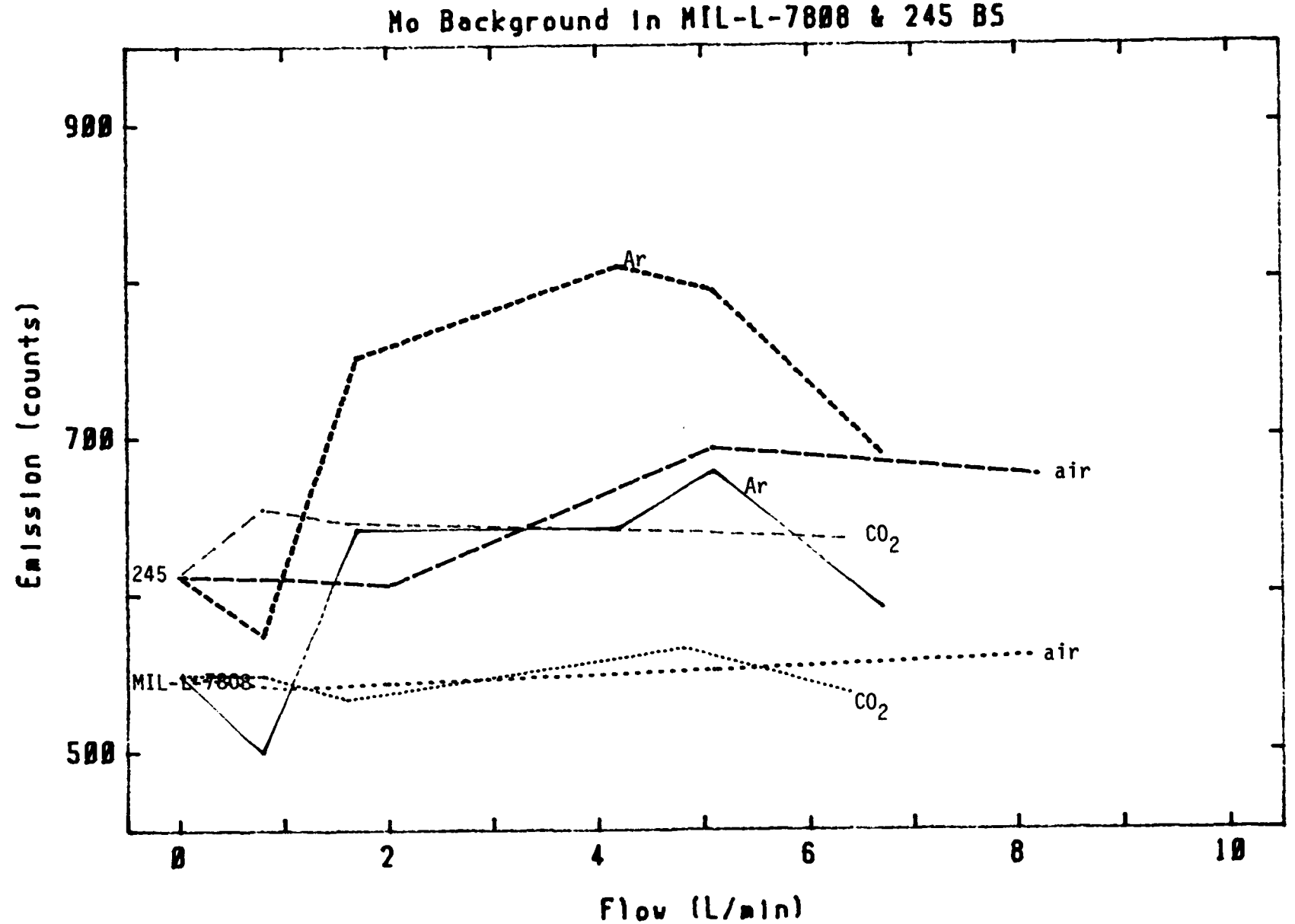


Figure 71. Gas Flow Rate Effect on Background Emission Signal for Mo in MIL-L-7808 and 245 Basestock

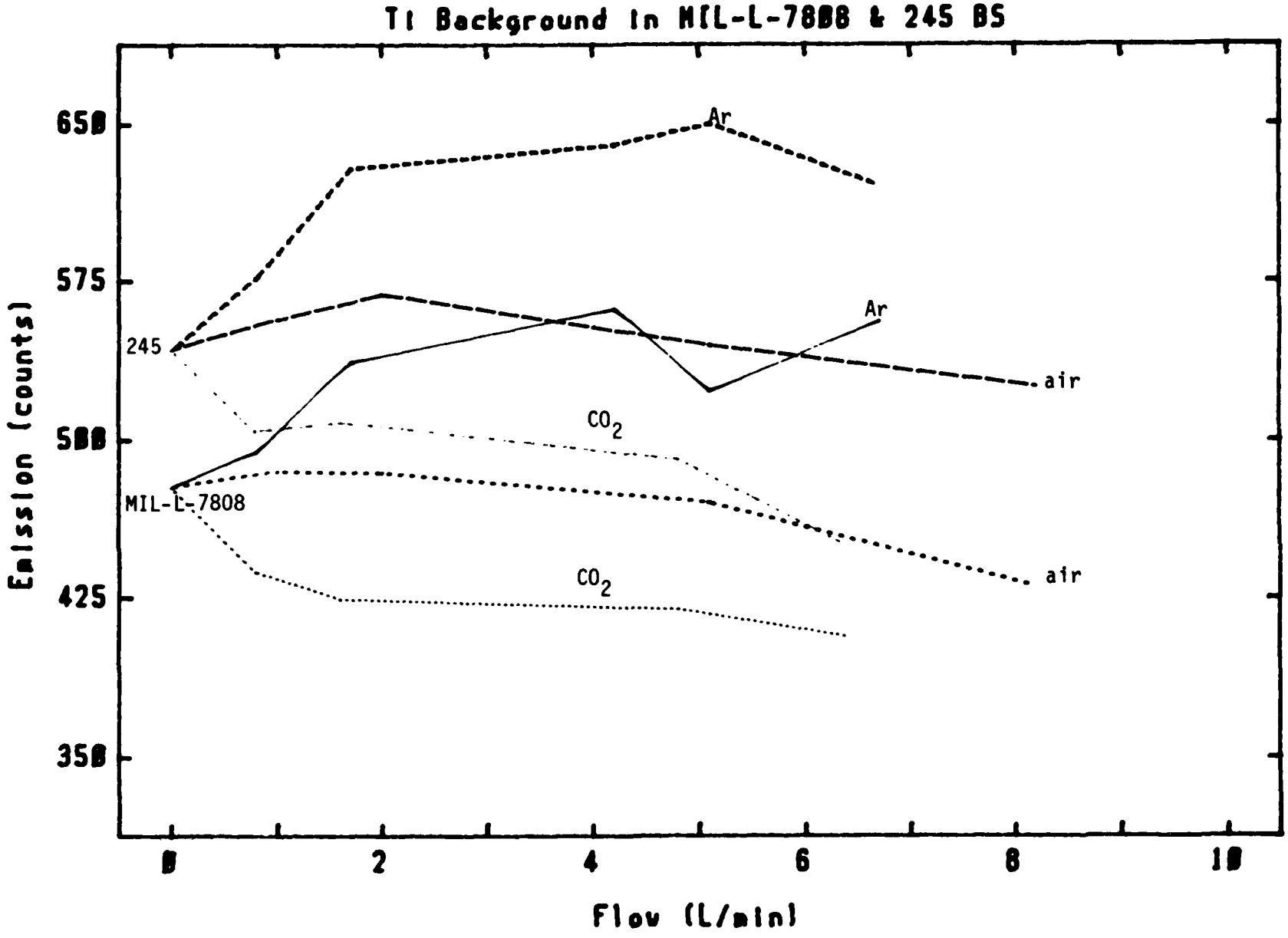


Figure 72. Gas Flow Rate Effect on Background Emission Signal for Ti in MIL-L-7808 and 245 Basestock

elements have been questionable and therefore other gases were studied. Argon and air along with carbon dioxide were used in order to determine which gas and at what flow the signal-to-background ratio is optimum for fourteen elements. A 900-ppm Conostan concentrate containing 19 elements (D19-900) was diluted with 245 basestock and MIL-L-7808 oils to prepare two 90 ppm standards (D19-90). The results reported are the averages of duplicate readings of the emission signal at several gas flow rates. The emission signal plotted is the emission signal in counts minus the background signal. The results are graphically shown in Figures 73-82 for only 10 elements. These figures compare the effect of Ar, air and CO_2 flow rates on the emission signal for Fe, Ag, Al, Cr, Cu, Mg, Ni, Si, Ti and Mo. Even though 14 elements were studied, only the data for the above 10 elements was considered, because the correlation program at TSC considers the same 10 elements.

According to Jarrell-Ash, it is desirable to analyze for trace metals with the gas jet on in order to minimize signal fluctuation. Therefore, only optimum gas flow rates greater than zero will be considered. It is clearly shown that CO_2 suppresses the emission signal for all elements at higher flow rates. Except for Mg, air enhances the signal for elements in 245 basestock at 5.1 LPM. The optimum gas for Mg in 245 oil is Ar at 5.1 LPM.

Since all samples analyzed by emission are normally used MIL-L-7808, optimum flow rates of Ar, air, and CO_2 for standards in MIL-L-7808 were also determined. Based on the curves shown in Figures 73-82, elements in MIL-L-7808 behave differently than the same elements in 245 basestock for the three gases studied. At a gas flow rate of 5.1 LPM or higher, air was the optimum gas for Ag, Al, Cu, Mo, Ni and Ti while Ar was

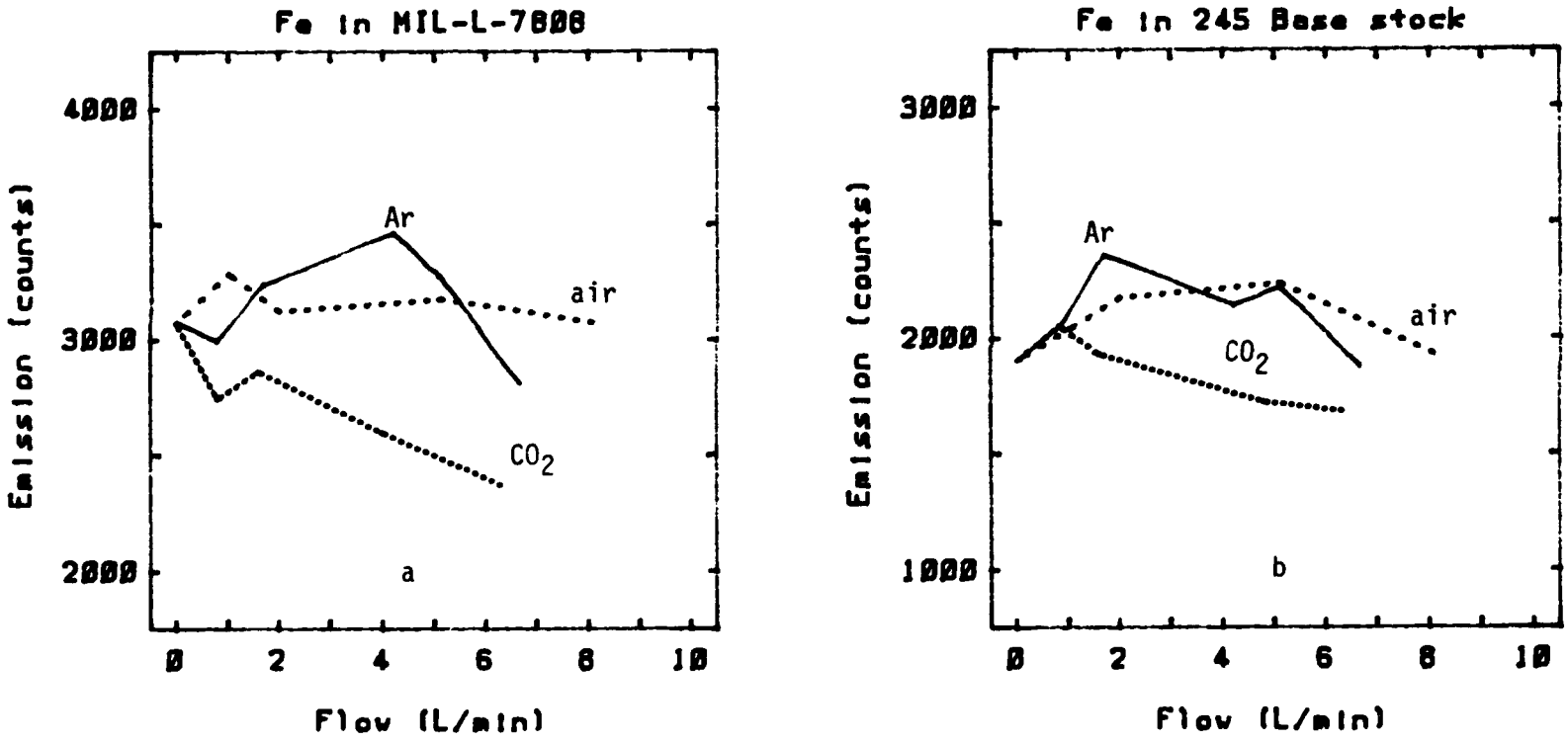


Figure 73. Gas Flow Rate Effect on Emission for Fe in a) MIL-L-7808 and b) 245 Basestock.

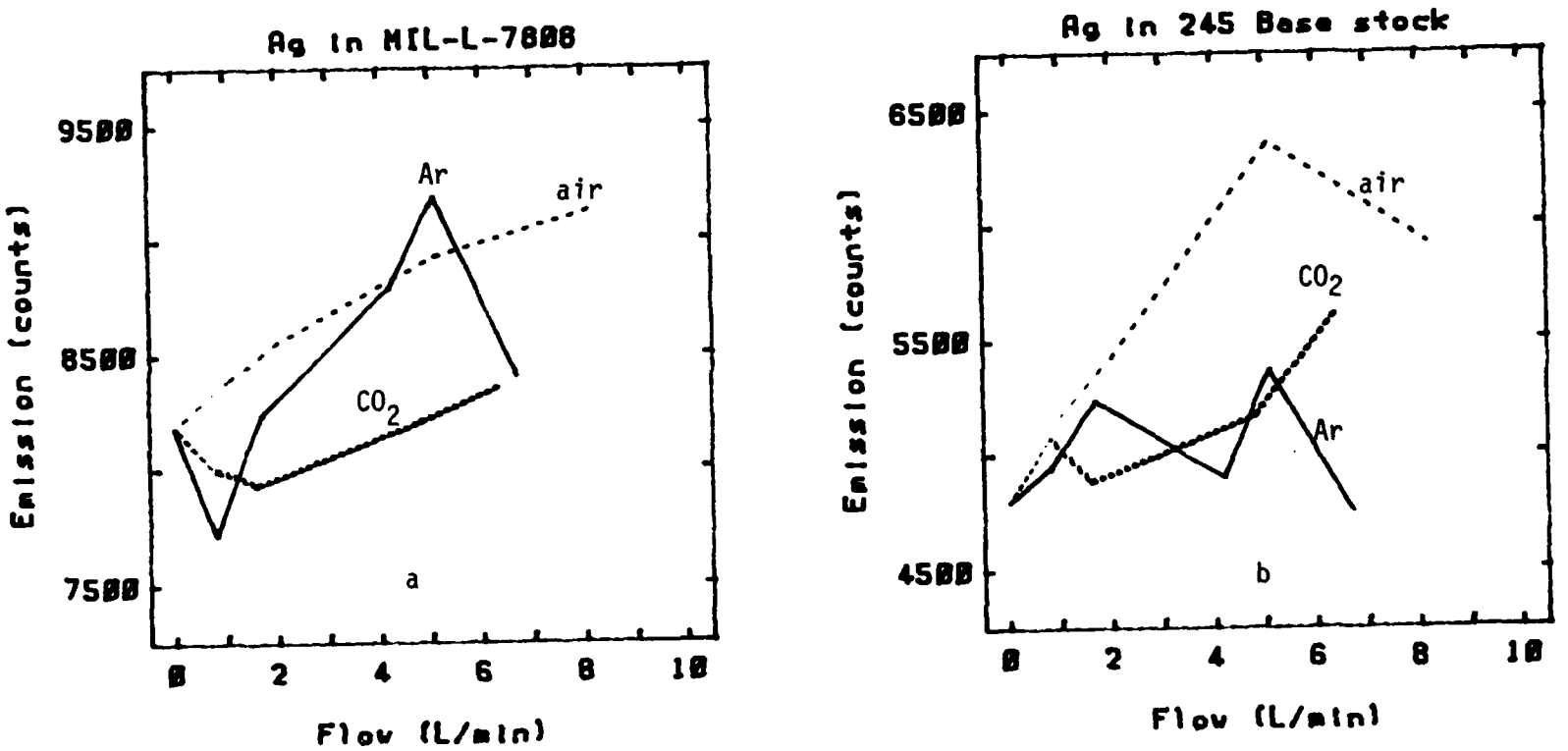


Figure 74. Gas Flow Rate Effect on Emission for Ag in a) MIL-L-7808 and b) 245 Basestock.

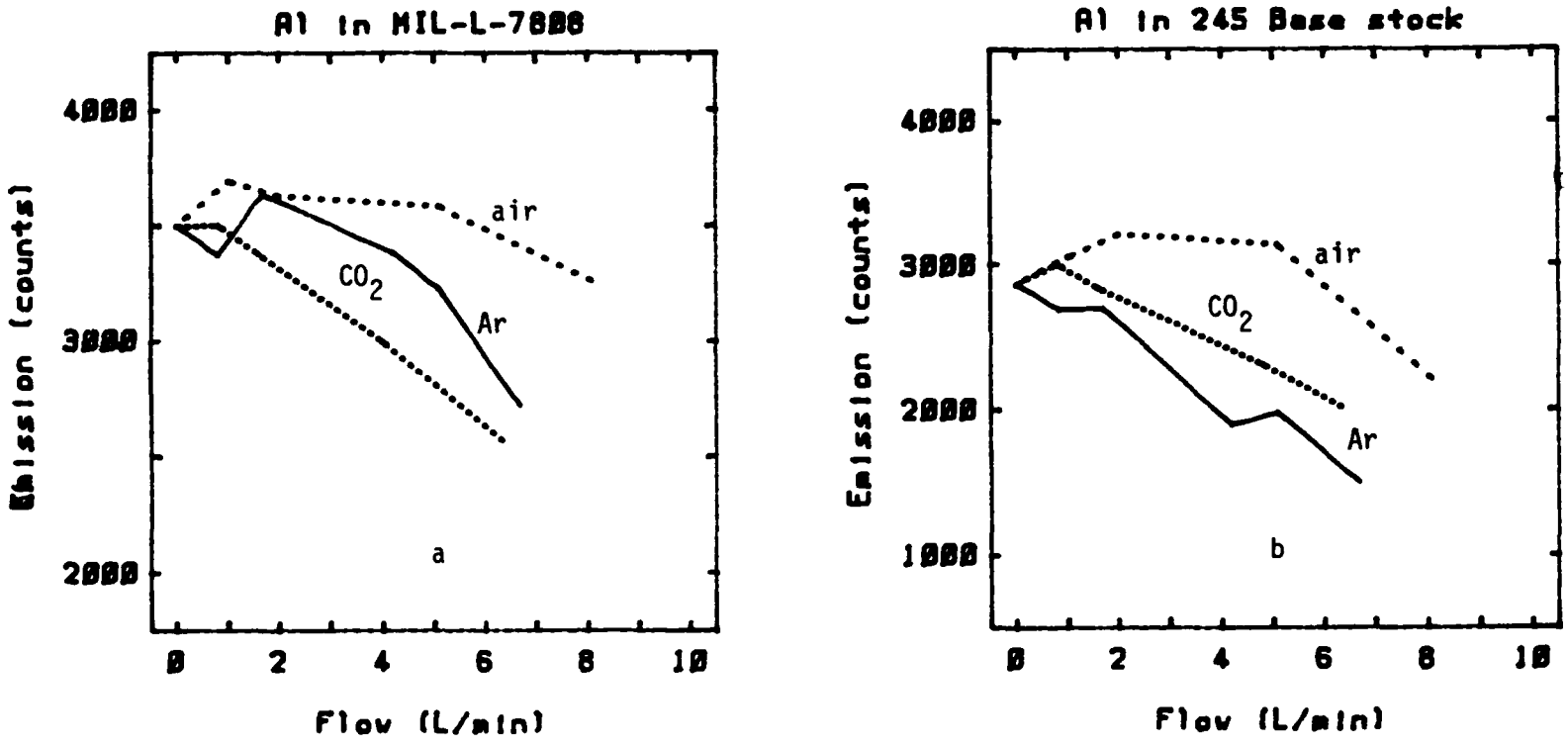


Figure 75. Gas Flow Rate Effect on Emission for Al in a) MIL-L-7808 and b) 245 Basestock.

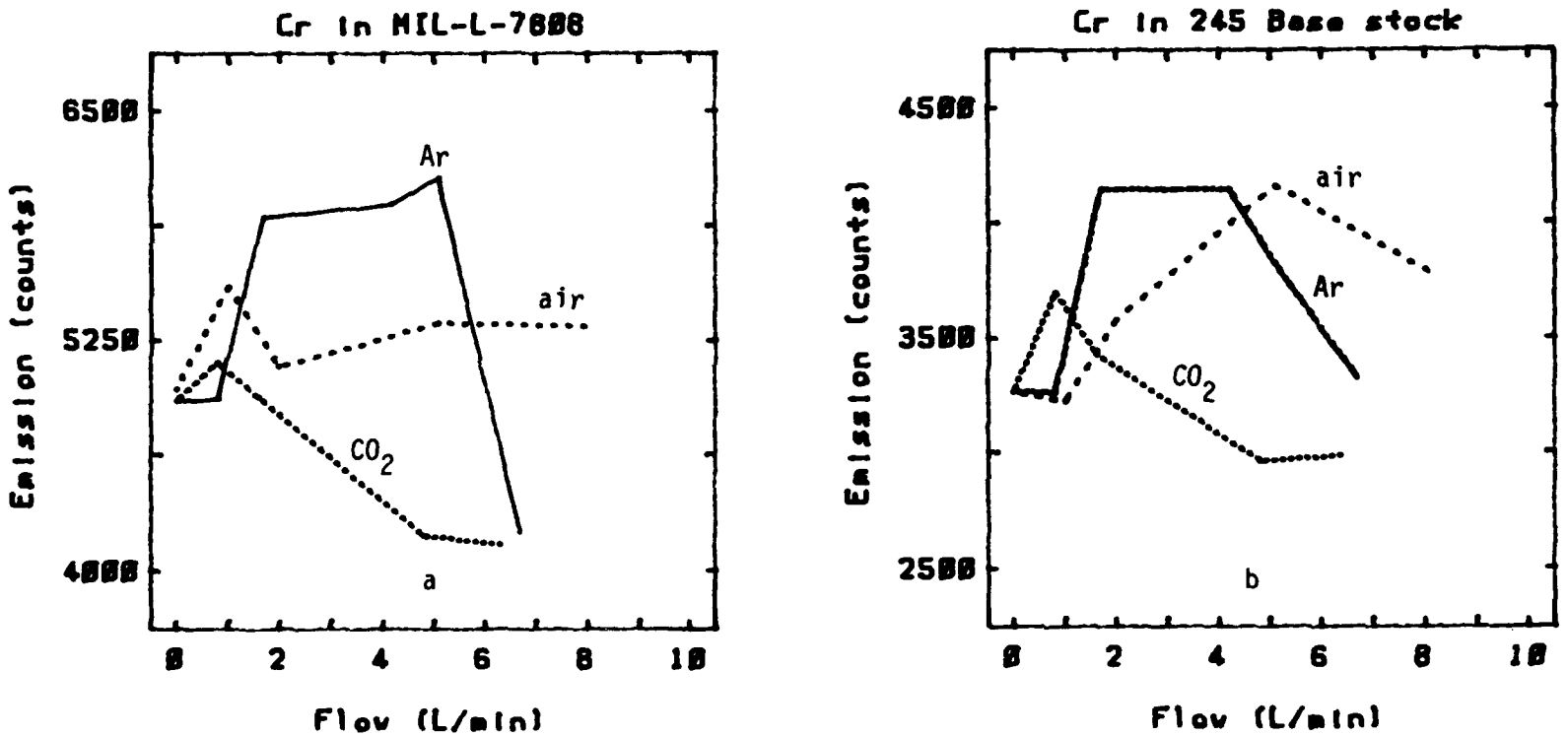


Figure 76. Gas Flow Rate Effect on Emission for Cr in a) MIL-L-7808 and b) 245 Basestock.

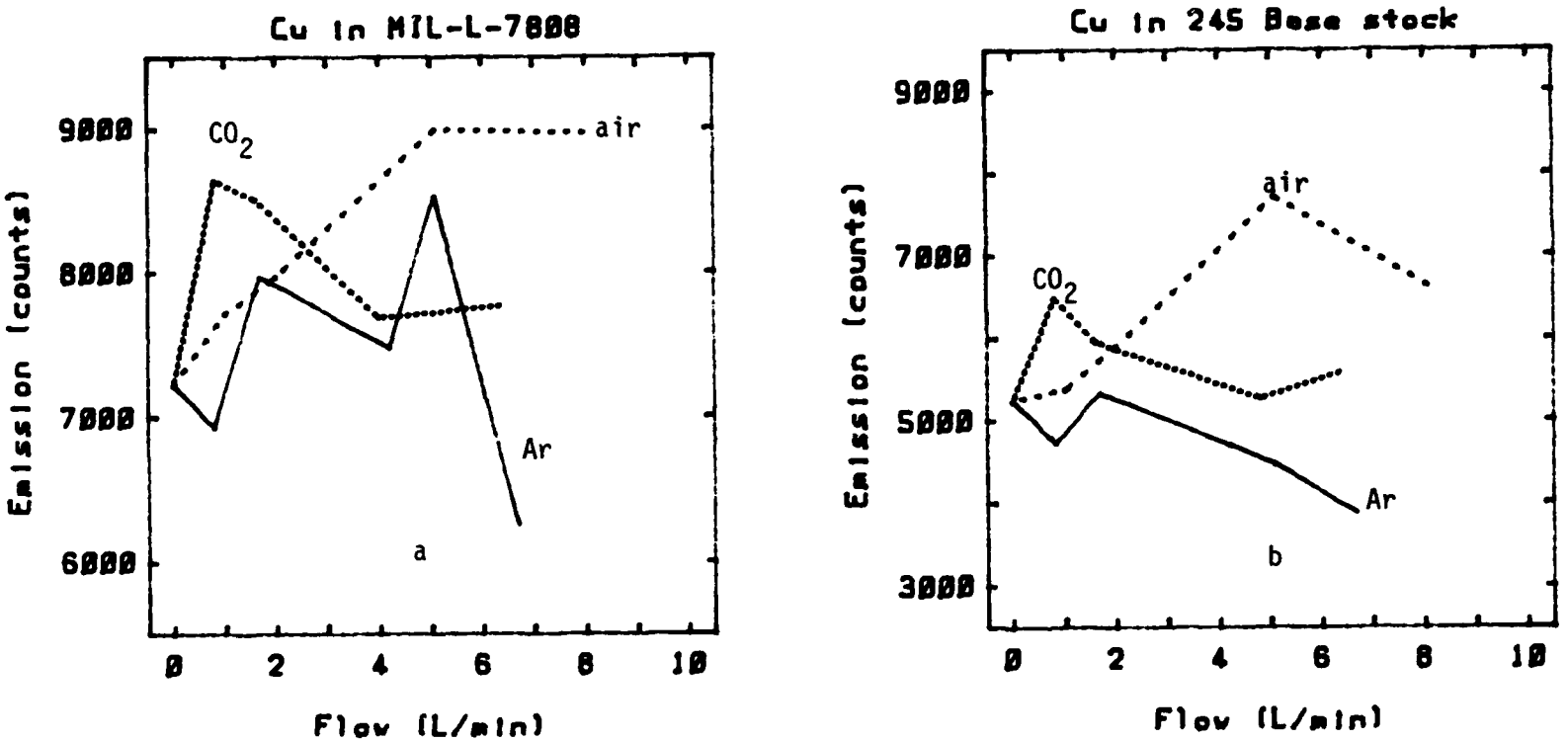


Figure 77. Gas Flow Rate Effect on Emission for Cu in a) MIL-L-7808 and b) 245 Basestock.

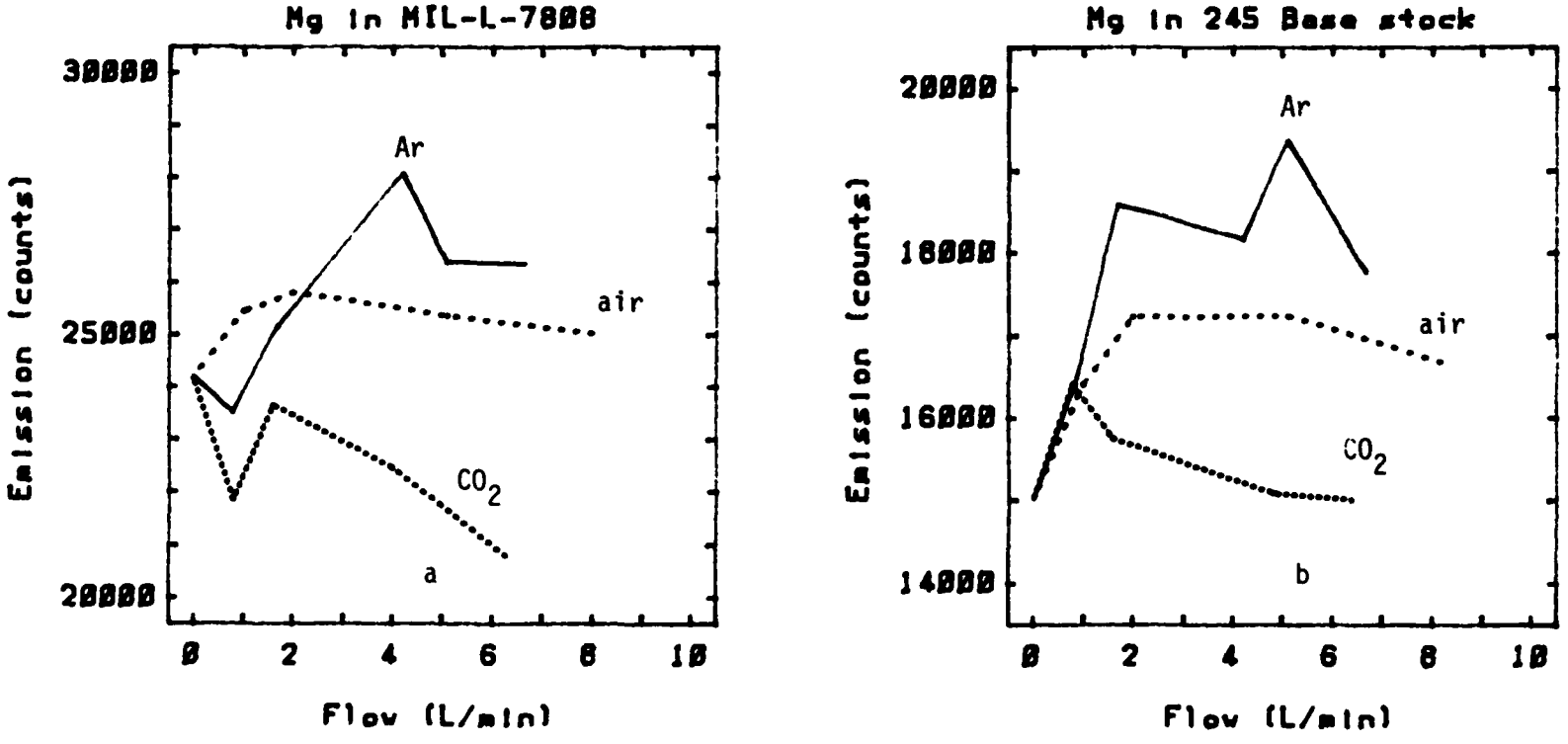


Figure 78. Gas Flow Rate Effect on Emission for Mg in a) MIL-L-7808 and b) 245 Basestock.

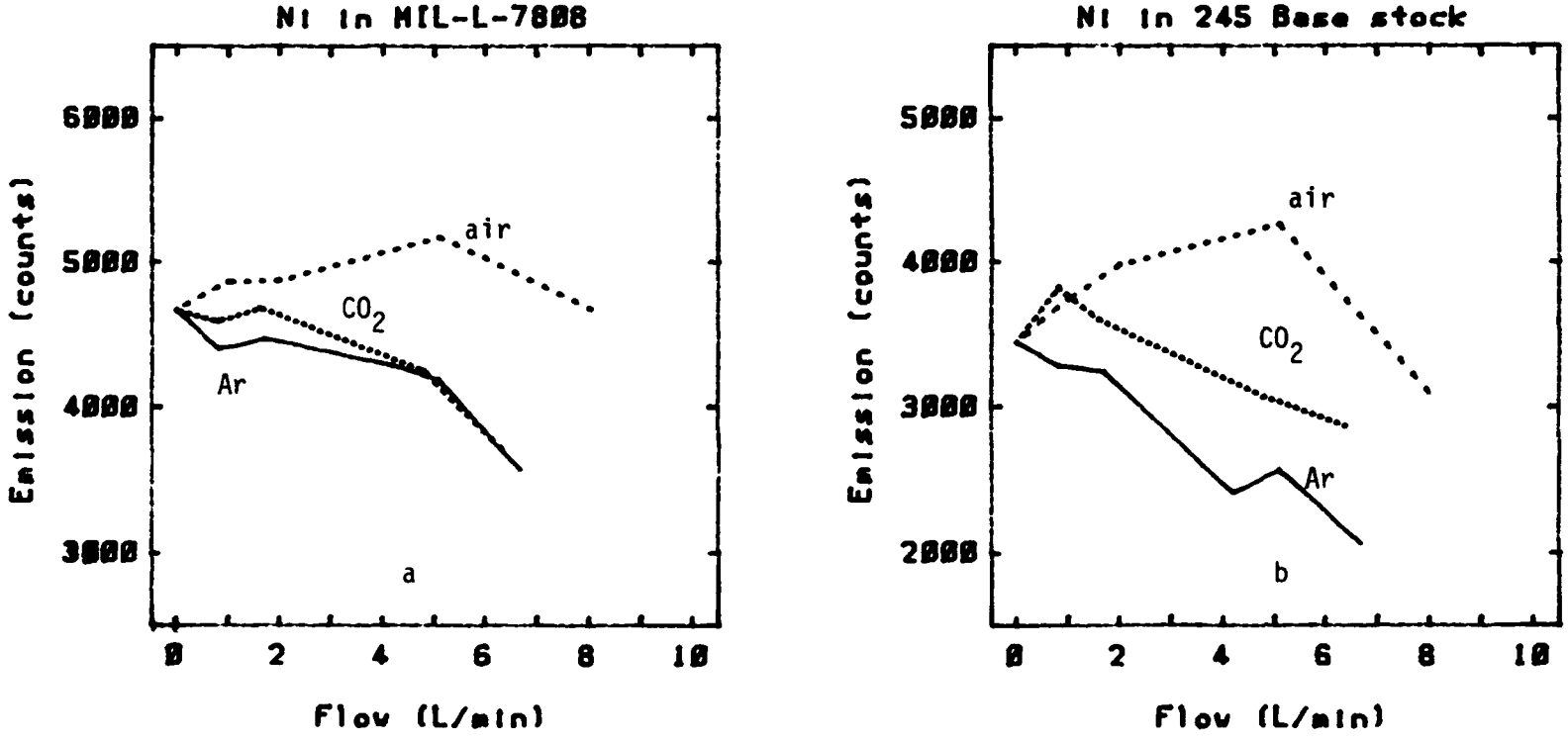


Figure 79. Gas Flow Rate Effect on Emission for Ni in a) MIL-L-7808 and b) 245 Basestock.

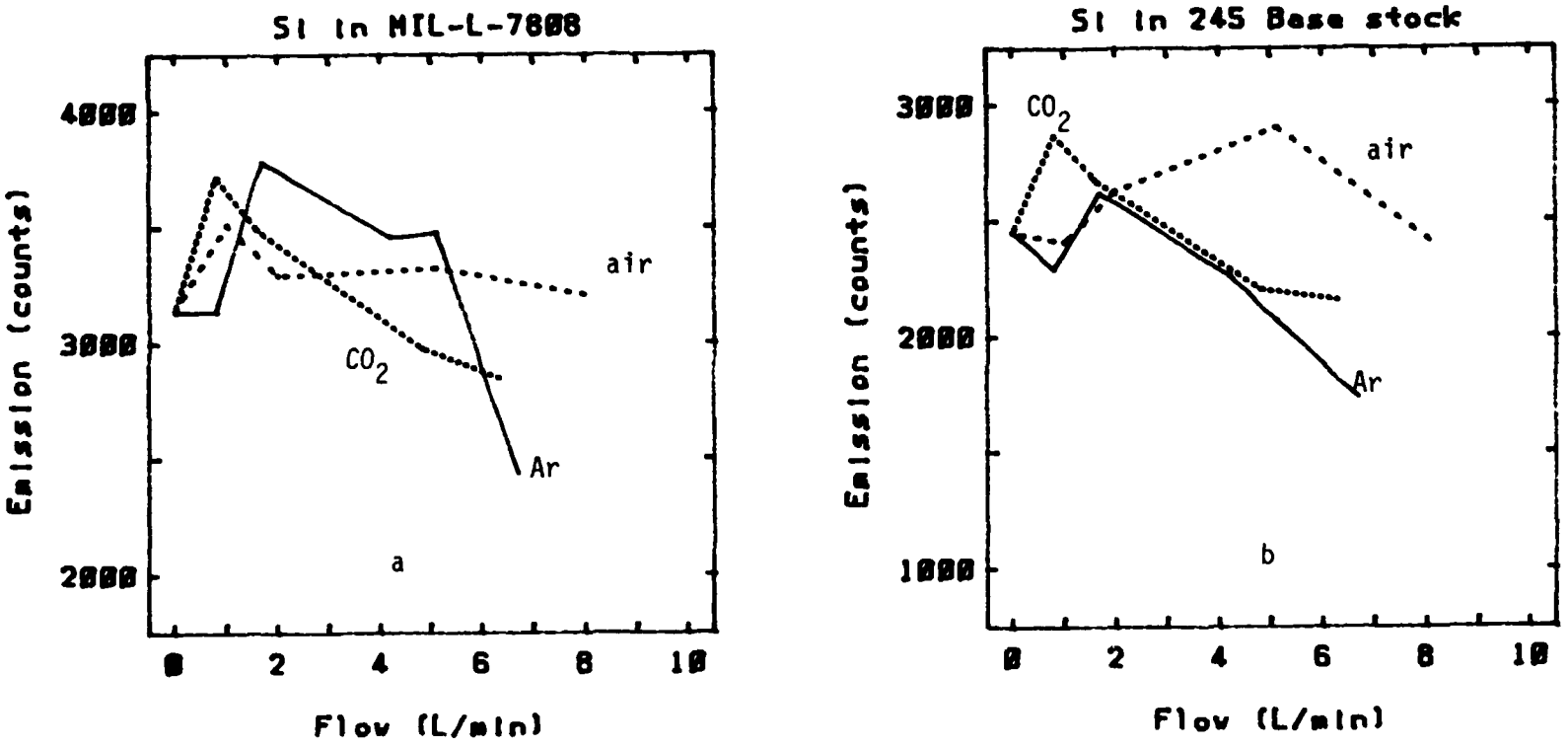


Figure 80. Gas Flow Rate Effect on Emission for Si in a) MIL-L-7808 and b) 245 Basestock.

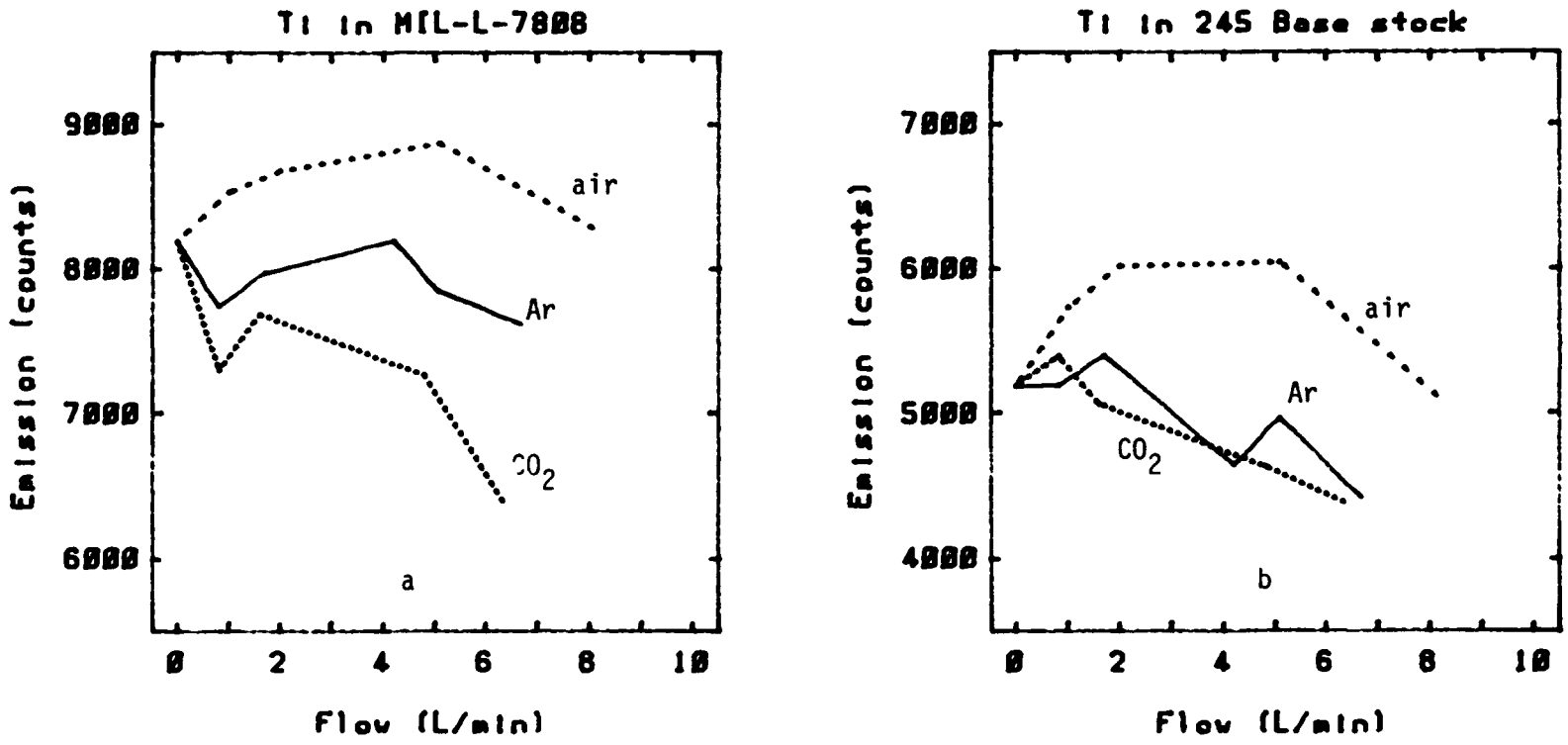


Figure 81. Gas Flow Rate Effect on Emission for Ti in a) MIL-L-7808 and b) 245 Basestock.

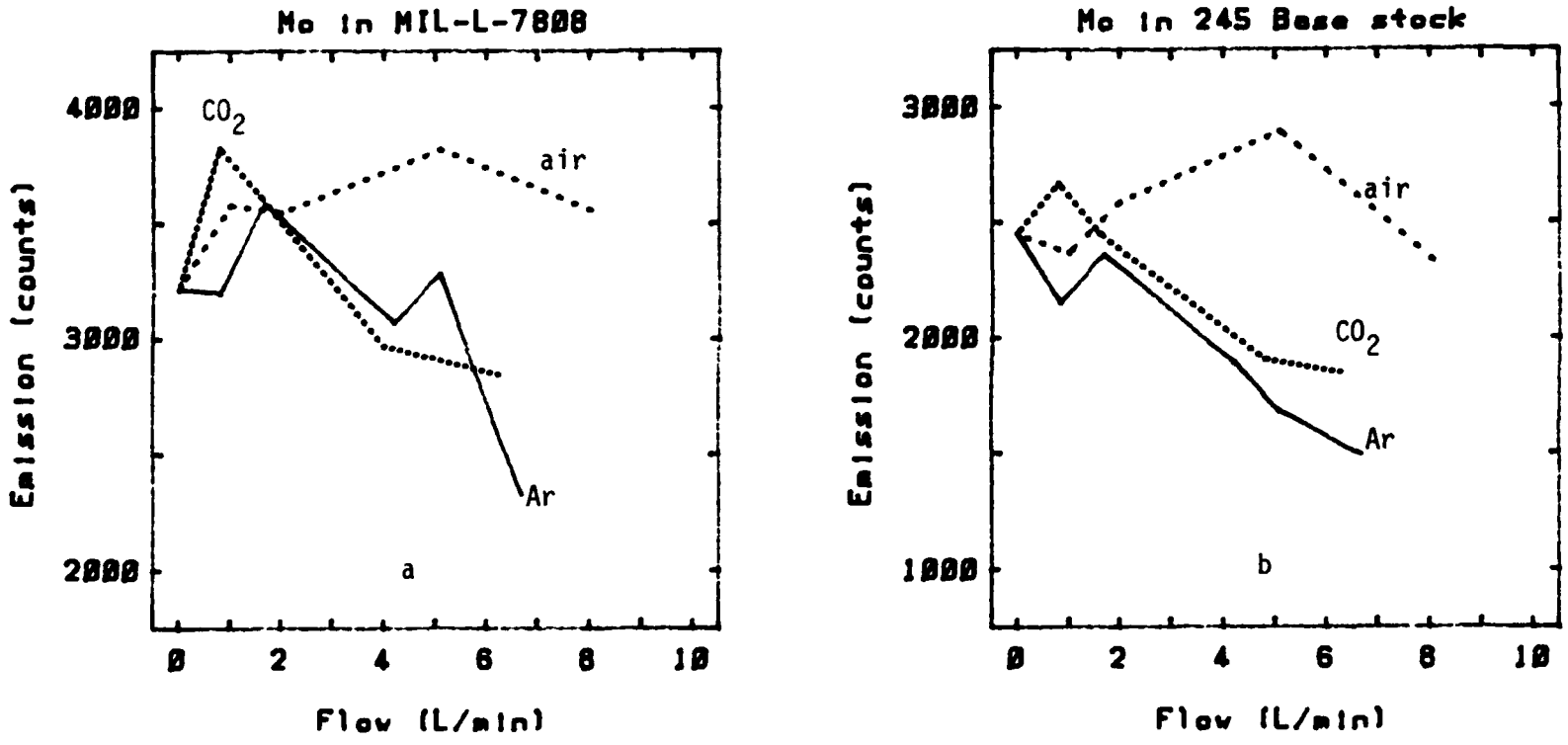


Figure 82. Gas Flow Rate Effect on Emission for Mo in a) MIL-L-7808 and b) 245 Basestock.

optimum for Fe, Cr, Mg, and Si.

d. Exposure Time

Exposure time study was performed at the optimum CO_2 flow of 1.7 LPM. Three burns were obtained for each exposure time setting using a 30 ppm Conostan standard. Exposure times of 20, 25, and 30 seconds were studied at a constant preburn time of 5 seconds. The results shown in Table 44 reveal that the longer exposure times, 25 or 30 seconds, yield better precision than the shorter time.

e. Spectrometer Calibration

After optimum conditions were established for the analytical gap gas flow (Ar), sample stand argon flow and exposure time, the spectrometer was then calibrated using standards in the range of 0 to 900 ppm. A new matrix was created under the file name "SOAP" for fourteen elements (Fe, Ag, Al, Cr, Cu, Mg, Na, Ni, Pb, Si, Sn, Ti, Mo, Zn).

f. Analysis of Used Oil Samples

(1) J57 Test Engine Stand and Engine Simulator Samples.

Samples from the J57 test engine stand and engine simulator were submitted for atomic emission analysis. The corresponding analytical results are shown in Tables 45 and 46. It is clearly seen that the spectrometer was responding to changes in wear metal concentration. As sample time increases the concentration of Fe increases.

(2) Correlation Samples

Four correlation samples are being received, monthly, from the Technical Support Center (TSC) to be analyzed on the Jarrell-Ash spectrometer. The results reported to TSC do not agree with the average in mean values of other labs using Baird Atomic A/E35U-3 rotating disk spectrometer. The average score for six months was 72%. Mainly, Cr failed

TABLE 44

EXPOSURE TIME EFFECT ON EMISSION READOUT[a]

	20 Sec (ppm)	25 Sec (ppm)	30 Sec (ppm)
Fe	26.77±0.74	31.49±0.79	30.14±6.2
Ag	24.19±3.53	26.97±6.79	26.28±5.36
Al	30.30+1.00	32.28±0.86	31.52±0.85
Cr	27.35±6.20	35.05±0.63	33.08±1.73
Cu	25.34±1.51	23.20±5.32	22.82±3.73
Mg	27.04±0.98	31.58±0.89	30.47±0.82
Ng	14.76±1.18	13.41±2.43	14.62±1.06
Ni	28.49±1.25	31.44±0.75	30.05±0.46
Pb	29.89±2.48	36.35±1.13	33.30±0.80
Si	24.37±3.56	31.17±0.45	27.00±5.68
Sn	27.09±1.89	31.09±2.07	31.33±0.97
Ti	26.16±5.07	31.42±1.02	31.04±0.71
Mo	29.66±1.95	33.48±0.19	31.33±1.42
Zn	26.83±1.13	25.98±4.94	31.24±3.88

(a) 5-second preburn time and D20-30 standard were used.

TABLE 45

ENGINE SIMULATOR TEST NO. 47-A1-WF WEAR METAL ANALYSES

SAMPLE NUMBER	SAMPLE TIME HOURS	Fe	Ag	Cr	Cu	Na	Pb	Zn
CAN	0							.1
SUMP	0							
OP-361-5	5	22.5				.1		1.6
OP-361-10	10	17.3	.4					1.3
OP-361-15	15	19.1	.8	1.1			7.0	1.5
OP-361-20	20	23.4	.9				4.4	1.3
OP-361-25	25	37.2	1.2				3.5	1.0
OP-361-30	30	39.6	1.5					.4
OP-361-35	35	51.3	2.1				4.7	1.2
OP-361-40	40	57.6	2.3				3.8	1.0
OP-361-45	45	64.2	2.8	.2		.2	5.8	1.5
OP-361-50	50	64.5	2.5					.7
OP-361-55	55	66.5	2.5					.5
OP-361-60	60	66.2	2.8	.3	.6			1.6
OP-361-68	65	81.0	2.7	.8	.7			1.7
OP-361-70	70	89.8	3.0	1.3	.8	.1		1.6
OP-361-75	75	84.2	2.6	.5	.6			.9
OP-361-80	80	92.7	2.9	.8	.8	.1		1.2
OP-361-85	85	94.1	2.6	.7	.8			1.1
OP-361-90	90	111.0	2.8	1.5	.8			1.6
OP-361-95	95	115.4	2.8	2.2	1.0	.1		1.7
OP-361-100	100	127.4	2.6	2.2	1.0	.1		1.2

TABLE 46

ENGINE TEST STAND WEAR METAL ANALYSES

SAMPLE NUMBER	SAMPLE TIME HOURS	Fe	Ag	Cr	Cu	Mg	Na	Pb	Si	Zn	
OP-360-1	0	28.1	.1	1.1	1.3	8.8	2.0	283.5		25.9	
OP-360-3	5	9.6		1.9	.5	1.1	.1	53.6		7.2	
OP-360-5	10	12.0	.2	1.0	.4	2.0	.6	53.4		10.6	
OP-360-7	15	13.4	.2	2.2	.8	2.0	1.3	53.7		11.4	
OP-360-9	20	12.8	.3	2.2	1.0	1.9	1.0	48.4	16.1	11.0	(1.9)*
OP-360-11	25	13.5	.4	2.6	1.1	1.9	.69	48.1		14.2	
OP-360-13	30	14.5	.6	4.2	2.0	2.4	1.5	46.1	3.0	15.7	
OP-360-15	35	14.9	.4	3.4	1.2	2.0	.7	44.2	3.4	13.3	
OP-360-17	40	11.8	.1	1.6	1.3	1.6	1.8	28.2	2.8	9.0	
OP-360-19	45	13.7	.2	2.5	1.3	1.7	1.7	31.7	4.1	9.6	
OP-360-21	50	14.9	.2	1.9	1.2	1.7	.6	29.7	4.1	9.5	
OP-360-23	55	14.9	.2	2.1	1.2	1.9	2.4	29.6	4.6	10.8	
OP-360-25	60	14.7	.3	1.7	1.1	1.9	1.9	28.1	5.1	9.5	
OP-360-27	65	16.3	.2	2.4	1.2	1.9	2.4	27.1	5.1	10.1	(1.3)
OP-360-29	70	16.4	.5	1.8	.8	3.3	1.0	24.0	5.6	31.4	(6.1)
OP-360-31	75	12.9	.3	.9	.5	6.1	2.4	11.1	2.8	31.1	
OP-360-33	80	23.1	.8	2.1	1.2	15.7	2.0	18.2	8.4	75	

*( ) AA values

the accuracy criteria in the synthesized oil and Mg, Si and Ni failed the accuracy criteria in the used oils. An investigation was conducted in order to determine the reason for these accuracy failures. It has to be noted that the AFWAL/POSL spectrometer is the only Jarrell-Ash instrument entered in the correlation program. All others are Baird Atomic A/E35U-3. Therefore, factors that may differently influence the emission response of these two spectrometers include source energy, choice of spectral lines and matrix effect. Since Baird Atomic does not have a gas jet flowing on the analytical gap, the Jarrell-Ash may or may not produce results similar to Baird Atomic. It was also found that the wavelengths of half of the elements used in Jarrell-Ash are different from Baird Atomic. Since wavelengths of the same elements have different intensities, it is conceivable that spectrometers measuring the same elements but using different spectral lines produce different results.

g. Summary

Like the A/E35U-3 spectrometer (Baird-Atomic) the Jarrell-Ash spectrometer suffers from poor particle detection capability. Significant improvement in sensitivity was obtained when using air as the stabilizing gas, replacing and profiling the refractor plates and optimizing preburn (5 sec) and exposure (25 sec) times. The Jarrell-Ash spectrometer results did not correlate with Baird Atomic when analyzing JOAP TSC correlation samples.

h. Future Effort

Since the present rotating disk atomic emission spectrometer sample introduction system is particle size dependent, plans are being made to implement an ashing technique developed by UDRI.[29] Ashing technique allows the sample to be deposited directly on the disk and ashed in a muffle furnace prior to analysis. Plans are being made to use coated graphite disks and

higher density graphite disks than presently used to prevent permeation and adsorption of trace metals beyond the disk surface.

DC arc is another capability that will be used. Jarrell-Ash is equipped with a DC arc stand which allows the analysis for trace metals in powders. The DC arc allows for a total consumption of the sample at higher temperature than the spark source. Therefore, the capability of this source to analyze for wear metal particles will be investigated.

4. WEAR PARTICLE ANALYZER

a. Introduction

The Spectrometric Oil Analysis Program (SOAP) is used primarily by the USAF to monitor changes in specific wear metal concentrations using atomic emission and atomic absorption spectroscopy. Magnetic plugs and chip detectors are also used as on-line engine health monitoring techniques. However, the sensitivity of these techniques is limited by particle sizes. The particle size sensitivity of SOAP is limited to 3-10 microns, depending upon the metal being analyzed, and the on-line monitoring devices are limited to large size particles. Therefore, a need exists for a technique to monitor wear metal debris having particle size sensitivity above SOAP and below the particles captured by magnetic plugs and chip detectors.

One of the techniques being considered is the Wear Particle Analyzer (WPA) manufactured by Tribometrics Inc., Berkeley, CA (Figure 83). Particle capture efficiency, particle size detection limit and filter fiber size effect are among the parameters which were evaluated.

b. Principle of Operation

The wear particle analyzer (WPA) is a technique which complements the spectrometric oil analysis by providing a measure of metallic iron, the form of iron most often associated with abnormal wear condition. The WPA is an

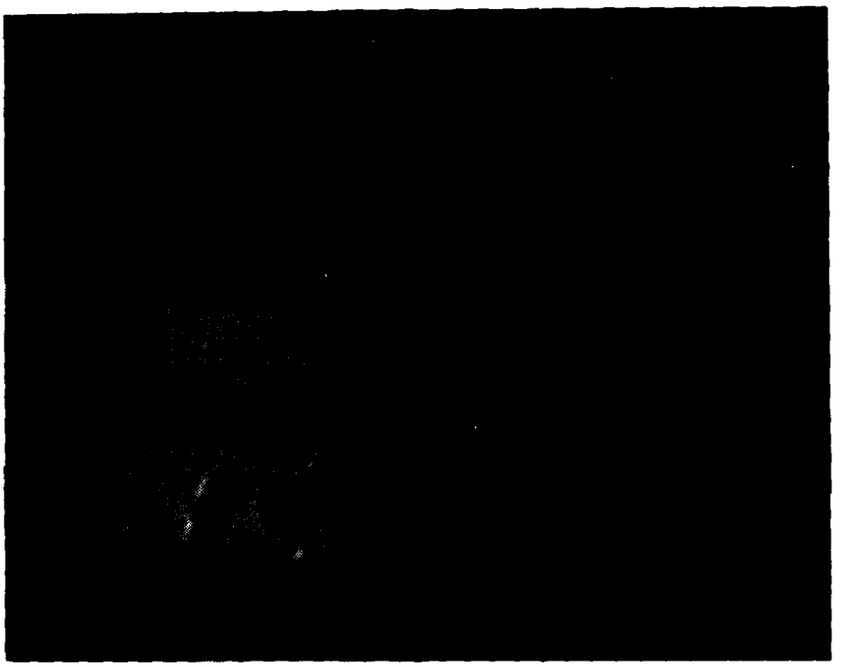


Figure 83. Wear Particle Analyzer

easy to use portable instrument for measuring the amount of ferromagnetic particles in fluids such as metallic iron or steel wear particles in a sample of used lubricating oil. It does not detect non-magnetic metals such as Cu, Ag, Sn, Pb or non-magnetic forms of iron such as ferric oxide and other corrosion products.

The WPA captures the particles in a filter consisting of a magnetic bed of ferromagnetic fibers using the principle of High Gradient Magnetic Separation (HGMS). This type of separation makes use of the very strong magnetic attractive forces, due to the high field gradients, around a magnetized ferromagnetic fiber. The amount of iron captured is determined from the increase in magnetic flux which is converted into ppm of iron by calibration with a 50 ppm standard. Even though the HGMS filter is efficient in capturing magnetic particles, it allows the fluid to pass readily through the filter.

Particle size distribution can be obtained by making use of the capturing efficiency in HGMS. For example, to capture the majority of the magnetic particles, the filter containing the finest fibers (grade 0000) should be used. However, if only larger particles are desired, a coarser grade filter should be used. Captured particles can be recovered for microscopic examination by removing the filter from the analyzer head and flushing with solvent in the reverse direction.

c. Apparatus and Procedure

A schematic of the Wear Particle Analyzer is shown in Figure 84. The sample is poured into the one ounce funnel. Sufficient vacuum is applied to allow the sample to flow through the ferromagnetic fiber which is placed in a brass tube. The tube is positioned in a way such that the fiber is aligned with the magnet N-S and the flux sensor.

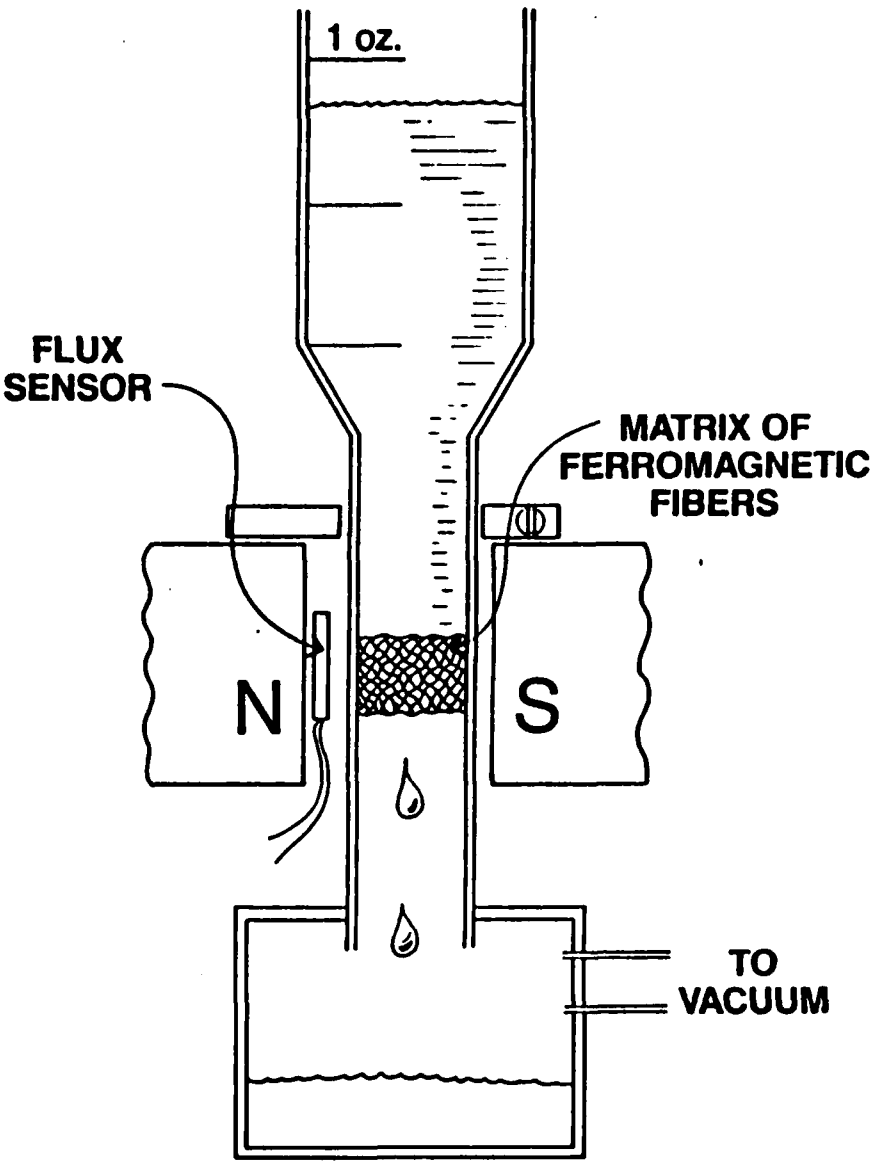


Figure 84. Schematic of the Wear Particle Analyzer

The capturing efficiency in HGMS decreases for particles whose diameter is less than half the fiber size.[30] The filters used in the WPA contain compressed steel wool which is available in several grades. Filter tubes provided with the WPA kit contain the following grades:

Grade	Nominal fiber size (micron)
0000	15-25
0	41-58
2	102-152

The pore size of the opening between fibers is determined by the compressive force used to pack the tubes. Larger openings are produced when a lower compressive force (3.5 lbs) is used. This type of filter is useful for fluids with high viscosity or when large sizes of non-magnetic debris is present. On the other hand higher compressive force (20 lbs) produces smaller openings. The currently used compressive force of 20 lbs is suitable for a viscosity of 2.2 cSt and the 3.5 lbs is suitable for a viscosity of 22 cSt for filters containing grade 0000 steel wool. Therefore, the magnetic filter or Firon will be referred to as "fiber grade:compressive force in lb". So the fiber grade 0000 with compressive force of 20 lb is termed as 0000:20.

d. Results and Discussion

(1) Effect of Particle Size and Filter Fiber Size

The efficiency of filters using various particle sizes of Fe powders and pin-on-disk used oil samples were studied and the results are shown in Table 47. This Table shows the filter type used, the stock concentration of the sample, the WPA results and the concentration of Fe in the filtrate. Except for the pin-on-disk sample (POD), the filtrates of all samples contained less than 2% Fe. The low Fe concentration indicates that

TABLE 47

EFFICIENCY OF FILTERS USING VARIOUS PARTICLE SIZES OF FE POWDERS

Size (um)	Fe Conc. (ppm)	WPA (ppm)[a]	Filter Type	Filtrate Fe Conc.(ppm)[c]
0-5	39	8 ± 1	0000:20	0.5 ± 0.1
0-5	39	17 ± 0	0000:3.5	0.4 ± 0.1
0-5	39	11 ± 1	0:20	0.4 ± 0.0
0-5	39	18 ± 2	2:20	0.5 ± 0.1
5-10	44	12 ± 1	0000:20	0.6 ± 0.2
5-10	44	25 ± 1	0000:3.5	0.5 ± 0.1
5-10	44	17 ± 0	0:20	0.5 ± 0.1
5-10	44	20 ± 2	2:20	0.3 ± 0.1
10-20	45	8 ± 1	0000:20	0.5 ± 0
10-20	45	31 ± 6	0000:3.5	0.6 ± 0
10-20	45	11 ± 1	0:20	0.9 ± 0
10-20	45	20 ± 2	2:20	0.6 ± 0
325 mesh	85	39 ± 5	0000:20	1.3 ± 0.1
325 mesh	85	48 ± 6	0000:3.5	1.6 ± 0.1
325 mesh	85	43 ± 3	0:20	1.3 ± 0
325 mesh	85	46 ± 3	2:20	1.4 ± 0.1
POD[b]	140	77 ± 2	0000:20	52.8 ± 0.3
POD	140	62 ± 4	0000:3.5	52.3 ± 0.7
POD	140	55 ± 8	0:20	54.4 ± 0
POD	140	40 ± 1	2:20	88.4 ± 0.8

[a] WPA Concentration is the average of two runs
[b] POD Wear metal generated from pin-on-disk wear test
[c] ADM concentrations

the filters' capturing efficiency of ferromagnetic debris is better than 98%. However, the same filters exhibited much lower efficiency for the POD sample with less than 63% of Fe being captured. Table 47 also indicates that the WPA results are not accurate and do not correspond to the actual values of Fe being captured. For the 0-5 micron sample, the WPA results were less than 50%.

The preliminary results shown in Table 47 indicate that for metal powder samples, as the particle size increases in the order of

$$0\text{-}5 < 5\text{-}10 < 10\text{-}20 \text{ micron},$$

the WPA readout does not significantly change for any size filter. Also, for any particle size i.e. 0-5 micron, the WPA readout does not appreciably increase with a decrease in the fiber size of the filter. However, for the pin-on-disk sample, the WPA readout increased in the order of decreasing filter fiber size as follows:

$$2{:}20 > 0{:}20 > 0000{:}20$$

(steelwool grade : compressive force, lb)

with the 0000:20 (15-25 micron) giving the highest WPA reading

(2) Effect of Sample Volume

Sample volume effect on the WPA readout was also studied using two oil samples containing Fe wear debris. Tables 48 and 49 show the WPA results for various sample volumes and different WPA filters. The results of the pin-on-disk sample reveal that WPA readouts were optimum for smaller sample volumes (less than 2.5 ml). Similar results were obtained for the TF39 used engine oil sample where the optimum sample volume for all filters considered was less than 1.5 ml (Table 49). The pin-on-disk sample was analyzed without dilution. Results in Table 50 indicate that the 0000:20 filter gave the highest readout and gave the best repeatability for volumes

TABLE 48

EFFECT OF SAMPLE VOLUME ON WPA READOUT USING PIN-ON-DISK WEAR SAMPLE AND VARIOUS SIZE WPA FILTERS

Sample Vol[a] (ml)	Filter[b] 0000:20	Sample Vol (ml)	Filter 0:20	Sample Vol (ml)	Filter 2:20
5.7	15.0	6.2	15.4	5.8	16.9
2.5	21.0	2.4	19.0	2.4	21.8
1.7	23.2	1.5	15.0	1.4	23.3

a. 1:2 pin-on-disk diluted 1:1 with kerosene
b. WPA results in ppm

TABLE 49

EFFECT OF SAMPLE VOLUME ON WPA READOUT USING USED ENGINE OIL SAMPLE AND VARIOUS SIZE WPA FILTERS

Sample Vol[a] (ml)	Filter[b] 0000:20	Sample Vol (ml)	Filter 0:20	Sample Vol (ml)	Filter 2:20
40.5	2.4	47.4	1.7	45.2	1.1
8.0	2.4	8.8	2.4	8.7	1.4
4.9	2.9	4.8	1.8	6.3	1.6
1.5	6.6	1.5	3.9	1.6	3.2

a. TF39 used engine oil, 50 ppm Fe, diluted 1:1 with hexane
b. WPA results in ppm

TABLE 50

REPEATABILITY OF THE WEAR PARTICLE ANALYZER FOR DIFFERENT SAMPLE VOLUMES AND FILTERS USING PIN-ON-DISK OIL SAMPLE

	Filter Type			
Sample Size[a] (ml)	0000:20 (ppm)	0000:35 (ppm)	0:20 (ppm)	2:20 (ppm)
2.0	22.7 $\pm$ 0.7[b]	20.7 $\pm$ 0.9	9.9 $\pm$ 1.3	9.8 $\pm$ 0.6
1.5	20.5 $\pm$ 1.2	16.5 $\pm$ 1.3	10.6 $\pm$ 0.3	8.8 $\pm$ 0.7
1.0	20.3 $\pm$ 4.1	18.8 $\pm$ 1.1	12.4 $\pm$ 2.0	8.4 $\pm$ 1.3

a. Pin-on-disk sample contained 53 ppm Fe
b. All samples were run 3 times

above 1.0 ml.

(3) Analysis of Used Oils

Fifteen samples from different operational engines, previously analyzed for metallic iron using a solvent extraction technique[31] were analyzed by the WPA. The results of these two techniques are reported in Table 51. In general, the two techniques gave comparable results. However, for samples where the WPA gave lower results than the solvent extraction method for total metal, one should consider that smaller particles (approximately less than one micron) are not captured by the WPA filter. Therefore, if the WPA results were compared with the solvent extraction results for iron particles greater than 0.6 micron, a better comparison will be realized for sample F. The WPA gave higher results for samples H and P containing particles greater than 0.6 micron.

Comparative results of atomic emission (AE), acid dissolution method (ADM) and WPA for Fe in Engine Simulator test samples are shown in Table 52. Even though the WPA results were much lower than either AE or AD, they exhibited similar trends in the increase of iron concentration in the 0 to 100 hour samples.

(4) Sample Introduction System

The sample introduction system of the WPA was modified in order to provide comparative data with the manufacturer's FIRON filters. Samples were filtered through a 13 mm diameter 0.22 micron Millipore filter. The filter containing the captured debris was then compressed and inserted in a brass tube similar to the FIRON tube.

The results of the WPA readouts for the modified filter and the 0000:20 FIRON filter are reported in Table 53. The data clearly shows that the Millipore filter gave much higher WPA results than the FIRON 0000:20

TABLE 51

COMPARATIVE RESULTS USING THE WEAR PARTICLE ANALYZER AND A SOLVENT EXTRACTION METHOD FOR IRON IN USED TURBINE JET ENGINE OILS

Sample[a]	WPA Fe Conc.[b] (ppm)	Solvent Extraction[d] Total Fe (ppm)	Solvent Extraction[d] >0.6μm (ppm)
H-1	2.4 ± 0.2	2.3	1.0
H-23	23.8 ± 0.8	28.8	15.7
H-47	3.4 ± 0.2	3.7	0.9
H-49	5.5 ± 0.2	7.9	5.8
P-7	3.0 ± 0.6	1.1	-
P-48	2.7 ± 0.5	4.5	-
P-58	20.3 ± 1.2	19.0	11.0
P-61	3.7 ± 0.4	7.2	-
P-83	2.7 ± 0.3	5.7	-
P-97	4.3 ± 0.7	7.7	-
R-5	4.3 ± 0.5[c]	1.4	-
R-445	5.5 ± 0.5	1.3	-
F-19	6.5 ± 0.7	19.0	12.0
F-22	4.9 ± 0.2[c]	14.1	6.5
F-39	17.7 ± 0.6[c]	22.5	20.0

a. Sample size used was 2.0 ml through 0000:20 filter type
b. Each sample was run 3 times
c. Sample was run 2 times
d. Method reported by Kauffman, Saba, Rhine and Eisentraut, ASLE Trans., 28, 400 (1985)

TABLE 52

COMPARATIVE RESULTS OF AE, ADM AND WPA FOR FE IN ENGINE SIMULATOR TEST SAMPLES

Sample (hours)	AE	ADM	WPA[c]
0[a]	0	0	0
0[b]	0	0	0
5	22.5	35.9	10 ± 1
10	17.5	12.0	6 ± 1
15	19.1	14.3	5 ± 1
20	23.4	22.0	8 ± 1
25	37.2	44.6	9 ± 1
30	39.6	32.8	10 ± 1
35	51.3	33.7	14 ± 3
40	57.6	42.5	11 ± 1
45	64.2	48.0	11 ± 1
50	64.5	54.3	13 ± 1
55	66.5	52.4	12 ± 0
60	66.2	72.1	14 ± 1
65	81.0	65.1	12 ± 1
70	89.8	74.8	17 ± 1
75	84.2	72.7	15 ± 1
80	92.7	87.7	18 ± 1
85	94.1	85.5	17 ± 3
90	111.0	90.7	19 ± 1
95	115.4	109.4	18 ± 1
100	127.4	111.1	18 ± 2

[a] Can
[b] Sump
[c] 1/2 ml sample was used, each sample run twice through 0:20 filter

TABLE 53

RESULTS OF MODIFIED SAMPLE INTRODUCTION SYSTEM IN THE WPA

Fe Sample	Stock Conc. (ppm)	Sample Size (ml)	Modified[a] Filter (ppm)	0000/20 Filter (ppm)
325 mesh	340	4.0	202 ± 14[b]	91 ± 28
325 mesh	340	1.0	180	157 ± 11
325 mesh	85	1.0	52 ± 7[c]	39 ± 5
POD #1[d]	35 ppm	1.0	18	20

a. Modified filter = 13 mm 0.22 µm millipore filter
b. Result of 4 runs
c. Result of 9 runs
d. POD sample was diluted 4 times

filter for the 325 mesh Fe powder. This is due to the higher particle capturing efficiency of the 0.22 micron Millipore filter. However, difficulty was experienced when pin-on-disk wear test sample and TF39 used engine oil sample were filtered through the Millipore filter. The filter was clogged and therefore, samples could not be filtered effectively. Filtering smaller volumes of these samples did not clog the filter but decreased the WPA sensitivity. The method has proven to be efficient provided that a different method of filtration is found which will allow all types of oil samples to be filtered without difficulty.

e. Conclusion

The Wear Particle Analyzer is a technique sensitive to metallic iron, the form of iron most often associated with abnormal wear in lubrication systems. Even though the instrument suffers from a lack of accuracy in the analysis of metal powders, it responds to changes in concentration of ferromagnetic wear debris. The WPA results for used turbine jet engine oils were comparable to those obtained using a solvent extraction technique.

f. Future Effort

The manufacturer has supplied only one standard (50 ppm) to calibrate the instrument only at the highest range. Other standards are needed to calibrate the instrument at lower ranges. Therefore, new standards for different ranges of the instrument are being developed and a calibration curve will be established for the 1, 30 and 100 ppm ranges.

Tribometrics will be providing a newly developed addition to the electronics of the WPA that should greatly improve the zero stability with changes in ambient temperature. Six samples were shipped to Tribometrics to be analyzed on their WPA. Their findings will be reported along with ours in a future report.

Plans are being made to manufacture filters with smaller size fibers in order to increase the efficiency of capturing the smaller sizes of metallic iron wear debris. Included also in future plans is the determination of the particle size detection limit of the instrument.

5. MICROFILTRATION

a. Introduction

The impact of using fine filtration (3-10 μm) in aircraft lubrication systems is of primary concern. Present Air Force wear metal monitoring techniques will be evaluated with respect to their capability in analyzing particles less than 3-10 μm. Another factor of concern is the effect of fine filtration on the threshold values already established in SOAP. The Air Force relies on these values as guidelines to determine the health of their operating engines. Any change within the lubrication system such as filtration might have an impact on the analytical results and the present threshold values and guidelines may have to be revised.

b. Background

Within the last decade ultrafine filtration has been introduced into several new engines by retrofitting micron size filters into these engines. The primary objective is to extend component life by reducing the amount and size of wear debris in the lubrication system. References obtained to date[32-47] are limited to the development, use and advantages of microfiltration in the oil system. The development of the first known 3-micron absolute oil filter for the development of the T53 gas turbine lubrication system is described by Lynch and Cooper.[46] The filter ran for approximately 300 hours without clogging. Its use provided cleaner lubrication environment and decreased the frequency of filter inspection and oil drains. As a result one of the major benefits immediately realized was

the prevention of abrasive wear caused by 1-5 micron particles. The result of this project provided the background data for the more extensive testing program at Fort Rucker which was established in 1978.[37] The Army established a research and development effort to develop an advanced oil debris discrimination and filtration system for the Bell UH-1/AH-1 helicopters. Thirty-eight UH-1's were fitted with 3 micron (β_3>200) filters on the engine and transmission lubrication systems in conjunction with full-flow debris monitoring chip detectors. A flight test program was run with more than 70,000 flight hours logged. The major results of this program were as follows:

(1) Engine oil and transmission oil change intervals increased by tenfold and threefold, respectively.

(2) The average filter life was over 1000 hours.

(3) Reduced seal wear and very clean oil wetted component conditions were realized.

(4) "Nuisance" chip lights were greatly reduced.

Wansong[47] deals with the maintainability benefits of 3 micron filtration on the General Electric T700 engine. The T700 engine powers the H-60 series and AH-64 Helicopters. This is the first production engine that incorporates 3 micron lube oil filters from its inception.

Since analysis for wear debris has proven to be a valuable diagnostic tool of the health of an engine, the use of fine filtration would raise an important question as to the effectiveness of spectrometric oil analysis due to the filter's "removal" of debris detectable by the spectrometer. Therefore, it is the requirement of this task to determine the impact of microfiltration on Spectrometric Oil Analysis Program (SOAP). For this effort a microfiltration test rig was developed in order to simulate wear debris generation and the engine filtration system.

c. Apparatus

(1) Test Rig Assembly

The microfiltration test rig was built on a rolling bench assembly. The bench was built from steel bench legs and a wooden top made of solid, kiln-dried wood core surface with high density tempered pressed wood. Several holes were made in the bench top to accommodate a stainless steel 5-gallon oil reservoir, 3/4" and 1/4" tubings. The high pressure gear pump with constant speed drive motor and the scavenger pump were installed at the lower level of the rolling bench assembly (Figure 85). The schematic in Figure 86 shows the configuration (not exact dimensions) of the actual construction of the rig including valves, flowmeters, pumps, thermocouples, pressure transducers, test filter and reservoirs. The Falex wear test device will be considered as one possible technique for generating wear metals. It was used to generate different types of wear metals using the pin-on-disk, gear and four ball wear test configurations.

(2) Filter Element and Filter Housing

Filter element and housing are made by the Aircraft Porous Media (APM). The filter element is made of Ultipor resin impregnated organic and inorganic fibers and is capable of withstanding temperatures in the range of -65°F to +350°F. The maximum clean pressure drop at the rated flow of 4 GPM is 4 psi at 100°F. The minimum element collapse differential pressure is 100 psi. Minimum dirt capacity at rated flow at 100°F is still to be determined using AC fine test contaminant to 14 psid when tested per MIL-F-8815. The filter element is a non-cleanable depth type filter whose effective filtration area is approximately 2.3 square feet. It is constructed of multi-layer graded filter media (4 layers) and rated as 3 micron absolute β_3>200, which allows one particle, >3 micron, for every 200 particles to pass

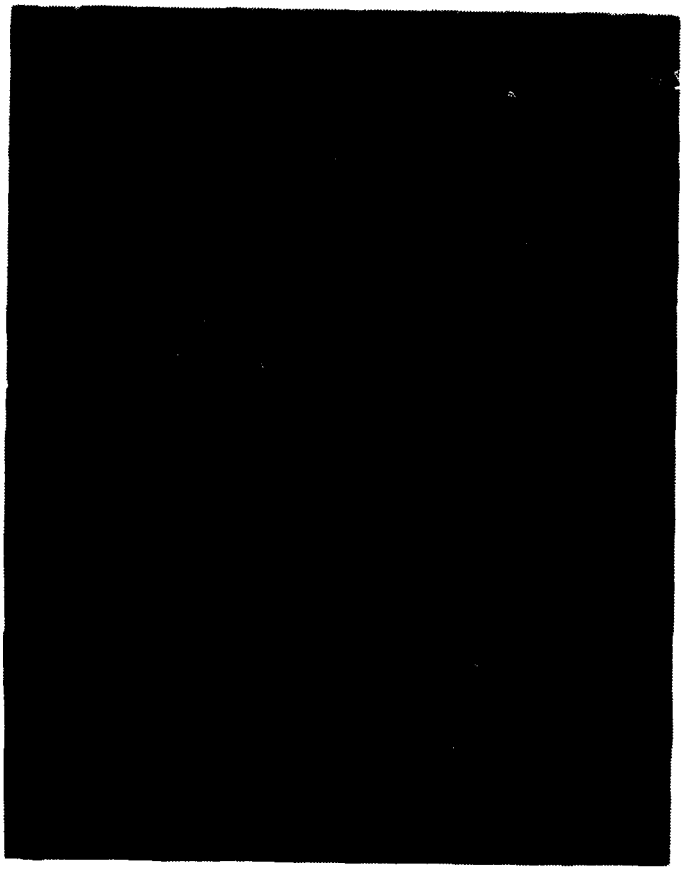


Figure 85. Microfiltration Test Rig Assembly

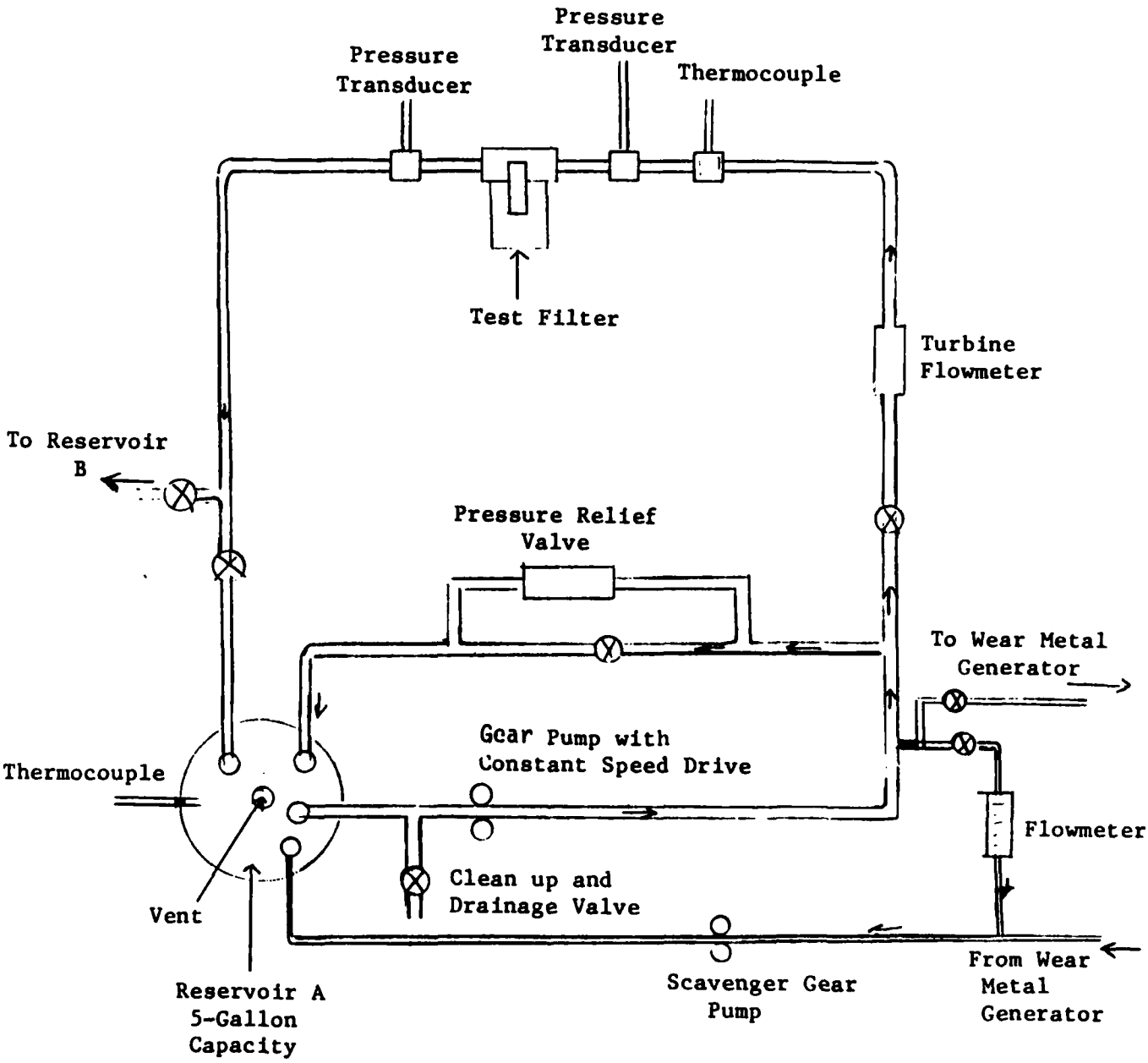


Figure 86. Schematic of the Microfiltration Test Rig

through the filter. The filter element is protected by an inner wire mesh support and a mesh outer wire. The element is optimized to meet system requirements of dirt capacity, pressure drop, fluid compatibility and wear particle removal. The protective wire mesh supports were removed from one of the filters and the filter layers were separated and examined using an optical microscope and SEM. The layers are constructed from fibrous type material integrally bonded to form a porous material. Microscopic examination revealed that the porosities of the layers were different. Layers 1 and 3 were identical, made of glass type fibers (Figure 87) and have much larger pore sizes than layers 2 and 4. The mesh sizes of layers 1 and 3 seem to be approximately between 35 and 40 micron (optical microscope). Layers 2 and 4 were also identical but the mesh size could not readily be determined from SEM (Figure 88).

Filter housing parts are made of anodized aluminum and its rated pressure is 60 psi, proof pressure of 90 psi and a burst pressure of 150 psi. The temperature range, rate flow capacity and clean pressure drop are the same as the filter element specifications. The relief valve cracking minimum and maximum (full flow) differential pressures are 14.0 psi and 18.0 psi, respectively, at 4 GPM of oil port to port.

(3) Test Rig Lubricant Flow

In order to simulate the lubricant flow in a real jet engine lubrication system we obtained information about the lubricant flow in several GE and Pratt and Whitney engines used by the Air Force. In general, GE engines operate at about 10 GPM while Pratt and Whitney's operate at least at twice this flow. Plans are to operate the test rig at lubricant flow of approximately 20 GPM or less.

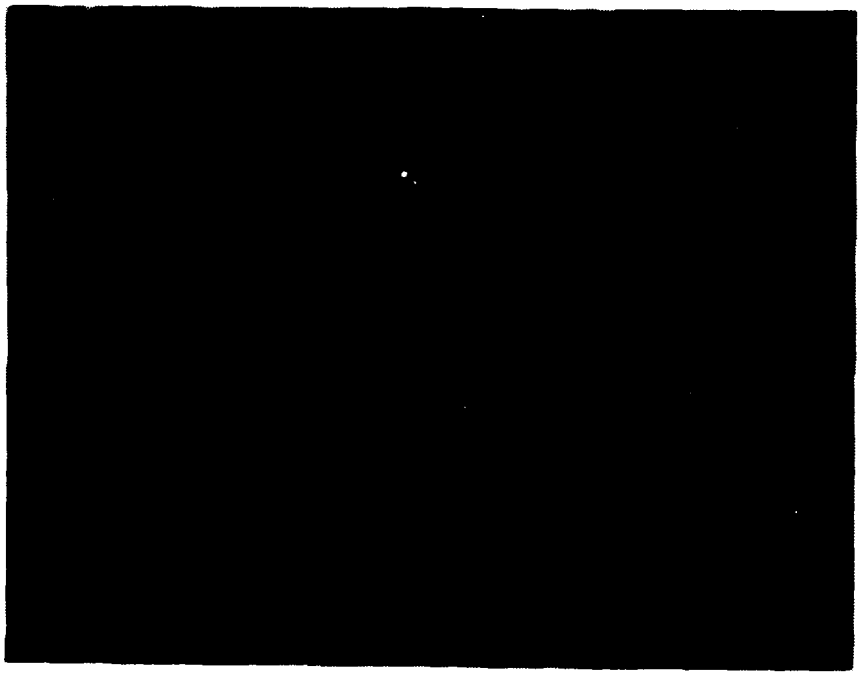


Figure 87. Photomicrograph of the Small Pore Size APM Graded Filter Medium (1000X)

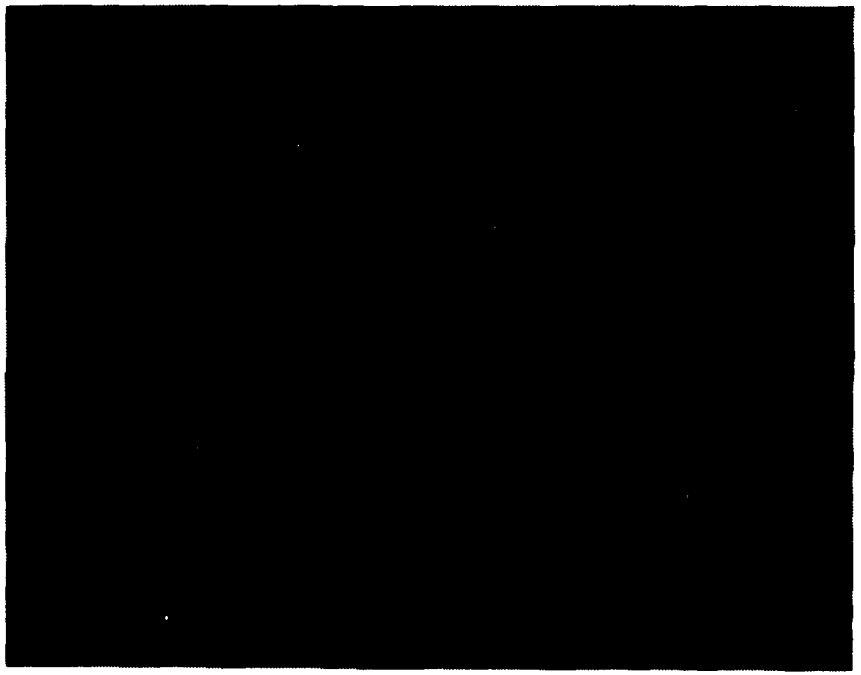


Figure 88. Photomicrograph of the Large Pore Size APM Graded Filter Medium (60X)

(4) Wear Metal Generator

The Falex wear test machine is considered as one possible technique for generating wear metals. Wear metals were generated using the pin-on-disk, gear and four ball wear test configurations. The particle size distributions were determined for the above wear debris from the three different tests using a filtration technique. Aliquots of the oil sample containing the wear debris were filtered through 0.4, 1-, 2-, 3-, 5-, 8-, 12- μm Nuclepore membranes. A 2-g aliquot of the oil sample was diluted with pentane prior to filtration. A total of 10 grams were filtered for each filter pore size. Pentane was allowed to evaporate and the remaining oil (filtrate) was analyzed for total Fe content using AA and the acid dissolution method. The particle size distribution of particles generated from the above wear tests are graphically shown in Figure 89. The bar graph shows that among the three types of wear the pin-on-disk wear test produced the smallest size particles while the gear test produced the largest particles. Initial filtration using the microfiltration test rig will be performed using the wear debris generated from the pin-on-disk wear test.

d. Efficiency of the APM Filter Element

Evaluation of the APM 3-micron absolute filter element was initially performed by determining its efficiency of capturing wear debris. The filter element removed from its protective wire mesh supports was cut into 47-mm diameter filters. The small filters were fitted in a Millipore stainless screen glass filter holder for vacuum filtration of oil samples containing metallic wear debris. Samples from the pin-on-disk (POD) wear tests containing various amounts of brass and steel wear particles were allowed to pass once through the APM filter. The results of this experiment reveal that the efficiency of filtration for brass ranged from 53.5 to 83.7 percent, and

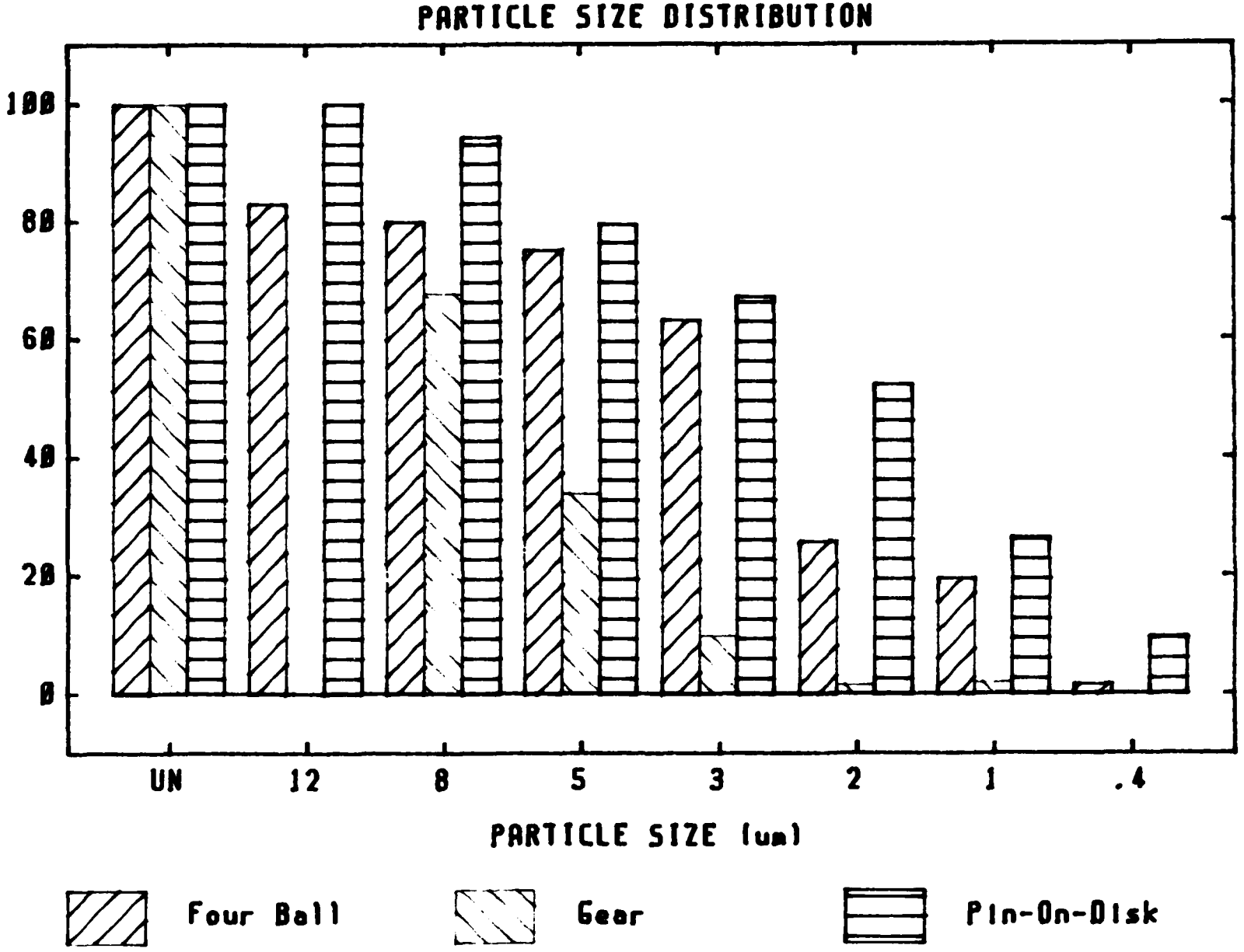


Figure 89. Particle size Distribution of Fe Particles Generated from Four Ball, Gear and Pin-On-Disk Wear Tests.

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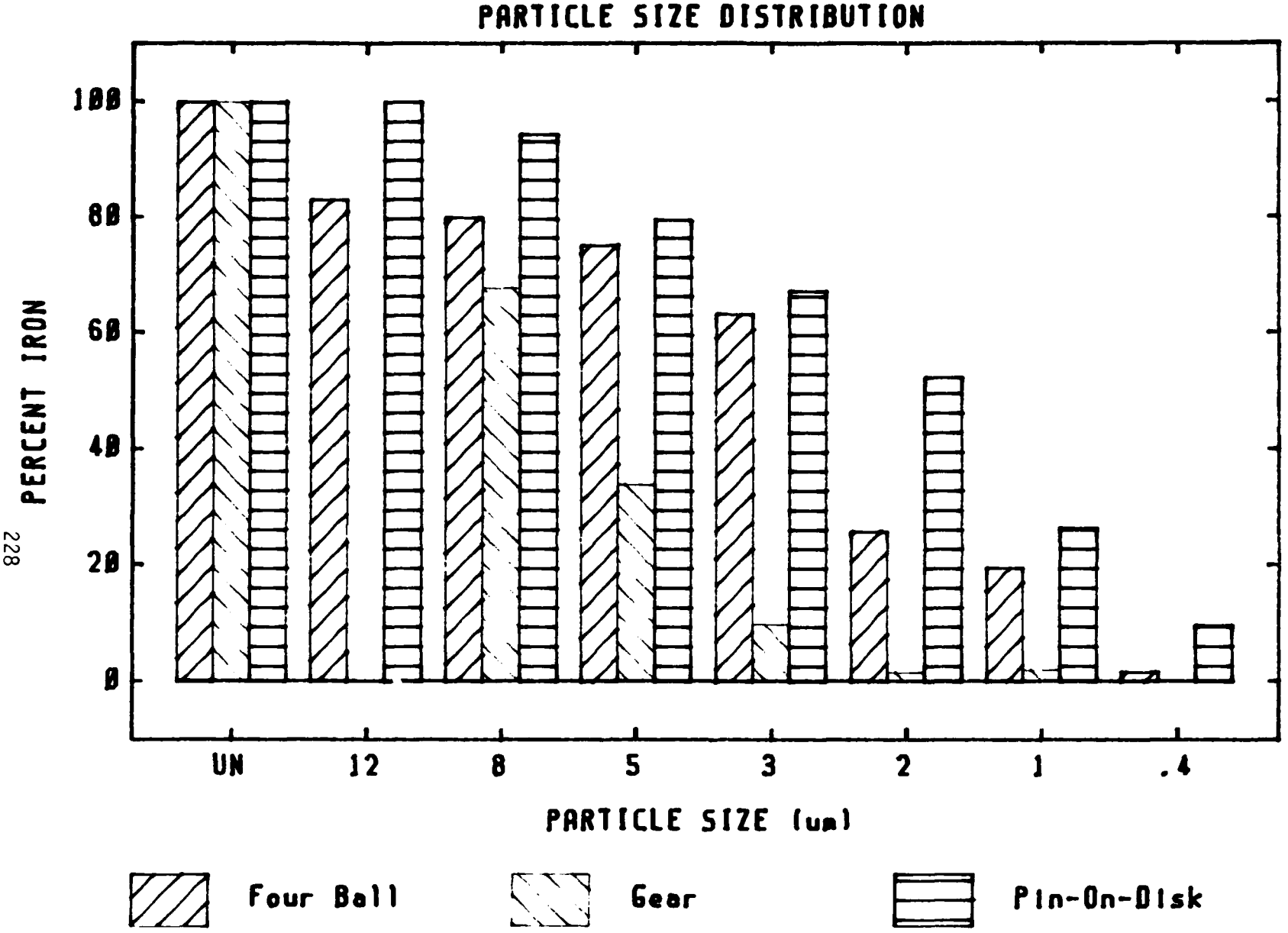


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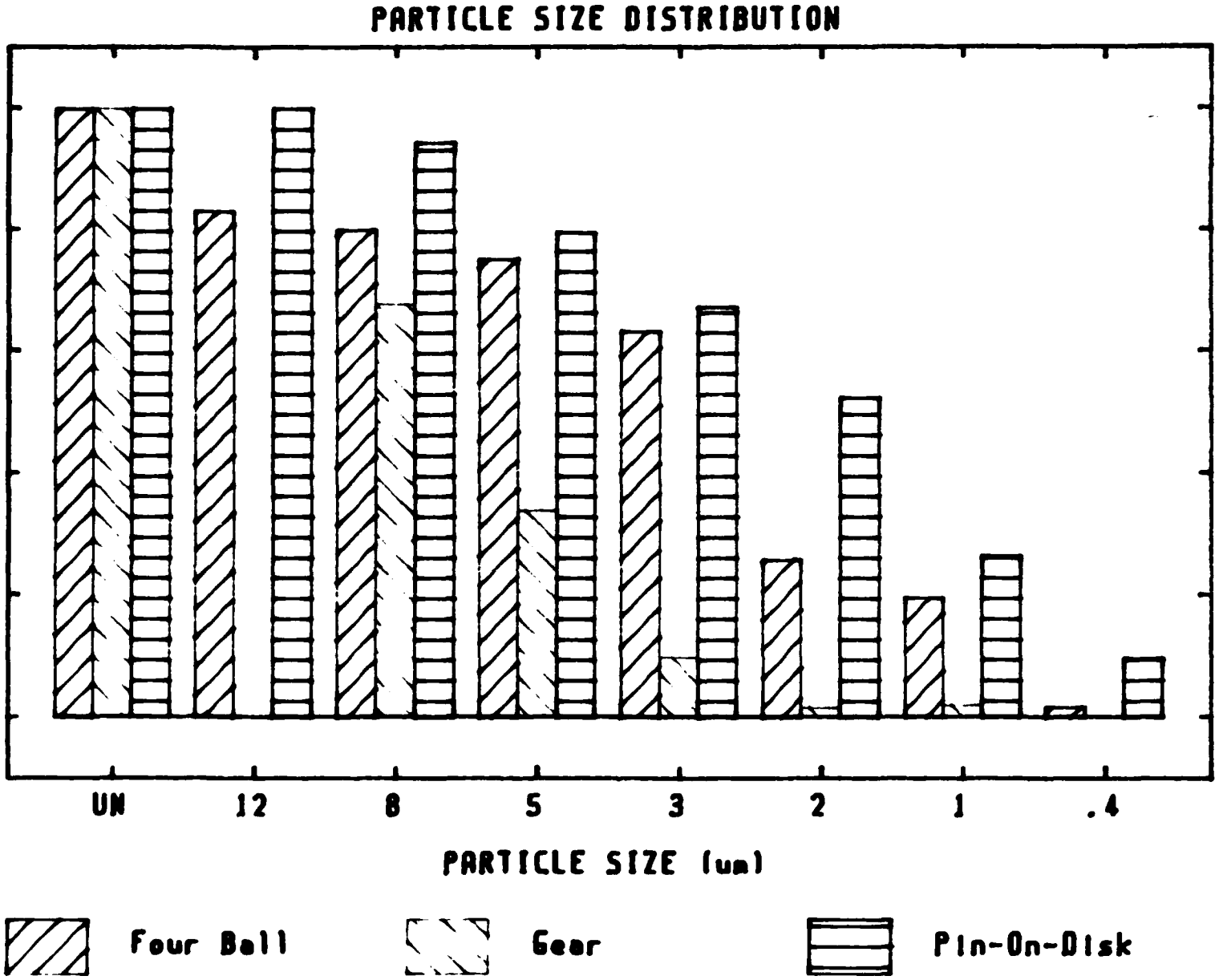


Figure 89. Particle size Distribution of Fe Particles Generated from Four Ball, Gear and Pin-On-Disk Wear Tests.

zero for Fe (Table 54). Microscopic examination of these samples revealed that the brass particles were much larger than the Fe particles and the Fe particles were less than 2 micron in size. The efficiency of the APM filter element was determined for a TF 39 used oil sample. A single pass of the sample through the filter yielded 31.7% capturing efficiency (Table 54).

The efficiency of the APM filter was also determined as a function of the number of passes of a POD sample through the filter. After the first pass, the filter element was replaced by a new one and the filtrate was passed through the new filter. The filter from the second pass was also replaced by a new one and the filtrate from the second pass was filtered through the third filter. The stock solution and aliquots of the filtrates from the first, second and third pass filtrations were analyzed for Fe using atomic emission spectrometry. The analytical data revealed that the capturing efficiency of the filter for the first, second and third pass were 27, 52 and 66%, respectively. Ferrographic micrographs of the stock solution and filtrates revealed that the quantity and size of wear debris decreased with each filtration pass (Figure 90).

Work is continuing towards the determination of the APM capturing efficiency as a function of the number of passes through the filter for turbine engine used oil samples and other wear debris as generated from the wear test machine.

e. Conclusion

A 5-gallon capacity microfiltration test rig was constructed in order to simulate fine filtration in turbine engine lubrication systems. The test rig allows the determination of the filtration efficiency of different micron size filters at ambient or elevated temperatures (175^{o}F). The gear pump with a constant drive allows the oil to circulate at 20 GPM through the test

TABLE 54

EFFICIENCY OF APM FILTERS FOR SINGLE PASS OF PIN-ON DISK SAMPLES

Sample[a]		Fe Conc.[b] (ppm)	Cu Conc.[b] (ppm)	% Efficiency
POD 20	Stock	7.0	177.2	
POD 20	Filtrate	6.4	82.4	53.5
POD 13	Stock	8.4	174.8	
POD 13	Filtrate	10.7	79.4	54.6
POD 19	Stock	5.8	144.8	
POD 19	Filtrate	5.3	24.3	83.2
POD 22	Stock	6.1	56.4	
POD 22	Filtrate	5.3	14.7	73.4
POD 14	Stock	4.0	43.6	
POD 14	Filtrate	5.2	7.1	83.7
POD 23	Stock	13.3	106.8	
POD 23	Filtrate	12.6	45.7	57.2
TF 39	Stock	75.9	-	
TF 39	Filtrate	51.8	-	31.7

a. POD = Pin-on-disk sample with brass pin on 52100 steel disk with 30-150 lb load and 1 m/sec speed except POD 23 which was run at 0.5 m/sec
b. Determined by acid dissolution method

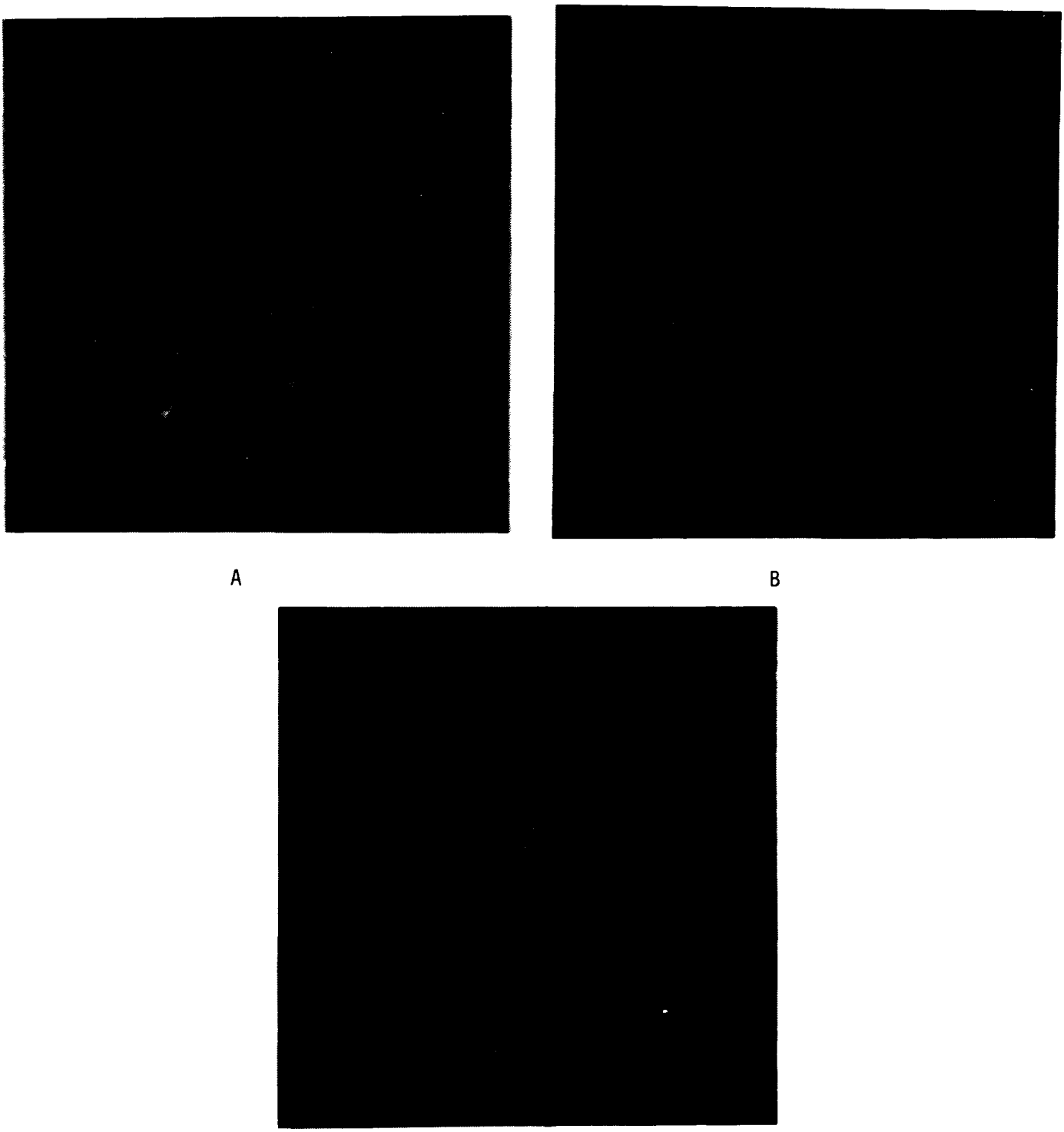


Figure 90. Ferrographic Micrographs of the Pin-on-Disk Wear Debris; (A) Stock Solution; (B) After the First Pass Through the APM Filter; (C) After the Third Pass Through the APM Filter

filter and less than 20 GPM with the aid of bypass valves. The Falex wear test machine was found to generate wear particles in an acceptable range (less than 20 micron) using the pin-on-disk wear test configuration.

f. Future Effort

A three-micron filter was obtained from another supplier UCC Amfac Fluid Power. This filter will be evaluated for its capturing efficiency. Plans are being made to determine the efficiency of filtration of larger pore size filter media (5,10,25, and 45 micron absolute). The results from these experiments will be compared with those determined for the 3 micron filter. Comparative results will also be established for various size filters using the microfiltration test rig. The test rig initial operation and testing will be performed during the second phase of this effort. Test results should reveal the impact of fine filtration on the Air Force Oil Analysis Program.

SECTION IV

INVESTIGATION OF LUBRICANT MONITORING TECHNIQUES

1. INTRODUCTION

The main purpose of this study was to investigate the electrochemical properties of degraded synthetic turbine lubricants and to relate these properties to specific chemical changes in the lubricant. To this end, the electrochemical properties of MIL-L-7808 lubricants and various laboratory formulations stressed in the Squires oxidative and confined heat tests at various temperatures were measured by several different methods. Also, an initial investigation into the specific degradation chemistry of these lubricants was made. Summarization of these efforts is as follows.

2. COMPLETE OIL BREAKDOWN RATE ANALYZER

The Complete Oil Breakdown Rate Analyzer (COBRA) is a portable electrochemical device that had been studied by the Air Force as a lubricant monitoring device and demonstrated success in identifying abnormally operating engines in aircraft.[2] In order to further evaluate this device, its electronic circuitry was analyzed, measurements of degraded lubricants were made, and some preliminary investigations into the specific degradation chemistry of the various lubricants made.

a. Electronic Circuitry Analysis

A COBRA instrument was submitted to University of Dayton Research Institute for analysis of its electronic circuitry. The objective of this analysis was to gain an understanding of how the COBRA functions in order to determine what physical property of the lubricant is being measured. An illurstration of the COBRA instrument is shown in Figure 91. With no sample present, a situation in which the electrodes are isolated from each other,

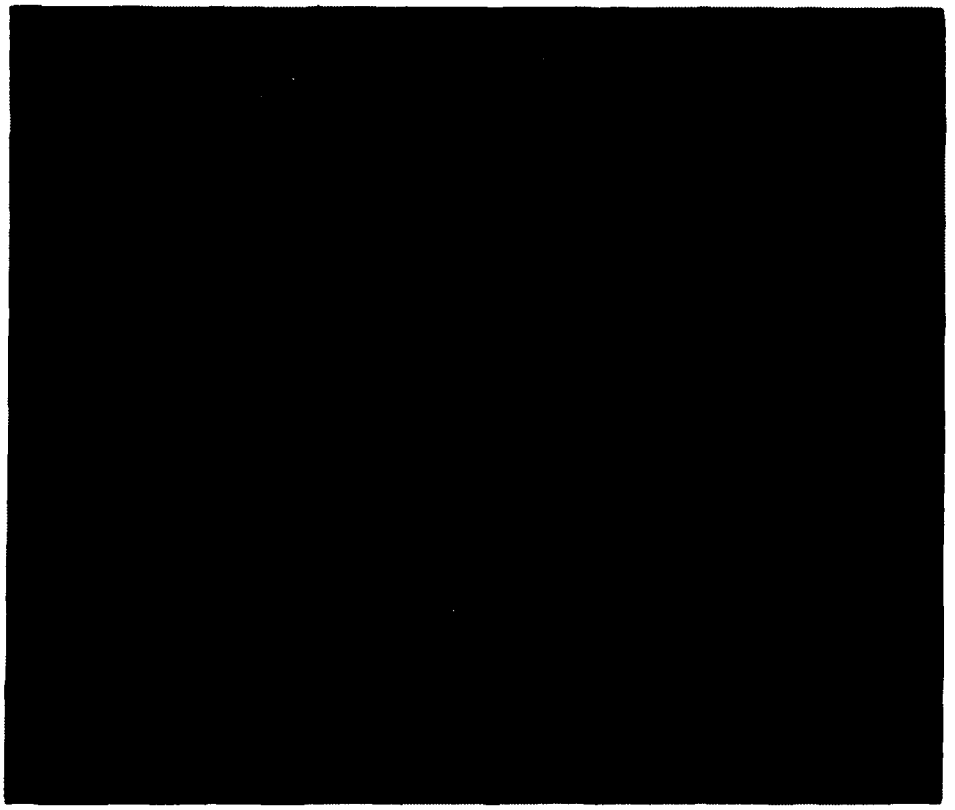


Figure 91. Complete Oil Breakdown Rate Analyzer (COBRA)

pushing the analysis button results in the application of a square wave with an amplitude of 0 to 0.4 volts. When a fluid is present between the electrodes that causes an off-scale (>200) reading (such as a badly degraded oil) the a.c. (square wave) voltage component becomes 0 and a d.c. voltage component becomes 0.3 volts. This d.c. offset is presumably due to voltage developing from the dissimiliar metals used in the electrodes (stainless steel and cadmium, top and bottom respectively). Any fluid present between the electrodes that gives a reading greater than 0 but less than off-scale causes a reduction in the square wave amplitude but an increase in the d.c. offset. The electrical equivalent of an oil between the electrodes would be a variable resistor (representing the buildup of conductive species in the oil) in series with a capacitor (representing the dielectric strength of the oil). It is interesting to note that application of resistance alone is not sufficient to obtain a COBRA reading. It appears also that the d.c. voltage offset is important in obtaining a COBRA reading as application of a voltage to the electrodes will also cause a reading. The importance of this voltage offset to the actual instrument reading is shown by the effect of changing the bottom electrode. The bottom electrode, which is cadmium, was replaced with a flat piece of metal that was grounded to the instrument. A standard degraded oil sample, that produces a COBRA reading of 83, was analyzed on this modified instrument using various metals for the bottom electrode and the results are summarized in Table 55.

TABLE 55

EFFECT OF ELECTRODE COMBINATIONS ON COBRA READINGS

Bottom Electrode Metal	COBRA Reading	Standard Reduction Potential E^{o}(volts)
Cadmium (Original Electrode)	83	-0.403
Cadmium	85	-0.403
Copper	10	+0.342
Stainless Steel	20	-0.447[a]
Nickel	40	-0.257
Zinc	95	-0.762
Magnesium	140	-2.372

a. $Fe^{+2} + 2e^{-} \text{---} > Fe^{o}$

As expected, the cadmium plate gave a reading very close to the original bottom electrode. Other metals gave COBRA readings that varied considerably and there appears to be rough correlation between the standard reduction potential E^{o} of the metal and the COBRA reading obtained, that is more negative E^{o} produced higher readings. It may be that the particular metal combination affects the previously discussed d.c. zero offset. It would appear then that the most important property of the oil sample is its resistance (or conductivity) which controls the extent to which the cell (capacitor) can charge, the final result being a change in the D.C. offset voltage to which the instrument is sensitive.

b. Analysis of Degraded Lubricants

COBRA readings have been recorded on the six MIL-L-7808 lubricants stressed in the Squires oxidative and confined heat tests at 175, 190 and 205°C. The plots of these COBRA readings vs. test hours are shown in Figures

92 to 97. It is apparent from the oxidative data (Figures 92 to 94) that four of the lubricants (O-79-16, O-79-17, O-79-20 and O-82-3) have similar rises in COBRA readings vs. test time while the other two lubricants (O-82-2 and O-82-14) show very rapid rises in COBRA reading. As the test temperature rises this relationship starts to breakdown probably due to large volatilization weight losses since this test does not use condensate return. The confined heat test data (Figures 95 to 97) show similar correlations except that the COBRA response of O-82-2 is more in line with the other four lubricants. In general the COBRA response vs. time (slope) of the confined heat test data is less than that of the the oxidative test data probably due to its limited oxygen availability and subsequent lesser formation of oxygenated degradation products. These trends would be meaningful only if all six lubricants degraded at the same rate. The fact that they do not degrade at equal rates emphasizes the importance of comparing COBRA values for different lubricants at points of equal deteriorative changes in the oils. Since the break point of the lubricant (where the physical properties rapidly deteriorate) is not easily calculable, due to the lack of a breakpoint for some oils and a limited number of sampling points, COBRA readings are compared at equal changes in total acid number (TAN) and viscosity. For the oxidative test, TAN changes of 1.5 and viscosity changes of 35% will be used while the confined heat test will use TAN changes of 4.0 and viscosity changes of 5%. The test hours required to reach these values had been calculated previously in preparation of the Arrhenius plots. The data for these comparisons are shown in Tables 56 and 57. The interpretation of these comparisons is somewhat dependent on which physical property is used. For the oxidative data (Table 56), examination of the COBRA readings for viscosity changes of 35% yield the same interpretation as that shown in

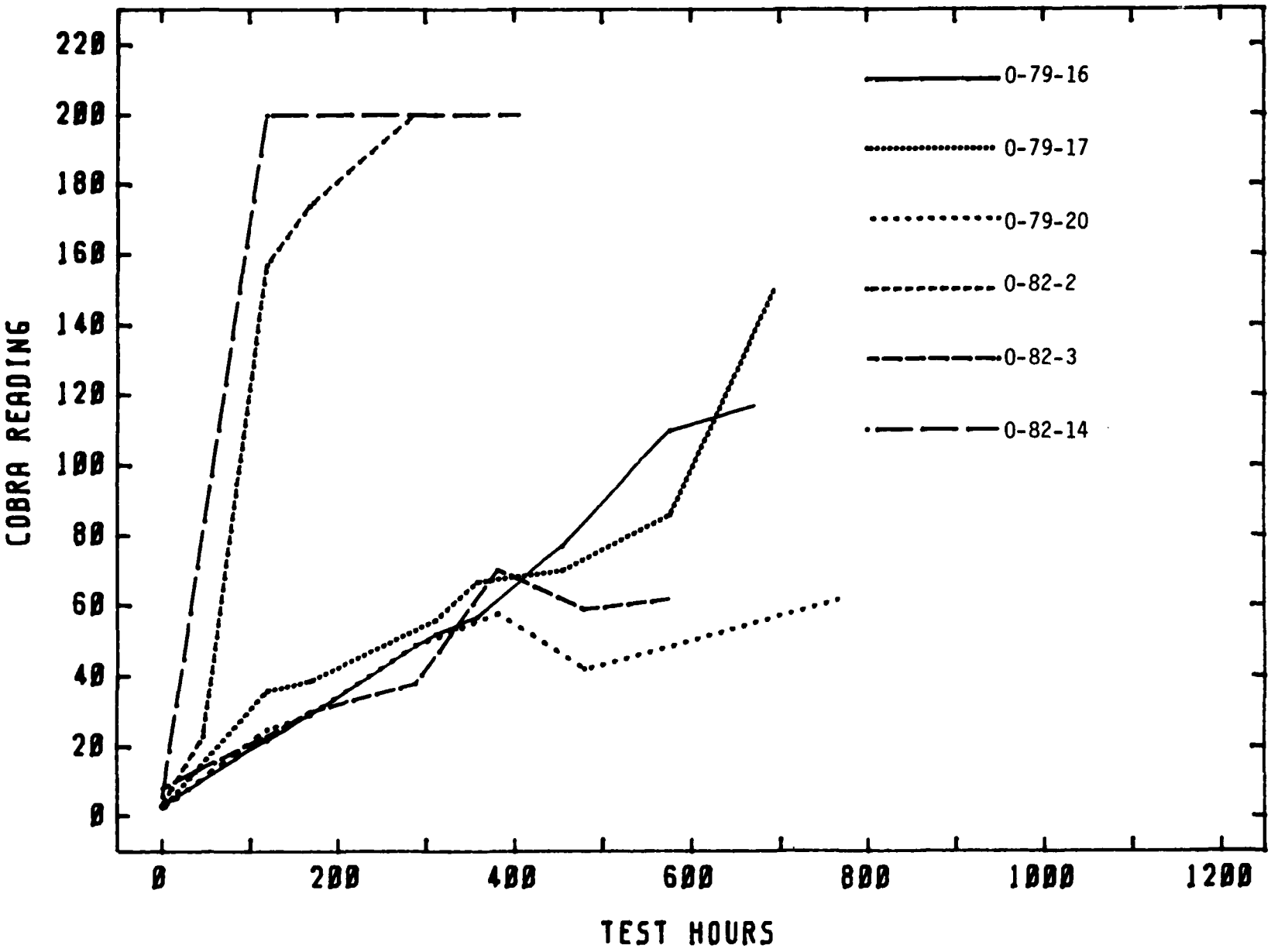


Figure 92. COBRA Readings of MIL-L-7808 Lubricants from the Squires Oxidative Test at 175°C

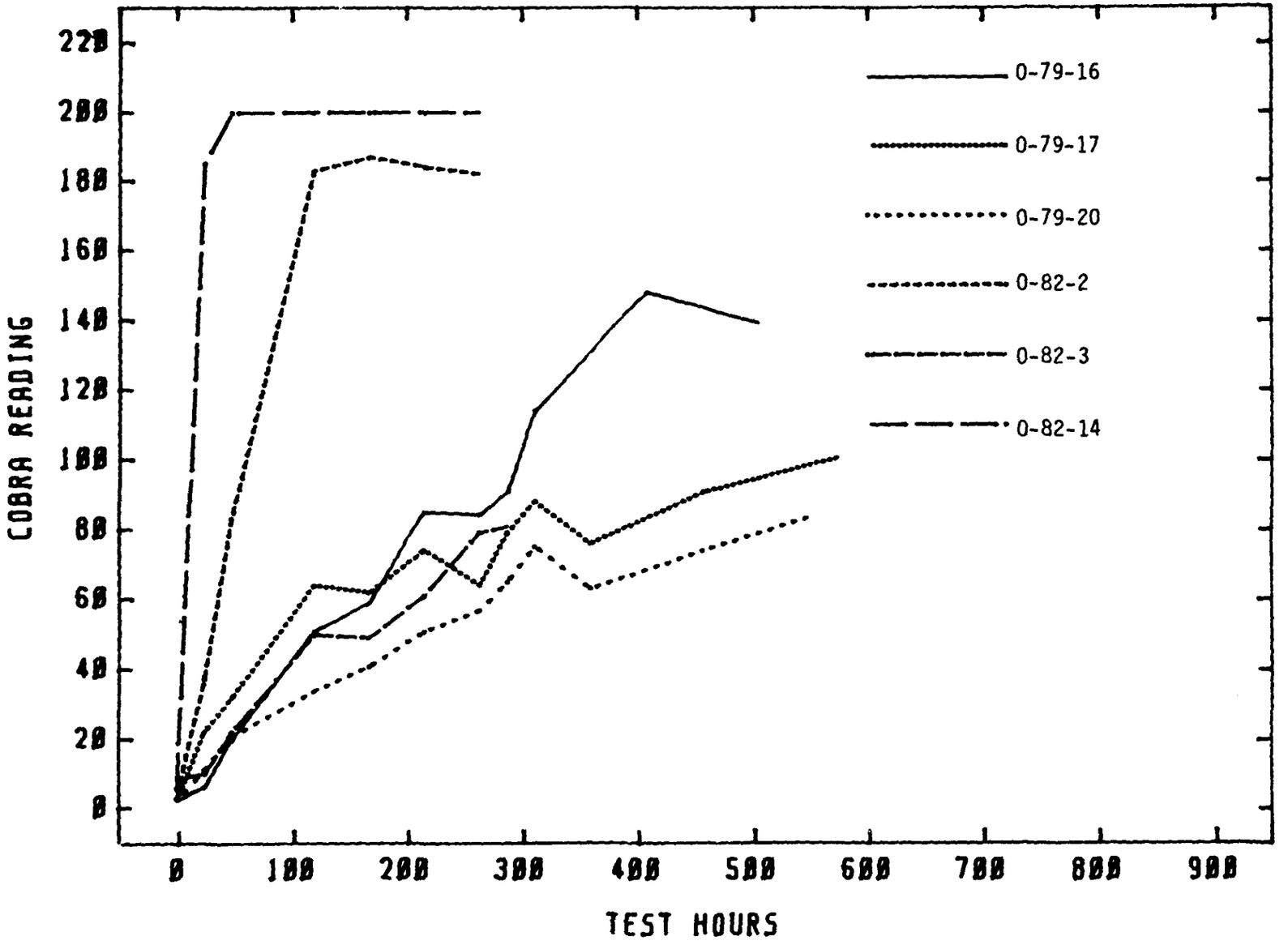


Figure 93. COBRA Readings of MIL-L-7808 Lubricants from the Squires Oxidative Test at 190°C

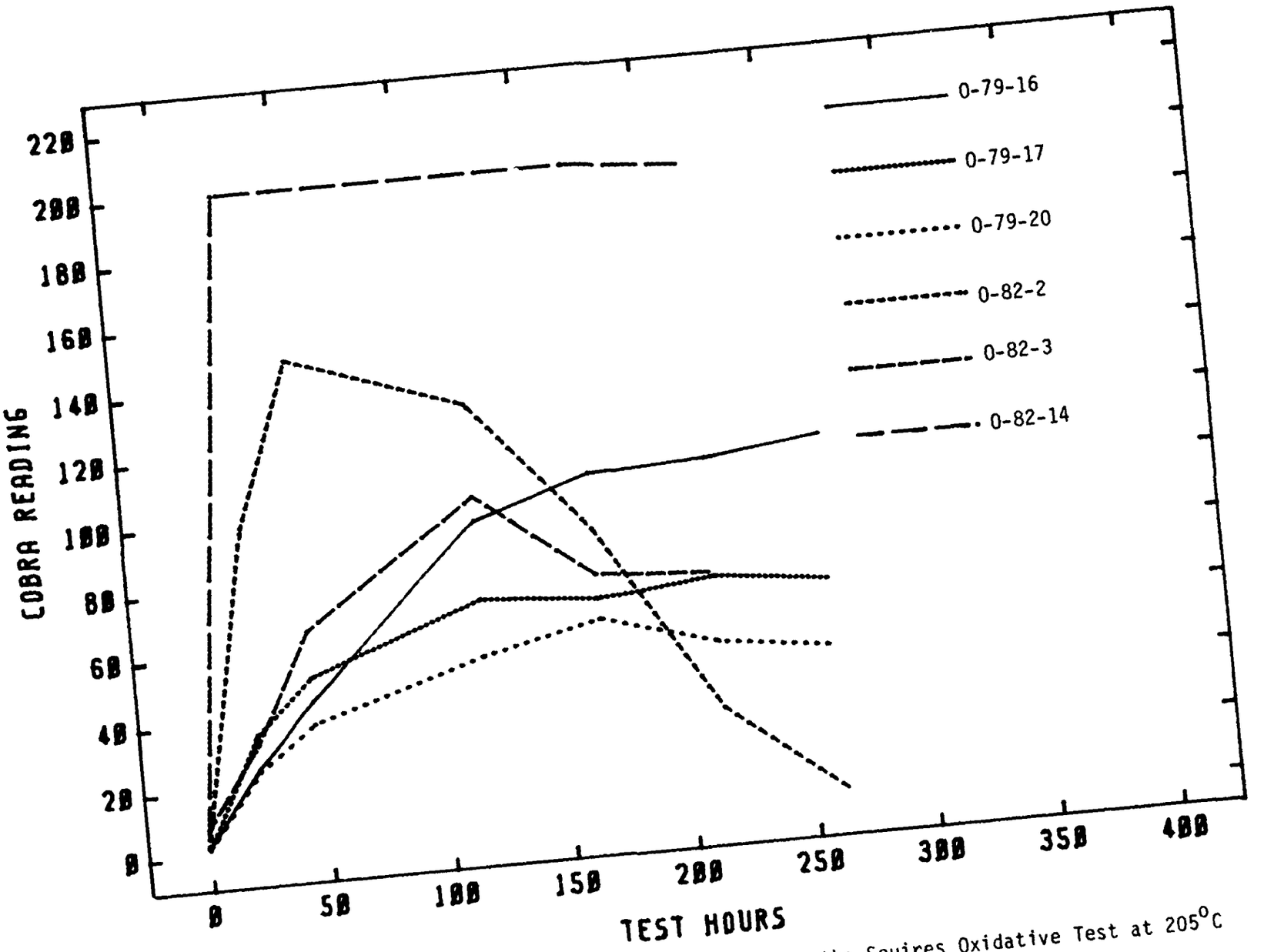


Figure 94. COBRA Readings of MIL-L-7808 Lubricants from the Squires Oxidative Test at 205°C

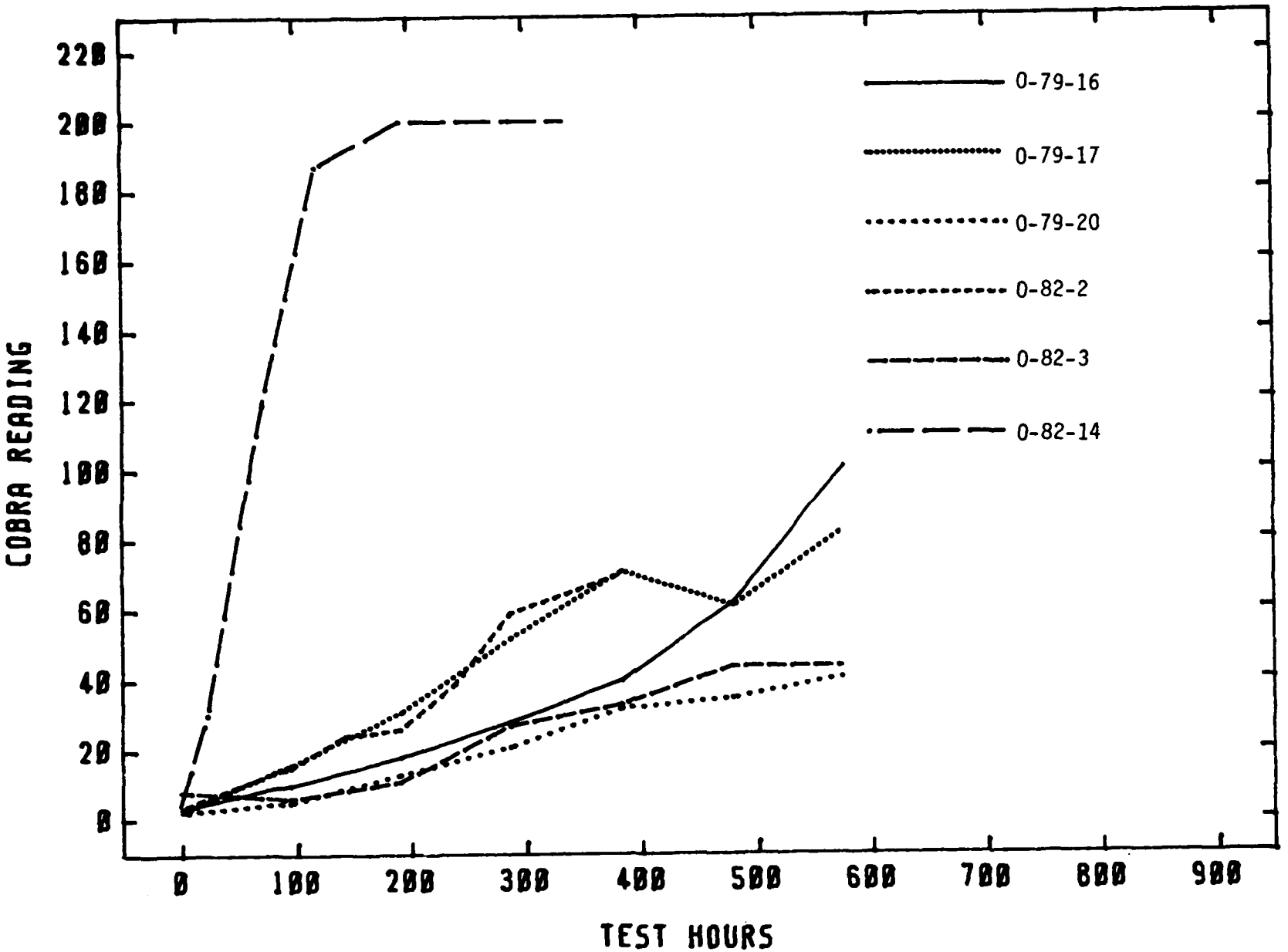


Figure 95. COBRA Readings of MIL-L-7808 Lubricants from the Squires Confined Heat Test at 175°C

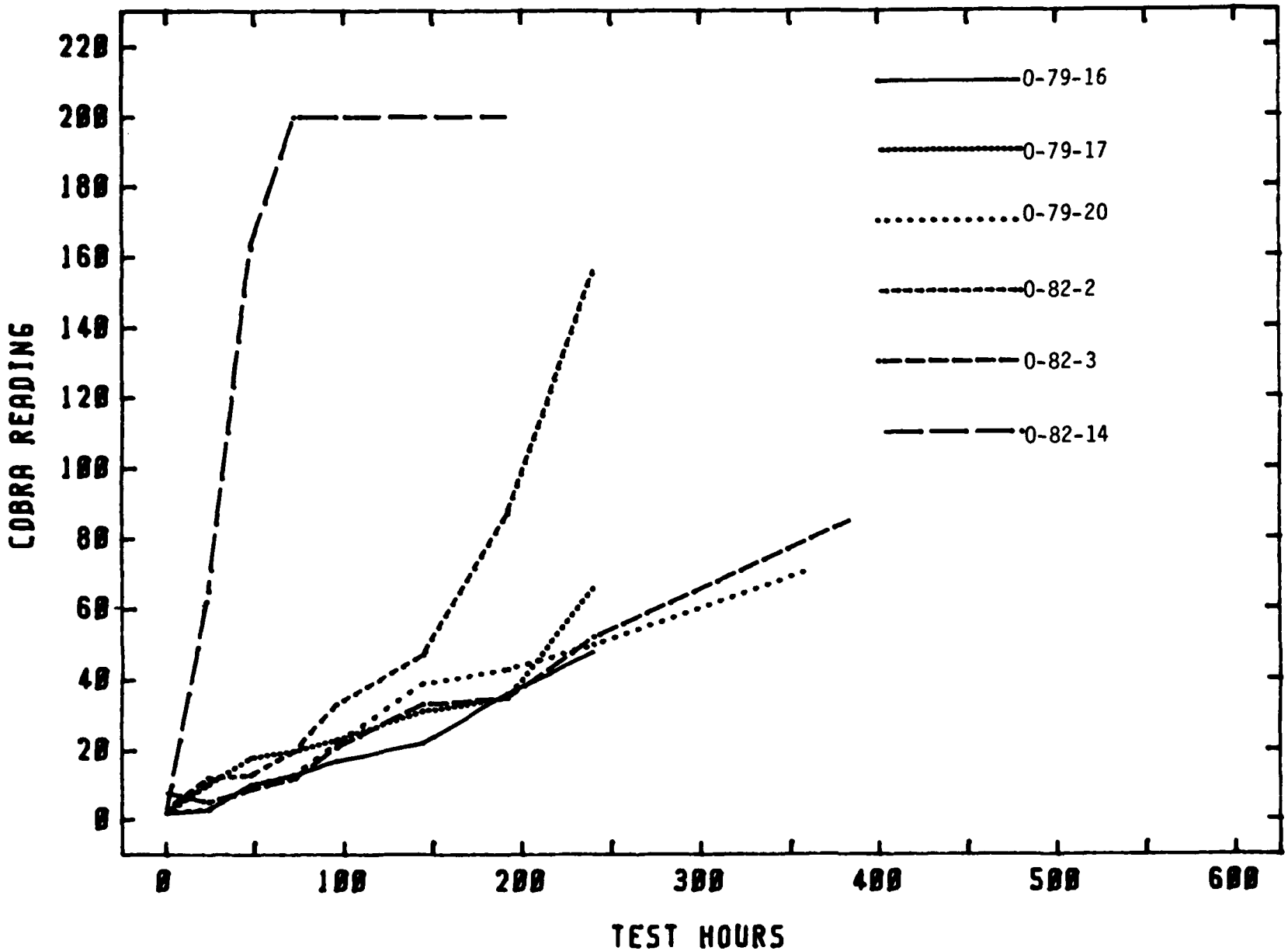


Figure 96. COBRA Readings of MIL-L-7808 Lubricants from the Squires Confined Heat Test at 190°C

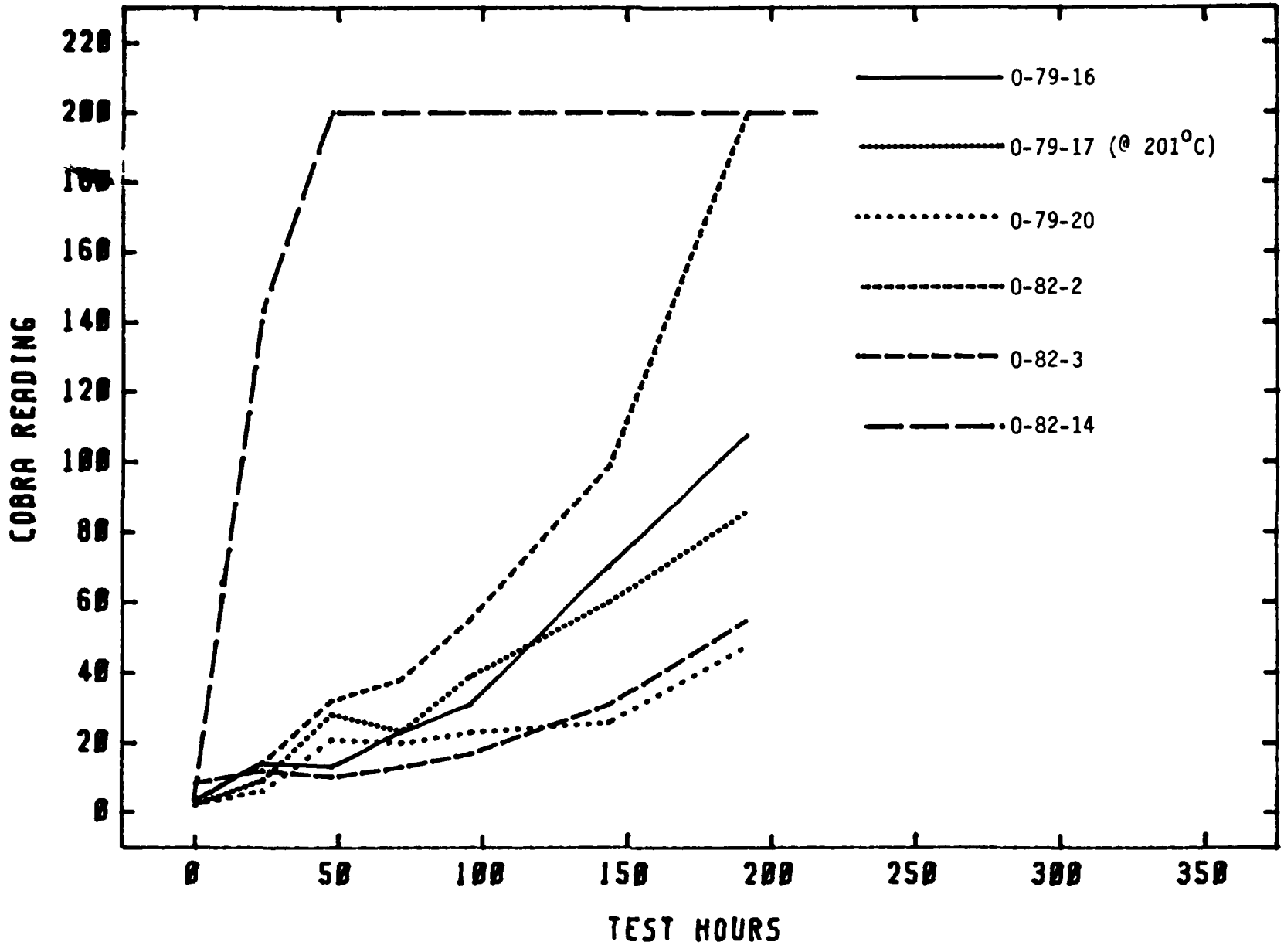


Figure 97. COBRA Readings of MIL-L-7808 Lubricants from the Squires Confined Heat Test at 205°C

TABLE 56

COBRA READINGS OF MIL-L-7808 LUBRICANTS FROM THE SQUIRES OXIDATIVE TEST AT POINTS OF EQUAL TAN AND VISCOSITY INCREASES

	175°C		190°C		205°C	
Lubricant	TAN Inc. of 1.5	Viscosity Inc. of 35%	TAN Inc. of 1.5	Viscosity Inc. of 35%	TAN Inc. of 1.5	Viscosity Inc. of 35%
0-79-16	a	62	146	63	101	79
0-79-17	a	a	100	80	71	71
0-79-20	a	a	80	63	51	54
0-82-2	126	200	78	186	85	153
0-82-3	a	42	84	51	80	81
0-82-14	200	200	200	200	200	200

a Oil did not reach value by end of test

TABLE 57

COBRA READINGS OF MIL-L-7808 LUBRICANTS FROM THE SQUIRES CONFINED HEAT TEST AT POINTS OF EQUAL TAN AND VISCOSITY INCREASES

	175°C		190°C		205°C	
Lubricant	TAN Inc. of 4.0	Viscosity Inc. of 5%	TAN Inc. of 4.0	Viscosity Inc. of 5%	TAN Inc. of 4.0	Viscosity Inc. of 5%
O-79-16	62	46	27	43	37	46
O-79-17	68	82	25	42	34[b]	73[b]
O-79-20	47	34	57	53	24	31
O-82-2	64	75	41	80	28	42
O-82-3	a	29	84	27	22	16
O-82-14	200	200	178	200	153	167

a Oil did not reach value by end of test

b Test run at 201°C

Figures 92 to 94. That is, two lubricants (O-82-2 and O-82-14) show disproportionately large rises in COBRA reading relative to the other four. For TAN changes of 1.5, the O-82-2 lubricant is more in line with the other four lubricants. The difficulty in interpretation arises from the influence of volatilization losses in this test on the physical properties. Such losses would be expected to increase viscosity changes and decrease TAN changes but not consistently for all six lubricants since their formulations, and thus degradation products, are different from one another. The confined heat tested lubricants do not undergo large volatilization weight losses and the interpretation of the results (Table 57) is more straightforward. Of the six lubricants only O-82-14 shows a very high COBRA rise relative to the standard increases of TAN and viscosity.

c. Structure/Property Relationships

It is of primary interest to investigate the relationship between the COBRA reading of a degraded lubricant and specific changes in its chemical composition. The chemical degradation of synthetic formulated ester basestock lubricants has been thoroughly investigated by a number of researchers using a variety of analytical techniques.[48-56] Such investigations have shown that oxidative degradation of the basestock proceeds via hydroperoxide intermediates to form various low molecular weight oxygenated compounds, such as ketones, aldehydes and carboxylic acids, which can polymerize to form high molecular weight compounds which increase the viscosity of the lubricant and can form insoluble deposits. To what extent these degradation products, as well as any additives and their degradation products, contribute to the COBRA reading of a stressed lubricant is not known. To investigate this relationship several methods were used to characterize degraded lubricants.

(1) Peroxide Number

Since hydroperoxides are intermediates in the oxidative degradation of ester lubricants their concentration in the six MIL-L-7808 lubricants stressed in the Squires oxidative test at 190°C was measured. The method used was ASTM D 3703-83, an iodine reduction method which is sensitive to easily reduced peroxides such as hydroperoxides and diacyl peroxides but is not sensitive to the more difficult to reduce peroxides such as dialkyl peroxides. Results are reported in units of peroxide number with a value of 1 equal to 8 meq peroxide/gram oil. Also, in order to allow for volatilization weight loss of the degraded lubricant an adjusted peroxide number was calculated which is equal to the peroxide number times the weight fraction of the lubricant remaining. The peroxide number plot (Figure 98) vs. test hours shows an initial rapid rise for each lubricant followed by a smaller, consistent rise. This secondary rise is greatly influenced by volatilization weight loss, as shown in the plot of the adjusted peroxide number (Figure 99) which reveals that after a sharp initial rise, the peroxides reach a steady state concentration (between 48 and 120 hours) for the remainder of the test. Previous antioxidant analysis by gas chromatography of these samples had shown that the leveling off phenomena generally occurred at the point where the primary antioxidant (PANA or Octyl-PANA) was depleted. The fact that the two Octyl-PANA containing lubricants (O-82-3 and O-82-14) show much larger initial rise in peroxide concentration is probably due to the lower volatility of this additive relative to PANA. Regardless, comparison of either plot to its corresponding COBRA reading plot (Figure 93) does not show any particular correlation between peroxide number and COBRA reading except that they both increase with time for all lubricants. This data does not explain the unusually high COBRA

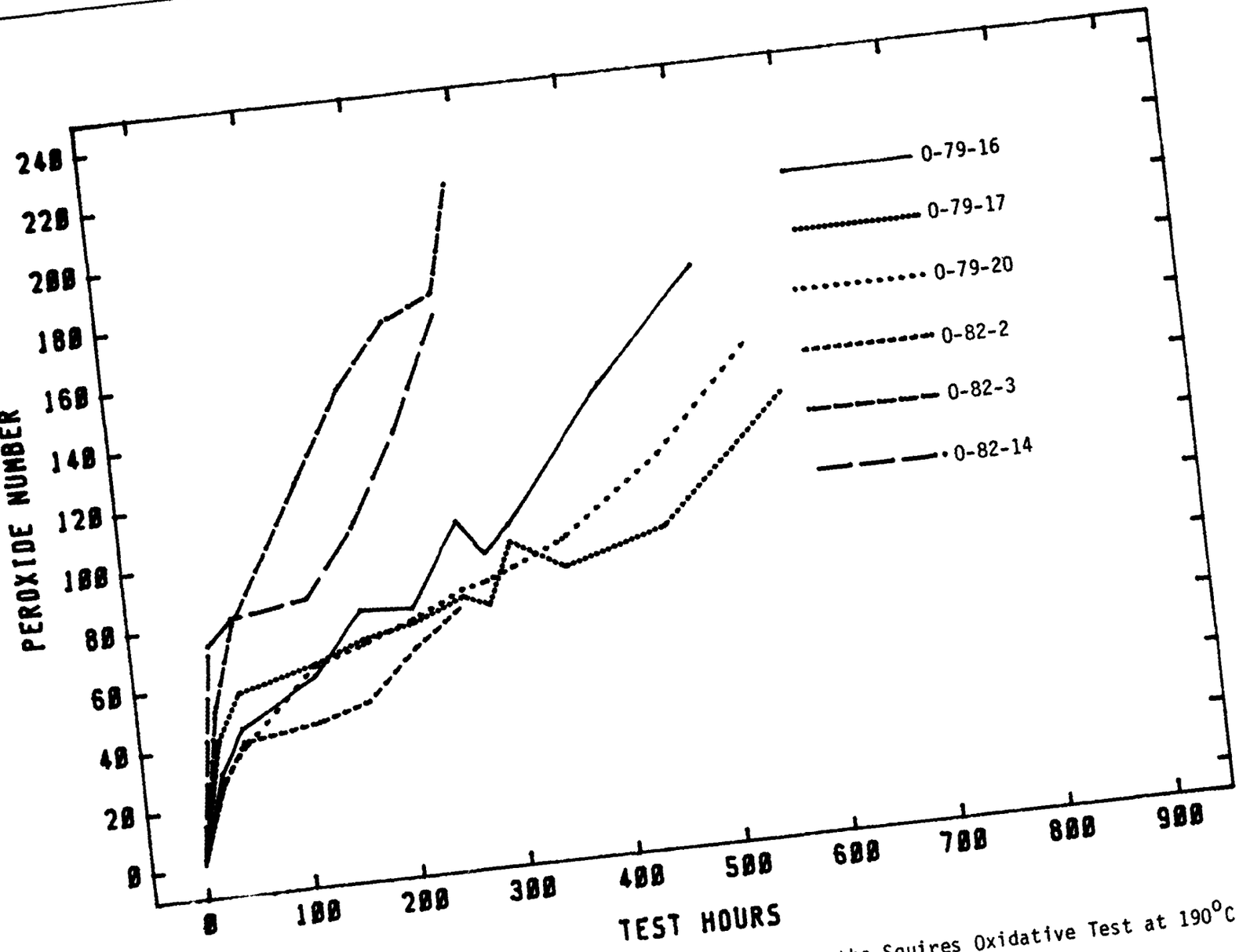


Figure 98. Peroxide Numbers of MIL-L-7808 Lubricants from the Squires Oxidative Test at 190°C

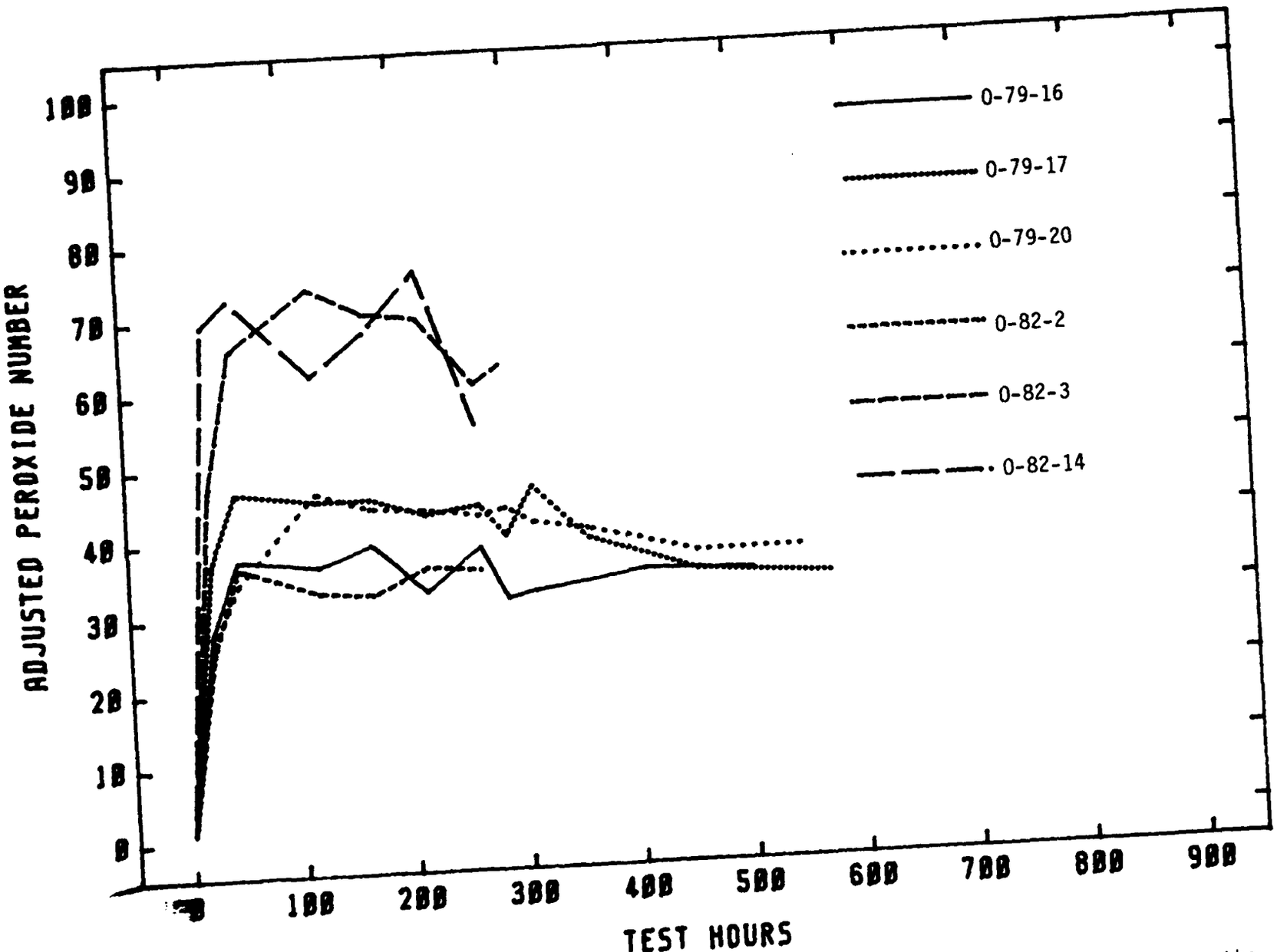


Figure 99. Peroxide Numbers (Adjusted for Volatility Loss) of MIL-L-7808 Lubricants from the Squires Oxidative Test at 190°C

readings that are seen in the O-82-2 and O-82-14 lubricants.

(2) Basestock Ester Analysis

Since it is expected that the oxidative and thermal degradation of the basestock esters of the MIL-L-7808 lubricants would yield products that would contribute to a rising COBRA reading, a measurement of the degree of ester breakdown in a degraded lubricant may reveal some correlation with COBRA readings. Because of the great complexity of the basestocks of MIL-L-7808 lubricants, initial efforts were concentrated on analysis of laboratory formulated lubricants containing either di-2-ethylhexyl adipate (D2EHA) or trimethylolpropane triheptanoate (TMP-777). A method was developed for quantitative analysis of these two esters by gas chromatography by an internal standard method. The conditions of analysis are listed in Table 58.

TABLE 58

CHROMATOGRAPHIC CONDITIONS FOR ESTER ANALYSIS

Instrument:	Varian 6000
Detector:	Flame Ionization
Column:	25 m X 0.53 mm ID Fused Silica Capillary (Quadrex)
Liquid Phase:	DB-5, 0.25 Micron Film Thickness
Injector Temp:	300°C
Detector Temp:	325°C
Oven Temp:	150-325°C at 8°C/minute, 5 minutes final hold

Formulations of these two esters with 1% PANA and 1% DODPA that had been stressed in both the Squires oxidative and confined heat tests at 205°C were analyzed for the percent of ester remaining in the sample. The plots of the percent ester and COBRA readings vs. test hours for the two formulations from

the Squires oxidative test at 205°C (Figures 100 and 101) reveal that at 24 test hours the basestock consumption is minimal. Likewise the physical properties and antioxidant levels were found to be very similar. But there is a considerable difference in the COBRA readings (20 for TMP-777 and 78 for the D2EHA). After this, comparison of the two is complicated by the rapid degradation (or volatilization loss) of the D2EHA basestock (and a resultant COBRA rise to 200). Similar results are seen with the corresponding confined heat test data (Figures 102 and 103). After 96 hours, percent basestock loss is much more for the TMP-777 lubricant than for the D2EHA lubricant (8.9 vs. 1.9% respectively) but the COBRA reading is less (15 vs. 51). This data seems to indicate that oxidative and thermal degradation of a dibasic ester produces decomposition products that contribute greatly to COBRA readings relative to that of a hindered triester. This implies that the disproportionately high COBRA rise of O-82-2 and O-82-14 is due to the fact that they both contain significant amounts of diesters. Exactly what role the antioxidants (PANA/DODPA) play in affecting the COBRA reading is not known but it may be that their oxidation products are of importance.

(3) Contribution of Basestock Degradation Products to COBRA Reading.

Since carboxylic acids and aliphatic alcohols are known decomposition products from oxidatively and thermally stressed ester lubricants it is of interest to determine their individual and combinative contributions to COBRA readings. The basestock oil O-77-1 (D2EHA) was formulated with certain degradation products including carboxylic acids and alcohols in order to determine their contribution to the COBRA readings. The half acid ester of D2EHA was not available so lauric acid ($n\text{-}C_{12}$) was used in its place. The COBRA readings of various combinations of added degradation products is shown in Table 59.

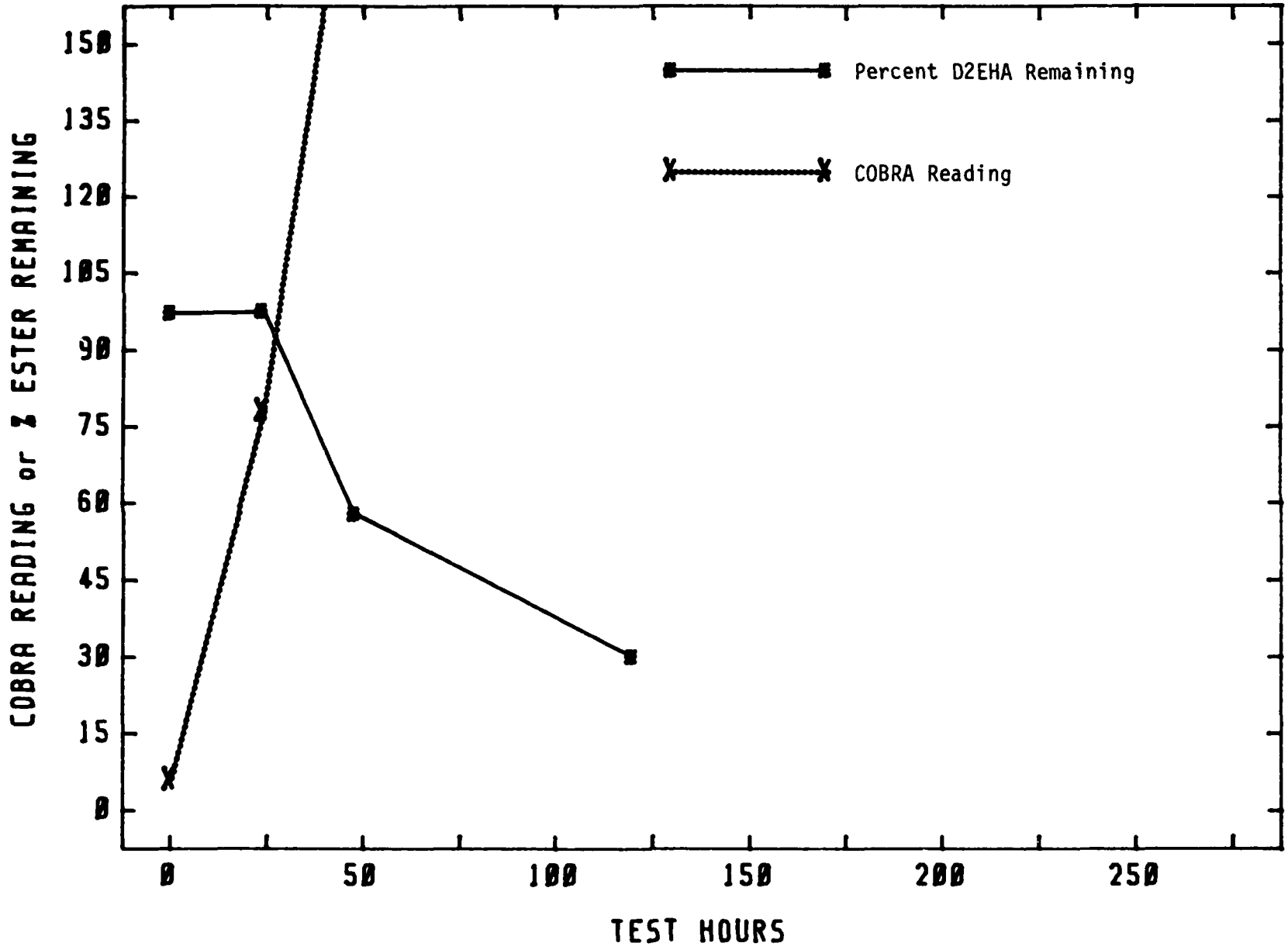


Figure 100. COBRA Reading vs. Ester Depletion for O-77-1 + 1% PANA + 1% DODPA from the Squires Oxidative Test at 205°C.

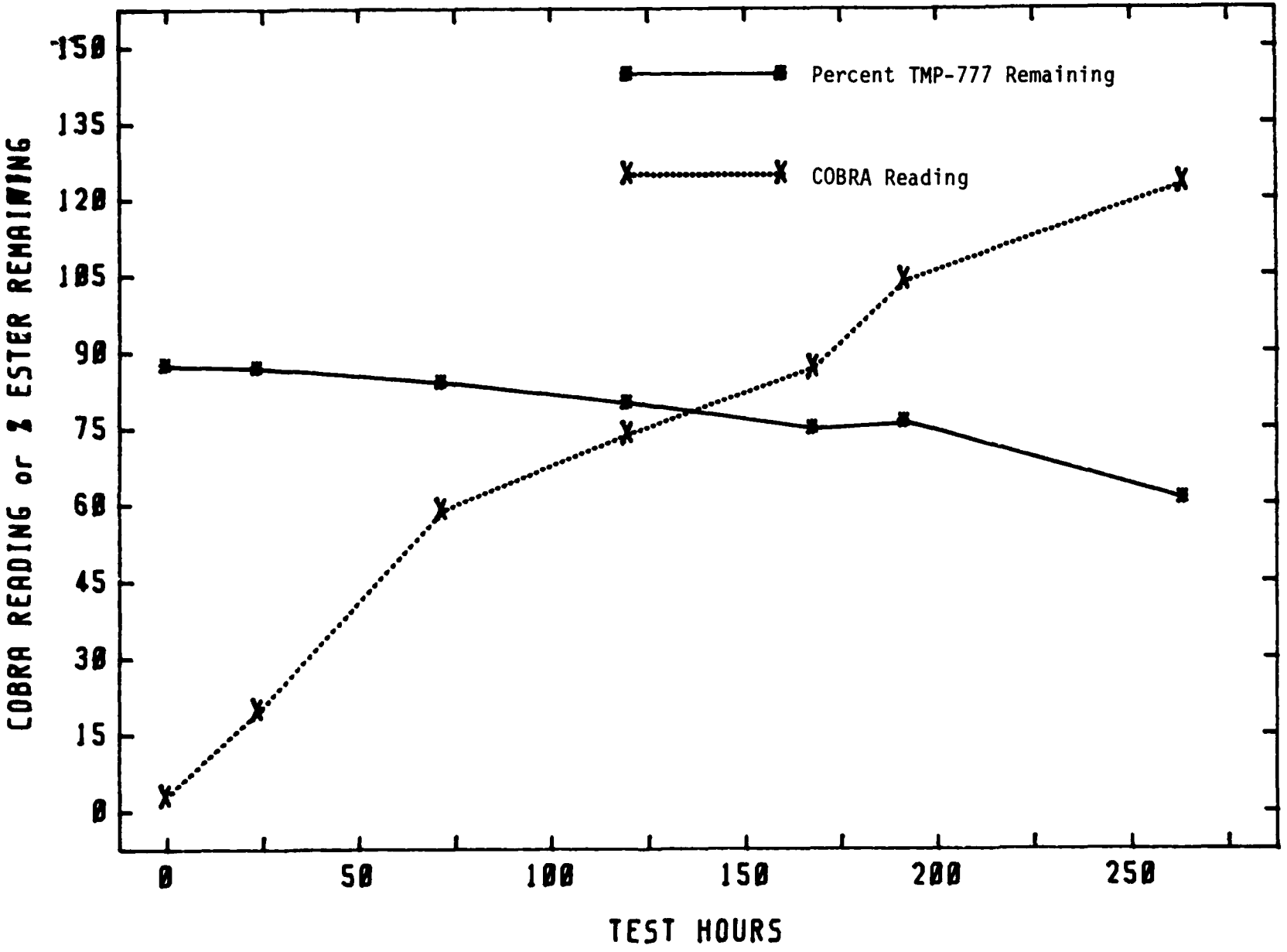


Figure 101. COBRA Reading vs. Ester Depletion for O-76-5A + 1% PANA + 1% DODPA from the Squires Oxidative Test at 205°C.

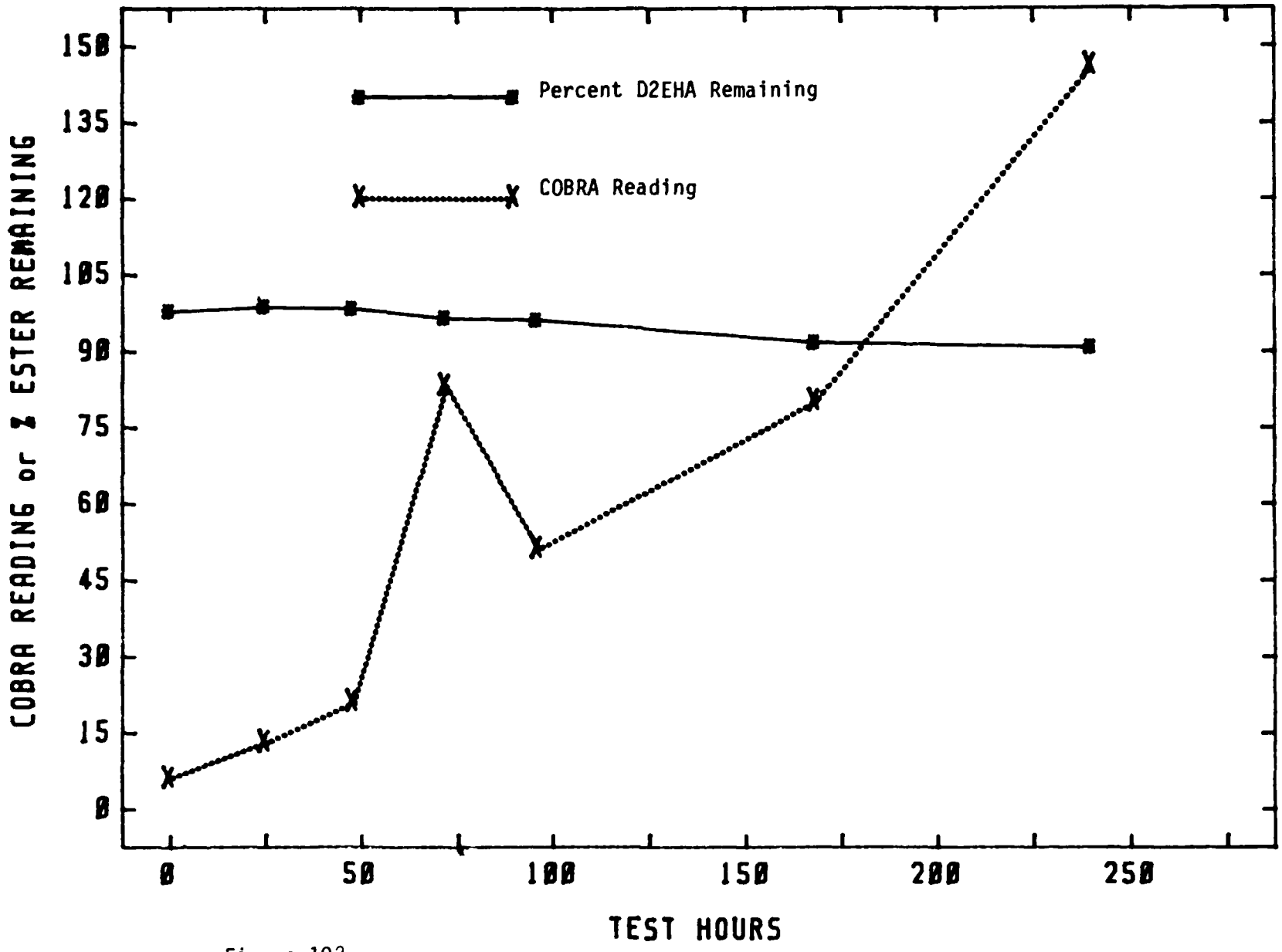
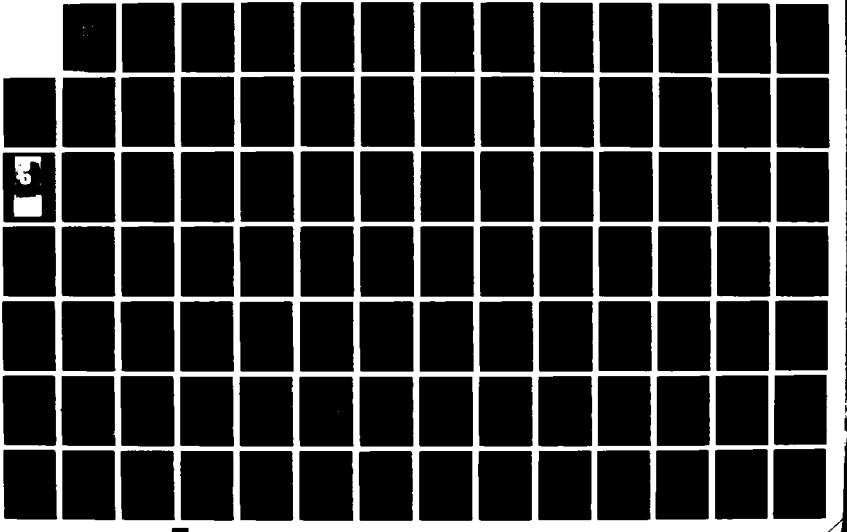
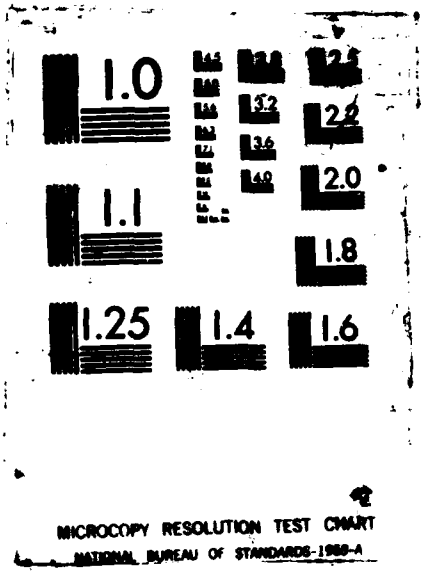


Figure 102. COBRA Reading vs. Ester Depletion for O-77-1 + 1% PANA + 1% DODPA from the Squires Confined Heat Test at 205°C.

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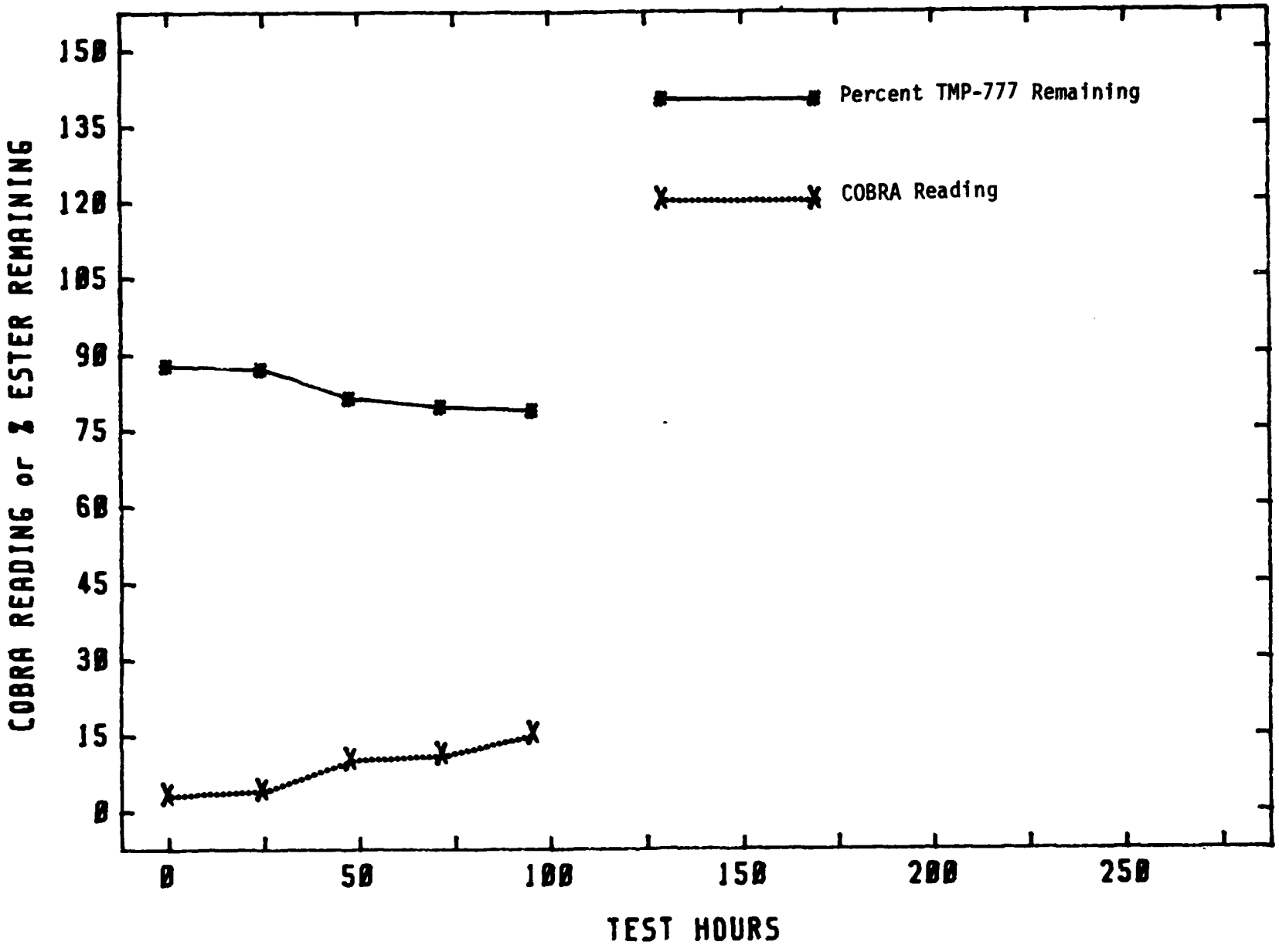


Figure 103. COBRA Reading vs. Ester Depletion for O-76-5A + 1% PANA + 1% DODPA from the Squires Confined Heat Test at 205°C

TABLE 59

EFFECT OF ADDED DEGRADATION PRODUCTS TO O-77-1 ON COBRA READING

	SAMPLE	COBRA READINGS
(1)	O-77-1 basestock	4
(2)	above plus lauric acid (TAN = 9.9)	5.5
(3)	above (2) plus 2% 2-ethylhexanol	5
(4)	O-77-1 plus 1% PANA and 1% DODPA	6
(5)	above plus lauric acid (TAN = 9.7)	13
(6)	above (5) plus 2% 2-ethylhexanol	11.5
(7)	O-77-1 with 1% PANA and 1% DODPA Squires oxidative test at 190°C, 48 hrs. (TAN = 0.50)	56
(8)	above plus lauric acid (TAN = 3.0)	53

As the data shows, none of the added materials affected the COBRA reading significantly. It is interesting to note that the addition of either the acid (2) or the antioxidants (4) separately has no effect on the COBRA reading but if combined (5) they more than double the reading. This may be due to ion pair formation (the antioxidants being weak bases) with a subsequent increase in conductive species. It does not seem to be a very significant effect since addition of acid to a degraded oil (8) produces no measurable COBRA reading change. The addition of 2-ethylhexanol produces no measurable effect on COBRA reading.

3. CONDUCTIVITY MEASUREMENTS

Conductivity measurements on degraded lubricants were made using a YSI Model 34 conductivity meter (Yellow Springs Instrument Co.). No commercially available conductivity cell proved to be adequate due to lack of sensitivity and large sample requirements. Instead, an electrode cell assembly from a COBRA instrument was wired to the conductivity meter. Because of the COBRA

cell's small electrode gap (low cell constant) the sensitivity of this system for the analysis of degraded lubricants proved to be excellent and gave very stable readings. And unlike the COBRA instrument itself, the conductance readings showed no great dependence on the composition of the lower electrode as shown in Table 60.

TABLE 60

EFFECT OF BOTTOM ELECTRODE ON CONDUCTANCE MEASUREMENTS

Bottom Electrode	Conductance (μMHOs)*
Cadmium	0.140
Magnesium	0.133
Nickel	0.122
Copper	0.131
Zinc	0.130
Stainless Steel	0.138

*Using a standard degraded lubricant

The lowest range of the meter (0-2 μmhos) was used for all measurements and all were made on the same uncalibrated electrode cell assembly.

Conductance measurements were made on six MIL-L-7808 lubricants that had been stressed in the Squires oxidative and confined heat tests at either 190°C or 205°C. The results (Figures 104 to 107) show trending that is virtually identical to what was seen in the COBRA readings of these same oils (see Figures 93, 94, 96 and 97). This is clearly shown in plots of all COBRA readings vs. their conductance readings for a given test and temperature (Figures 108 to 111). These plots reveal the basically linear relationship between conductance and COBRA readings. Some of the data scatter can be explained by the fact that the COBRA readings are subject to considerable

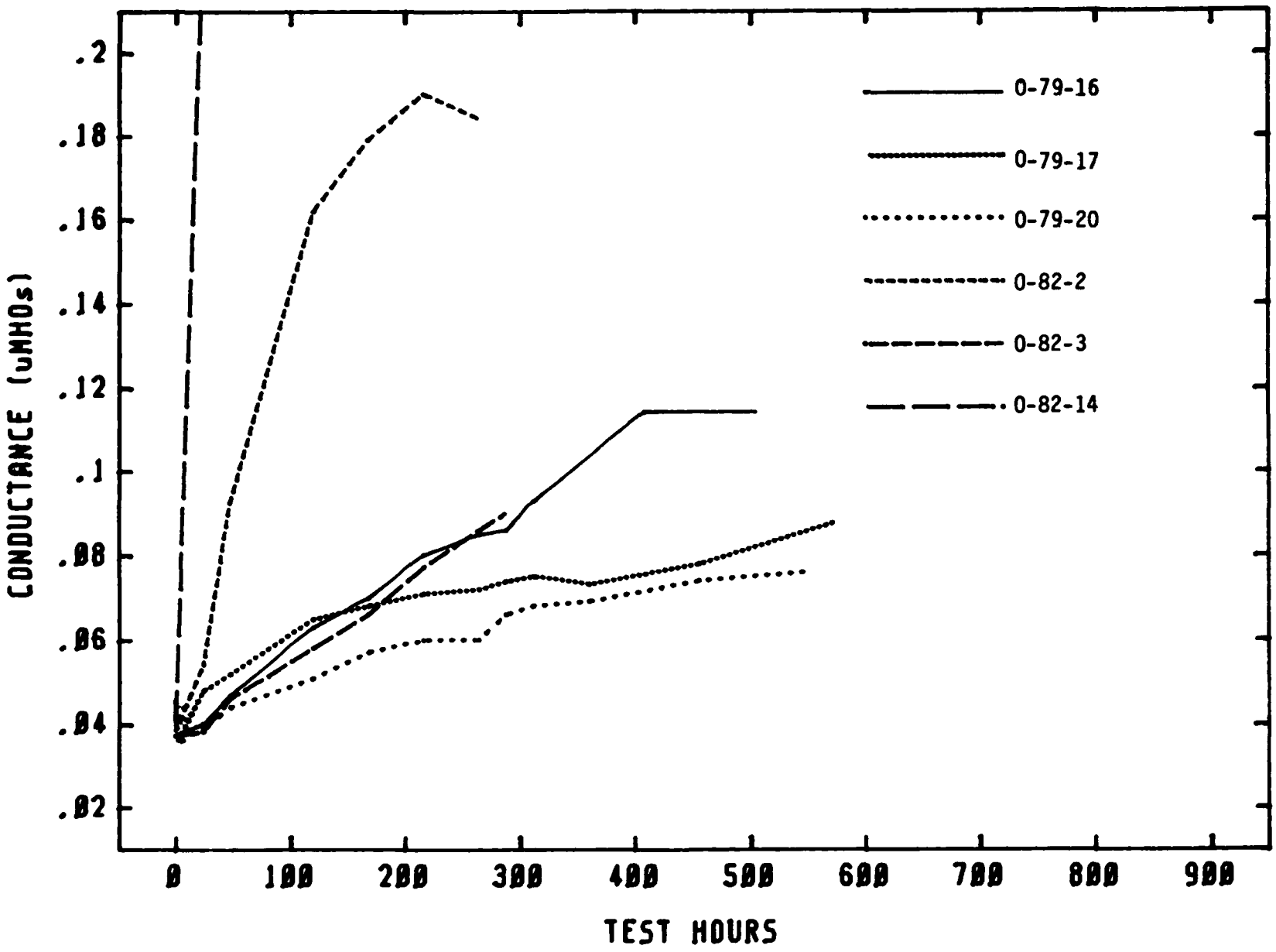


Figure 104. Conductance of MIL-L-7808 Lubricants from the Squires Oxidative Test at 190°C

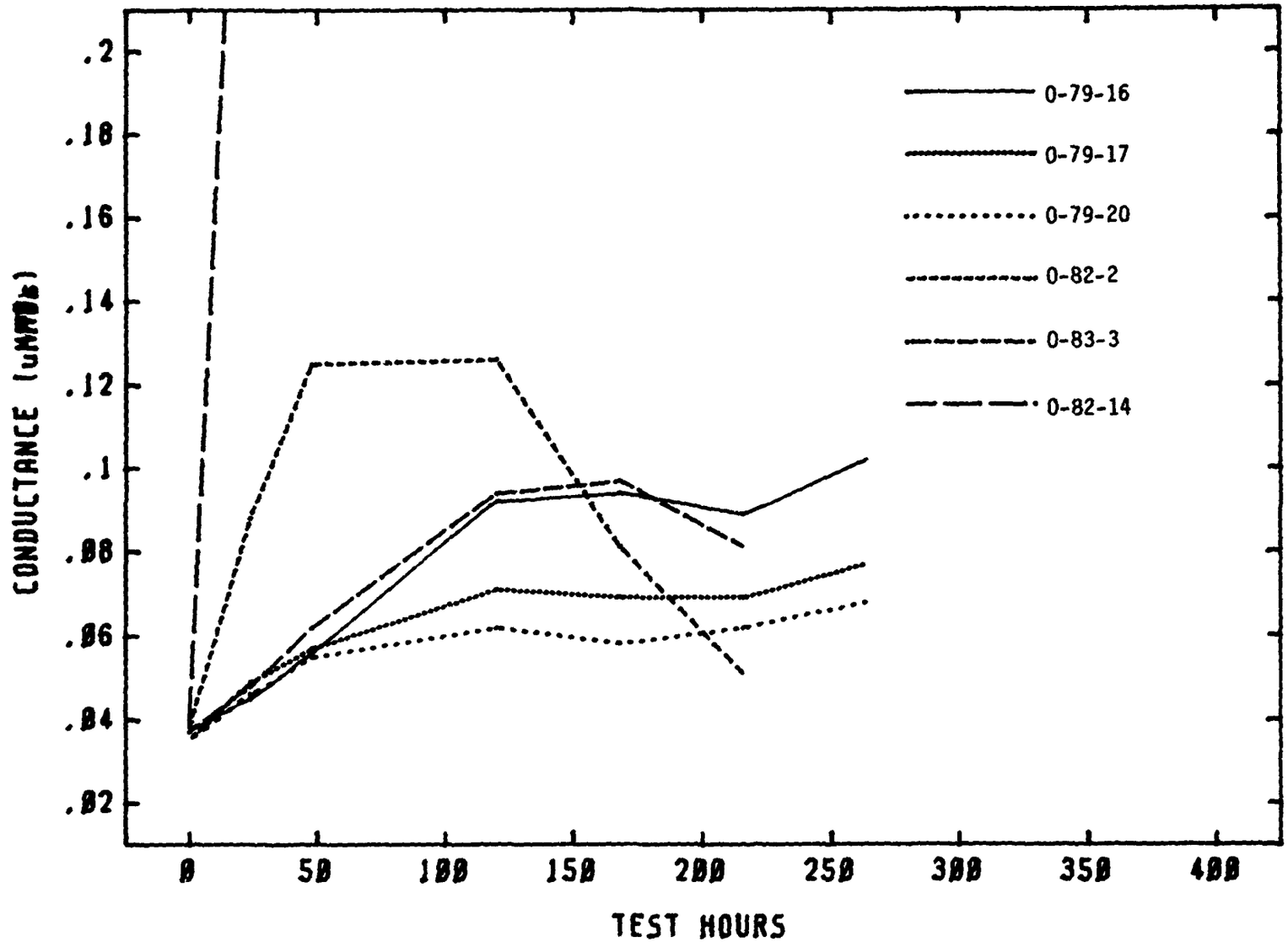


Figure 105. Conductance of MIL-L-7808 Lubricants from the Squires Oxidative Test at 205°C

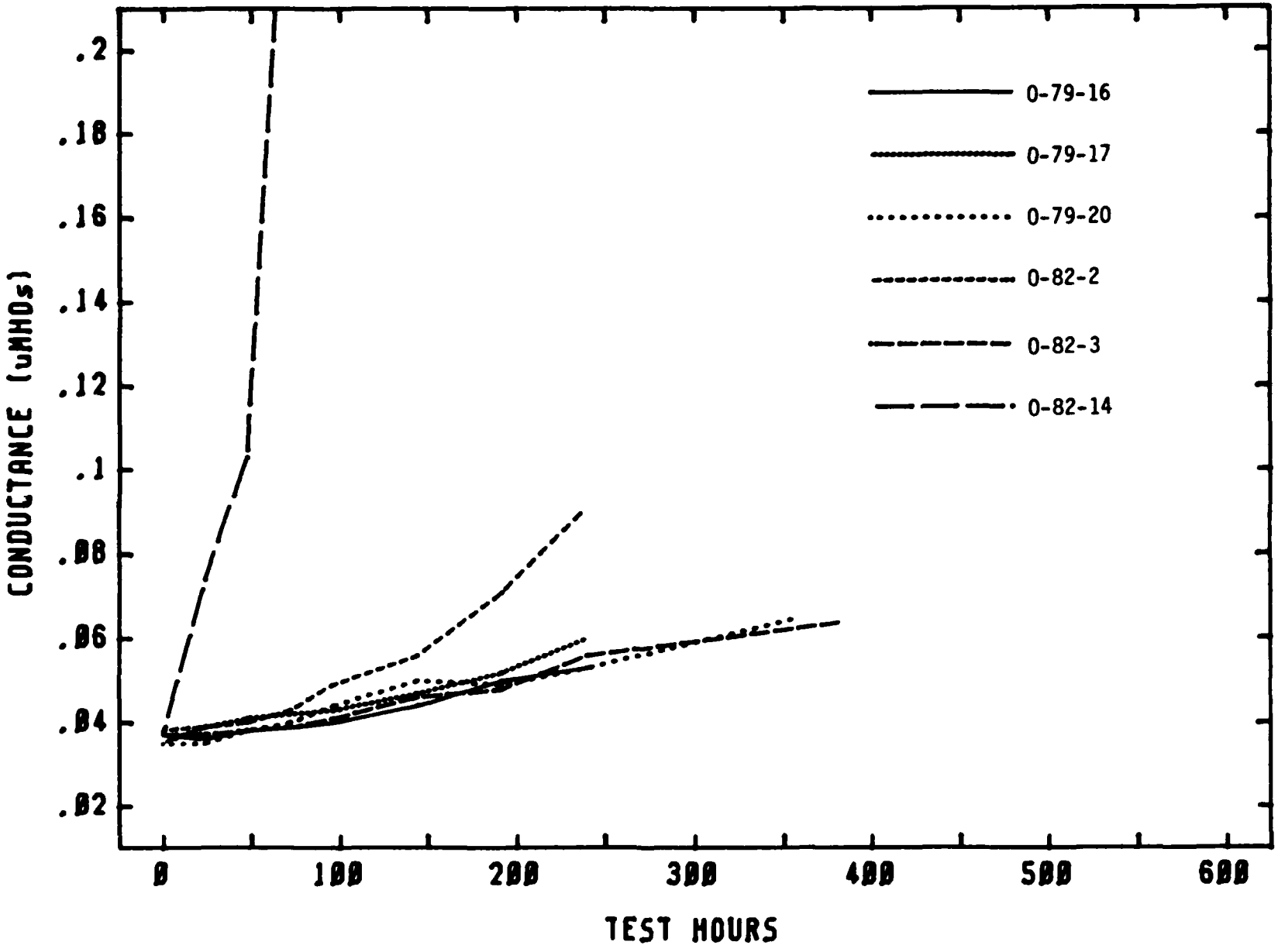


Figure 106. Conductance of MIL-L-7808 Lubricants from the Squires Confined Heat Test at 190°C

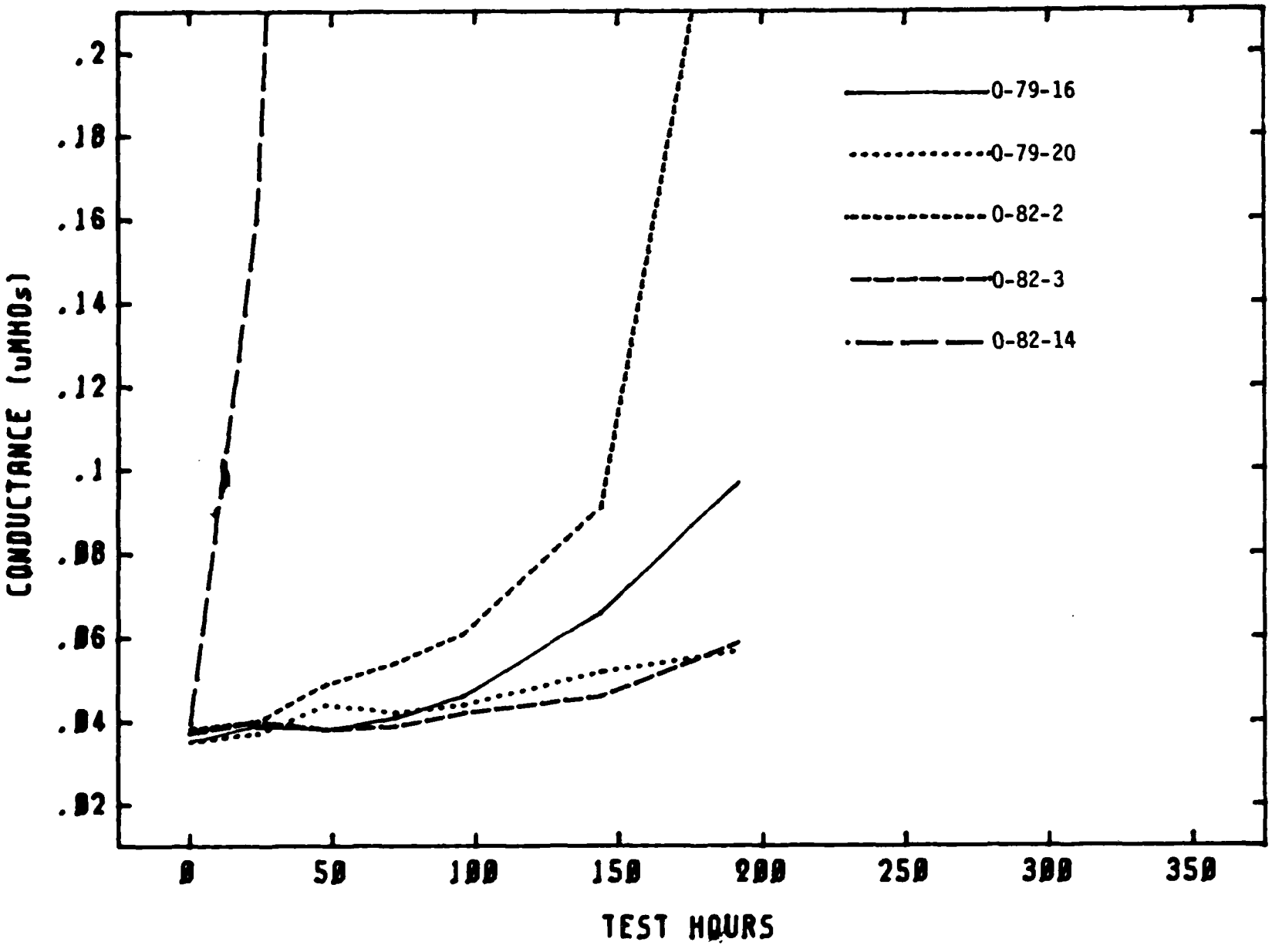


Figure 107. Conductance of MIL-L-7808 Lubricants from the Squires Confined Heat Test at 205°C

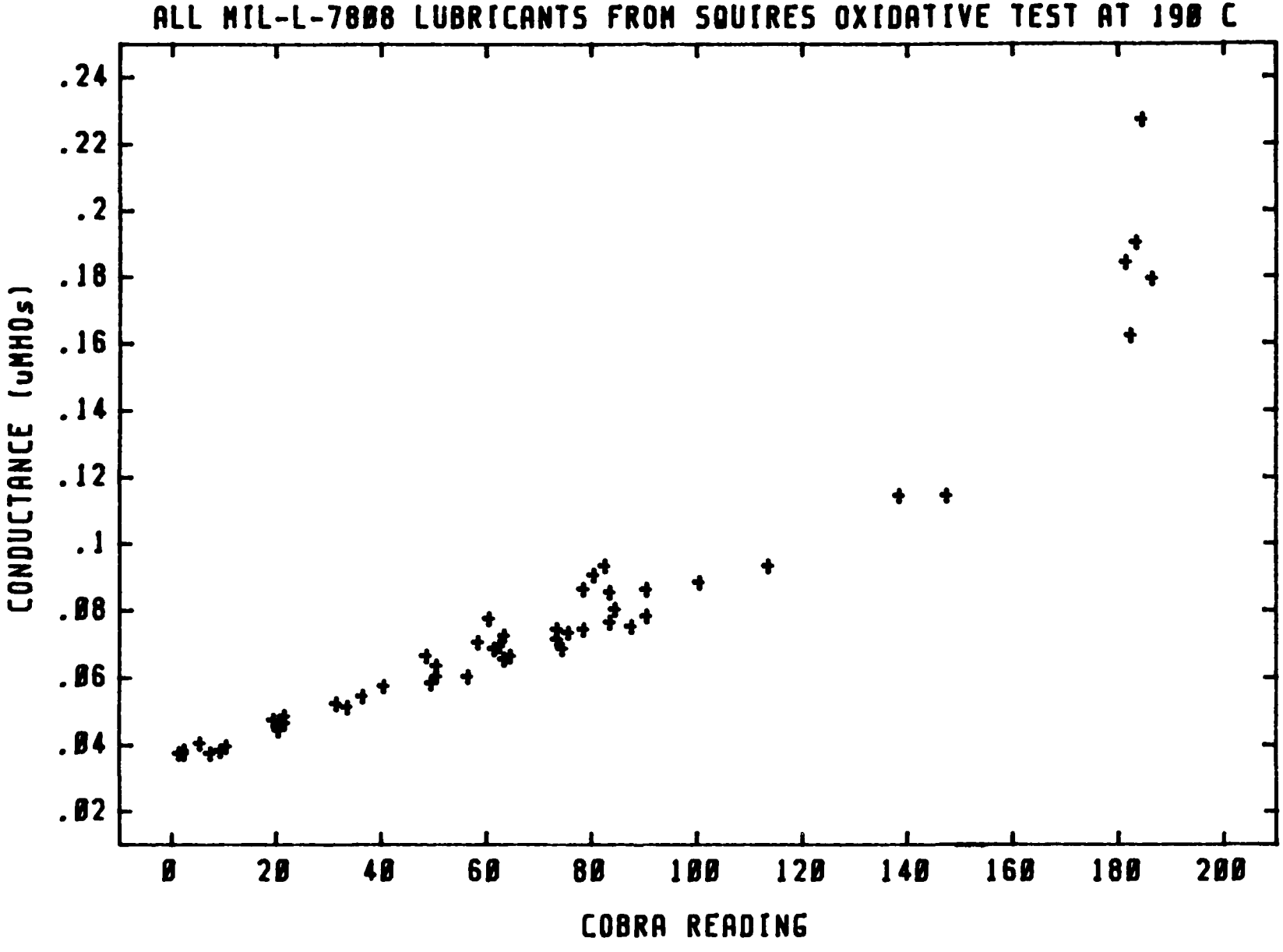


Figure 108. Conductance vs. COBRA Readings for Lubricants from Squires Oxidative Test at 190°C.

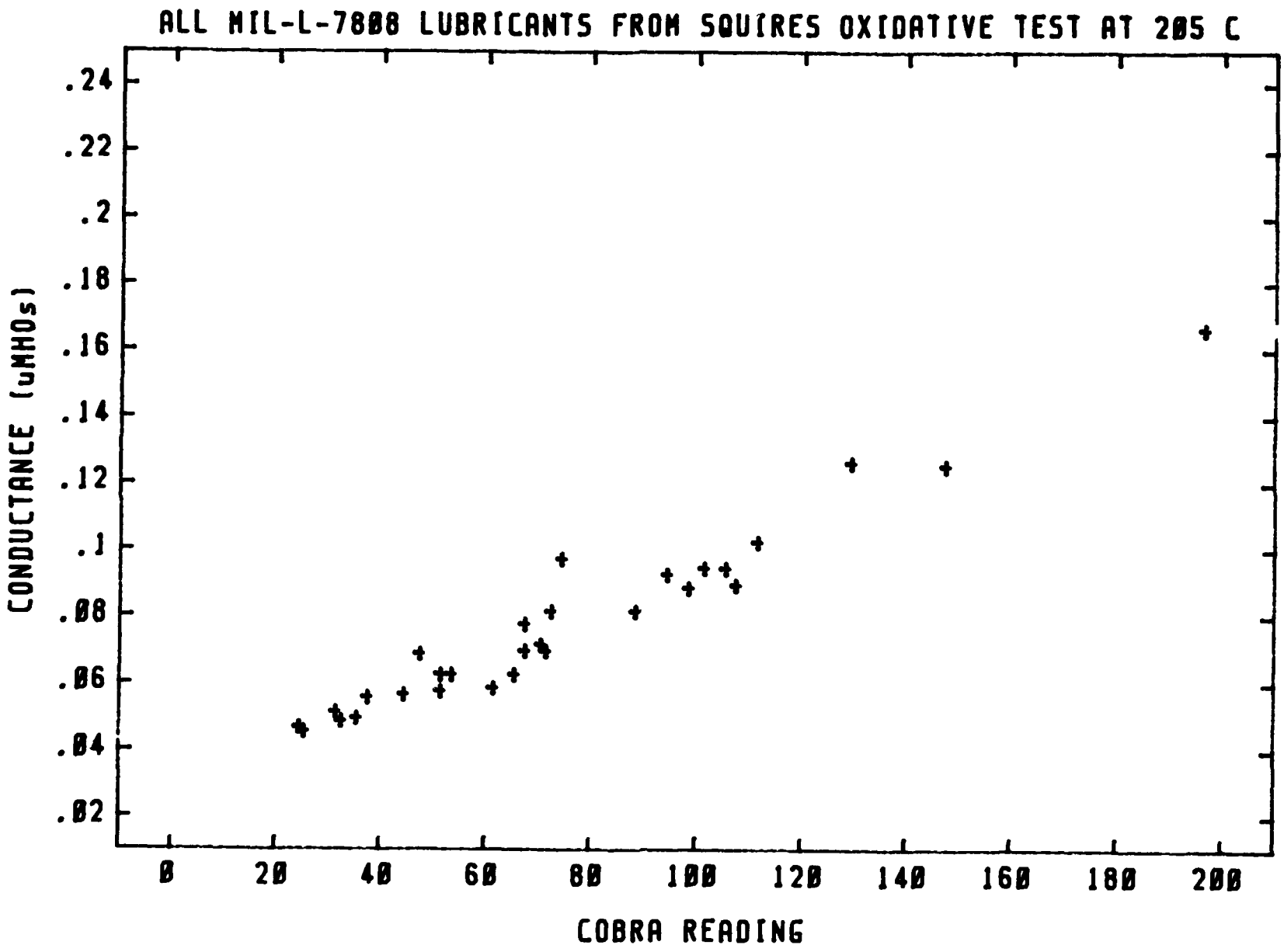


Figure 109. Conductance vs. COBRA Readings for Lubricants from Squires Oxidative Test at 205°C.

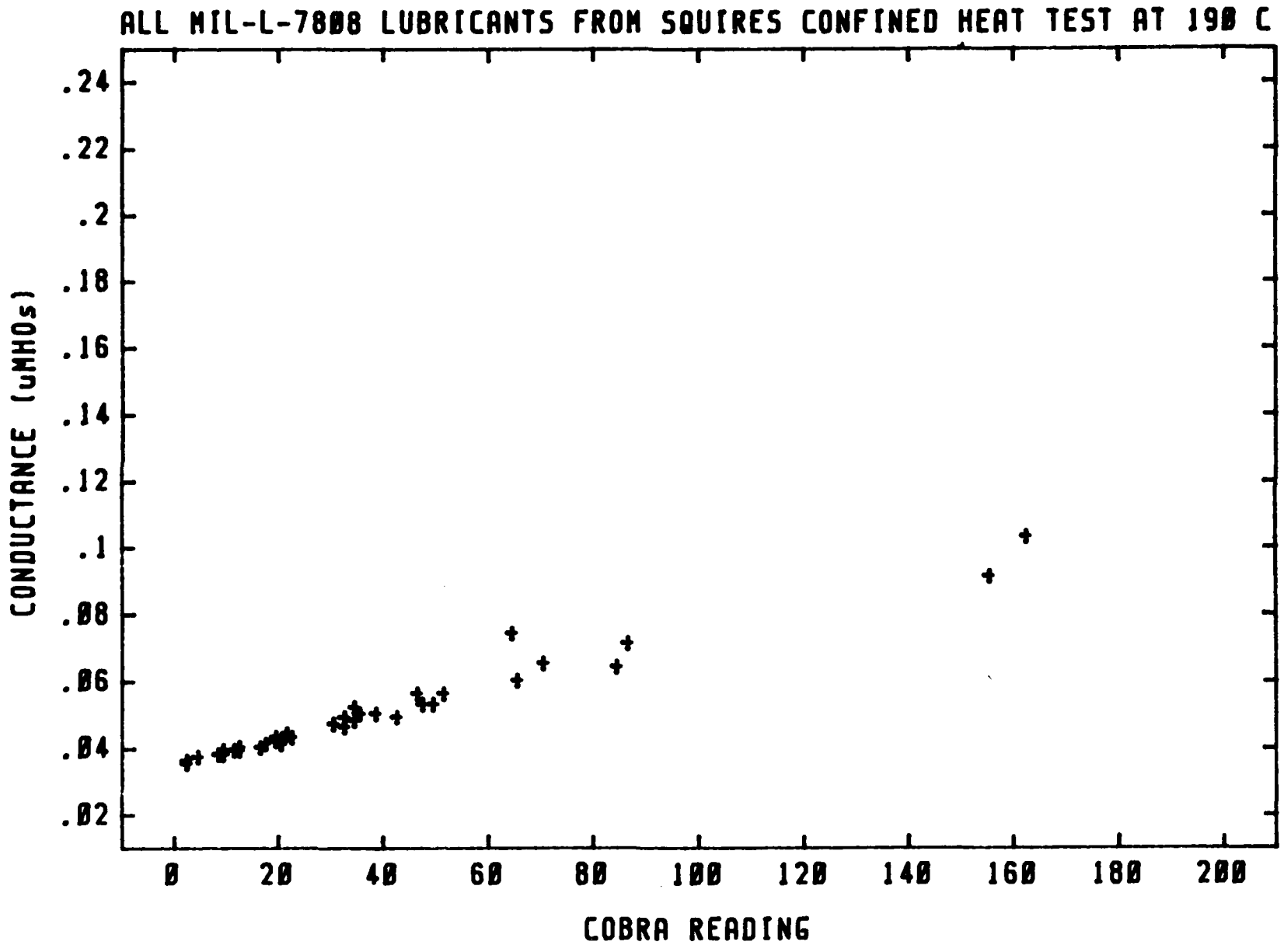


Figure 110. Conductance vs. COBRA Readings for Lubricants from Squires Confined Heat Test at 190°C.

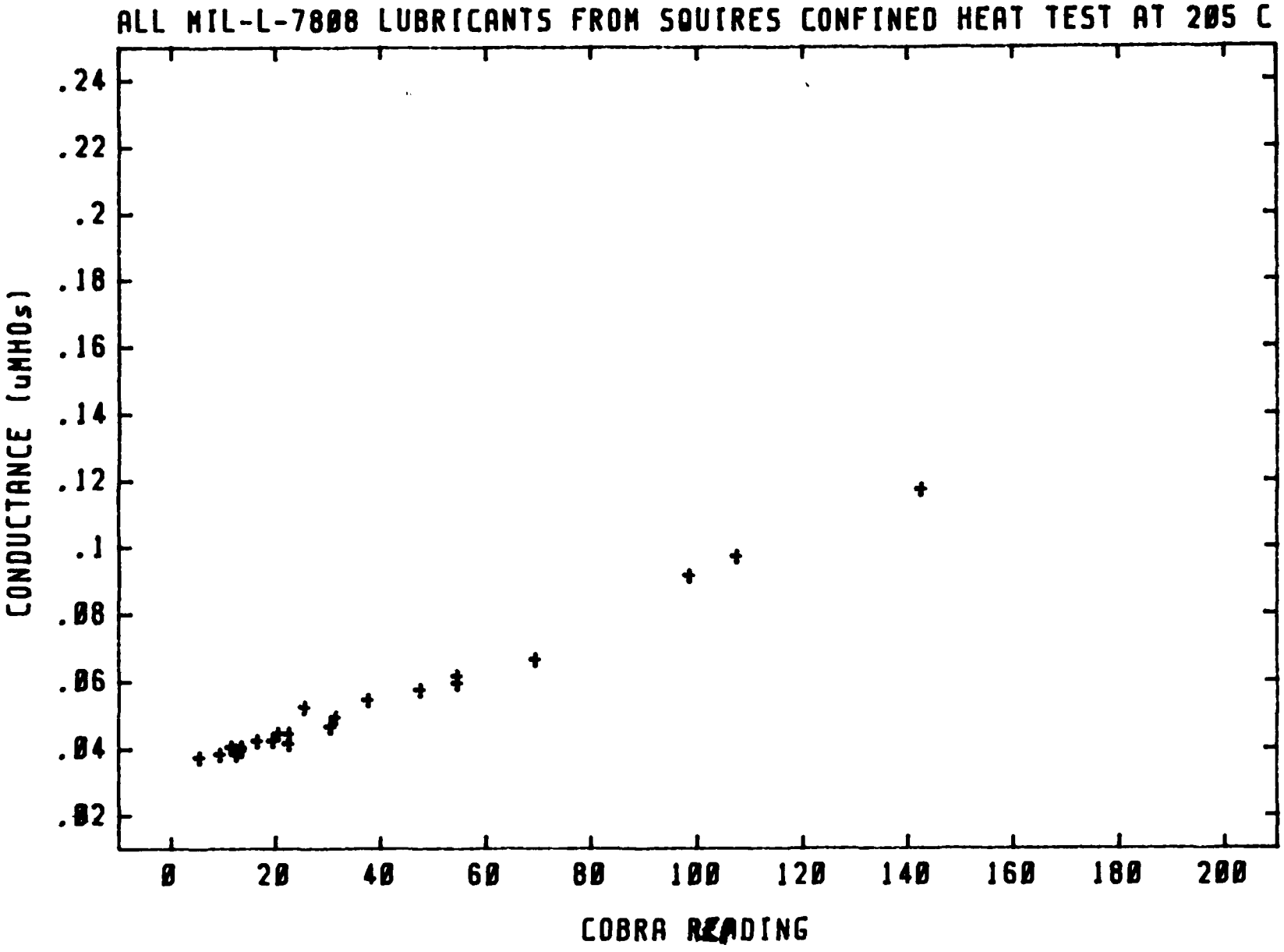


Figure 111, Conductance vs. COBRA Readings for Lubricants from Squires Confined Heat Test at 205°C.

drift and that the actual COBRA readings may have changed somewhat in time since they were not remeasured at the time of the conductance measurements. Despite the relationships found here, it is not claimed that the COBRA readings are equivalent to conductivity but that both methods yield essentially the same type of information, at least for the degraded oils that were measured. While the conductance measurements may offer a greater range of sensitivity and more stable readings relative to the COBRA, the response of both instruments to oxidatively and thermally stressed MIL-L-7808 lubricants show a large formulation dependence.

4. OIL MAINTENANCE TESTER

A device had been recently described[57] that was developed for determination of the degradation level of automotive engine oils. This oil maintenance tester measures the steady state current decay in the lubricant after application of a DC step voltage of 300 volts through nickel electrodes. Changes in the current and current decay parameters were related to the presence of contaminants and charged particles. A device similar to this was constructed in order to determine its usefulness as a lubricant degradation level device for laboratory stressed MIL-L-7808 lubricants. The device itself consisted of a simple electrical loop containing a 300 volt DC power supply, a 100K resistor and a test cell containing the lubricant. The current is determined by using a millivolt recorder to measure the voltage drop across the calibrated resistor. Initial results using a nickel electrode assembly, consisting of two 3 x 1/2 inch plates separated by 1 mm, were inconsistent and non-reproducible. Instead, a YSI model 3417 conductivity cell (Yellow Springs Instrument Co.), containing platinized platinum-iridium electrodes, was used and yielded much more reproducible data although sensitivity was reduced. In addition to the 0.5 second current and

the current decay between 0.5 and 3.5 seconds normally measured[57], the stable current after 12 seconds was also measured. The results of the analyses of the six MIL-L-7808 lubricants stressed in the Squires oxidative test at 190°C are shown in Table 61. In general it is observed that the 0.5 second current increases and the 3 second current decay decreases with stressing time though only slightly and somewhat erratically. The 12 second current however shows a very definite and consistent increase vs. stress time (Figure 112). Furthermore, the latter current shows an excellent correlation with the COBRA readings (Figure 93).

5. CONCLUSIONS

The COBRA device has been evaluated with respect to six MIL-L-7808 lubricants stressed in the Squires oxidative and confined heat tests. The COBRA readings of these stressed lubricants show, in general, a constant rise in value vs. stressing time. When the data is compared at equal values of physical deterioration (TAN and viscosity) for each lubricant, four of the lubricants (O-79-16, O-79-17, O-79-20 and O-82-3) give comparable COBRA readings in the oxidative test while the other two (O-82-2 and O-82-14) show disproportionately large rises in COBRA readings. For the confined heat tested lubricants, only O-82-14 gave unusually large rises in COBRA readings relative to physical property deterioration. Attempts to relate this formulation dependence to the level of peroxides, basestock ester consumption or the contribution of certain known ester basestock degradation products did not reveal a definite relationship. However, analysis of the data of stressed laboratory formulated lubricants containing di-2-ethylhexyl adipate (diester) basestock indicates that this formulation dependence may be related to the presence of large amounts of diesters in O-82-2 and O-82-14.

Two alternative electrochemical devices were evaluated as lubricant

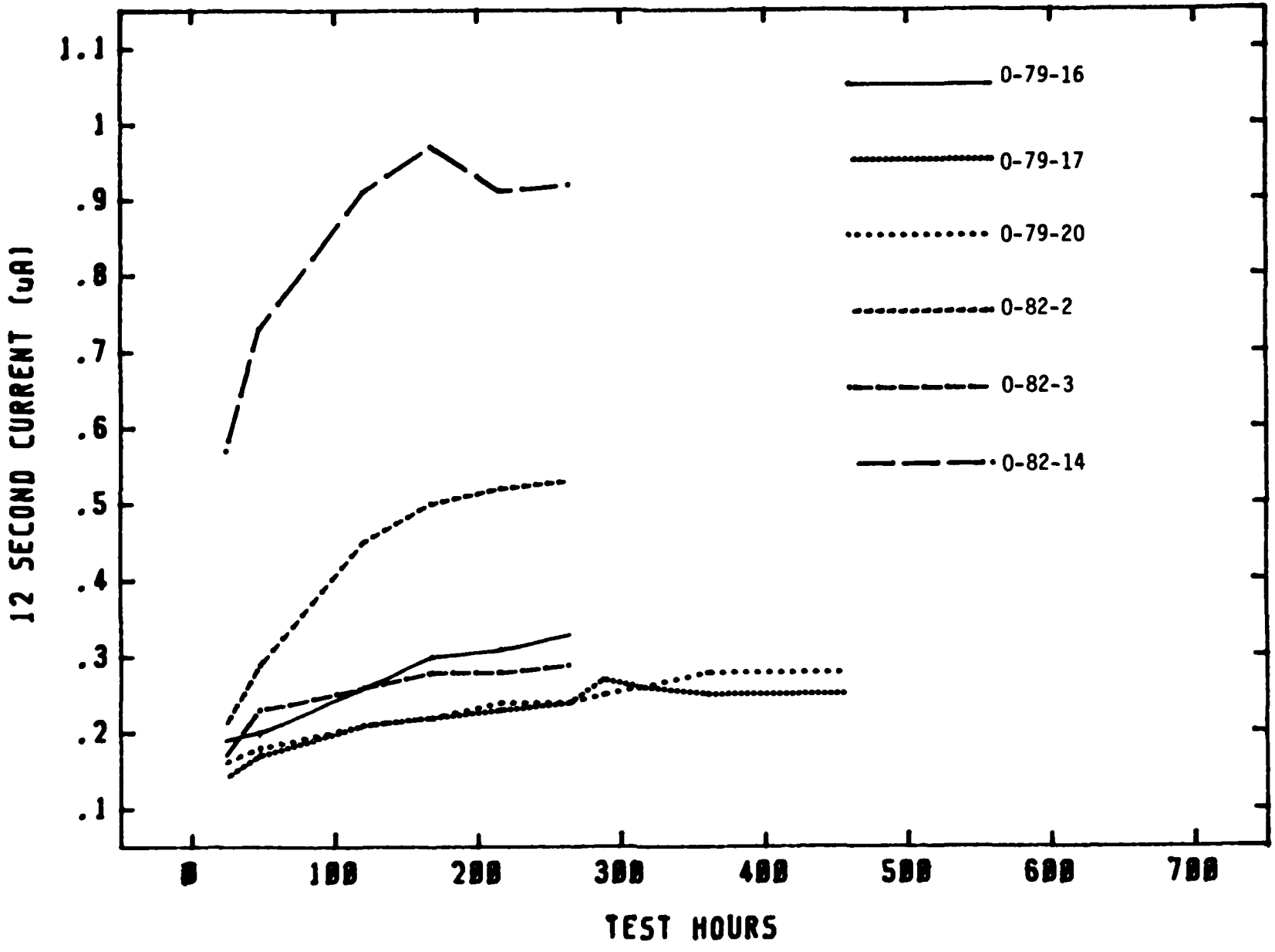


Figure 112. Oil Maintenance Tester 12 Second Current Readings of MIL-L-7808 Lubricants from the Squires Oxidative test at 190°C

TABLE 61

OIL MAINTENANCE TESTER DATA FROM SQUIRES OXIDATIVELY STRESSED LUBRICANTS AT 190°C

0-79-16J

Time (hrs)	1/2 Second Current (μA)	Current Decay (μA)	12 Second Current (μA)	COBRA
24	5.56	4.14	0.19	6
48	5.56	4.14	0.20	20
120	5.56	4.07	0.26	51
168	5.64	4.15	0.30	59
216	5.64	4.09	0.31	85
264	5.72	4.07	0.33	84

0-79-17E

Time (hrs)	1/2 Second Current (μA)	Current Decay (μA)	12 Second Current (μA)	COBRA
24	5.51	4.19	0.14	22
48	5.50	4.12	0.17	32
120	5.56	4.12	0.21	64
168	5.61	4.16	0.22	62
216	5.56	4.12	0.23	74
264	5.61	4.10	0.24	64
288	5.71	4.20	0.27	79
312	5.53	4.06	0.26	88
360	5.56	4.10	0.25	76
457	5.63	4.12	0.25	91

TABLE 61 (CONT'D)

0-79-20A

Time (hrs)	1/2 Second Current (μA)	Current Decay (μA)	12 Second Current (μA)	COBRA
24	5.59	4.19	0.16	22
48	5.58	4.17	0.18	32
120	5.64	4.17	0.21	64
168	5.67	4.20	0.22	62
216	5.59	4.11	0.24	74
264	5.58	4.10	0.24	64
288	5.64	4.16	0.25	79
312	5.67	4.16	0.26	88
360	5.69	4.17	0.28	76
457	5.59	4.10	0.28	91

0-82-2

Time (hrs)	1/2 Second Current (μA)	Current Decay (μA)	12 Second Current (μA)	COBRA
24	5.59	4.20	0.21	37
48	5.61	4.10	0.29	83
120	5.69	4.06	0.45	183
168	5.67	4.15	0.50	187
216	5.61	3.93	0.52	184
264	5.71	3.99	0.53	182

TABLE 61 (CONCLUDED)

0-82-3

Time (hrs)	1/2 Second Current (μA)	Current Decay (μA)	12 Second Current (μA)	COBRA
24	5.46	4.08	0.17	10
48	5.54	4.12	0.23	22
120	5.67	4.20	0.26	50
168	5.67	4.17	0.28	49
216	5.67	4.15	0.28	61
264	5.67	4.14	0.29	79

0-82-14D

Time (hrs)	1/2 Second Current (μA)	Current Decay (μA)	12 Second Current (μA)	COBRA
24	5.69	3.96	0.57	185
48	5.69	3.82	0.73	200
120	5.67	3.66	0.91	200
168	5.67	3.63	0.97	200
216	5.77	3.75	0.91	200
264	5.74	3.72	0.92	200

monitoring devices. A device used to measure the conductance of the lubricants proved to be stable, sensitive and reproducible although it gave the same formulation dependence as the COBRA instrument. An oil maintenance tester, which measures the current decay in an oil after application of a DC step voltage at 300 volts, yielded similar data although it was somewhat less sensitive.

6. FUTURE EFFORT

The effect of condensate return in the Squires oxidative test on COBRA readings for MIL-L-7808 lubricants will be determined. COBRA readings on new classes of stressed lubricants, such as 4 cSt ester lubricants, polyphenyl ethers and perfluoroalkylethers, will be measured. Investigation into the formulation dependence of MIL-L-7808 lubricants on COBRA readings will continue. Initial efforts to isolate and concentrate species from stressed lubricants that produce the COBRA readings by adsorption chromatography has been successful and will be used in conjunction with chromatographic and spectroscopic analysis to identify these components. A device that measures the dielectric breakdown strength of fluids will be investigated as a potential lubricant monitoring device.

SECTION V

LUBRICANT LOAD CARRYING CAPABILITY TEST ASSESSMEMT

1. INTRODUCTION

Three variations of gear test methods are available for determining load carrying capability of lubricants with the basic principles involved in each case being similar. These tests are the FZG test used in Germany, the IAE test used in England, and the Ryder gear test used in the United States. Of the three, the Ryder test is run under the severest conditions, i.e. highest load and speed. The FZG test machine has been chosen by the CEC (Coordinating European Council) for the development of performance tests for lubricants for use as it's standard test rig. A comparison has been made between the IAE and FZG test data obtained under their respective recommended operating conditions. No correlation in test results were observed. When each test is operated under similar conditions, there seems to be good agreement in test results. A problem with this correlation is that the FZG rig is incapable of running at higher loads which are possible with the IAE rig.

Load carrying capacity (LCC) of a test fluid, determined by the Ryder gear test method, is defined as the tooth load, in lb/inch of face width, which results in an average tooth face scuffing of 22.5%. The ASTM test D-1947-83 outlines the procedure for the determination of LCC using the Ryder gear test rig. A set of test gears is run under a constant load and speed for 10 minutes. The test is stopped and the operator examines, through a microscope probe, each face of the 28 tooth test gear for percent area scuffed and records the observation. The load is increased, and the test is

run for 10 minutes and the gear faces are examined again. This process continues until the operator determines that 40% of the tooth surfaces are scuffed. A graph of load versus percent area scuffed is plotted and the load corresponding to 22.5% scuffed area is obtained. The Ryder gear tests have several shortcomings. They are expensive, time consuming, and several tests must be conducted in order to get a representative value of load carrying capacity. These ratings involve a great deal of subjectivity on the part of the operator to determine the percentage of scuffed area of each tooth face. The tests are sensitive to factors which are difficult to control such as gear tooth tip relief, variation in surface finish and hardness, and alignment of gear driveshafts not to mention the operator's skill. It has been noted that the test results are not reproducible between operators and labs.[58] Some of the differences between the various gear tests are listed in Table 62.[59]

TABLE 62

COMPARISON OF RYDER, IAE AND FZG TEST PARAMETERS

Parameters	Ryder	IAE	FZG
Pinion speed, rpm	10K	6K	4.4K
Test oil temp, ^{o}C	74	110	90
Failure criteria	22.5% Scuff	60% Scuff	Wt. loss and wear rate
Time at load, min	10	5	15
Lube system	Jet	Jet	Dip
No. of teeth	28	16	24
Gear width, mm	6.4	4.8	20.1
Tooth tip sliding Velocity, m s^{-1}	13.2	11.8	11.3

The primary failure mode of gears in this type of testing is scuffing. Scuffing is caused by localized welding of the surface of the gear caused by local breakdown of the lubricant boundary layer. The scuffing load has been found to be proportional to the sliding speed at the gear tooth tip and is

related by the function $WN^X=C$ where: W=load on tooth, N=rpm, C=constant, and x=scuffing index. Scuffing load has been found to be proportional to the square root of lubricant viscosity and inversely proportional to the square root of surface roughness. EP additives in the lubricant have helped to increase LCC by forming mono-layers of sacrificial metal compounds when surfaces of the gears reach operating temperatures.

2. LITERATURE SEARCH

There are several reported studies of efforts to correlate results obtained on the Ryder, IAE and FZG test rigs. All have been relatively unsuccessful for various reasons. Benzing reported correlations between the Ryder and the IAE machines.[60] Good correlations have been difficult to obtain since all rigs operate under different loads and speeds. Good correlation of test results using the IAE and FZG machines was obtained when using similar sliding velocities and oil temperatures.[61] The gear geometries in the various tests are also different as well as the gear materials. A correlation of results between the FZG, IAE, and Ryder tests was also reported by Niemann.[59] The scuffing loads of the three test methods were related by a quantity termed "Almen factor". This factor is a function of sliding speed, load, and tooth geometry. Finally, criteria of what constitutes failure with each rig is different. As mentioned earlier, the IAE rig is limited in speed (6000 rpm) and is more appropriate for evaluating the high viscosity oils used in British turboprop gearing. Criteria for failure is a decrease in motor speed with the formation of vapors from the oil. The FZG rig offers promise in that it is a scaled down rig which can be less expensive. It is capable of varied speeds and loads. Failure can be observed visually, as in the case of the Ryder test, or from the weight loss of the gears. Determination of weight loss is difficult since the weight

lost through wear is rather insignificant compared to the total gear weight.

Recent literature indicates other factors which could have influence on the selection of a test alternative to the Ryder apparatus. Researchers in Japan have been very active in studies of the scoring of gear teeth. Terauchi has reported studies of factors influencing the scoring of spur gear teeth including the effects of gear geometry, tooth material, and lubricants on scoring.[62] Voitik and Heerdt have reported the use of a commercially available Falex machine to evaluate gear lubricants.[63] The particular geometry of the test specimens used by the Falex test provides promise for an alternate gear test. The specimens are inexpensive, small, and are assessed by weight loss. Only a small amount of oil is required per test.

3. DEVELOPMENT OF LOAD CARRYING CAPACITY TEST

Development of a repeatable, objective, and inexpensive test method to determine the tribological characteristics of oils is desirable due to the problems and costs of the Ryder, IAE, and FZG gear tests. Several methods including pin-on-disk, pin-on-ring, disk-on-washer, rotating crossed cylinders, the Falex gear simulator, the rolling four ball, and the sliding four ball test can be used for investigating tribological properties. The use of the sliding four ball test to determine the LCC of oils is currently being considered. The major advantage of this test is that the real area of contact between the balls during sliding can be accurately determined resulting in the calculation of average pressure on the oil film in real time. The development of the four ball test for determining LCC is covered in Task V along with development of specification wear test.

4. CONCLUSIONS

Time and experience with gear rig testing (Ryder, IAE and FZG) have shown that gear test rigs do not provide satisfactory test methods for determining

lubricant load carrying capacity. Factors contributing to the problems of gear rig testing include lack of precision due to uncontrollable parameters, supply of identical test gears, and the high and continuing increasing cost of conducting gear rig testing.

The sliding four ball geometry has been used in tribological testing for over forty years. The wealth of papers on the subject provide added assistance in understanding the processes that occur in four ball tests. The simplicity of the test rig and the availability of low cost test balls contribute to make this a very economical test. The objective nature of recording the test results (measuring the scars) combined with the ability to monitor wear in real time are very significant advantages to this method.

5. FUTURE EFFORT

Future work calls for examining closer the repeatability of the tests using four ball geometry. Other oils will be tested such as MIL-L-23699 oils. If time permits, the bulk oil temperature will be varied. Efforts will be made to correlate the results of the four ball tests with Ryder tests results using the Hertzian stresses as the basis for correlation. This includes trying combinations of higher speeds and temperatures in an effort to simulate conditions in the gear tests.

In addition, the Falex gear simulation test will be investigated more closely. This test produces a combination of sliding and rolling of the surfaces being tested to simulate the motion of meshing gear teeth. This method does not have the four ball test's advantage of a constantly increasing real contact area.

SECTION VI

DEVELOPMENT OF SPECIFICATION WEAR TEST

1. INTRODUCTION

This study was directed towards developing a repeatable, objective, and inexpensive test method for determining the tribological characteristics of oils. Several methods including pin-on-disk, pin-on-ring, disk-on-washer, rotating crossed cylinders, the Falex gear simulator, the rolling four ball, and the sliding four ball test can be used for investigating tribological properties.

Historically, researchers have used different types of wear tests to determine the tribological characteristics of lubricants. Begelinger and de Gee[64-67] used a pin-on-ring machine in their investigations of lubrication of concentrated contacts between E-52100 steel members in oils. They stopped the tests when the coefficient of friction reached a steady value (around 0.1). This usually occurred after five minutes although some of their tests ran for up to one hour. They observed three wear regimes (Figure 113); the region of low wear and friction (Region I), the region of low wear and high friction (Region II), and the region of high wear (two orders of magnitude greater) and high friction (Region III). The transition from the region II to I is dependent on lube viscosity and contact pressure. The transition from Region III to II is associated with a particular conjunction temperature. Even at speeds as low as 0.7 mm s^{-1} load carrying capacity was strongly dependent on lubricant viscosity.

Czichos[68-69], who performed tests using both a four ball and a ball on

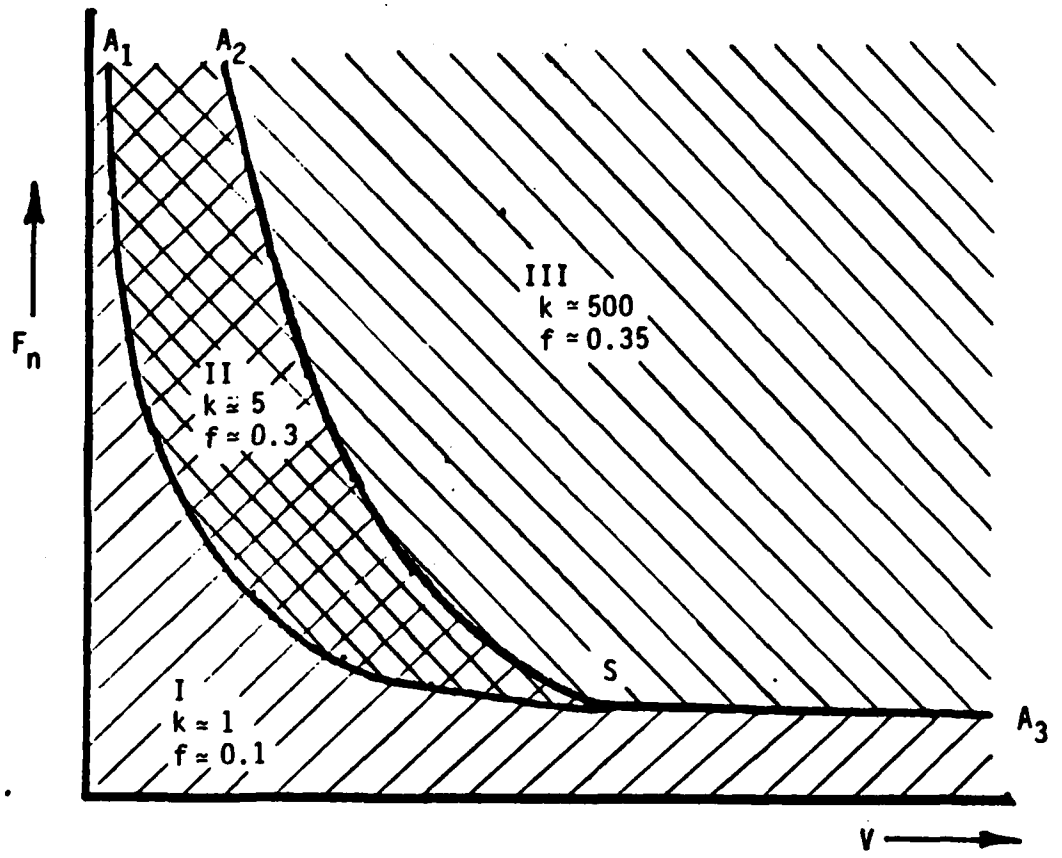


Figure 113. Three Regimes of Friction and Wear (Reproduced from Ref. 67). k is mm^3/Nm; f is Coefficient of Friction

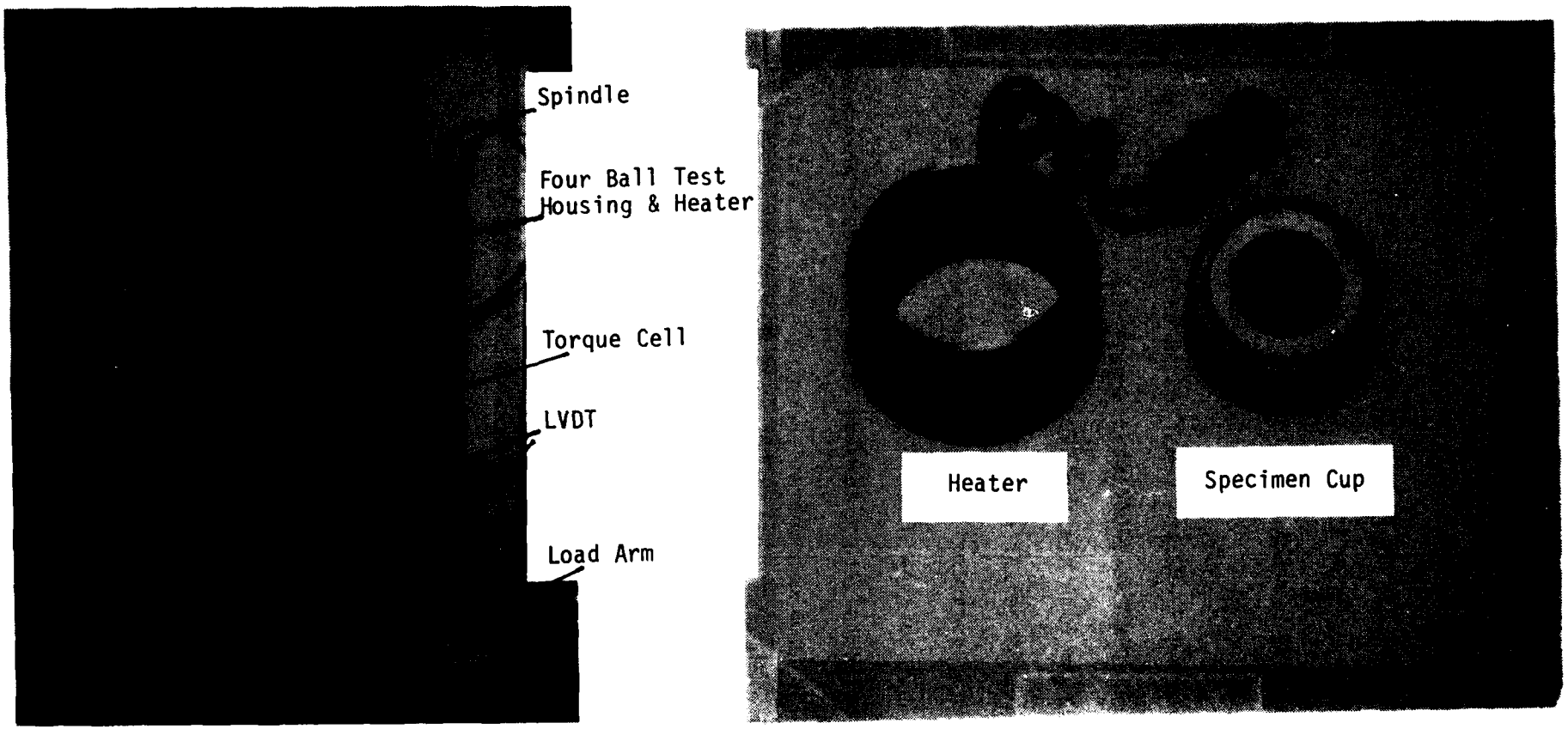
three pegs configuration to investigate the load carrying capacity of oils, observed that there was a combination of speed, load, temperature, and time required for an oil to fail. He used the concept of a "failure surface" to predict lubricant failure. He also noted the presence of three wear regimes similar to Begelinger, et al.[70]

Smith[71] utilized a ball-on-disk machine using castor oil and 52100 steel. He concluded that the frictional force is transmitted through a film of lubricant acting as a plastic solid whose shear strength decreases with increasing temperature. The coefficient of friction was essentially a measure of the temperature rise in this zone. At high sliding speeds (140 cm s^{-1}), the scatter in wear data was large. At lower sliding speeds (17 cm s^{-1}) it was found that the scar diameter on the balls was the same after six hours of sliding as it was after 4 minutes of sliding.

During the course of this study, work was performed to determine the effect of load, speed and lubricant formulation on the tribological characteristics of MIL-L-7808 type lubricants. Wear parameters such as wear scar size and linear variable differential transformer (LVDT) outputs were determined to study the above effects. The use of the sliding four ball test was initially investigated. The major advantage of this test is that the real area of contact between the balls during sliding can be determined, resulting in the determination of average pressure of the oil film.

2. EXPERIMENTAL PROCEDURES

Testing was performed using a four ball test machine shown in Figure 114. Load is applied using dead weights on a lever with a 10:1 ratio. The coefficient of friction was calculated from the torque measured by a load cell attached to the stationary shaft of the machine. Wear of balls during the test was monitored with a spring loaded linear variable differential



(a) (b)

Figure 114. Four Ball Wear Test Machine with LVDT (a), Resistance Heater and Three Balls Specimen Cup (b)

transformer attached to the lever arm. Friction, wear, and temperature data were collected using a data acquisition system based on an IBM PC/XT microcomputer. The system uses a 12-bit analog to digital converter supplied by Data Translation. The signal conditioning was performed, to minimize the effect of noise, by averaging the data. The data was stored on a floppy disk.

ASTM Method D-4172-82 was modified in order to study the tribological characteristics of MIL-L-7808 lubricants. The effect of load and spindle speed on wear scar size and the LVDT output were investigated for developing a wear test that is reliable, easy to perform, inexpensive, repeatable and suitable for incorporating into a lubricant specification wear test.

Seven MIL-L-7808 type oils were used in this work. These were synthetic ester base lubricants with tricresyl phosphate (TCP, antiwear additive) as determined by GC with concentration being shown in Table 63.

TABLE 63

TCP CONCENTRATIONS IN VARIOUS MIL-L-7808 TYPE LUBRICANTS

Fluid Type	TCP(wt.%)
0-72-9	1.5
0-76-1	0.0
0-79-16	2.4
0-79-20	0.5
0-82-2	2.3
0-82-3	0.0
0-82-14	2.2

The ASTM D 4172-82 test procedure was modified and used to perform the sliding four ball wear tests. The sliding between the balls was performed at 145, 245, and 392 N Load and 200, 1200, and 2000 rpm spindle speed to obtain a spectrum of lubricant performance. All the tests were conducted at a bulk lubricant temperature of 75°C. For lubricant 0-79-20, tests were repeated at loads varying from 22 to 400 N and speeds from 200 to 2000 rpm to verify the LVDT data.

Steel balls (AISI E-52100) 1/2 inch in diameter (tolerance $\pm$ 0.00005), case hardened to R_c 64-66, grade 25 (extra polish) were used in the tests. The balls were cleaned using a low boiling point petroleum solvent (pentane, pet. ether, etc.) in an ultrasonic bath and allowed to air dry for about 15 minutes. The balls were covered with 35 cm^3 of the test lubricant. After the test was completed the balls were cleaned in the solvent. The wear scars were measured on the three stationary balls using an optical microscope as per ASTM D 4172-82 test method. This involved measuring the diameter along and normal to the direction of sliding on each of the three stationary balls. The average of the six measurements is reported as the wear scar diameter.

3. RESULTS AND DISCUSSION

In order to develop a methodology for determining the wear characteristics of MIL-L-7808 type lubricants, oil 0-79-20 was extensively investigated. A series of tests were run on this oil using various combinations of load, speed, and duration.

a. Wear Scar Diameter as a Function of Time

Figure 115 shows a plot between wear scar size obtained by measurement and LVDT output with time. The test was run at 245 N load and

1200 rpm spindle speed. The increase in LVDT output with time indicates an increase in wear scar size. Initially the rate of increase is rapid and then the rate decreases with time. The curve levels off at around 10 hours, indicating that the wear scar size increase was negligible and could not be measured after this time. This phenomenon is termed in this report as "leveling-off", and the time it takes to reach this condition is termed "transition period". Since LVDT output and measurement of wear scar diameter give similar results it was concluded that the LVDT data is reliable and can be used to accurately determine the transition period.

b. Transition Period

In order to verify the above behavior, tests were run at other loads and speeds. Figures 116 and 117 show the variation of LVDT output with time, at different loads and speeds, respectively, for the same oil. Here, again the same trend can be noted where the LVDT output leveled off after approximately 10 hours. In addition, the transition period was found to be a function of load and speed and decreases as load or speed or both increase. This behavior may not be justified by a hydrodynamic action because of the relatively high coefficient of friction (0.1) but rather could be explained based on the chemical reaction of TCP or materials acting similar to TCP with the metal surface in the contact zone. Furthermore, of the oils tested, those without TCP did not exhibit similar behavior.

c. Precision

The repeatability of the wear scar size was verified by running several four ball tests at 1200 rpm and 75^{o}C for one hour and at various loads. In each test a new set of four balls was used. Even though one hour is not sufficient for the scar size to level off (see Figure 115), the data shows the scar diameter is reproducible. The effect of load on scar size is

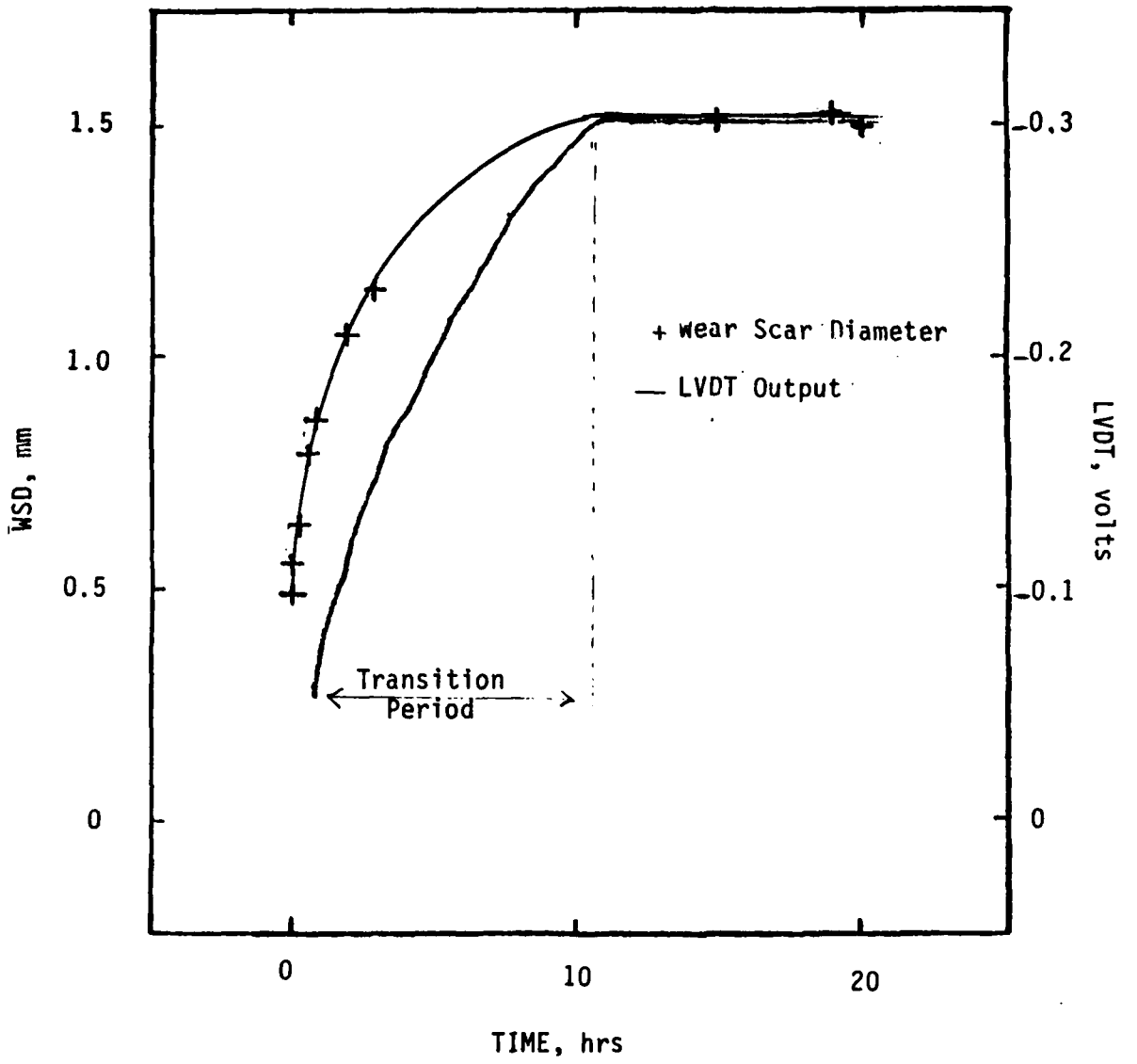


Figure 115. Variation of Wear Scar Diameter and LVDT Output with Time in O-79-20 Oil. Test Conditions: Load 245 N; Speed 1200 RPM

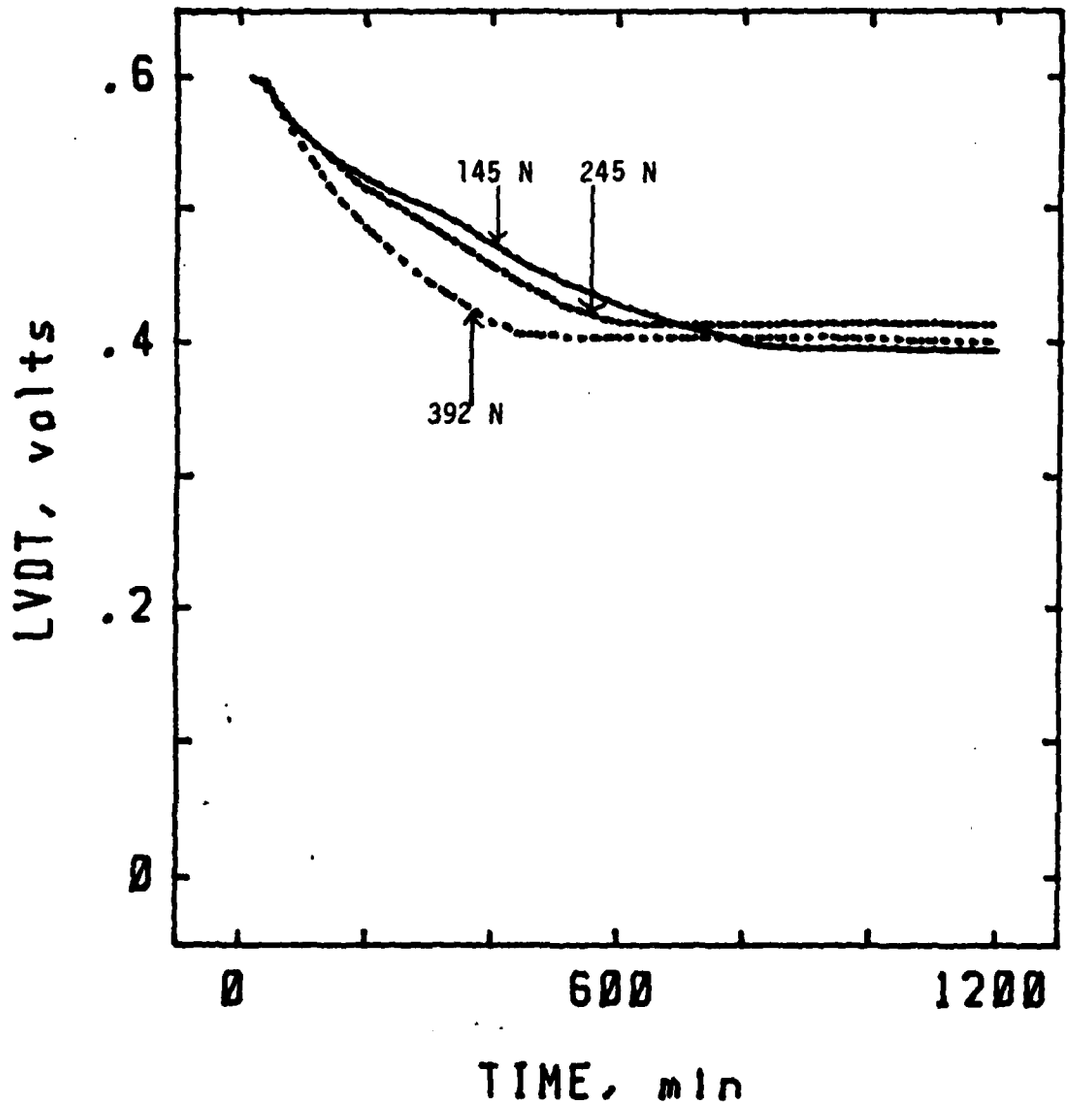


Figure 116. Variation of LVDT Output with Time for O-79-20 Oil at 1200 RPM and Loads of 145, 245, and 392 N

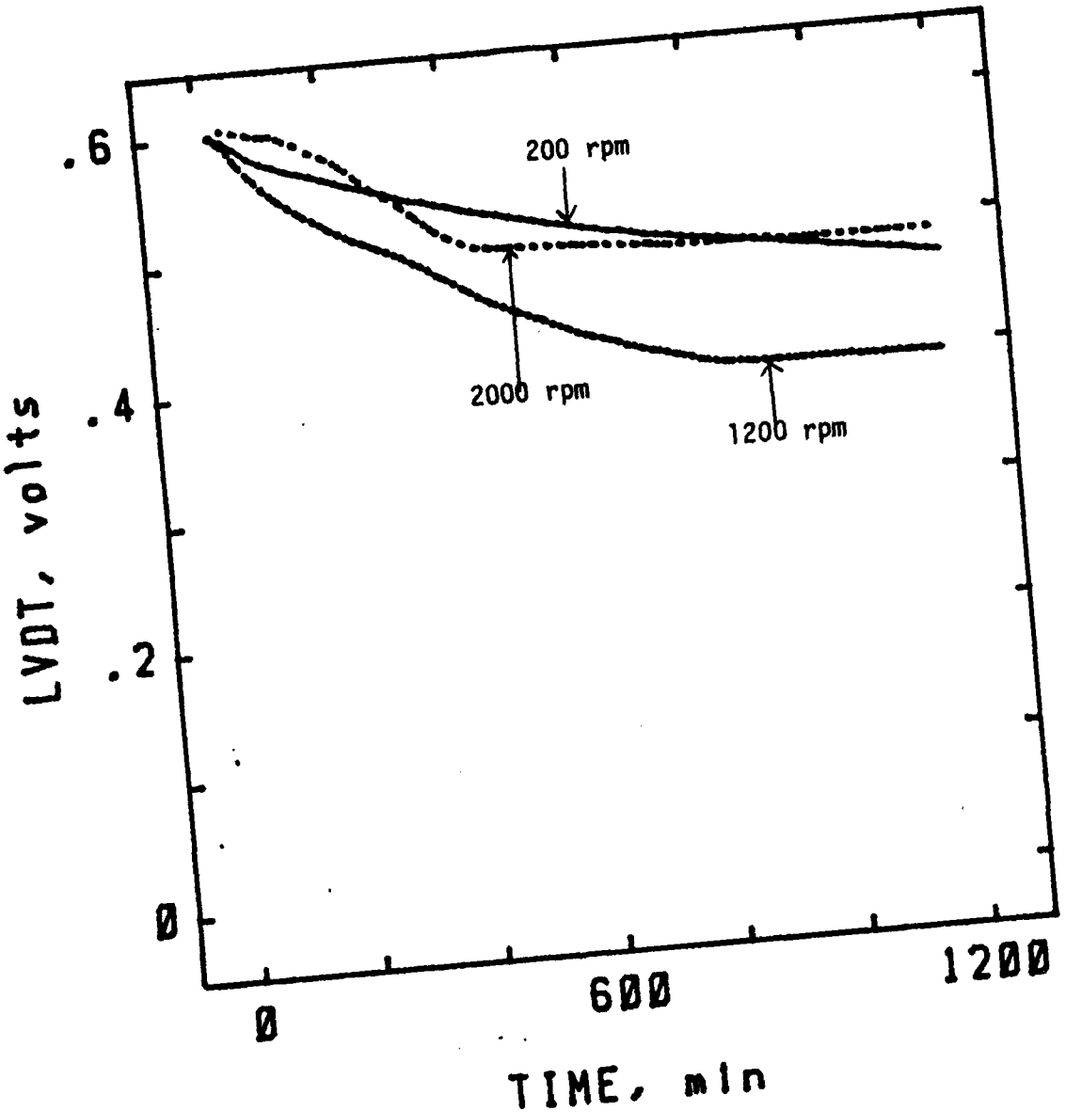


Figure 117. Variation of LVDT Output with Time for 0-79-20 Oil at 145 N Load and Spindle Speeds of 200, 1200, and 2000 RPM

more pronounced below 110 N than above this value (Figure 118).

d. Effect of Spindle Speed

The variation of wear scar size with spindle speed is shown in Figure 119. The scar size as a function of speed is shown for two different loads (145 and 245 N). The two curves overlap indicating the scar size diameter for both loads is similar over the speed range tested. The data shows that the wear scar diameter does not change over spindle speeds of 1000 to 1400 rpm. However, it tends to significantly decrease with increasing speed above 1400 rpm. No conclusive remark can be at this time for the small scar diameter produced at 200 rpm. More data is needed to establish the shape of the curve below 1000 rpm.

e. Testing with Other Lubricants

In order to learn if the above wear behavior is peculiar to O-79-20, or if this is the general behavior for formulated lubricants, six other oils (Table 63) were tested. Of the total seven oils studied, two oils did not contain TCP. The oils with TCP exhibited behavior similar to O-79-20 oil. The tests were run for 20 hours at three speeds and three loads only, unlike the previous case of O-79-20 where roughly forty different tests were run. The wear scar size as a function of load and speed is shown in Figures 120 and 121. These figures show that after the transition period, scar size does not vary significantly with either load or speed.

Boundary film lubrication is the most likely action occurring as a result of TCP chemical reactivity in the contact zone. TCP reacts with the metal surface under high temperature to form a deformable boundary film that can be replenished quickly to prevent metal to metal contact.

In the case of oils without TCP a different behavior was observed. Wear in oil O-76-1, as shown by the LVDT output curve, did not level off with

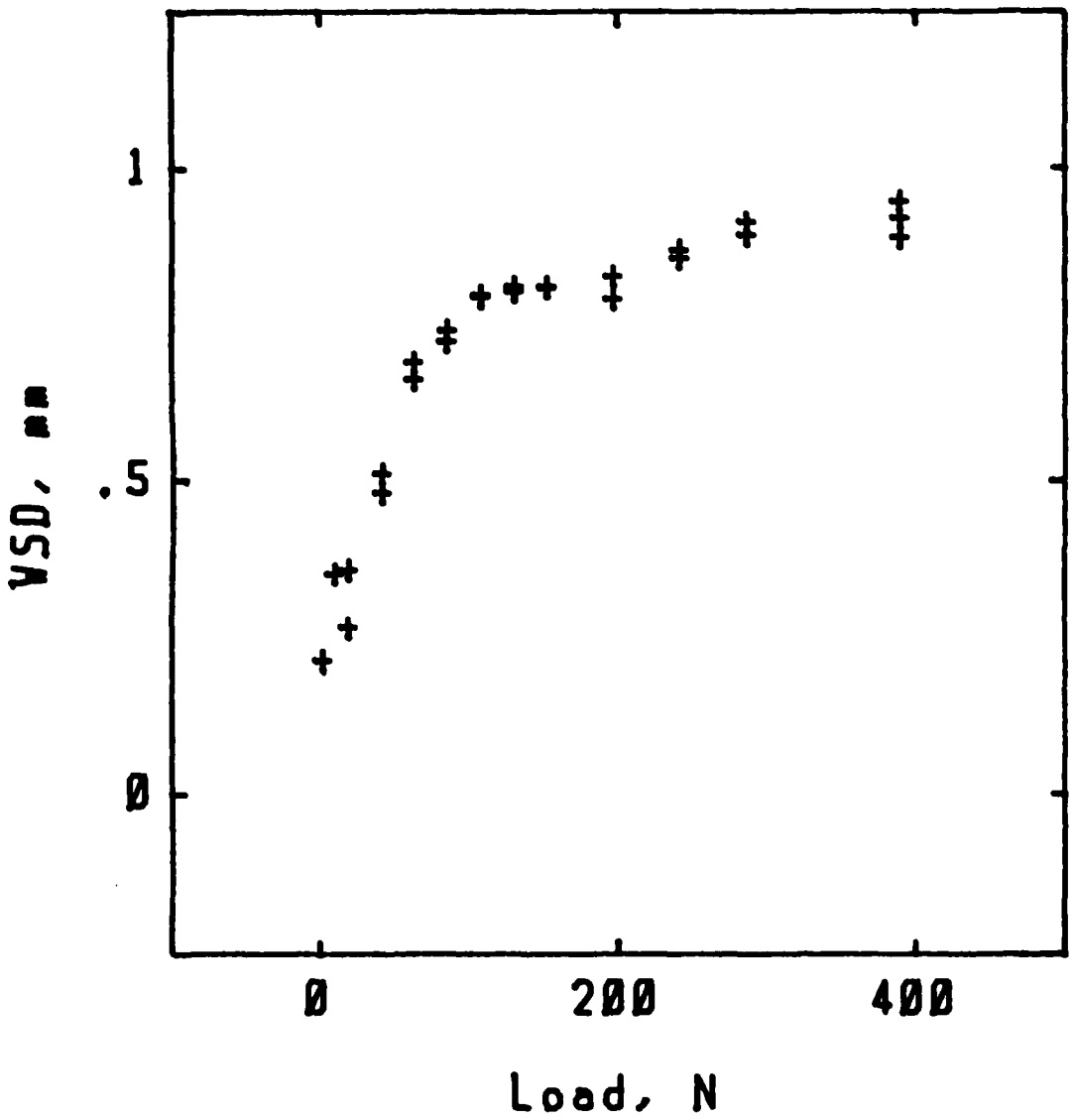


Figure 118. Effect of Load on Wear Scar Diameter (WSD) in O-79-20, at One Speed, 1200 RPM, for a Test Duration of One Hour

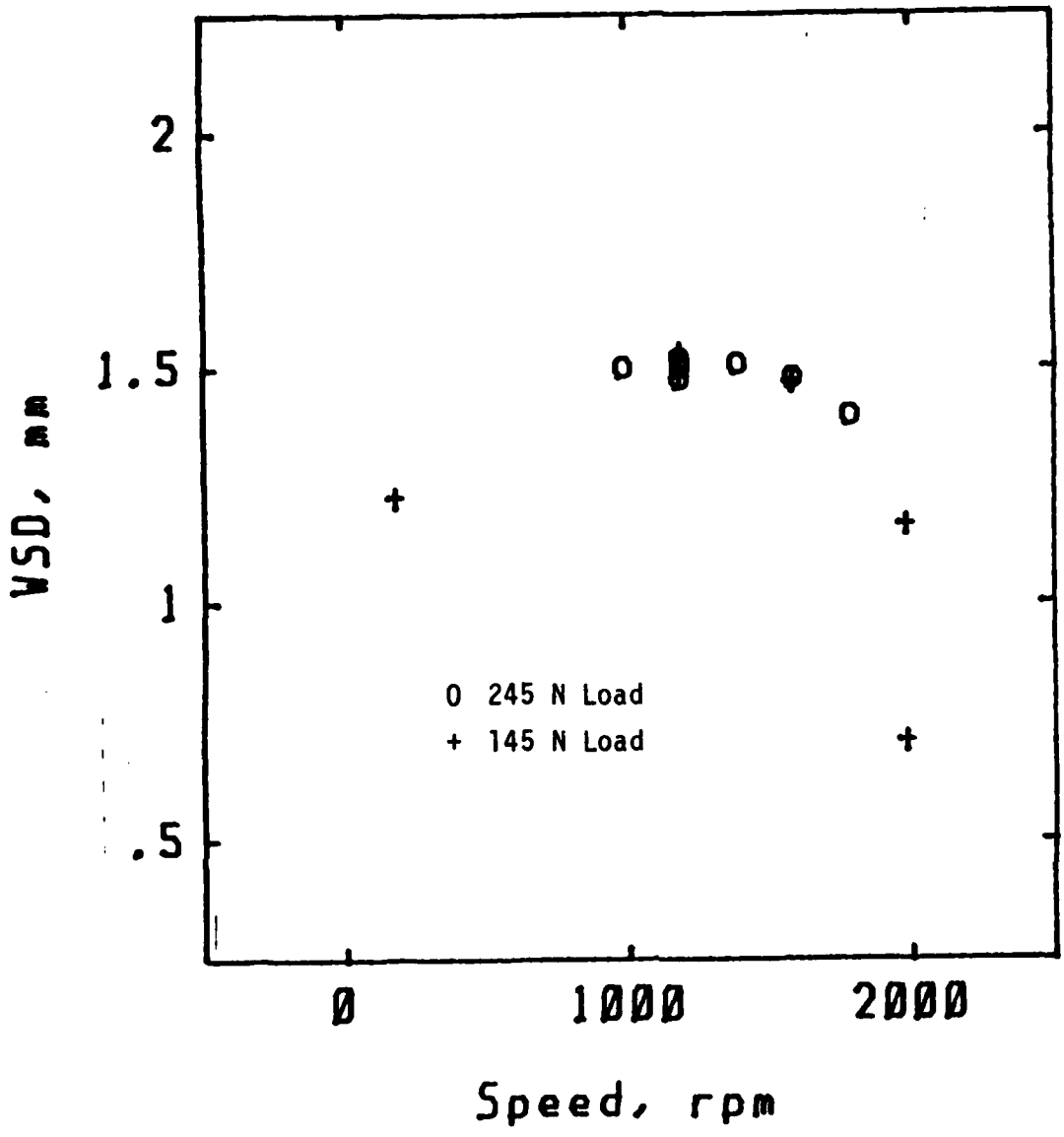


Figure 119. Effect of Spindle Speed on Wear Scar Diameter (WSD) in Oil 0-79-20, for 20 Hour Test Duration

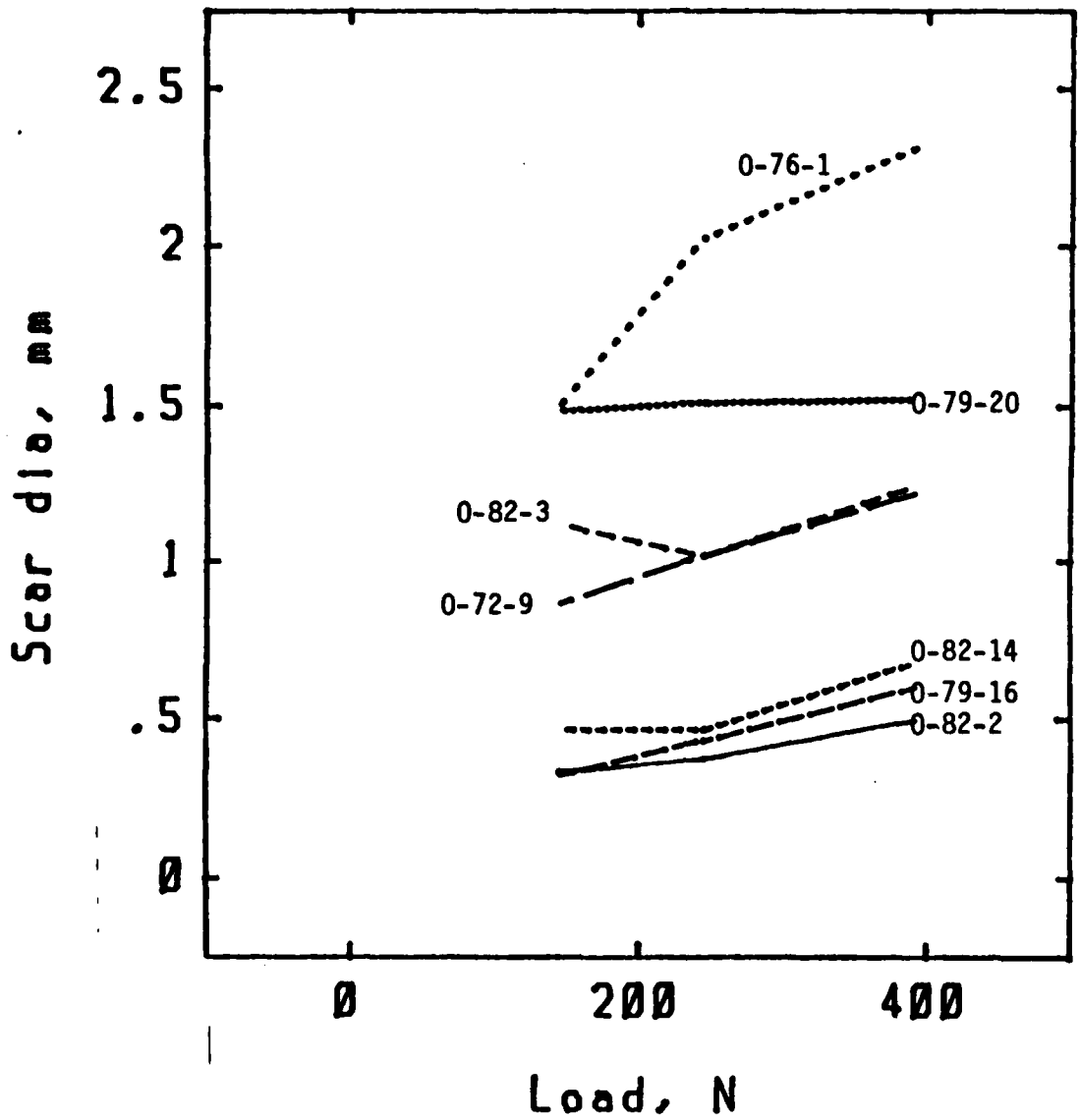


Figure 120. Effect of Load on Final Scar Diameter for Various MIL-L-7808 Type Lubricants

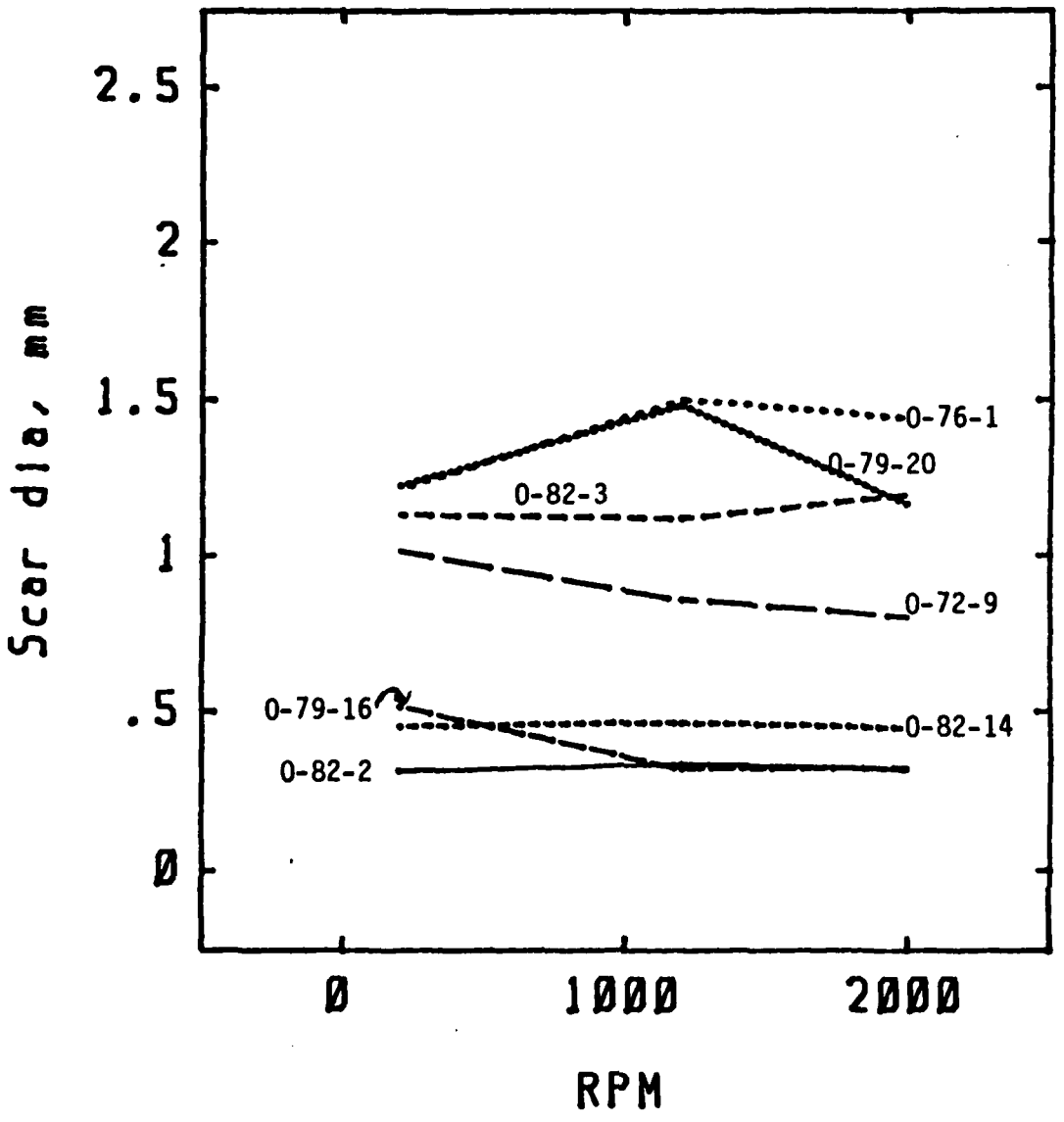


Figure 121. Effect of Spindle Speed on Final Scar Diameter for Various MIL-L-7808 Type Lubricants

time (Figure 122). The tests were extended for longer periods in an effort to determine if the leveling off phenomenon would occur. After 68 hours, leveling in LVDT output was not attained. Therefore the wear rate did not significantly decrease during this testing period.

Table 64 shows the wear scar diameters determined for all MIL-L-7808 lubricants after 20 test hours. Percent TCP as determined by GC is also shown. Oils containing approximately 2% TCP produced scar size diameter smaller than scars produced by oils having 0 to 0.5% TCP. However, oils with little or no TCP may contain other antiwear additives. Even though a limited number of lubricants were tested, the wear scar diameter data which is generated as the average result of five independent tests shows that lubricants containing 2% TCP had significantly reduced the wear scar size and that TCP plays an important role in the leveling off phenomenon when wear scar size is studied as a function of load and speed. Therefore, the resulting wear scar size produced after the transition period may be used to evaluate wear characteristics of lubricants in the test geometry and under these test conditions.

TABLE 64
VARIATION OF WEAR SCAR SIZE WITH OIL

Oil	TCP, %Wt	Scar Size *,mm (after 20 test hours)
0-72-9	1.5	1.02 ± 0.20
0-76-1	0.0	1.70 ± 0.50
0-79-16	2.4	0.45 ± 0.15
0-79-20	0.5	1.34 ± 0.29
0-82-2	2.3	0.37 ± 0.09
0-82-3	0.0	1.14 ± 0.11
0-82-14	2.2	0.50 ± 0.11

* Average of 5 tests

Obviously, an oil producing a smaller scar would be rated higher as compared

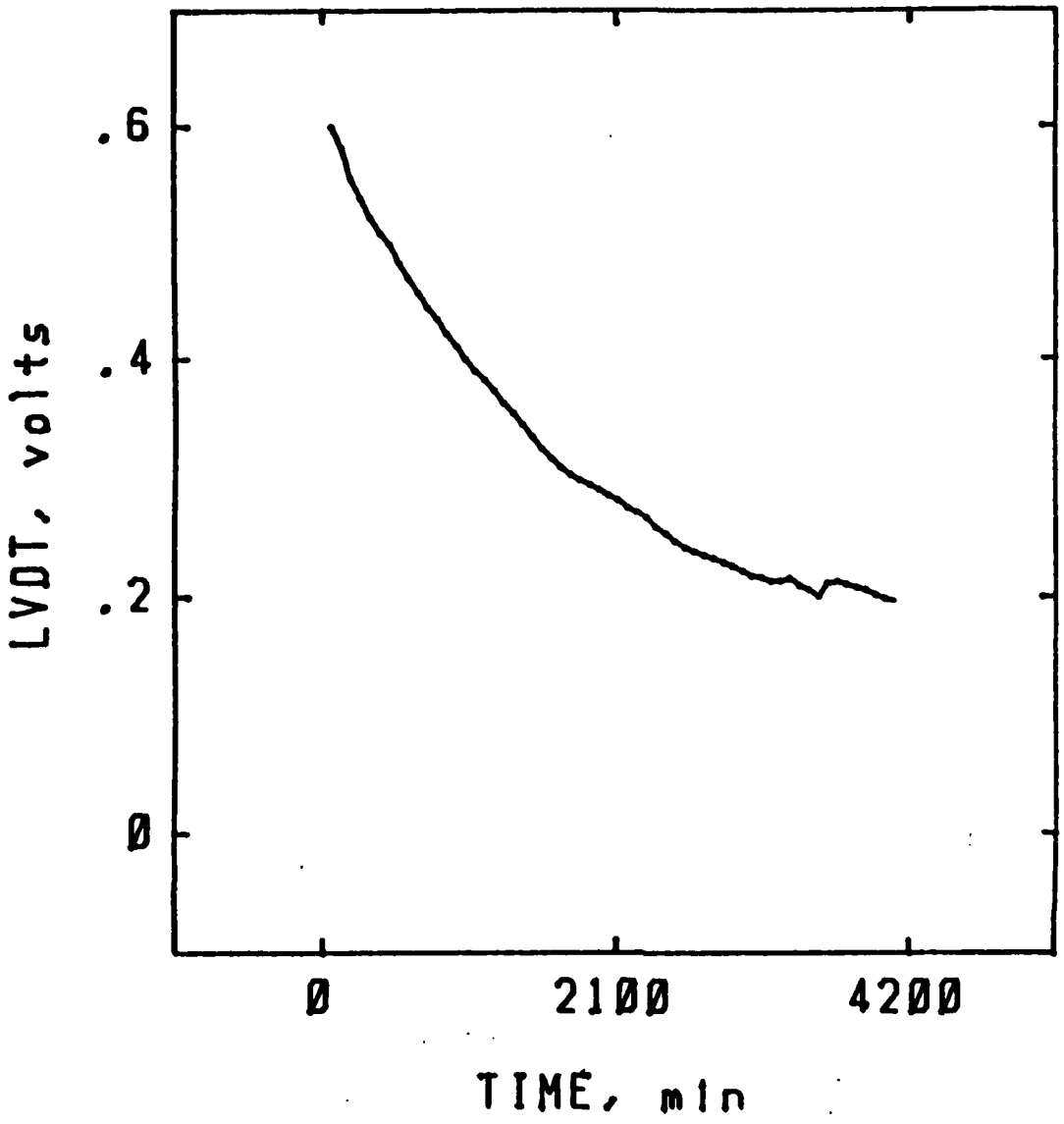


Figure 122. LVDT Output During a 68 Hour Test of O-76-1, Using 145 N Load and 2000 RPM Spindle Speed

to the one producing larger scar. Lubricants evaluated on this basis may produce results that could be used for ranking of lubricants based on their wear prevention characteristics.

4. CONCLUSIONS

Based on the above work, the following conclusions can be drawn regarding the use of the four ball test as a method to determine the tribological properties of lubricants.

a. Under certain load and speed conditions, the wear scar size produced after the transition period was significantly smaller for oils containing an appreciable amount of TCP (2%).

b. The concentration of TCP plays a major role in limiting the wear scar size.

c. The size of wear scar after the transition period is almost independent of the load and speed for oils containing higher concentrations of TCP.

d. The wear scar diameters produced after the transition periods may be used to help rank the oils with respect to their wear characteristics.

e. The use of the four ball test for evaluating lubricants is warranted since it is inexpensive and the results are reproducible.

5. FUTURE EFFORT

Future studies with the four ball wear test will include testing to determine the precision of the method. Since small increases in wear scar size may not be totally detected by LVDT, ferrographic analysis will be performed on oils after the transition periods in order to determine the extent of wear with time in the region of "constant wear". Statistical analysis will be conducted to determine a reliable parameter to evaluate oils by this type of test. If proven reliable, this method will be recommended

for inclusion as a specification wear test. Other MIL-L-7808 and MIL-L-23699 type oils will be tested. If possible, modifications will be made to the test equipment to enable testing oils at much higher temperatures. Attempts will be made to develop an analytical model of the wear process in an effort to predict wear based on operating conditions.

SECTION VII

DEVELOPMENT OF LUBE STORAGE AND RETRIEVAL SYSTEM

The purpose of this effort is to implement a software system on the Zenith Z-100 microcomputer for storage, retrieval and correlation of MIL-L-7808 lubricant qualification data. Of the software available for the Z-100 microcomputer, dBASE II (Ashton-Tate) was easily the most suitable data base management software for this application. Its general data file handling capabilities plus its programmability allows for the development of a menu driven software package that should be user friendly. The software programs that were written for this application allow the user to:

(1) Enter new records

(2) Correct or add to old records

(3) Conditionally search data

(4) Print reports

(5) Print selected data

At the present time data for six lubricants selected by AFWAL/POSL have been entered into the computer. Format of the oil data sheet obtained from the microcomputer storage is shown in Appendix C.

Software programs for the storage and retrieval of contractually generated lubricant data including the Squires oxidative and confined heat test data, foaming data and coking data. The software programs developed for these data sets are similar in function to those described above for the lubricant qualification data.

As part of future work, more lubricant qualification data sets will be entered as they become available. A manual for using the lubricant

qualification software will be written. Development of any additional features for existing software packages will be done as needed.

SECTION VIII

RULLER DEVELOPMENT

1. INTRODUCTION

While lubricating and cooling the oil-wetted components of the turbine engine, the lubricating oil experiences various environmental stresses which cause the oil's basestock to undergo thermal and oxidative degradation. As long as the lubricant is adequately protected by an antioxidant system, the oxidative degradation of the oil's basestock and the changes in the lubricating oil's properties are minimal. Since the antioxidants are depleted with engine operating time, they eventually become ineffective allowing large changes to occur in the physical properties of the lubricant's basestock. As the physical properties change, the useful life of the lubricant ends resulting in excessive component wear and eventual failure of the engine. The length of engine operating time from the time a lubricant is sampled until the large changes in the basestock's physical properties occur is referred to as the "Remaining Useful Life" of the lubricant (Figure 123).

To ensure that a lubricant is not used past the end of its useful life, the Air Force uses scheduled oil changes. Because the scheduled oil changes are inherently conservative, lubricants with remaining useful lives (RUL) are discarded. Therefore, the ability to predict the RUL of lubricants would eliminate the need for scheduled oil changes providing savings in material and labor costs to the Air Force.

However, the only tests currently available to the Air Force for determining the RUL of a lubricant are long term stability tests. The analytical techniques used to detect changes in the physical properties (viscosity, total acid number, color, etc.) of a lubricant are incapable of

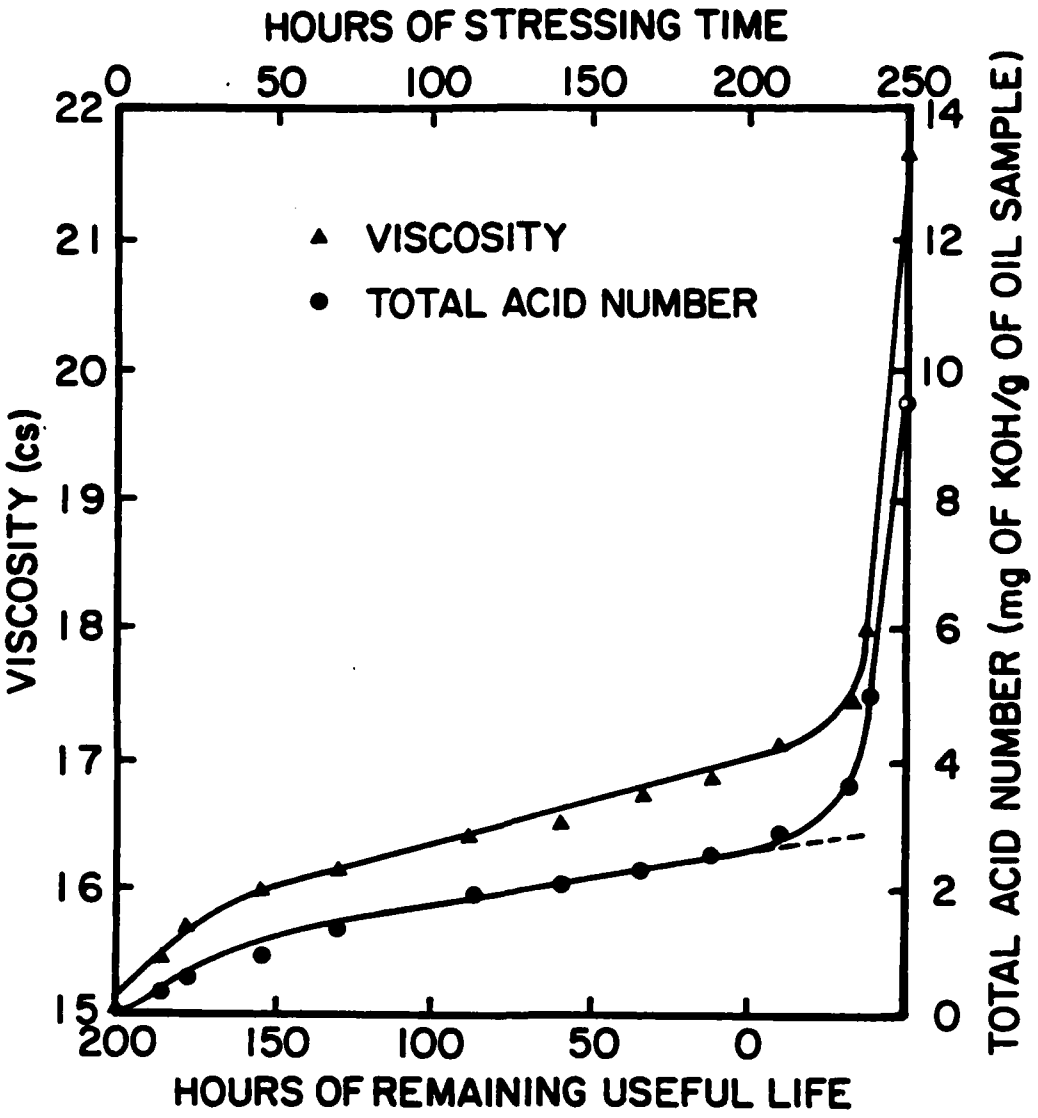


Figure 123. Determination of Useful Life by Plotting Physical Properties Versus Stressing Time for Long Term Stability Test

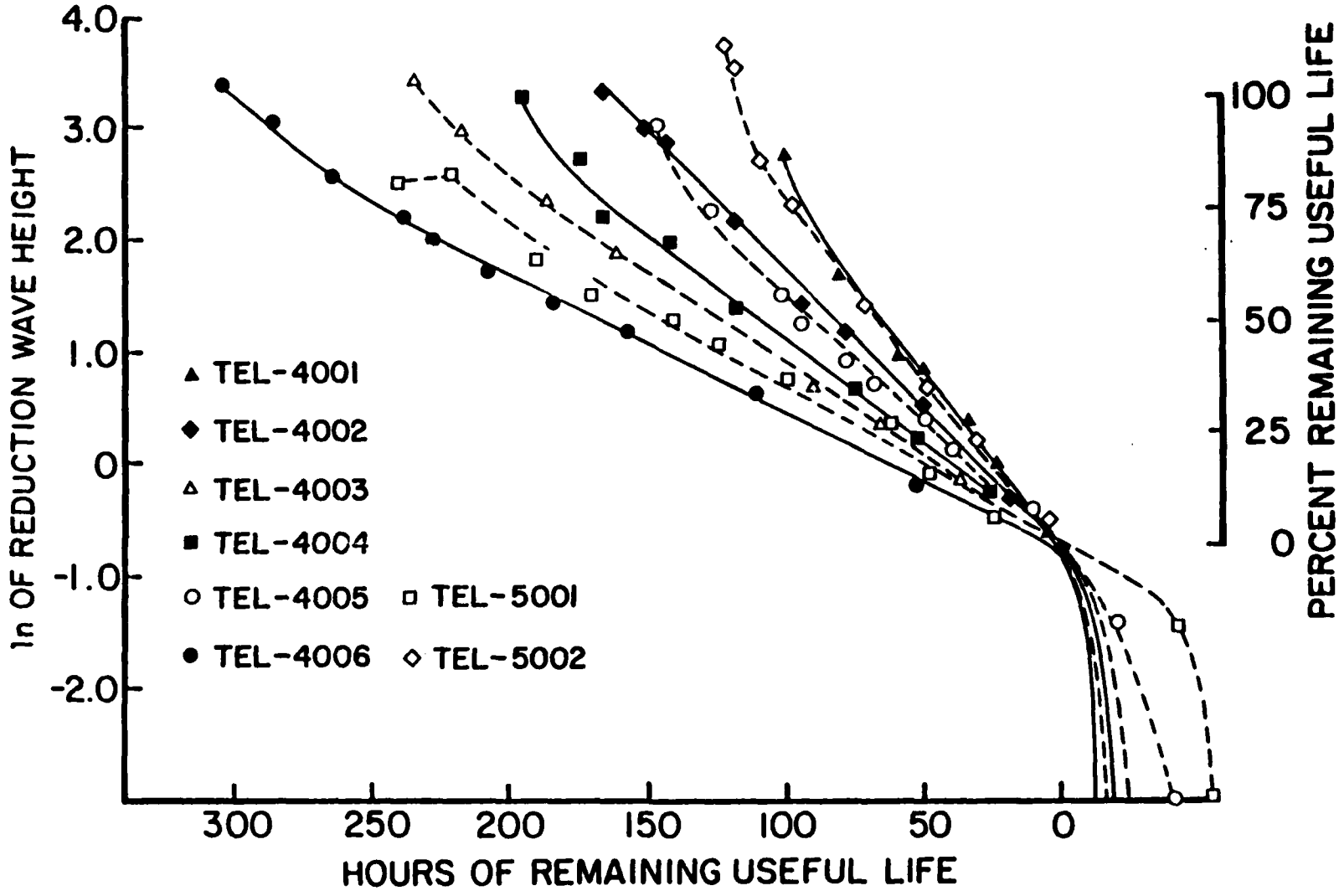


Figure 124. Plots of the ln of the Reduction Wave Height and Percent Remaining Useful Life of the RCV Technique Versus Hours of Remaining Useful Life at 370°F for the Fresh and Stressed MIL-L-7808 Oils

determining the lubricant's RUL.

Therefore, the University of Dayton Research Institute conducted research to develop techniques (Table 65) capable of determining the RUL of different lubricants(Reference 74). The RUL determining capabilities of numerous analytical techniques were evaluated using fresh and stressed (Federal Test Method Standard 791B Method 5307.1 at 370° and 392°F) MIL-L-7808 oil samples. In addition to determining the RUL of a lubricant, the remaining useful life of a lubricant evaluation rig (RULLER) candidate must be low cost, rapid, and base-level in operation and in interpretation of the results. The techniques developed for evaluation as a RULLER candidate could be categorized into three main groups (Table 65) and were ranked for development into a RULLER in the following order: voltammetric > thermal stressing > chemical stressing. The experimental parameters used to rank the potential of each analytical technique for development into a RULLER candidate are listed in (Table 66).

Of the techniques studied during this investigation, the cyclic voltammetric (CV) techniques are the lowest in cost, easiest to operate, and require the shortest analytical time (Table 66). The CV techniques also produce the most accurate and precise RUL determinations for fresh and used MIL-L-7808J oil samples. Examples of the reductive-CV (RCV) result versus RUL plots are shown in Figure 124 for the different MIL-L-7808 oils stressed at 370°F. The CV techniques can also be used to identify the type of antioxidants used in the MIL-L-7808 oil formulation, differentiate fresh or slightly stressed oil samples from moderately stressed oil samples, and estimate the total concentration of hydroperoxides in the oil sample.

Therefore, the CV techniques, in particular the RCV technique, were determined to be the best candidates for development into a RULLER.

To increase the potential of the RCV technique for development into a

TABLE 65

SUMMARY OF ANALYTICAL TECHNIQUES WITH RUL EVALUATION CAPABILITIES

Type of Technique	Specific Technique	Basis of Technique	Oil Property Measured by Technique
Voltammetric	Voltammetry-reduction only Scan from +1.0V to 0.0V	Quantify generated antioxidant species and oil's potential to generate new antioxidant species	Measures concentration of antioxidant species thought to be responsible for lubricant's oxidative stability.
	Cyclic Voltammetry Scan between 0.0V and +1.0V	Detect and approximate concentration of original antioxidant and generated antioxidant species	Estimates degree of antioxidant depletion
	Scan between 0.0V and and -1.0V	Approximate concentration of hydroperoxide	Estimates degree of lubricant degradation
Thermal Stressing	High Pressure-Differential Scanning Calorimetry (HP-DSC)	Uses thermal-oxidative stressing to rapidly degrade oil samples then quantifies energy and time of exothermic reaction that occurs at the end of the oil's induction period	Measures total oxidative stability of lubricant. Energy of exothermic reaction may also be related to oxidative stability
	High Pressure-Differential Thermal Analyzer(HP-DTA)	Uses thermal-oxidative stressing to rapidly degrade oil samples then detects time of exothermic reaction that occurs at the end of the oil's induction period	Measures total oxidative stability of lubricant
Chemical Stressing	Colorimetric Method	Uses cumene hydroperoxide to deplete antioxidant species and then detects decoloration of reaction system that occurs at the end of the oil's induction period.	Measures hydroperoxide decomposing capacity of antioxidant species
	Modified Ford Method	Uses generated free radicals to deplete antioxidant species and then detects rapid pressure decrease of reaction system that occur's at the end of the oil's induction period.	Measures radical trapping capacity of antioxidant species

TABLE 66

COMPARISON OF THE ANALYTICAL TECHNIQUES' POTENTIALS FOR DEVELOPMENT INTO A RULLER

Type of Technique	Advantages	Disadvantages
Voltammetric	- Analysis time: 2 to 10 seconds - Easy to operate - Low cost - less than $2000 - Obtain supplemental information to aid in prediction of lubricant life	- Requires sample dilution - Relationship between lubricant life and technique not fully established - Dependent on antioxidants used
Thermal Stressing	- No sample preparation - Relationship between lubricant life and technique established for MIL-L-7808 oils by other researchers - DTA: easy to operate low cost-less than $3000	- Analysis Time - 1 to 20 minutes - DSC: Hard to operate : High cost-greater than $15-20,000
Chemical Stressing	- Low cost-less than $3000 - Relationship between lubricant life and technique established for various lubricants by other researchers	- Requires extensive sample preparation - Requires use of unstable, toxic chemicals - Hard to operate - Analysis time - 1 to 20 minutes - Degree of oil degradation affects results
Other Methods	- Low cost-less than $3000	- Unable to predict lubricant life
Feasible Methods	- Used by other researchers in long and short term oxidation tests	- Offer no apparent advantage over thermal stressing techniques

RULLER, an Apple IIe microcomputer with associated hardware and software was used to control the voltammograph, acquire the voltammetric data, and then calculate the percent RUL of the oil sample. The Apple IIe data management system made the RCV technique a "one-step, push-button" technique. In the present setup of the RCV-Apple IIe data management system, the total analysis time (the time from when the start button is pushed until the percent RUL of the oil sample is printed) is approximately 30 seconds. Thus, the incorporation of a data management system makes the RCV technique rapid and base-level in operation and in interpretation of the results.

To further evaluate the RCV technique for development into a RULLER candidate, the RCV technique was used to determine the RUL of used MIL-L-23699 oil samples taken from abnormally operating turbine engines. The RCV result versus flight time plots shown in Figure 125 indicate that when the engine is operating normally (low Fe concentrations in Figure 125), the RUL of the used oil samples reach a steady state value or decrease slowly with engine operation time. However, when severe wear begins (large increases in Fe concentrations in Figure 125), the RUL of the oil samples decrease rapidly prior to engine failure. Similar results were obtained for a series of used MIL-L-7808 oil samples taken from an abnormally operating engine (Figure 126). Thus, the RCV results can be used to detect abnormally operating engines to supplement the SOAP of the Air Force.

In contrast to the RCV result versus flight time plots for the used MIL-L-23699 oils in Figure 125 and for the used MIL-L-7808 oils in Figure 126, the RCV plots of the used MIL-L-23699 oils in Figure 126 indicate that the oil samples have 0% RUL several hours prior to the initiation of severe wear. Therefore, it appears that the severe wear modes experienced by the turbine engines in Figure 126 were a result of the MIL-L-23699 lubricating

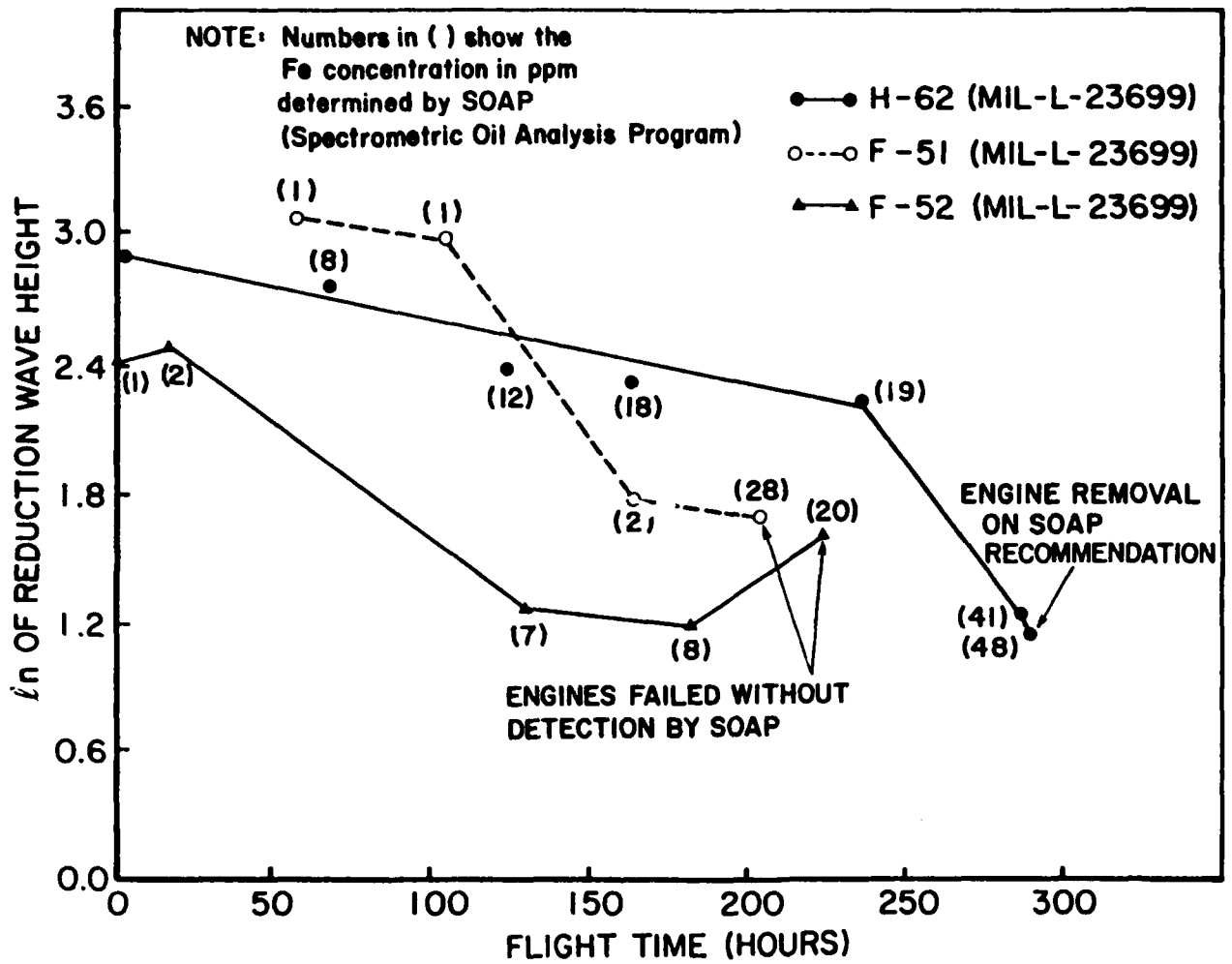


Figure 125. Plots of the ln of the Reduction Wave Height and the Fe Concentration (ppm) Versus Flight Time (Hours) for Used MIL-L-23699 Oil Samples

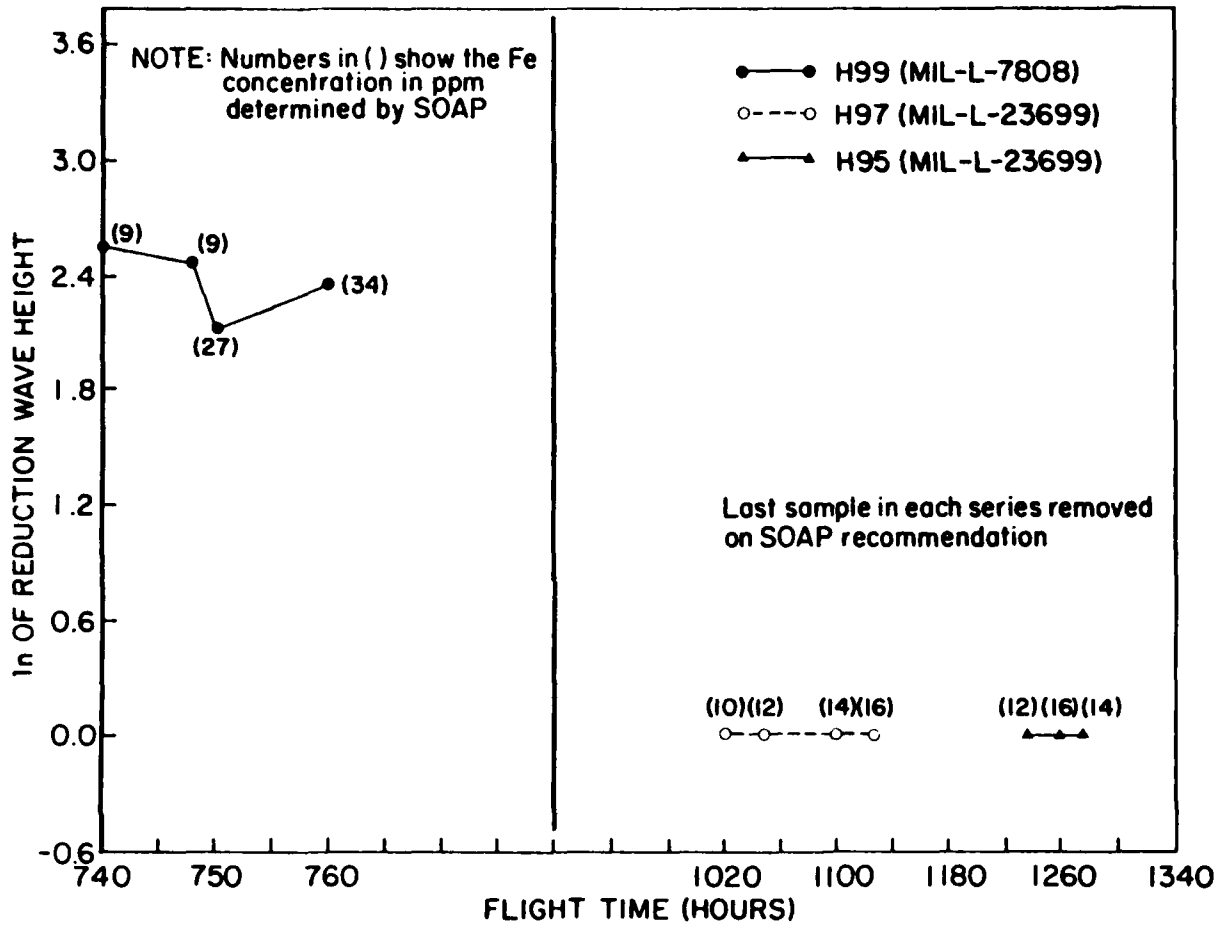


Figure 126. Plots of the ln of the Reduction Wave Height and the Fe Concentration (ppm) Versus Flight Time (Hours) for Used MIL-L-7808 and MIL-L-23699 Oil Samples

oils being used past the ends of their useful lives. Used MIL-L-7808 oil samples with 0% RUL were not observed during the investigation.

The results of the RULLER candidate based on RCV indicate that a RULLER could be used to significantly reduce the number of the engines that fail without prior detection by the Air Force SOAP as well as eliminating the need for scheduled oil changes. In fact, the results of the RCV candidate indicate that the scheduled oil change intervals are incorrect for these particular MIL-L-23699 oil systems resulting in severe component wear caused by degraded lubricating oil. Therefore, a RULLER could be used to increase fleet reliability and decrease labor and equipment costs by lowering the number of engines that fail without SOAP detection, by lowering the number of engines experiencing severe wear modes caused by lubricant degradation, and by lowering the number of premature oil changes which discard lubricating oils with RUL.

Although the previous investigation showed that the RCV technique with a data management system was suitable for development into a RULLER, the experimental parameters of the RCV technique were not optimized for use by the Air Force and the bulkiness of the data management system limited the suitability of the RCV technique for development into a RULLER. Also, only preliminary RUL results were obtained for the authentic used MIL-L-7808 and MIL-L-23699 oil samples.

Therefore, a follow-on effort is being conducted to develop a RULLER candidate from the RCV technique. The first part of the research is concentrating on optimizing the experimental parameters of the RCV technique, on optimizing the RUL determinations of the RCV technique for authentic used MIL-L-7808 and MIL-L-23699 oil samples, and on miniaturizing the data management system. The second part of the research will be concerned with

field testing the developed RULLER candidate.

The work reported herein discusses the preliminary results of the research to optimize the experimental parameters of the RCV technique and to optimize the data acquisition and handling techniques of the data management system.

2. EXPERIMENTAL

a. Instrumentation

(1) CV-1B Voltammograph

The initial reductive-cyclic voltammetry studies were performed on a CV-1B Voltammetry Electronics Control Module [Bioanalytical Systems, Inc. (BAS), West Lafayette, Indiana]. The auxiliary electrode was a platinum wire electrode and the reported potentials were referenced to a RE-1 Ag/AgC1 reference electrode (BAS). The working electrode was a glassy carbon (GCE) voltammetry electrode (BAS).

(2) Microcomputer System

The microcomputer system used in Task VII was an Apple IIe microcomputer (Apple Computer, Inc., Cupertino, California). The output of the CV-1B module was converted to digital form by a 16 channel, 12 byte analog/digital converter, Model AI13 (Interactive Structures, Malvern, PA), which was interfaced with the Apple IIe microcomputer. The sampling rate was controlled by a Speed-Demon card (Microcomputer Technologies, Santa Monica, California). The reductive-cyclic voltammograms and the results of the data analyses were displayed on the Apple IIe monitor and printed on a µ92 Microline Printer (Okidata, Mt. Laurel, New Jersey).

b. Supplies

(1) Chemicals

The pyridazine (97% purity) was obtained from Aldrich Chemical Corporation, Milwaukee, Wisconsin. The acetone (ACS certified) and lithium perchlorate [$LiClO_4$ (ACS certified)] were obtained from Fisher Scientific, Cincinnati, Ohio.

(2) Antioxidants

The generic samples of the N-phenyl-α-naphthylamine (PANA) and dioctyl diphenylamine (DODPA) antioxidants were obtained from AFWAL/POSL.

(3) Lubricating Oils

The fresh MIL-L-7808 lubricating oils (TEL-4001 and TEL-4004) were obtained from AFWAL/POSL.

The stressed MIL-L-7808 lubricating oils (TEL-4001 and TEL-4004) were prepared during the previous contract (Reference 74) using Federal Test Method Standard No. 791B Method 5307.1 at a test temperature of 370°F. Oil samples (30-40 ml) were withdrawn at 16-48 hour intervals to establish a well defined useful life for each MIL-L-7808 lubricating oil. Each withdrawn oil sample was characterized by COBRA, viscosity (40°C), and total acid number (ASTM Method 664) measurements and by magnesium concentrations (atomic emission spectrometry).

The values obtained for the COBRA, viscosity, and total acid number measurements and the Mg concentrations were then plotted versus stressing time to determine the useful lives of the MIL-L-7808 oils at 370°F as shown in Figure 123.

(4) Polyester/Polyethylene Sample Tubes

The sample container used in this research was a 3-ml tube and is manufactured by Nalgene Company, Rochester, N.Y. The tube is sealed on

one end and is made from a two-ply composite film, a polyester outer layer and a meltable polyethylene inside layer.

The heat sealer used in this research is also manufactured by Nalgene. The jaws of the heat sealer are spring-loaded and serrated to produce a seal in 2 to 3 seconds. The operating temperature of 177°C is well below the autoignition temperature of acetone (537°C).

The dialyzer tubing closures used in this research are one piece polypropylene clamps with a 37 mm gripping width manufactured by Spectrum Medical Industries, Los Angeles, California.

c. Sealing Procedure of Sample Tubes

The appropriate amount of voltammetric solution (3 ml) is pipetted into the tube through its open end. The open end of the tube is then heat sealed for 2-3 seconds with the ribbed edge of the tube laying parallel with the jaws and the ribbed closed end of the tube perpendicular to the jaws.

When ready for analysis the sealed end of the tube is cut off and the appropriate amount of oil sample (50 µl) is pipetted into the tube through the reopened end. The dialyzer tubing closure is then used to form a liquid tight closure of the tube so that the oil sample can be dissolved in the voltammetric solution through agitation (hand shaken). To obtain the liquid tight closure, the tube must be laid in the dialyzer tubing closure so that the ribbed closed end of the tube is perpendicular to the jaws of the closure and the ribbed edge of the tube is parallel with the jaws.

The dialyzer tubing closure is opened and removed from the tube. The tube is then opened and the electrode system inserted through the opened end into the solution for RCV analysis. After analysis the solution is immediately disposed of or the tube is resealed for later disposal.

d. Reductive-Cyclic Voltammetric Technique

All of the RCV analyses were performed on a 50 µl oil sample diluted with 3 ml of acetone containing 0.05M $LiClO_4$ and 375 pm of pyridazine. The RCV voltammograms were produced by cycling in a voltage range of -0.2 V to 1.2 V at voltage scan rates of 1.0 to 20.0 V/sec. The reduction linear sweep voltammograms were produced by applying a potential of 1.1 V to the working electrode for 0-5 seconds and decreasing the potential of the working electrode at a scan rate of 1 V/sec.

e. Systems to Perform the RCV Technique

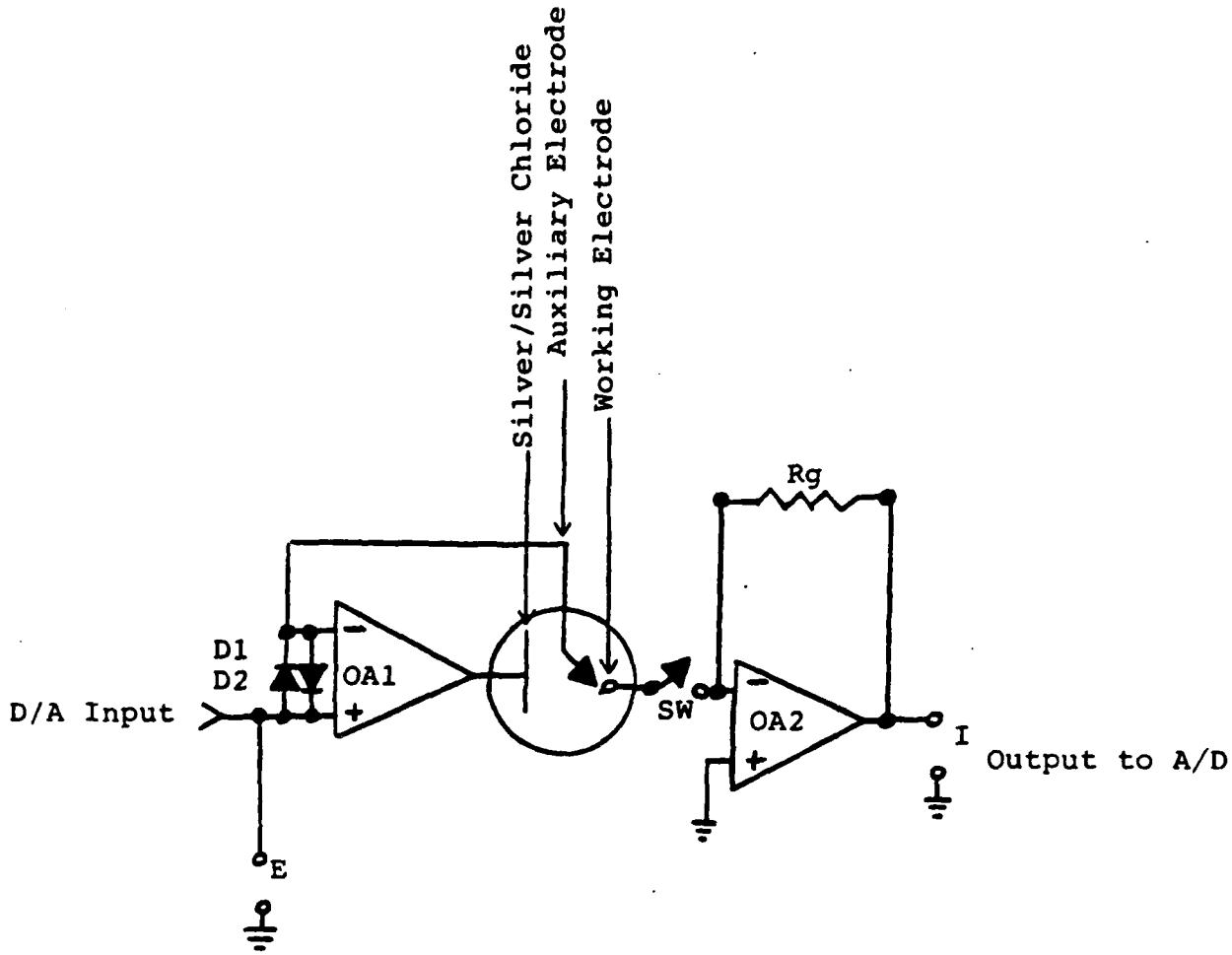
(1) CV-1B Voltammograph/Apple IIe Microcomputer System

To record the RCV voltammograms on the CV-1B Voltammograph/Apple IIe microcomputer system, the Apple IIe microcomputer was programmed to obtain 3000 samples of the CV-1B output at rates of 0.88 to 8.8 milliseconds per sample. The signal of the oxidation wave was nullified by having the computer set its gain to zero. The computer recorded data was obtained by pushing the "A" key of the Apple IIe keyboard. The Apple IIe was programmed to simultaneously start the data acquisition procedure and close the scan switch (start scanning) of the CV-1B Module when the "A" key was pushed. The data acquisition was performed for eleven cycles at scan rates of 1.0 to 10.0 V/sec. The maximum height of each reduction wave was calculated and printed out on the µ92 Microline Printer. The computer program used to control the data acquisition and data handling of the Apple IIe microcomputer system was written in Basic language and is listed in Appendix D.

(2) Single Board Voltammograph/Apple IIe Microcomputer System

(a) Single Board Voltammograph

The schematic of the developed single board voltammograph is depicted in Figure 127. The design of the single board voltammograph was based on the CV-1B voltammograph and on single board voltammographs described



SW = Working Electrode Switch
OA1, OA2 = Op Amp 1 and 2 on LF347N
R_g = Gain Control Selected for Specific Application
D1, D2 = Signal Diodes

Figure 127. Schematic of Single Board Voltammograph

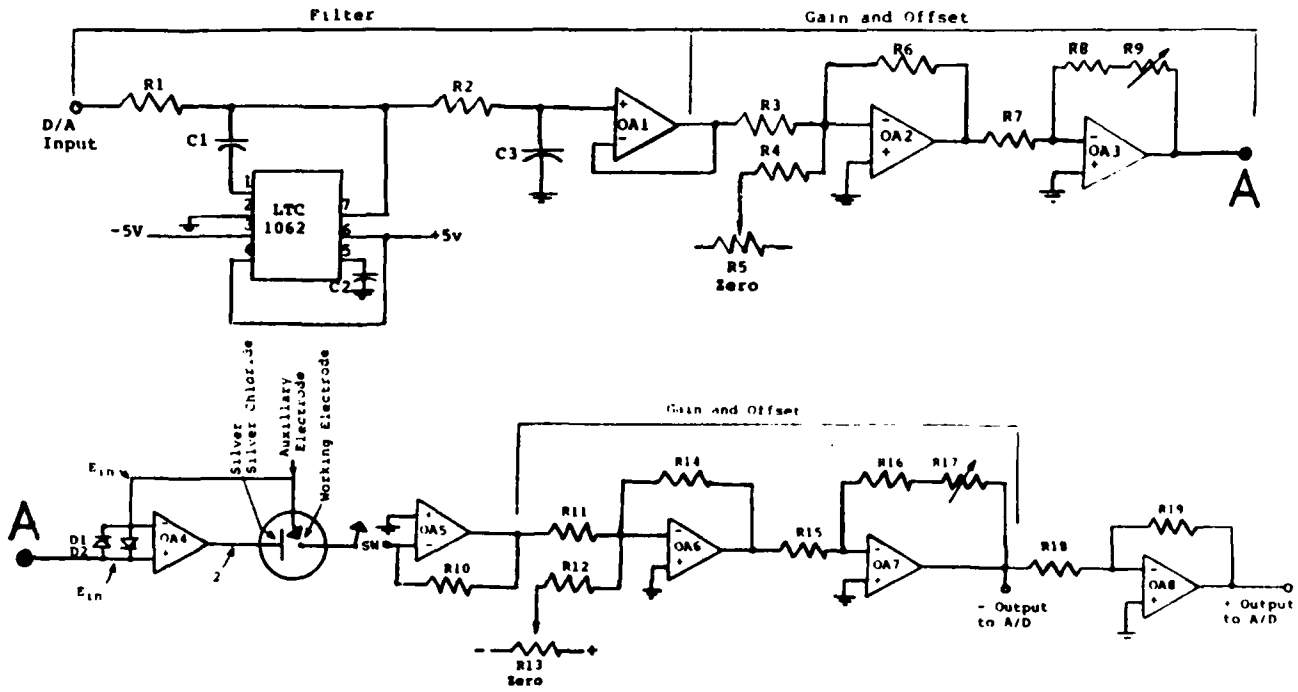
in the literature (References 75-77). The analog signal from the Apple IIe microcomputer is applied at the D/A input (Figure 127) and the output of the working electrode is applied at the A/D output (Figure 127) of the single board voltammograph. The working electrode switch (SW in Figure 127) is controlled by a relay which is driven by a TTL signal from the Apple IIe microcomputer.

(b) Active Filtering and Gain Control Systems of the Single Board Voltammograph

To provide the single board voltammograph with independently variable maximum and minimum voltage scan limits, the components designated gain and offset in Figure 128 were added to the single board voltammograph. To provide the single board voltammograph with an active filtered input, the timing clock and associated electronics (designated filter in Figure 128) were added to the single board voltammograph. To obtain the oxidation or reduction waves of the cyclic voltammograms, negative and positive outputs, respectively, were added to the single board voltammograph as shown in Figure 128.

(c) Apple IIe Microcomputer Hardware

The voltage scan rate, voltage scan range, and output of the single board voltammograph were managed and displayed by the Apple IIe microcomputer. The analog output of the working electrode (applied to A/D output in Figure 128) was converted to digital form by a 16 channel, 12 bit analog/digital converter, Model AI13, which was interfaced with the Apple IIe microcomputer. The output of the Apple IIe microcomputer used to control the voltage ramp of the single board voltammograph (applied at D/A input in Figure 128) was converted from digital to analog form by a 8 channel, 12 bit digital/analog converter, Model Apple Digital to Analog Converter Board



OA1 - OA8	= OP AMP1 - OP AMP7 ON LF 347N
SW	= Working Electrode Switch
LTC 1062	= Filter Chip
D1, D2	= Signal Diodes
C1	= .1uf
C2	= 250 pf
C3	= 150 pf
R1	= 16.2 KΩ
R2	= 1 MΩ
R3, R11	= 10 K
R4, R12	= 100 R
R5, R13	= 10 K
R6, R14	= 10 K
R7, R15	= 10 K
R8, R16	= 9090
R9, R17	= 2 K
R18, R19	= 10 K

Figure 128. Schematic of Single Board Voltammograph Equipped with Active Filtering and Gain Control Systems

(Tecmar Inc., Cleveland, Ohio), which was interfaced with the Apple IIe microcomputer. The TTL signal used to control the relay of the working electrode switch (SW in Figure 128) was produced by the game paddle port of the Apple IIe microcomputer. The reductive-cyclic voltammograms and results of the data analyses were displayed on the Apple IIe monitor and printed on a 92 Microline Printer.

(d) Apple IIe Microcomputer Software

The computer program used to control the voltage ramp of the single board voltammograph and to sample the output of the working electrode was written in a machine code due to the speed requirements of the RCV technique. The machine code is listed in Appendix D and a flow diagram of the machine code program is listed in Figure 129.

As the flow diagram in Figure 129 delineates, the machine code program consists of two loops. The first loop increases the voltage ramp of the single board voltammograph by 5 mV increments, delays a few milliseconds, and then checks the voltage of the ramp after the 5 mV increment. If the voltage of the ramp is below the maximum limit of the ramp, the program returns to the first statement of the loop to continue the incremental increasing of the voltage ramp. Thus, the first loop performs the oxidation sweep of the cyclic voltammetric analysis.

Once the voltage of the ramp reaches the maximum limit of the ramp, the program proceeds to the first statement of the second loop which decreases the voltage ramp of the single board voltammograph by 5 mV decrements. After each 5 mV decrement, the output of the working electrode is sampled and the obtained data is stored after a few millisecond delay. Thus, the second loop performs the reduction sweep of the cyclic voltammetric analysis and records the data points produced by the reduction wave.

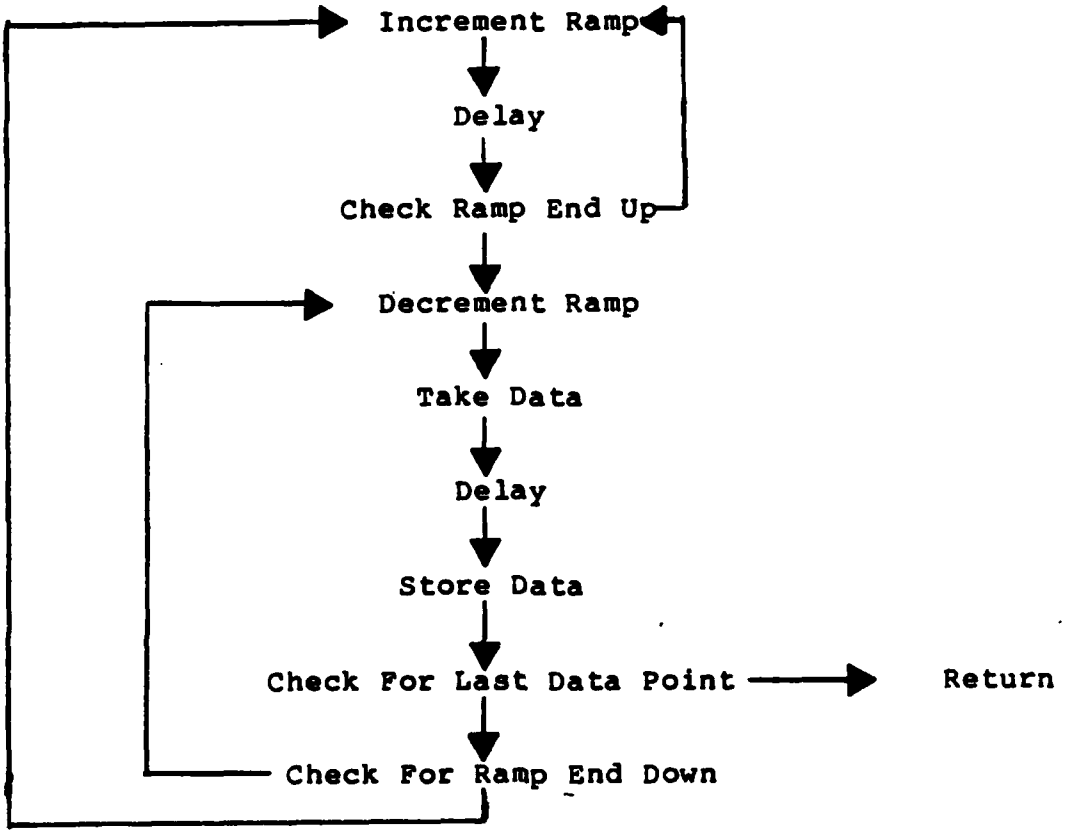


Figure 129. Flow Chart of the Machine Code Program Used to Operate the Single Board Voltammograph

The program then checks to see if the stored data point is the last data point to be obtained, i.e., last data point of the 10th cycle. If the answer is yes, the loop is ended and the machine code program returns to the data analysis portion of the data management program (Appendix D).

If the answer is no, the program then checks to see if the voltage is above the minimum limit of the ramp. If the voltage of the ramp is above the minimum limit of the ramp, the program returns to the first statement of the second loop to continue the incremental decreasing of the voltage ramp. Once the voltage of the ramp is equal to the minimum limit of the ramp, the cycle is complete and the program returns to the first statement of the first loop to begin the incremental increasing of the voltage ramp for the next cycle.

The RCV voltammograms were produced by cycling in a voltage range of -0.2V to 1.2V at voltage scan rates of 1 to 20 V/sec.

The data management program was written in BASIC language to facilitate the numerous changes that may be made in the program during future development. The data analysis program, variables and locations, and memory map of the Apple IIe microcomputer are listed in Appendix D.

3. RESULTS AND DISCUSSION

a. Introduction

During the previous investigation (Reference 74), a data management system was developed for use with the reductive-cyclic voltammetric (RCV) technique. Although the data management system was capable of acquiring and handling the data produced by the RCV technique, the bulkiness of the data management system limited its suitability for use in the RULLER. Also the experimental parameters of the RCV technique and of the voltammograph used to perform the RCV technique were not optimized for the different type gas

turbine engine lubricating oils used by the Air Force.

Therefore, research was performed to develop RULLER candidates based on miniaturized data management systems and voltammographs. The developmental research of the RULLER candidates was performed in two parts. In the first part of the research, the experimental parameters of the RCV technique were optimized for use by the Air Force. Single board voltammographs were then developed based on the optimized RCV technique. In the second part of the RULLER development, the developed single board voltammographs were incorporated into miniaturized data management systems based on microcomputers and microprocessors.

b. Development of a Data Acquisition System for Use in the RCV Optimization Studies

(1) Introduction

During the previous investigation (Reference 74), a data acquisition system based on an Apple IIe microcomputer was developed to monitor the output of the CV-1B voltammograph for the RCV technique. The data acquisition software was designed to take 279 samples at a rate of 70 msec per sample. The 70 msec sampling rate was capable of accurately monitoring the output of the CV-1B voltammograph for voltage scan rates of less than 1.0V per second. However, voltage scan rates of up to 20 V per second will be investigated during the RCV optimization studies.

Therefore, research was conducted to develop data acquisition software capable of taking up to 3000 samples at rates of 0.1 to 70 msec per sample to accurately monitor the CV-1B output for scanning rates of less than 10 V/second. The 10 V/second scanning rate is the maximum scanning rate of the CV-1B voltammograph. A voltage scan range of -0.2 to 1.0 V was used in this study.

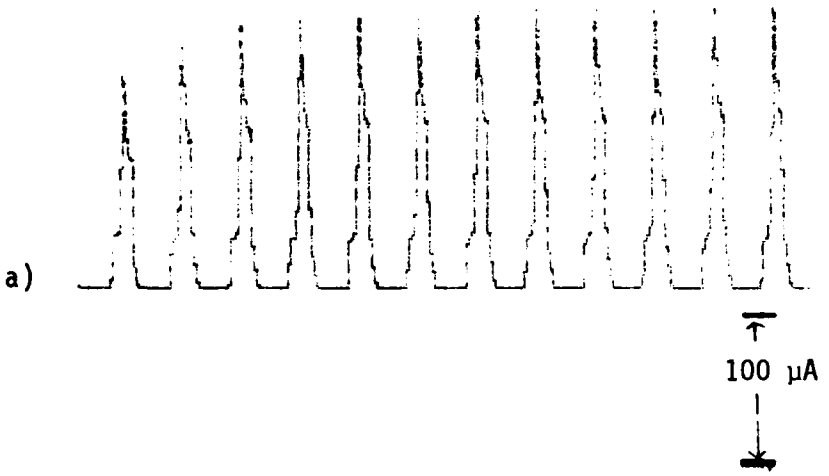
(2) Development of Data Acquisition Software

In order to perform accurate analyses of the CV-1B voltammograph's output at voltage scan rates of 1,5, and 10 V/sec for the RCV technique, data acquisition software (Appendix D) capable of taking and storing 3000 samples at rates of 8.8, 1.8, and 0.88 msec per sample, respectively, was developed.

To demonstrate the capability of the developed software to accurately monitor the CV-1B voltammograph output at different voltage scan rates for the RCV technique, fresh TEL-4001 MIL-L-7808 oil, a stressed TEL-4001 oil with 50% RUL, and the blank solution were analyzed at 1,5 and 10 V/sec scan rates. The voltammograms produced by the 1, 5, and 10 V/sec scan rates are shown in Figures 130, 131, and 132 respectively.

The voltammograms in Figures 130-132 show that the developed data acquisition software is capable of accurately monitoring the output of the CV-1B voltammograph for voltage scan rates of up to 10 V/sec. The voltammograms in Figures 130-132 also show that the shape of the waves produced by the 1,5 and 10 V/sec scan rates of the RCV technique are similar. For the fresh TEL-4001 oil, the heights of the waves increase during the initial cycles of the RCV technique and then level off at a steady state height, regardless of the voltage scan rate (Figures 130-132). Whereas, the heights of the waves produced by the RCV technique for the stressed TEL-4001 oil (Figures 130-132) and the blank (Figures 130-132) remain fairly constant as the number of cycles increases regardless of the voltage scan rate.

However, the heights of the waves in Figures 130-132 are dependent on the voltage scan rate of the RCV technique. The steady state wave heights of the fresh TEL-4001 oil increase from 15 to 61 μA as the voltage scan rate increases from 1 to 10 V/sec. Also, the (fresh TEL-4001





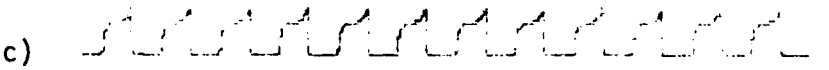


Figure 130. Raw Data Plots (3000 points: 8.8 msec per point) of the Voltammograms Produced by the RCV Technique Using a 1V/Sec Scan Rate for the Fresh TEL-4001 Oil (a), the Stressed TEL-4001 Oil with 50% RUL (b), and the Blank Solution (c)

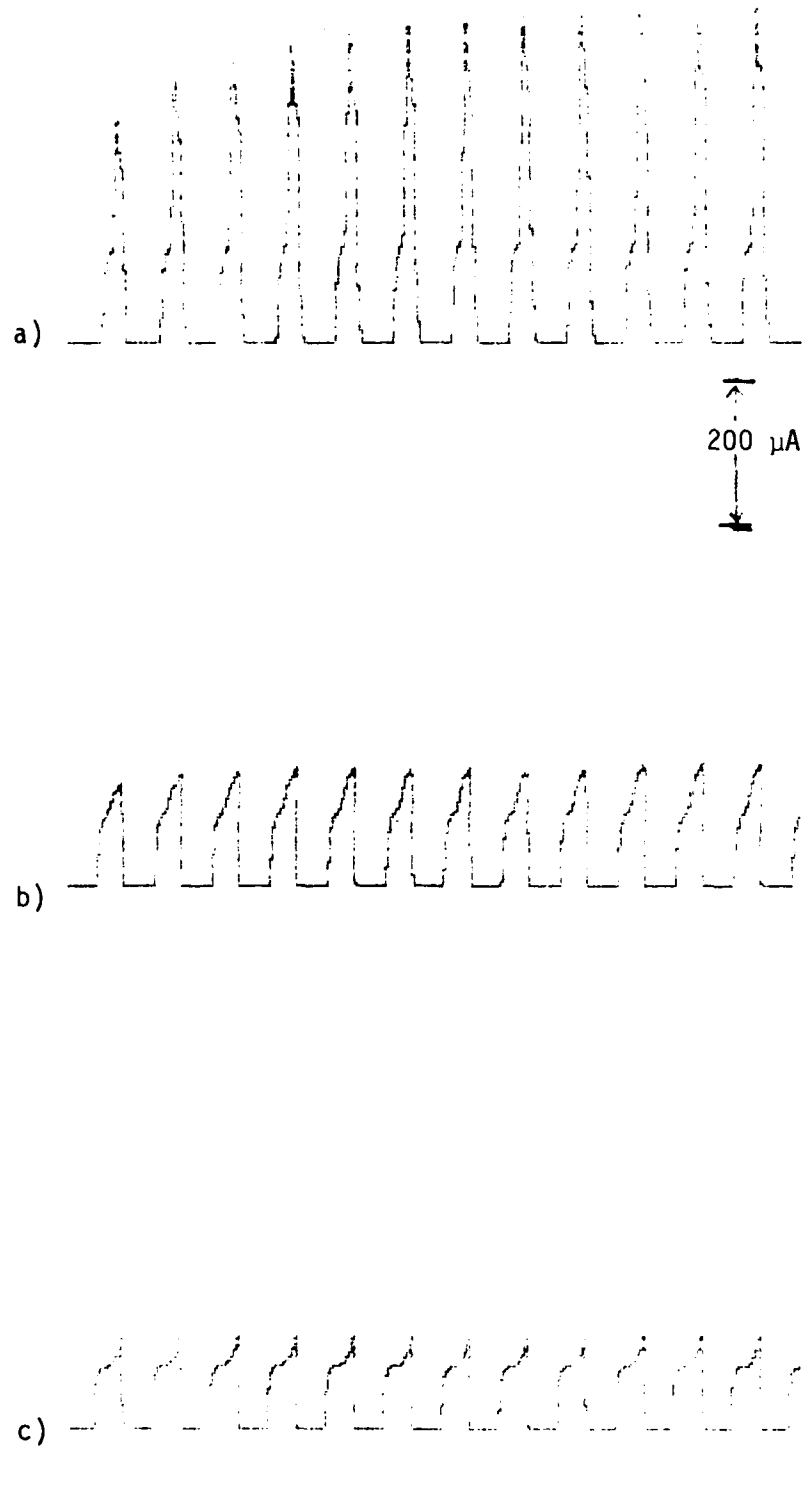


Figure 131. Raw Data Plots (3000 points: 1.8 msec per point) of the Voltammograms Produced by the RCV Technique Using a 5V/Sec Scan Rate for the Fresh TEL-4001 Oil (a), the Stressed TEL-4001 Oil with 50% RUL (b), and the Blank Solution (c)

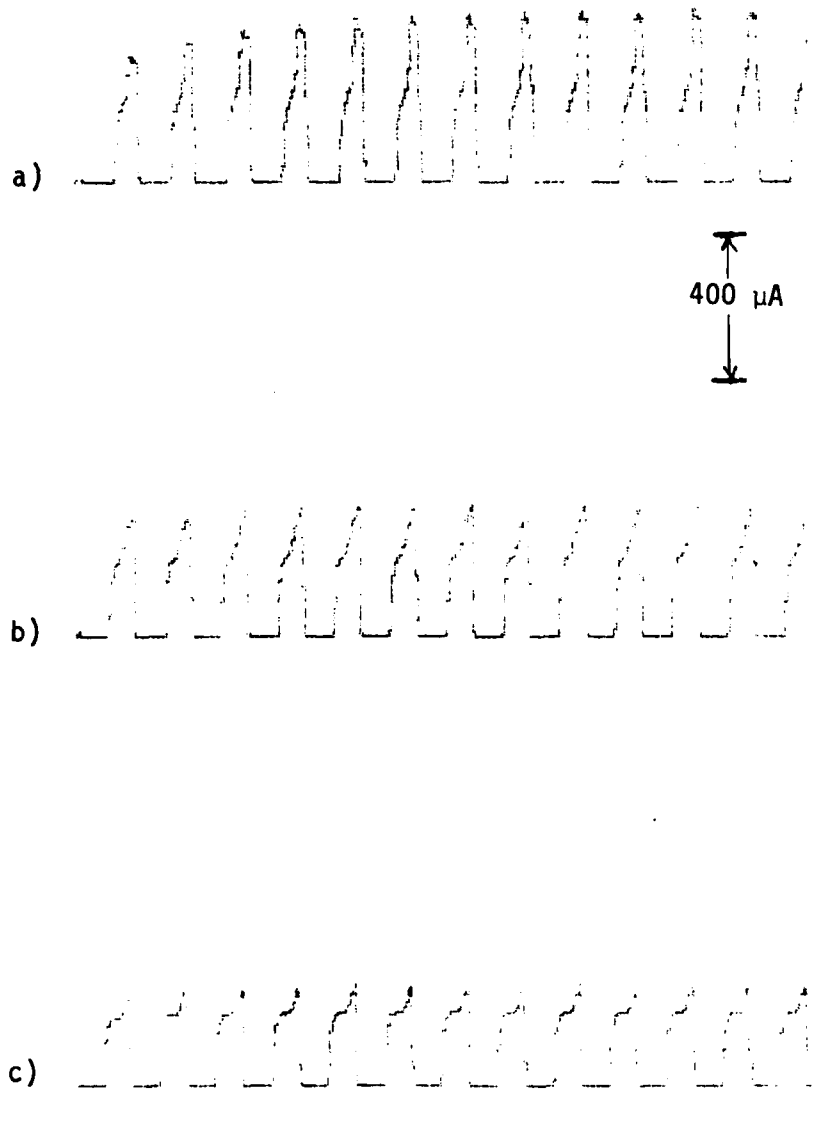


Figure 132. Raw Data Plots (3000 points: 0.88 msec per point) of the Voltammograms Produced by the RCV Technique Using a 10V/Sec Scan Rate for the Fresh TEL-4001 Oil (a), the Stressed TEL-4001 Oil with 50% RUL (b), and the Blank Solution (c)

oil/stressed TEL-4001 oil) ratio of the steady state wave heights is 6.4 for the 1 V/sec scan rate but decreases to 2.5 for the 10 V/sec scan rate.

(3) Summary

The initial results of the data acquisition software developed for the RCV technique demonstrate that the software is capable of accurately monitoring the output of the CV-1B voltammograph for voltage scan rates less than 10 V/sec. Therefore, the differences in the voltammograms produced by the 1-10 V/sec voltage scan rates in Figures 130-132 are due to voltage scan rate effects and not data acquisition software inefficiencies.

c. Effects of Voltage Scan Parameters on RUL Evaluations of RCV Technique

(1) Introduction

During the development of the data acquisition software, it was shown that the voltammograms produced by the RCV technique at 1,5, and 10 V/sec scan rates (Figures 130-132) are similar in shape, but differ in height. The increased sensitivity and decreased analysis time of the 10 V/sec scan rate in comparison to the 1 V/sec scan rate, make the 10 V/sec scan rate more suitable for use in the RULLER. However, the relationship between the maximum height of the reduction wave and the RUL of the TEL-4001 oil was different for each scan rate (Figures 130-132).

Therefore, a study was performed to study the effects of the voltage scan parameters on the RUL evaluations of the RCV technique. The parameters studied during this investigation were the voltage scan rate, the voltage scan range, and the electrochemical oxidation time of the RCV technique.

(2) Effects of Voltage Scan Rate

A study was performed to determine the effects of the voltage

scan rate on the RUL evaluations of fresh and stressed TEL-4001 oils by the RCV technique. Voltage scan rates of 1, 5, and 10 V/sec were used to analyze the series of fresh and stressed (Method 5307.1 at 370°F) TEL-4001 oils prepared in Reference 74. The maximum heights of the reduction waves (minus the maximum height of the blank wave) produced by the seventh through tenth cycles of the RCV technique were determined by the data management program listed in Appendix D. The average maximum height of the seventh through tenth reduction waves was then calculated for each TEL-4001 oil. The ln of the average maximum height of the reduction wave was plotted versus the hours of RUL of the oil sample at 370°F for the fresh and stressed TEL-4001 oils using voltage scan rates of 1, 5, and 10 V/sec as shown in Figure 133.

The ln plots of the reduction wave height versus the hours of RUL of the oil sample for the 1,5, and 10 V/sec scan rates in Figure 133 show that the voltage scan rate affects the RUL evaluations of the RCV technique for the TEL-4001 oils. For the 1 and 5 V/sec scan rates, the ln plots were linear after the first 24 hours of stressing. These plots are similar to those produced during the previous investigation (Reference 74) as shown in Figure 124. In contrast to the 1 and 5 V scan rates, the ln plot of the 10 V/sec scan rate is linear during the first 48 hours of stressing and is linear during the last 48 hours of stressing containing an inflection point at 48 hours of stressing (Figure 133).

The main difference between the ln plots of the 1 and 5 V/sec scan rates and of the 10 V/sec scan rate occurs during the first 24 hours of stressing. The ratio of (the new TEL-4001 oil wave height/the 24 hour stressed TEL-4001 oil wave height) is 4.3 for the 1 V/sec scan rate, but is only 1.7 for the 10 V/sec scan rate. Similar results were seen in Figures 130-132.

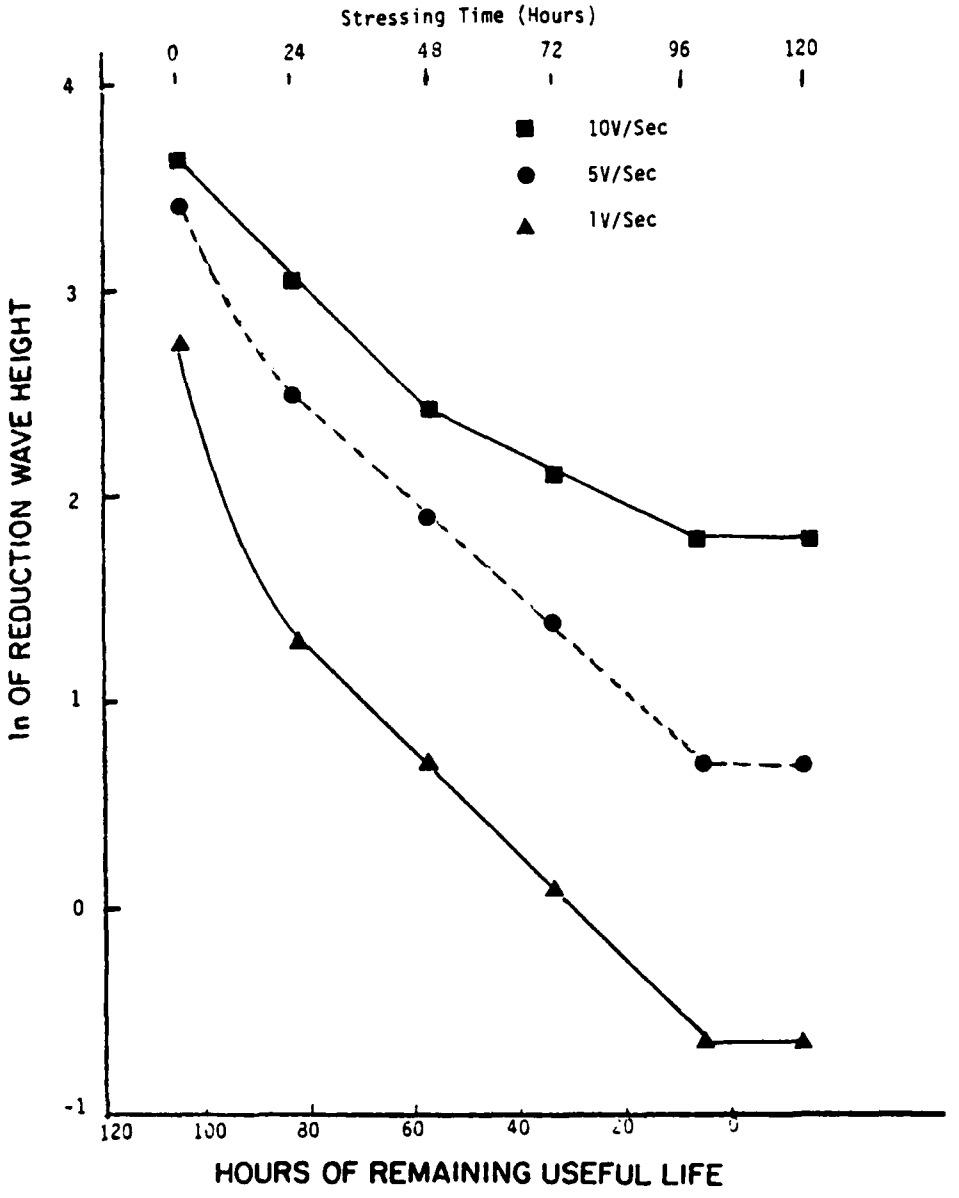


Figure 133. Plots of the ln of the Reduction Wave Height of the RCV Technique Versus Hours of Remaining Useful Life at 370°F for the Fresh and Stressed TEL-4001 Oils Using 1, 5, 10V/Sec Scan Rates

These results indicate that the 10 V/sec scan rate is less efficient at electrooxidatively generating the species which produce the reduction wave used in the RCV technique than the 1 and 5 V/sec scan rates.

(3) Effects of Voltage Scan Range

Since the results in Figure 133 indicate that the 10 V/sec scan rate is inefficient at electrooxidatively generating the species which produce the reduction wave used in the RCV technique, the effects of the voltage scan range on the RUL evaluations of the 10 V/sec scan rate were studied. To study the effects of the voltage scan range on the RUL evaluations of the 10 V/sec scan rate, the voltage scan range was increased from a range of -0.2 to 1.0V to a range of -0.2 to 1.2V. By increasing the positive limit of the voltage scan from 1.0 to 1.2 V, the potential of the working electrode remains above the oxidation potential of the antioxidants for a longer period of time. Thus, the -0.2 to 1.2 V scan range increases the ability of the working electrode to generate the species which produce the reduction wave used in the RCV technique.

To determine the effects of the voltage scan range on the RUL evaluations of the 10 V/sec scan rate, the ln of the maximum height of the reduction wave was plotted versus the hours of RUL of the oil sample at 370^{o}F for the fresh and stressed TEL-4001 oils using the -0.2 to 1.0V and -0.2 to 1.2 V scan ranges as shown in Figure 134.

The ln plots of the reduction wave height versus the hours of RUL of the TEL-4001 oil sample using the -0.2 to 1.0 V and -0.2 to 1.2 V scan ranges in Figure 134 show that increasing the positive limit of the voltage scan shifts the inflection point to a lower hours of RUL. The ln plot of the -0.2 to 1.2 V scan range is linear after the first 24 hours of stressing (90 hours of RUL) which is in full agreement with the 1 and 5 v/sec scan rates

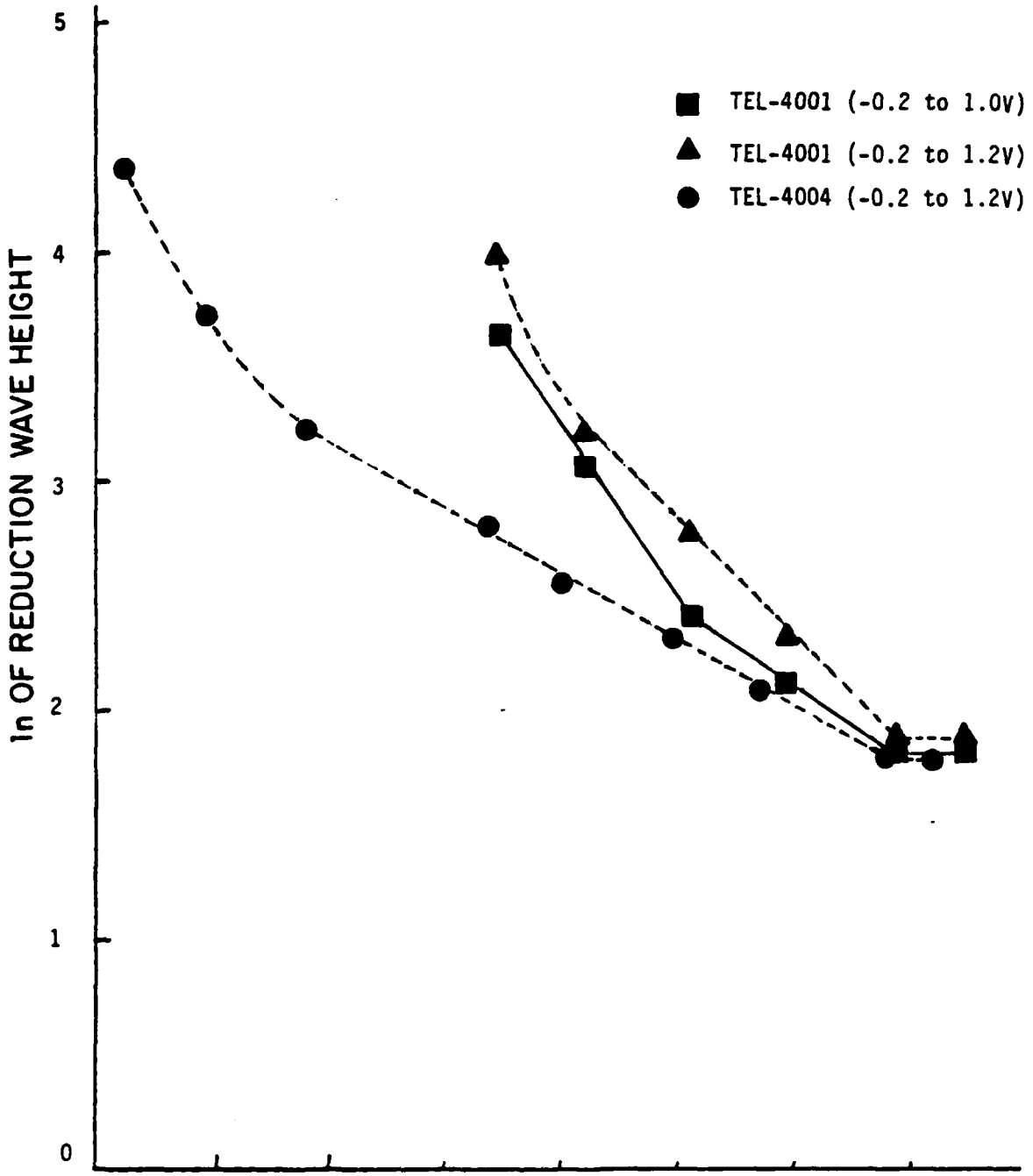


Figure 134. Plots of the ln of the Reduction Wave Height of the RCV Technique Versus Hours of Remaining Useful Life at 370°F for the Fresh and Stressed TEL-4001 and TEL-4004 Oils Using a 10V/Sec Scan Rate and -0.2 to 1.0V and -0.2 to 1.2V Scan Ranges

(Figure 133) and the previous investigation (Figure 124).

To ensure that the linearity of the 10 V/sec scan rate was not dependent on the antioxidant system of the MIL-L-7808 oil, fresh and stressed TEL-4004 oils were also analyzed by the RCV technique using a 10 V/sec scan rate and a -0.2 to 1.2 V scan range. The antioxidant system of the TEL-4001 oil contains octyl-PANA, DODPA, and an unknown antioxidant; while the antioxidant system of the TEL-4004 oil contains PANA and DODPA. The ln plot of the reduction wave height versus the hours of RUL of the oil sample at 370^{o}F for the fresh and stressed TEL-4004 oils is included in Figure 134.

The ln plot of the reduction wave height versus the hours of RUL of the oil sample for the fresh and stressed TEL-4004 oils in Figure 134 is linear after the first 48 hours of stressing (160 hours of RUL) in full agreement with the previous investigation (Figure 124). Also, the ln values of the reduction wave heights of the stressed TEL-4001 and TEL-4004 oils with 0% RUL are 1.7-1.8, i.e., the RUL evaluations of the RCV technique using a 10 V/sec scan rate and a voltage scan range of -0.2 to 1.2V are formula independent.

(4) Effects of Electrooxidation Time

Since the results in Figure 134 show that increasing the positive limit of the voltage scan range from 1.0 to 1.2 V affects the RUL evaluations of the RCV technique, the effects of the electrooxidation time on the RUL evaluation of the RCV technique were studied. The electrooxidation time is the length of time the potential of the working electrode is greater than or equal to the oxidation potential (1.0 V) of the antioxidant species in the lubricating oil sample. It was postulated that the length of the electrooxidation time would affect the RUL evaluations of the RCV technique. The use of an electrooxidation time would also reduce the number of

voltammetric cycles from 10 to 0.5 (1.1 to -0.15V) shortening the voltammetric scanning time (1 V/sec scanning rate) from 25 to 1 seconds, respectively, and would simplify the design and software of the developed voltammograph.

To determine the effects of the electrooxidation time on the RUL evaluations of the RCV technique, a voltage of 1.1 V was applied to the working electrode prior to the voltammetric scan. After an electrooxidation time of 0, 1, 2, or 5 seconds, a linear voltage scan was performed from 1.1 to -0.15 V at a 1 V/sec rate and the height of the produced reduction wave, determined. The ln plots of the reduction wave height versus the RUL of the oil sample are shown in Figure 135 for the fresh and stressed TEL-4004 MIL-L-7808 lubricating oil samples using the 0, 1, 2, and 5 second electrooxidation times.

The ln plots of the reduction wave height versus the RUL of the oil sample in Figure 135 demonstrate that the length of the electrooxidation time affects the linearity of the plots, and consequently, the RUL evaluations of the RCV technique. The 0 second electrooxidation time produces a plot which goes off scale after 168 hours of stressing (40 hours of RUL). The 1, 2 and 5 second electrooxidation times produce smaller inflection points after 48 hours of stressing (Figure 135). As expected, the reduction wave heights produced by the TEL-4004 oil samples increase as the electrooxidation time increases, i.e., the species which produce the reduction wave are produced during the electrooxidation time.

These initial results indicate that the electrooxidation time can be used to affect the RUL evaluations of the RCV technique and to increase the heights of the produced reduction waves. However, the electrooxidation time was unable to eliminate the inflection point from the

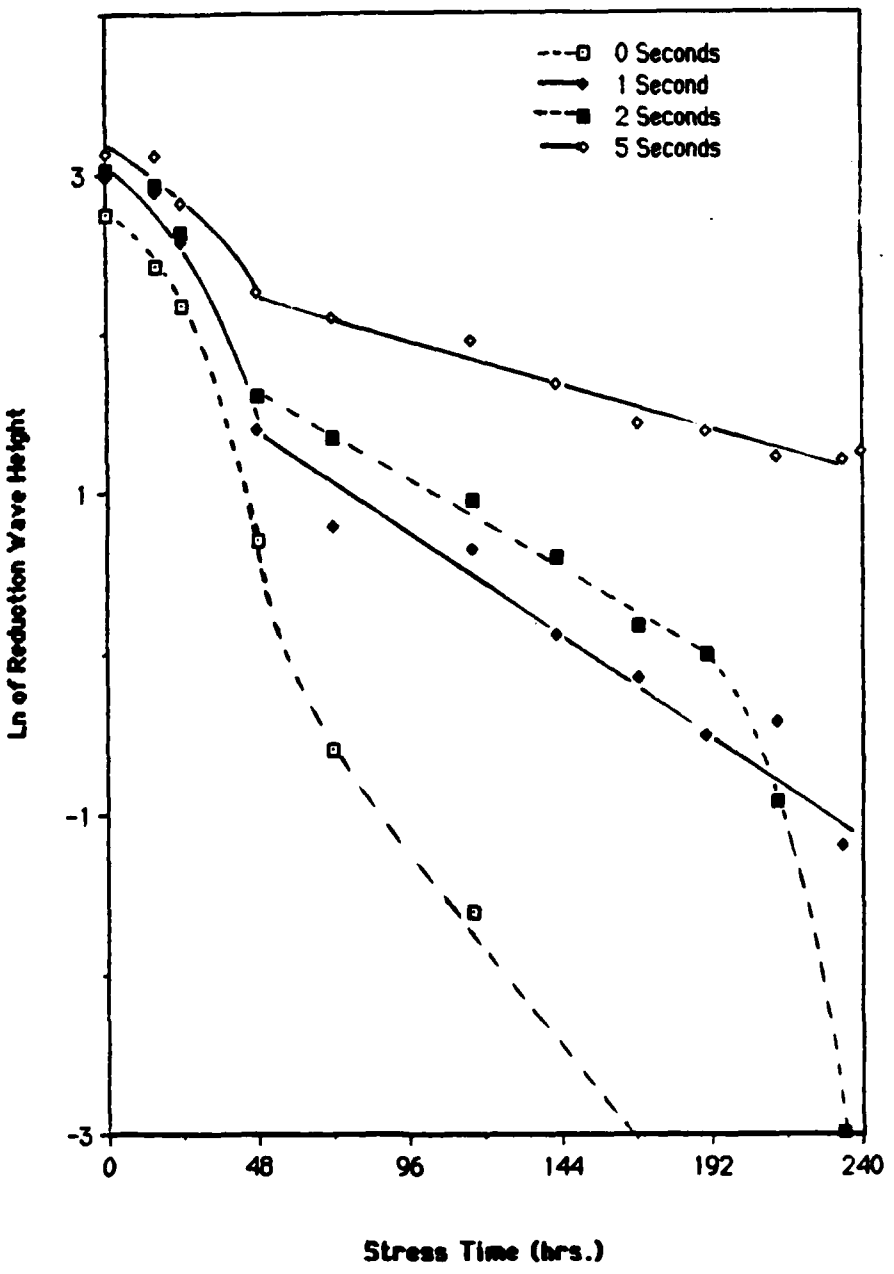


Figure 135. Plots of the ln of the Reduction Wave Height of the RCV Technique Versus Stressing Time (Hours) at 370°F for the Fresh and Stressed TEL-4004 Oils Using Electrooxidation Times of 0, 1, 2 and 5 Sceconds at 1.1V, a 1 V/Sec Scan Rate, and a 1.1V to -0.15V Scan Range

ln plots of the RCV results (Figure 135) and the electrooxidation time (2 to 5 seconds plus voltammetric scan time) increases the analysis time in comparison to the 10 V/sec scanning rate (2.5 second analysis time). Thus, the electrooxidation time does not have any apparent advantages in comparison to the cyclic scanning procedure for use in the RCV technique.

(5) Summary

The initial results of the voltammetric parameter study indicate that the linearity of the ln plot of the reduction wave height versus the RUL of the oil sample is affected by the voltammetric scanning rate, scanning range, and electrooxidation time. The results indicate that a voltage scan rate of at least 10 V/sec and the correct voltage scan range would enable the RCV technique to produce accurate RUL evaluations using analysis times of less than 2.5 seconds. The exact voltammetric parameters will be determined using the developed RULLER candidate and actual used MIL-L-7808 and MIL-L-23699 lubricating oils.

d. Development of Single Board Voltammograph

(1) Introduction

In order to miniaturize , to lower susceptibility to supplier product changes, and to lower the instrumental cost of the RULLER candidate, a single board voltammograph was developed to replace the CV-1B voltammograph. The characteristics of a single board voltammograph and the CV-1B voltammograph are listed in Table 67. As the characteristics in Table 65 delineate, a single board voltammograph has numerous advantages over the CV-1B voltammograph.

Therefore, a single board voltammograph was developed and incorporated into the hardware of the Apple IIe data management system of the RULLER candidate. The single board voltammograph equipped with different

TABLE 67

COMPARISON OF SINGLE BOARD AND CV-1B VOLTAMMOGRAPHS

	Single Board	CV-1B
Size (l x w x d) (in.)	5.75 X 4.75 X 0.75	7 X 5 X 3
COST	$100	$675
Maximum Scan Rate 100	100 V/Sec	10 V/Sec
Long Term Availability	Unlimited	Limited, Manufacturer Considering Ending Production in Next Five Years
Rugged	Yes, Single Board No Moving Parts	No, Multicomponent Moving Parts
Relationship with Data Acquisition	Completely Interactive (Data Acquisition Accurate Regardless of Sampling Rate)	Independent (Accuracy of Data Acquisition Decreases as Sample Rate Decreases)

filtering systems was then used to analyze fresh MIL-L-7808 oils and the blank to evaluate the performance of the single board voltammograph-Apple IIe microcomputer system. The results of the developmental research of the single board voltammograph are described herein.

(2) Unfiltered Single Board Voltammograph

To initially evaluate the performance of the unfiltered single board voltammograph (Figure 127), fresh TEL-4001 MIL-L-7808 lubricating oil and the blank were analyzed using a 1 V/sec scan rate and a -0.1 V to 1.15 V scan ran-e. The voltammograms produced by the reduction sweep are shown in Figure 136. Although the overall shapes of the reduction voltammograms produced by the unfiltered single board voltammograph for the fresh TEL-4001 oil and the blank in Figure 136 are as expected, the noise in the voltammograms is unacceptable. The noise in the voltammograms would greatly limit the accuracy of the reduction wave height or area determination, and consequently, limit the accuracy of the RUL determinations of the developed RULLER candidate.

(3) Single Board Voltammograph with RC Filtered Output

In an attempt to eliminate the noise from the reduction voltammograms, a capacitor (0.1 μf) was placed in parallel with resistor Rg (Figure 127) in the output of the single board voltammograph. The reduction voltammograms produced by the modified single board voltammograph for the fresh TEL-4004 oil and the blank in Figure 137 show that the addition of the capacitor to the output circuitry of the working electrode effectively eliminated the noise from the output of the voltammograph.

However, when successive RCV analyses of the fresh TEL-4004 oil were performed, the reduction waves produced by the voltammograph decreased in height and shifted to a lower voltage (Figure 138). In fact, by the

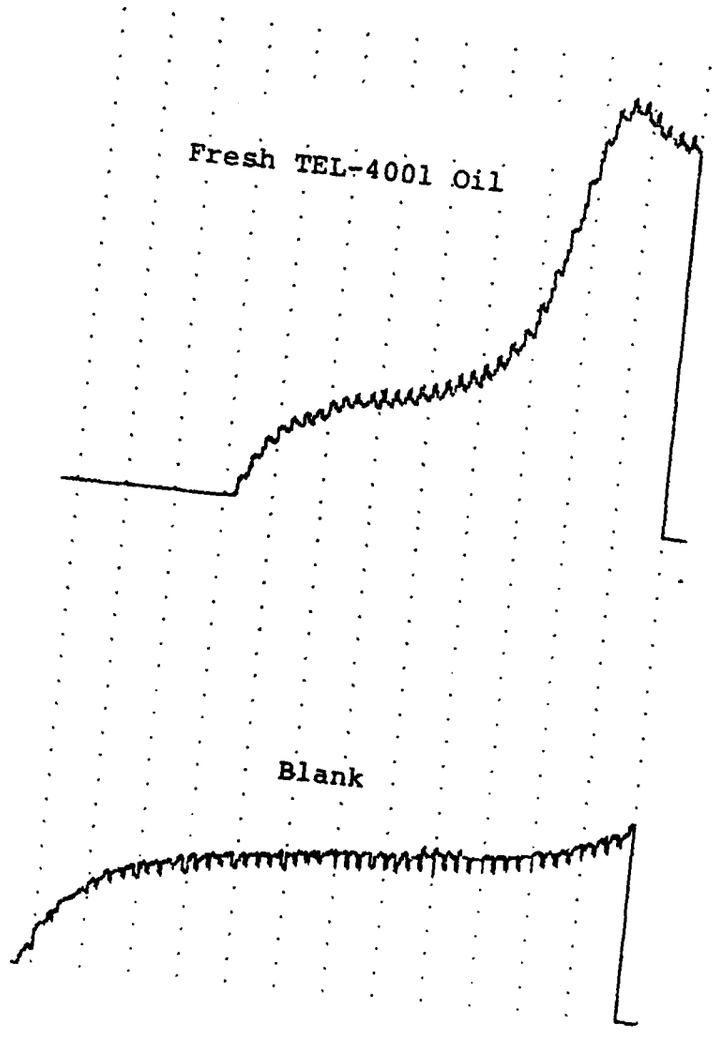


Figure 136. Reduction Voltammograms Produced by the Single Board Voltammograph for the Fresh TEL-4001 Oil and the Blank

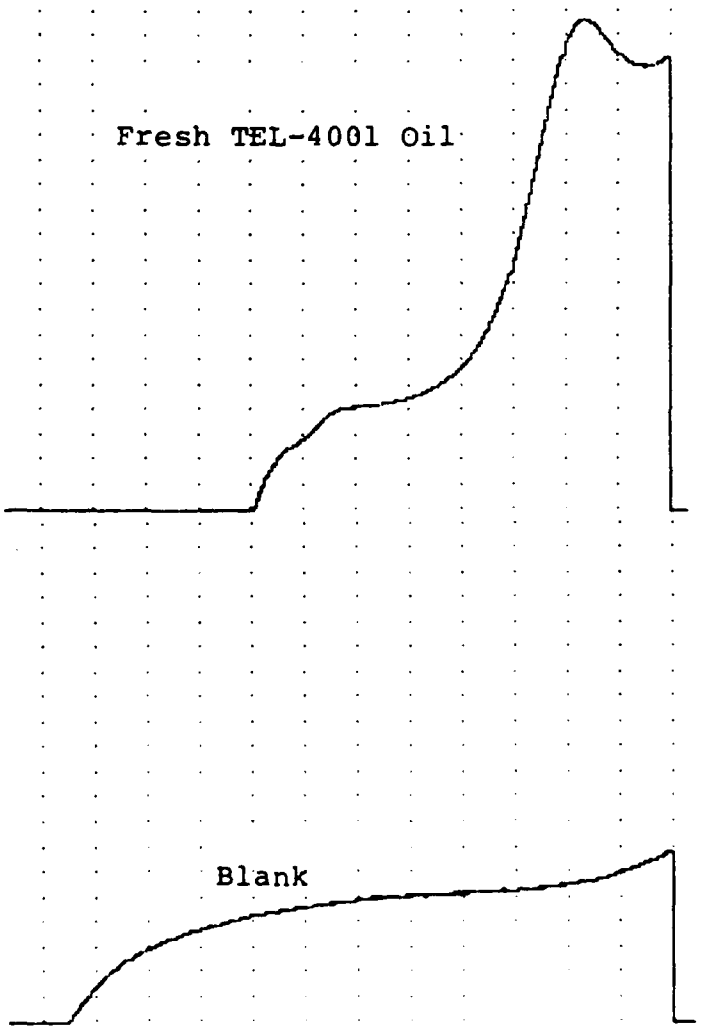


Figure 137. Reduction Voltammograms Produced by the Single Board Voltammograph with the RC Filtered Output for the Fresh TEL-4001 Oil and the Blank

fourth successive analysis, the RCV technique became nonresponsive to the fresh TEL-4004 oil (RCV technique analyses using the CV-1B voltammograph performed directly before and after the single board voltammograph analyses did not show any response decrease with successive analyses).

(4) Single Board Voltammograph with RC Filtered Input

The initial results of the single board voltammograph with and without RC filtered output demonstrate that, although the developed voltammograph has potential for use in the RCV technique of the RULLER candidate, the response of the developed voltammograph decreases with successive analyses. The decreased response of the single board voltammograph is indicative of film formation on the surface of the working electrode.

The circuitries of the CV-1B and single board voltammographs are very similar. The only main difference between the voltammographs is that the CV-1B uses a linear voltage ramp and the single board voltammograph uses a staircase (5 mV) voltage ramp. The noise observed in Figure 136 may be a product of the staircase voltage ramp.

Therefore, to determine if the staircase voltage ramp is responsible for the decrease in the single board voltammograph's response with successive analyses and for the noise in Figure 136, a RC system was placed into the circuitry of the D/A input of the single board voltammograph (Figure 127).

To evaluate the performance of the RC system (capacitor = 0.4 μf and resistor = 65 Kohms) used to filter the staircase voltage ramp of the single board voltammograph, fresh TEL-4004 MIL-L-7808 lubricating oil was analyzed with and without a filtered staircase voltage ramp. The RCV analyses were performed using a 1 V/sec scan rate and a -0.1 V to 1.15 v scan

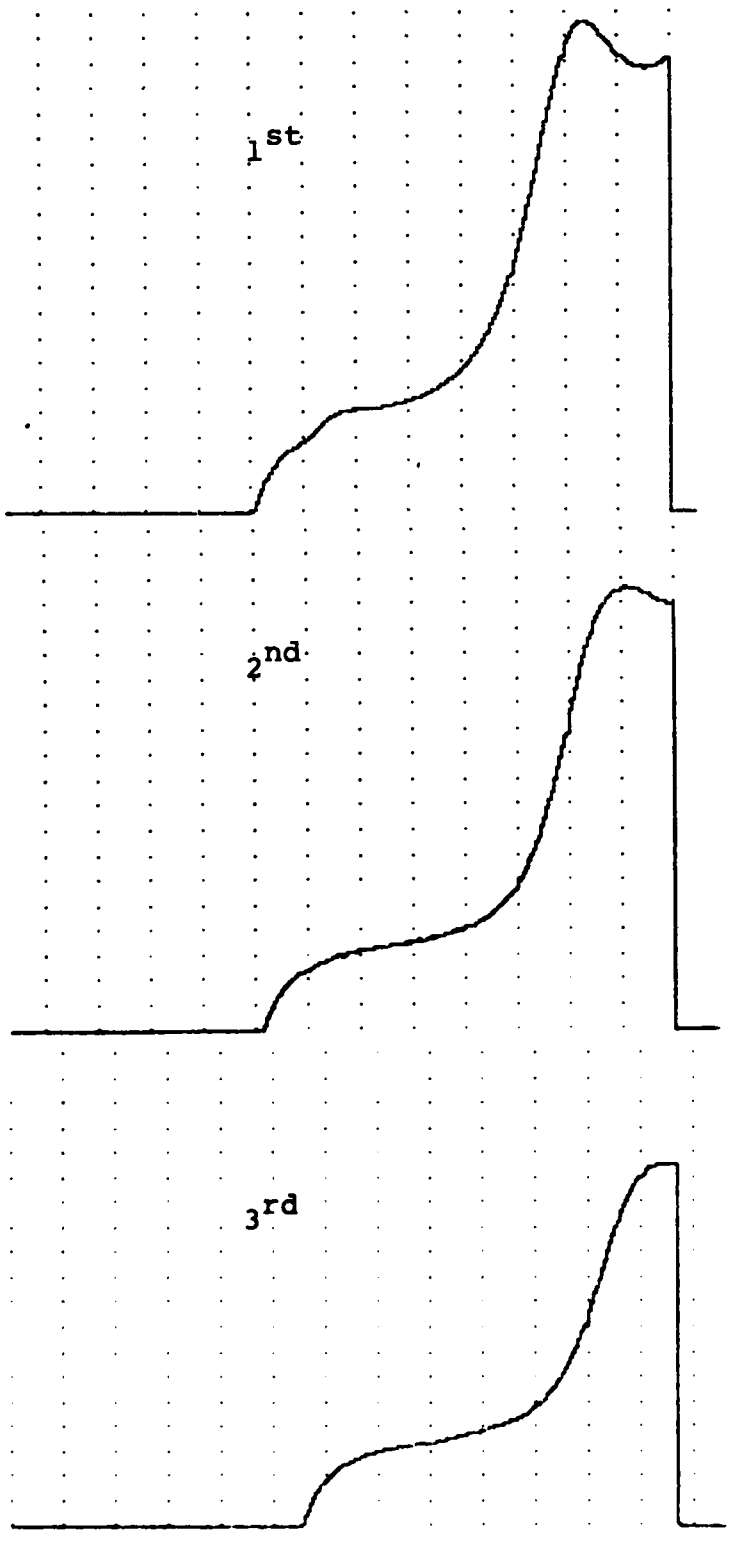


Figure 138. Effects of Successive Analyses on the Reduction Voltammogram Produced by the Single Board Voltammograph for the Fresh TEL-4004 Oil

range. Examples of the reduction waves produced by the filtered and non-filtered staircase voltage ramps for fresh TEL-4004 oil are shown in Figure 139. The reduction voltammograms in Figure 139 show that the RC filtering system effectively reduces the noise of the single board voltammograph.

To further evaluate the performance of the single board voltammograph with a RC filtered input, triplicate RCV analyses of the fresh TEL-4004 oil were performed using 1, 2, and 4 V/sec scan rates and a -0.1 V to 1.15 V scan range. The first two reduction waves produced by the triplicate RCV analyses using 1, 2, and 4 V/sec scan rates are shown in Figures 140-142, respectively.

The series of reduction waves in Figures 140 and 141 show that the RC filtering system eliminates film formation at the surface of the working electrode for the 1 and 2 V/sec scan rates, i.e., reduction waves' heights and shapes not affected by successive analyses.

However, the series of voltammograms in Figures 140-142 show that the reduction waves shift to lower voltages as the voltage scan rate is increased from 1 to 4 V/sec. In fact, the reduction waves for the 4 V/sec scan rate are incomplete. By shifting the voltage scan range of -0.1 to 1.15 V to a range of -0.2 to 1.05 V, complete reduction waves were produced at the 4 V/sec scan rate as shown in Figure 143. The positive voltage limit had to be reduced from 1.15 to 1.05 V when the negative voltage limit was decreased from -0.1 to -0.2 V because the total voltage range of the single board voltammograph is constant (1.25 V).

(5) Single Board Voltammograph with Active Filtered Input

The initial results of the single board voltammograph with RC filtered input demonstrate that the RC filtering system effectively

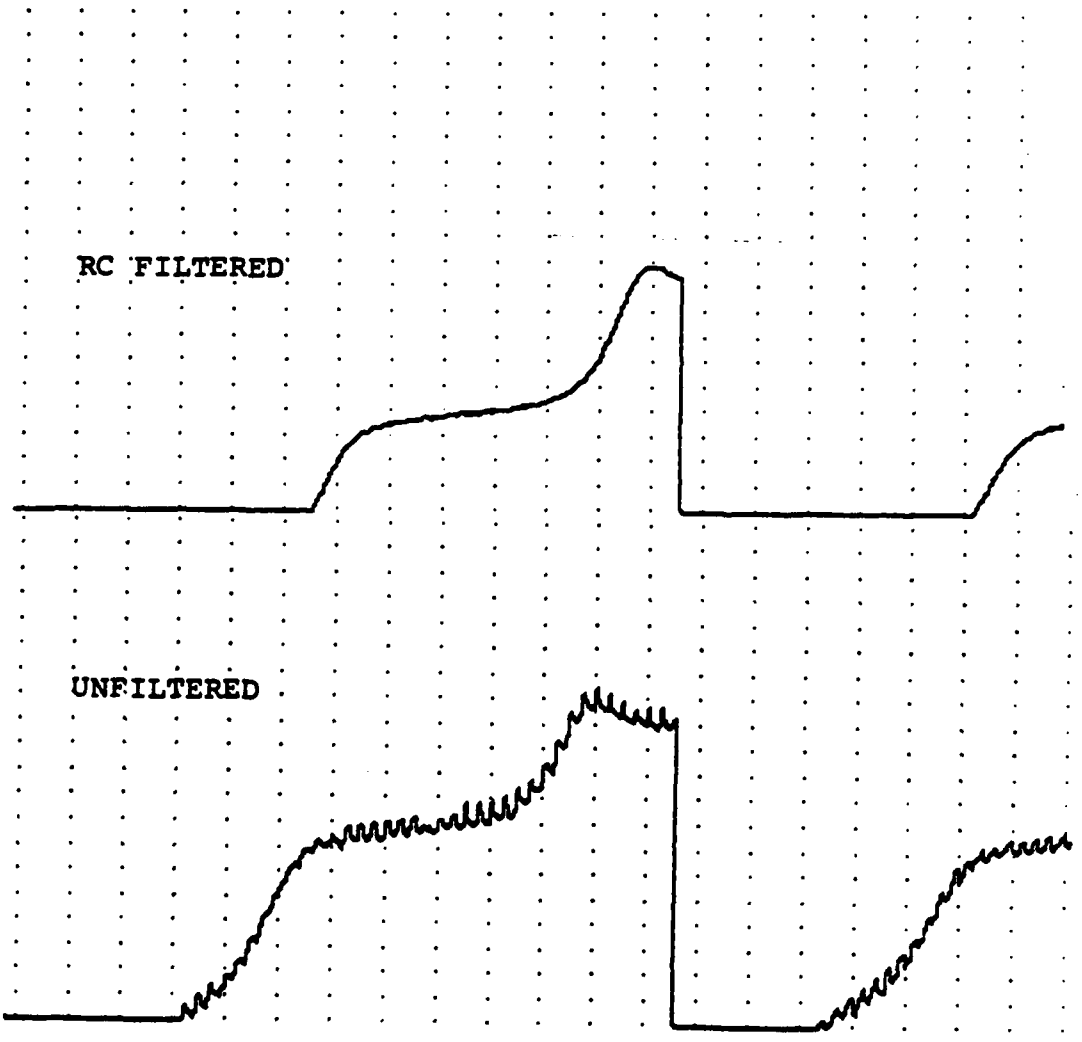


Figure 139. Reduction Voltammograms Produced by the Single Board Voltammograph with and without RC Filtered Input for the Fresh TEL-4004 Oil

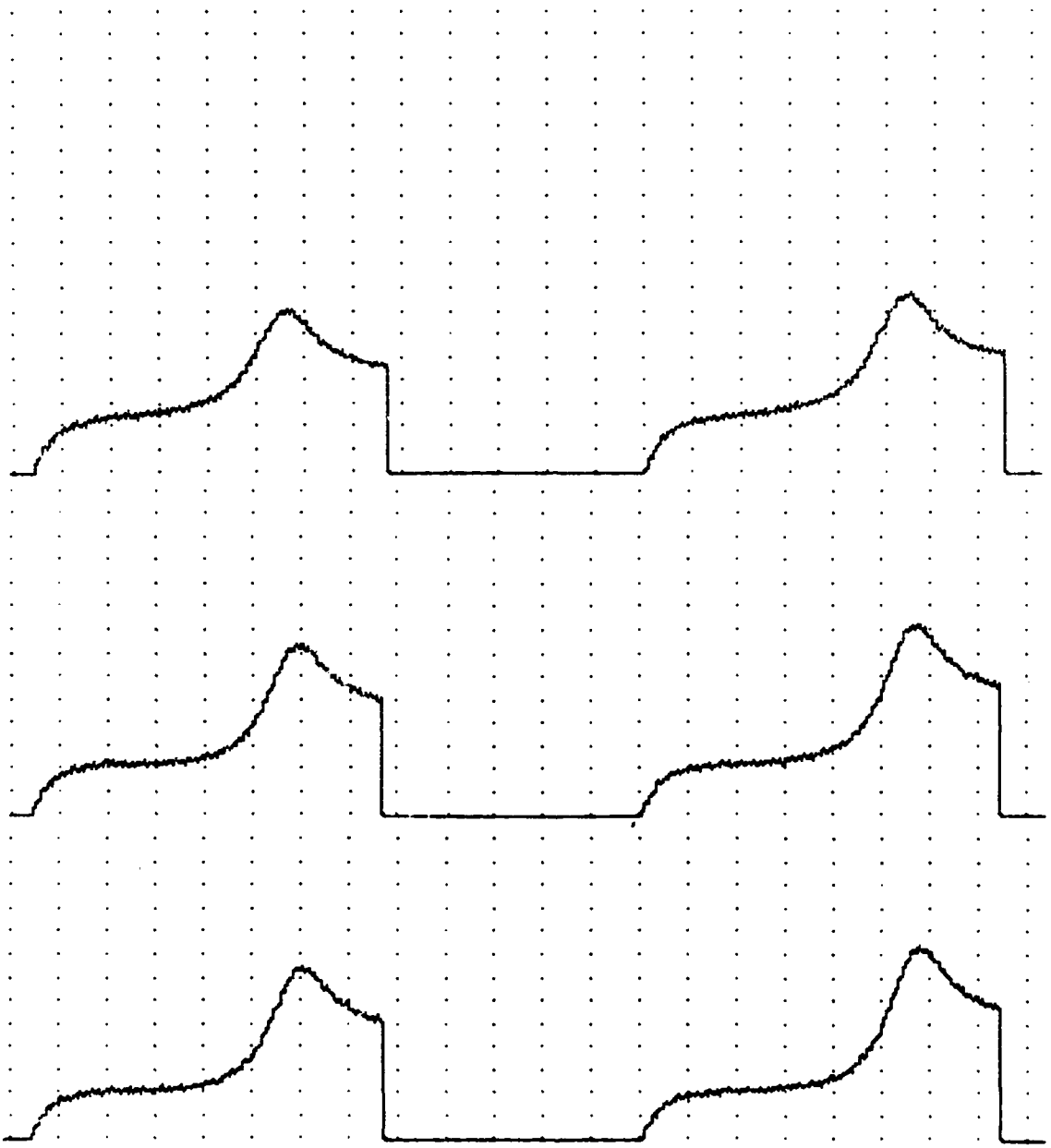


Figure 140. Effect of Successive Analyses on the Reduction Voltammograms Produced by the Single Board Voltammograph with RC Filtered Input for the Fresh TEL-4004 Oil Using a 1 V/Sec Scan Rate and a -0.1 to 1.15 V Scan Range

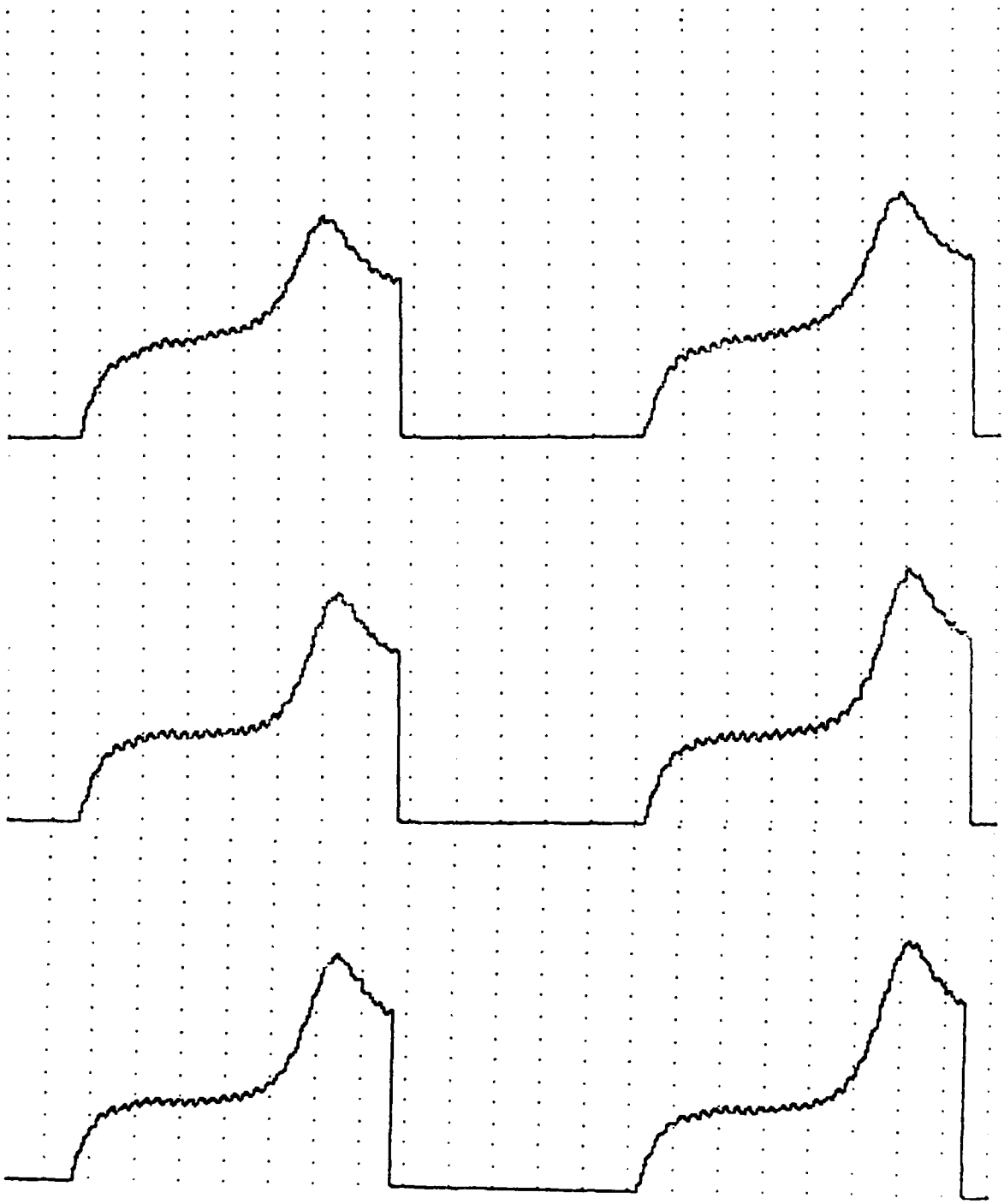


Figure 141. Effects of Successive Analyses on the Reduction Voltammograms Produced by the Single Board Voltammograph with RC Filtered Input for the Fresh TEL-4004 Oil Using a 2 V/Sec Scan Rate and a -0.1 to 1.15 V Scan Range

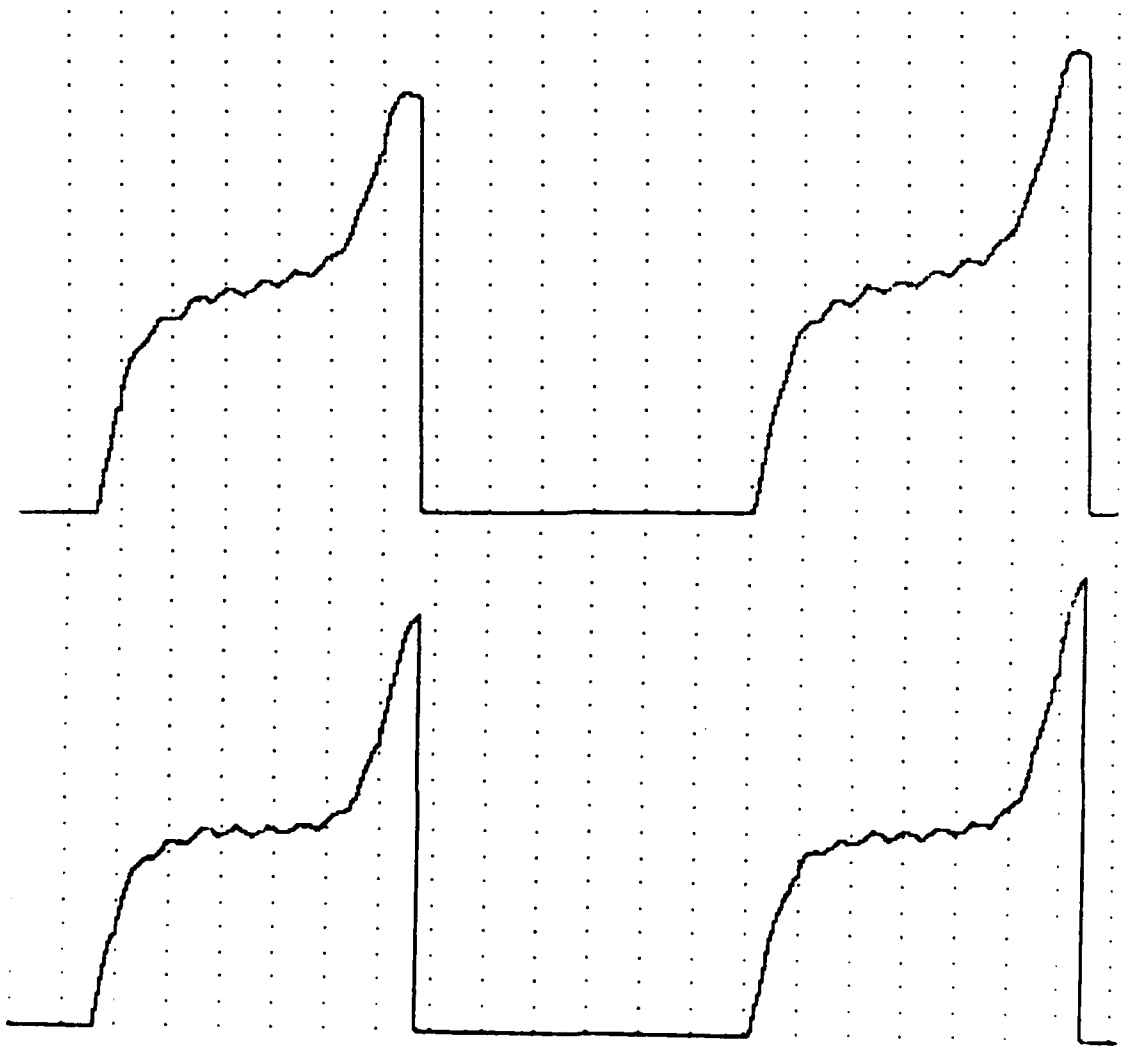


Figure 142. Effects of Successive Analyses on the Reduction Voltammograms Produced by the Single Board Voltammograph with RC Filtered Input for the Fresh TEL-4004 Oil Using a 4 V/Sec Scan Rate and a -0.1 to 1.15 V Scan Range

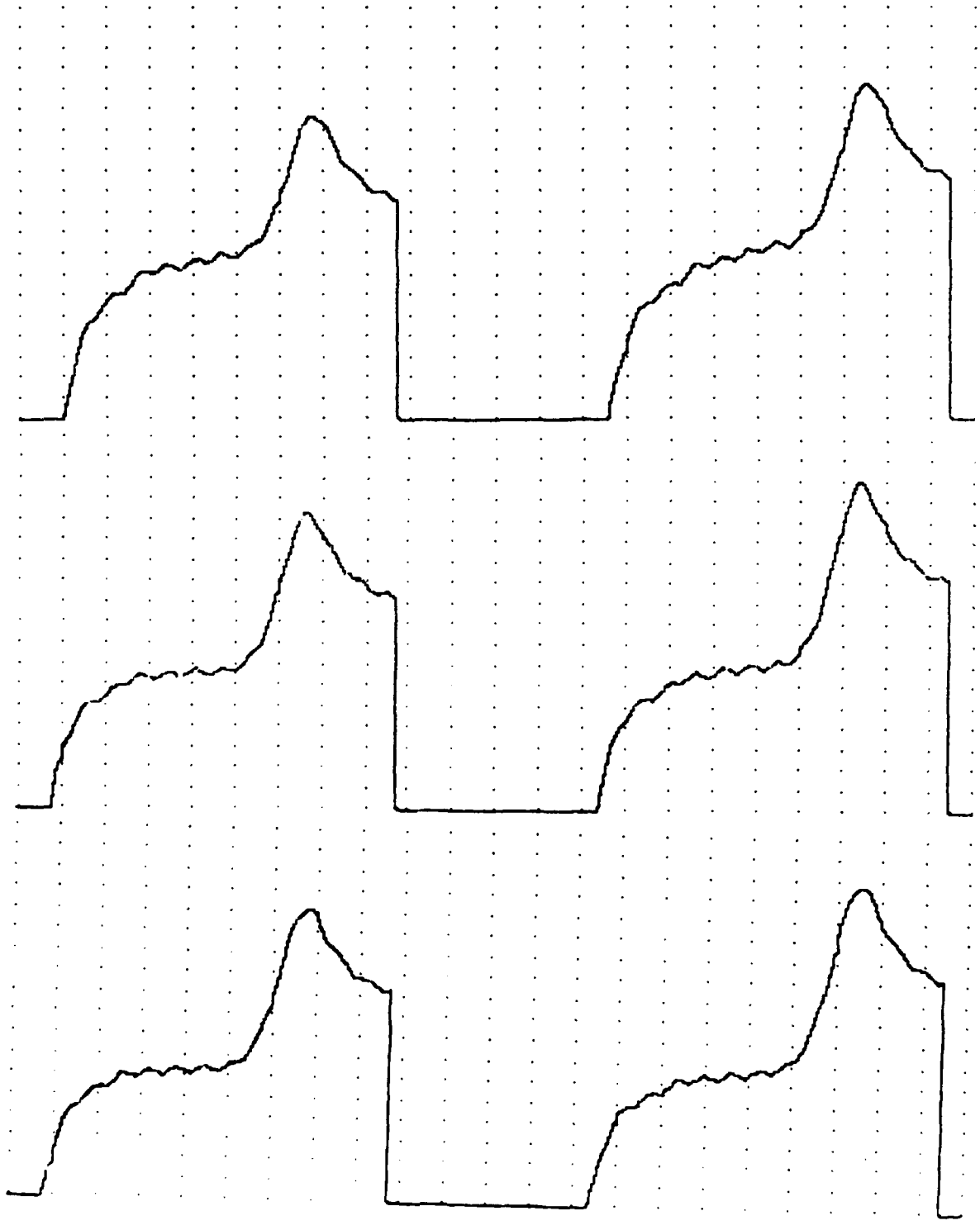


Figure 143. Effects of Successive Analyses on the Reduction Voltammograms Produced by the Single Board Voltammograph with RC Filtered Input for the Fresh TEL-4004 Oil Using a 4 V/Sec Scan Rate and a -0.2 to 1.05 V Scan Range

eliminates film formation at the surface of the working electrode and reduces the noise of the voltammograph's output. However, the RC filtering system causes the reduction waves to shift to lower voltages as the voltage scan rate is increased. It is believed that the RC filtering system is "rounding" (Figure 144) the triangular waveform of the single board voltammograph. Thus, the potential of the voltammetric electrode does not reach the limits of the voltage scan range causing the reduction wave to be incomplete.

Therefore, a more efficient filtering system, an active filtering system (Figure 128), was used to filter the staircase voltage ramp without affecting the shape of the triangular waveform of the single board voltammograph. Also, operational amplifiers with adjustable gains were added to the circuitry of the single board voltammograph to provide it with independently adjustable voltage limits (Figure 128).

To initially evaluate the performance of the active filter system (Figure 128), duplicate RCV analyses of the fresh TEL-4004 MIL-L-7808 lubricating oil were performed using a 2 V/sec scan rate and -0.1 V to 1.15 V scan range. The first two reduction waves produced by the duplicate RCV analyses of the fresh TEL-4004 oil are shown in Figure 145 and indicate that the active filtering system eliminates film formation at the surface of the working electrode, i.e., reduction wave heights and shapes are not affected by successive analyses.

To further evaluate the performance of the single board voltammograph with an active filtered input, the fresh TEL-4004 oil was analyzed using 4, 10, and 20 V/sec scan rates and a -0.1 V to 1.15 V scan range. The first three reduction waves produced by the 4, 10, and 20 V/sec scan rates are shown in Figure 146 and indicate that the active filtering system does not round (Figure 144) the triangular waveform of the the single

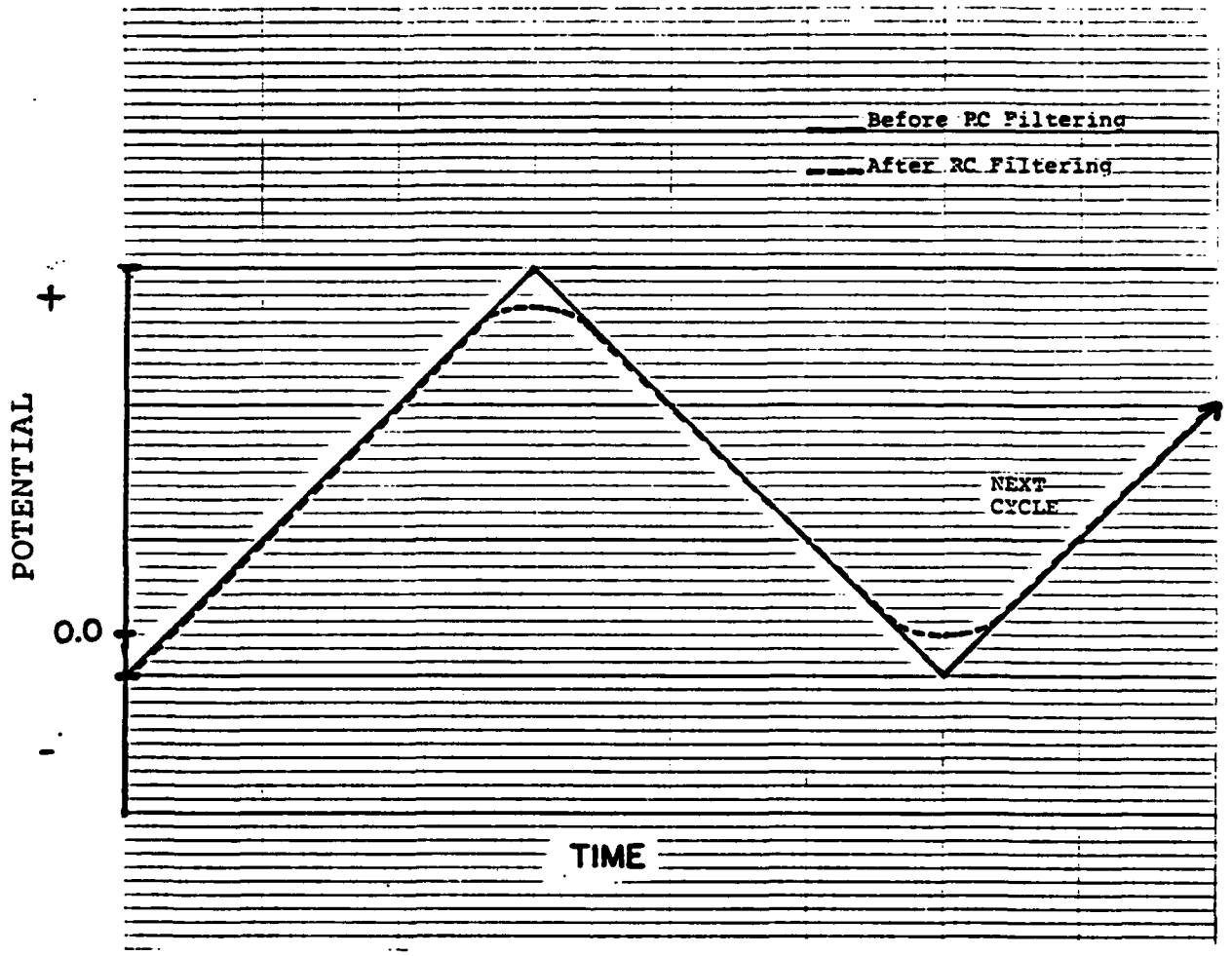


Figure 144. RCV Technique Voltage Waveform Before and After Filtering

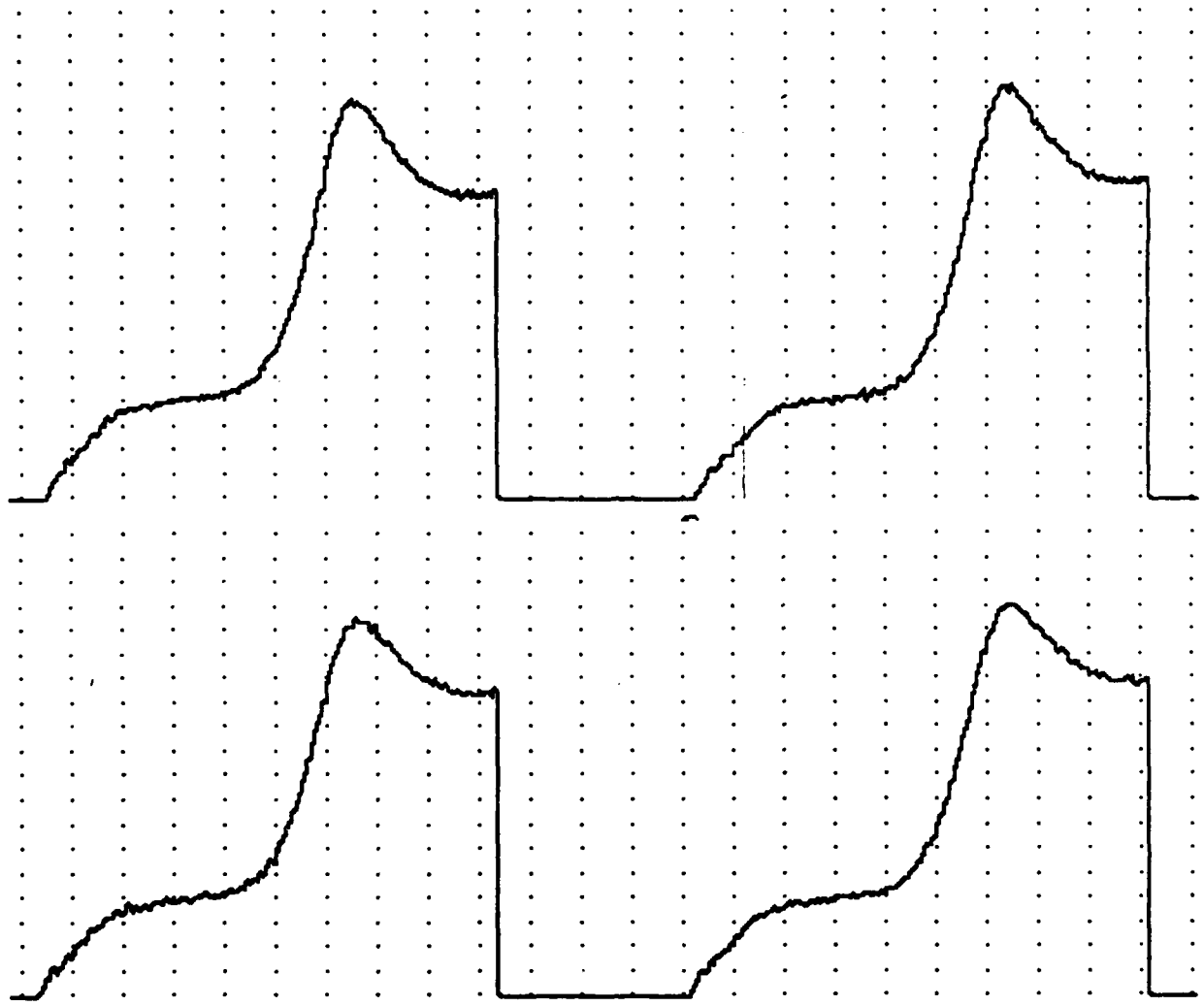


Figure 145. Effects of Successive Analyses on the Reduction Voltammograms Produced by the Single Board Voltammograph with Active Filtered Input for the Fresh TEL-4004 Oil Using a 2 V/Sec Scan Rate and a -0.1 to 1.15 V Scan Range

board voltammogram, i.e., complete reduction waves produced for scan rates up to 20 V/sec. Thus, the active filtering system is a more efficient filtering system than the RC filtering system because the RC filtering system produces incomplete reduction waves for voltage scan rates greater than 4 V/sec (Figure 142), while the active filtering system produces complete reduction waveform voltage scan rates greater than 20 v/sec.

(6) Summary

A single board voltammograph (Figure 128) has been developed which is suitable for use in the RULLER candidate. The active filtering system used to eliminate the noise of the voltage ramp input from the Apple IIe microcomputer is highly efficient and enables the RCV technique to be performed at scan rates of up to 20 V/sec in the -0.1 V to 1.15 V scan range. The single board voltammograph is also equipped with independently adjustable maximum and minimum range limits so that the voltage scan range can be adjusted to produce accurate RUL evaluations. Also, the negative limit can be lowered to 0.4 V to enable the voltammograph to produce complete reduction waves at voltage scan rates greater than 20 V/sec.

e. Single Board Voltammograph-Apple IIe Microcomputer RULLER Candidate

A RULLER candidate based on the single board voltammograph with an active filtered voltage ramp and independently adjustable voltage scan limits (Figure 128) and the Apple IIe microcomputer system has been developed. The computer programs to perform the voltage ramp and acquire RCV data (machine code) and to perform the data analysis and prompting of the user (Basic language) are listed in Appendix D. To further miniaturize the Apple IIe microcomputer system, the full size monitor is replaced by a flat screen monitor or a single line display. A printer could be used in place or in addition to the visual displays.

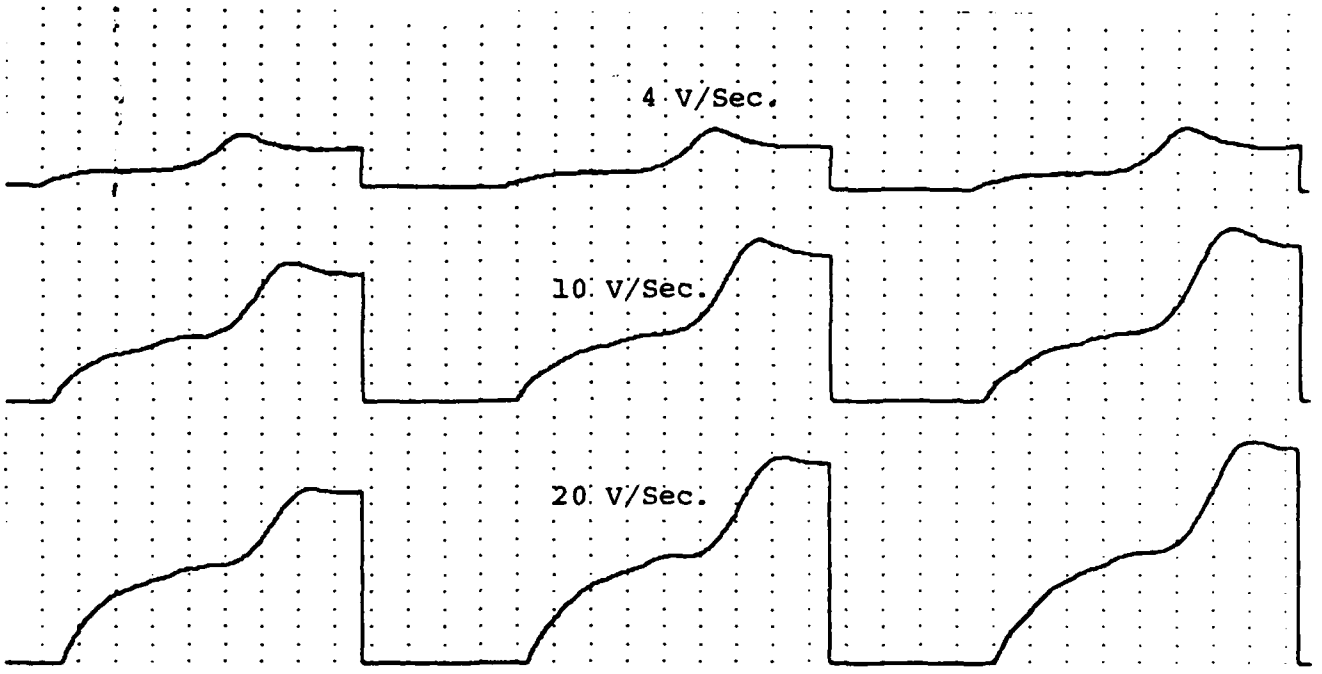


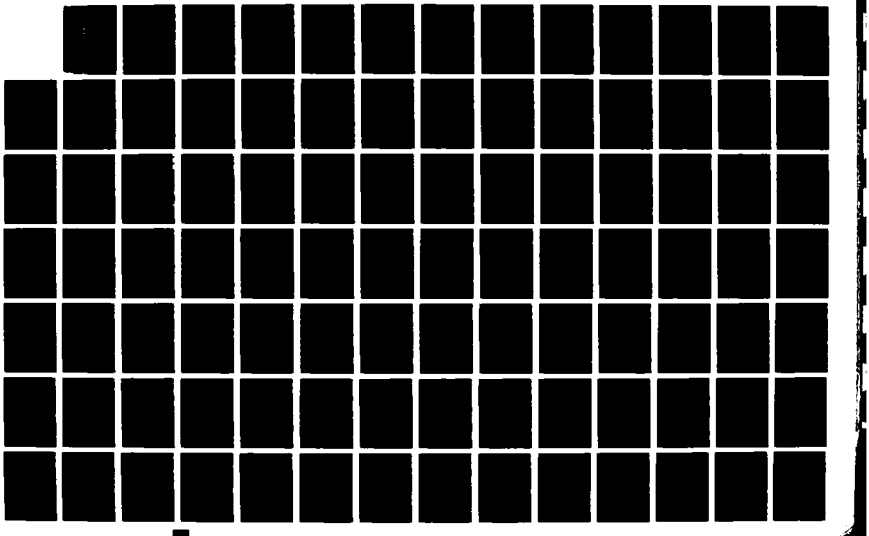
Figure 146. Reduction Voltammograms Produced by the Single Board Voltammograph with Active Filtered Input for the Fresh TEL-4004 Oil Using 4, 10, and 20 V/Sec Scan Rates and a -0.1 to 1.15V Scan Range

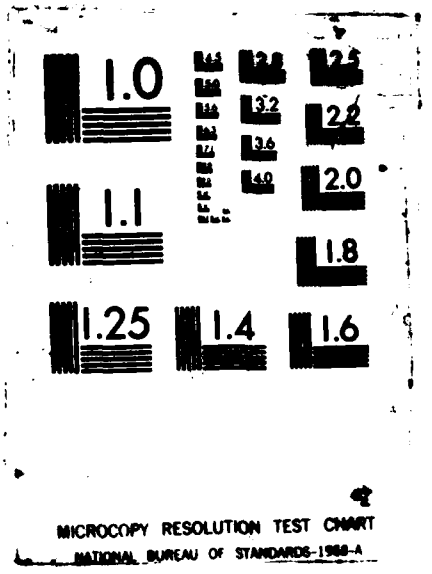
The developed single board voltammograph-Apple IIe microcomputer based RULLER candidate is more compact and less expensive than the CV-1B voltammograph-Apple IIe microcomputer based system developed in the previous investigation (Reference 74). The use of the Apple IIe microcomputer or any other suitable microcomputer system provides the RULLER candidate with permanent memory and flexibility through the use of a floppy disk system. The developed RULLER could be used to store the RUL and other pertinent information of previously analyzed oil samples so that operator independent trending analyses can be performed. The developed RULLER could also be used to study the antioxidant systems of new oil formulation or oil samples of unknown formulation which produce inaccurate RUL evaluations.

However, the main purpose of the developed single board voltammograph-Apple IIe microcomputer based RULLER candidate is to determine the optimum voltammetric parameters and other experimental conditions to be used for the development of the single board voltammograph-microprocessor based RULLER candidate. The single board voltammograph-Apple IIe microcomputer based RULLER candidate will also be used by UDRI personnel during the field testing program of the selected RULLER candidate.

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APPENDIX A

LUBRICANT PERFORMANCE TEST DATA

Appendix A contains lubricant test data obtained during the development of improved methods for measuring lubricant performance and presented in Section III of this report. This test data is tabulated as follows:

TABLE A-1. SQUIRES OXIDATIVE TEST DATA

Table A-1 contains all the Squires Oxidative test data arranged as follows:

a. Lubricants listed in order of specification and lubricant code number sequence.

b. Each lubricant's test data listed in order of increasing temperatures.

c. Esters listed in sample code sequence.

d. Esters containing antioxidants listed in order of increasing additive concentration.

TABLE A-2. SQUIRES CONFINED HEAT TEST DATA

Table A-2 contains all the Squires Confined Heat test data and is arranged in the same order as described for Table A-1.

TABLE A-3 AFAPL STATIC COKER TEST DATA

Table A-3 contains all the AFAPL Static Coker test data arranged in test number sequence.

TABLE A-4. LUBRICANT COKING PROPENSITY TEST DATA

Table A-4 contains all the Coking Propensity test data and is arranged in order of increasing test temperature.

TABLE A-5. DESCRIPTION OF COKING PROPENSITY TEST DATA

Table A-5 contains the description of Coking Propensity deposits arranged in the order of type test dish, type fluid, increasing code sequence and increasing test temperature.

TABLE A-6. LUBRICANT FOAMING TEST DATA

Table A-6 contains all Lubricant Foaming test data arranged in test number sequence.

TABLE A-1

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	120	168	312	360	456	577
	Weight Loss, %		22.2	28.1	46.3	50.4	58.6	67.2
O-79-16J	COBRA Reading	3	22	29	52	57	77	110
(a)	Total Acid No.	0.20	0.16	0.23	0.48	0.53	0.55	0.55
175°C	Viscosity @100°C,cs	3.16	3.55	3.65	4.05	4.16	4.47	4.81
	Viscosity Change, %		12.3	15.5	28.2	31.6	41.4	52.2
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	Slight Varnish	Slight Varnish	Slight Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	672					
	Weight Loss, %		ND					
O-79-16Jex	COBRA Reading	3	117					
	Total Acid No.	0.20	0.60					
175°C	Viscosity @100°C,cs	3.16	5.06					
	Viscosity Change, %		60.1					
	Toluene Insol, % wt	0.00	0.00					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		9.3	17.9	39.2	51.2	58.6	63.9
O-79-16J	COBRA Reading	2	6	20	51	59	35	84
(b)	Total Acid No.	0.20	0.23	0.23	0.65	0.86	0.90	0.93
190°C	Viscosity @100°C,cs	3.16	3.35	3.48	3.89	4.24	4.54	4.83
	Viscosity Change, %		6.0	10.1	23.1	34.2	43.7	52.8
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	Slight Varnish	None	Slight Varnish

(a) - see O-79-16Jex for extended testing results.
(b) - See O-79-16Jex for extended testing results.

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	288	312	408	505		
	Weight Loss, %		66.8	68.7	76.5	81.4		
O-79-16Jex	COBRA Reading	2	91	114	148	139		
	Total Acid No.	0.20	1.11	1.01	1.58	2.19		
190°C	Viscosity @100°C,cs	3.16	4.97	5.08	6.00	6.71		
	Viscosity Change, %		57.3	60.8	89.9	112.3		
	Toluene Inscl, % wt	0.00	ND	ND	ND	0.02		
	Visual Appearance of Deposits		Slight Varnish	Slight Varnish	Slight Varnish	Slight Varnish		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		15.1	29.8	59.0	69.0	76.0	80.4
O-79-16J	COBRA Reading	3	26	45	95	106	108	112
	Total Acid No.	0.20	0.51	0.78	1.49	1.79	1.98	2.36
205°C	Viscosity @100°C,cs	3.16	3.43	3.72	4.63	5.23	5.94	6.77
	Viscosity Change, %		8.5	17.7	46.5	65.5	88.0	114.2
	Toluene Insol, % wt	0.00	0.01	0.00	0.00	0.00	0.00	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	96			
	Weight Loss, %		22.4	34.2	70.8			
O-79-16	COBRA Reading	3	30	51	70			
	Total Acid No.	0.20	1.00	1.84	7.83			
210°C	Viscosity @100°C,cs	3.16	3.59	3.89	17.14			
	Viscosity Change, %		13.6	23.1	442.4			
	Toluene Insol, % wt	0.00	ND	ND	ND			
	Visual Appearance of Deposits		None	None	Slight varnish			

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		23.2	32.4	53.1			
O-79-16	COBRA Reading	2	77	163	85			
	Total Acid No.	.20	6.57	13.40	8.60			
215°C	Viscosity @100°C,cs	3.16	4.04	8.22	16.44			
	Viscosity Change, %		27.8	160.1	420.2			
	Toluene Insol, % wt	0.00	ND	ND	0.04			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	120	168	312	360	456	577
	Weight Loss, %		20.3	23.3	32.2	36.0	40.3	46.0
O-79-17E	COBRA Reading	2	36	39	56	67	70	86
(a)	Total Acid No.	0.08	0.30	0.26	0.34	0.35	0.59	0.37
175°C	Viscosity @100°C,cs	3.35	3.92	3.97	4.08	4.11	4.18	4.29
	Viscosity Change, %		17.0	18.5	21.8	22.7	24.8	28.1
	Toluene Insol, % wt	0.01	ND	ND	ND	ND	ND	0.02
	Visual Appearance of Deposits		None	None	Slight Varnish	Varnish	Varnish	Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	696					
	Weight Loss, %		53.8					
O-79-17Eex	COBRA Reading	2	151					
	Total Acid No.	0.08	0.41					
175°C	Viscosity @100°C,cs	3.35	4.39					
	Viscosity Change, %		31.0					
	Toluene Insol, % wt	0.01	0.03					
	Visual Appearance of Deposits		Varnish					

(a) - see O-79-17Eex for extended testing results.

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
0-79-17E (a) 190°C	Weight Loss, %		11.3	18.8	29.4	35.9	42.1	45.5
	COBRA Reading	2	22	32	64	62	74	64
	Total Acid No.	0.08	0.23	0.33	0.69	0.81	0.66	0.64
	Viscosity @100°C,cs	3.35	3.66	3.87	4.08	4.18	4.27	4.37
	Viscosity Change, %		9.3	15.5	21.8	24.8	27.5	30.4
	Toluene Insol, % wt	0.01	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	Tacky Crystals	Tacky Crystals	Tacky Crystals

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	288	312	360	457	576	
0-79-17Eex 190°C	Weight Loss, %		48.6	52.5	55.3	65.0	76.1	
	COBRA Reading	2	79	88	76	91	101	
	Total Acid No.	0.08	0.89	1.08	0.93	1.03	1.52	
	Viscosity @100°C,cs	3.35	4.42	4.47	4.58	5.05	6.22	
	Viscosity Change, %		31.9	33.4	36.7	50.7	85.7	
	Toluene Insol, % wt	0.01	ND	ND	ND	ND	0.04	
	Visual Appearance of Deposits		Tacky	Tacky	Tacky Varnish	Tacky Varnish	Tacky Varnish	

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	168	216	264			
0-79-17E 201°C	Weight Loss, %		56.8	66.4	69.3			
	COBRA Reading	2	77	74	81			
	Total Acid No.	0.08	2.02	3.91	2.47			
	Viscosity @100°C,cs	3.35	4.80	5.40	5.60			
	Viscosity Change, %		43.3	61.2	67.2			
	Toluene Insol, % wt	0.01	0.00	0.01	ND			
	Visual Appearance of Deposits		None	None	None			

(a) - See 0-79-17Eex for extended test results.

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		14.9	25.2	42.0	61.5	70.2	79.8
O-79-17E	COBRA Reading	2	36	52	71	68	72	68
	Total Acid No.	.08	.52	.77	1.05	2.43	2.90	4.04
205°C	Viscosity @100°C,cs	3.35	3.79	4.03	4.31	5.11	6.21	8.29
	Viscosity Change, %		13.1	20.3	28.7	52.5	85.4	147.5
	Toluene Insol, % wt	0.01	0.00	0.00	0.00	0.00	0.00	0.00
	Visual Appearance of Deposits		None	None	Slight Varnish	Slight Varnish	Slight Varnish	Slight Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	144			
	Weight Loss, %		22.1	30.0	64.0			
O-79-17	COBRA Reading	2	44	44	52			
	Total Acid No.	0.08	1.22	1.05	4.17			
210°C	Viscosity @100°C,cs	3.35	4.00	4.22	6.33			
	Viscosity Change, %		19.4	25.5	88.9			
	Toluene Insol, % wt	0.01	ND	ND	ND			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		23.2	49.8	63.5			
O-79-17	COBRA Reading	2	68	108	89			
	Total Acid No.	.08	1.65	10.55	8.14			
215°C	Viscosity @100°C,cs	3.35	4.07	8.74	12.09			
	Viscosity Change, %		21.5	160.9	260.9			
	Toluene Insol, % wt	0.01	ND	ND	0.04			
	Visual Appearance of Deposits		None	None	None			

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	120	168	288	383	480	768
	Weight Loss, %		12.9	17.2	28.8	36.0	41.9	53.9
O-79-20C	COBRA Reading	2	25	29	49	58	42	62
	Total Acid No.	0.20	0.22	0.30	0.48	0.42	0.50	0.67
175°C	Viscosity @100°C,cs	3.47	3.74	3.81	3.99	4.11	4.23	4.50
	Viscosity Change, %		7.8	9.8	15.0	18.4	21.9	29.7
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	Varnish on tube head	Varnish on tube head	Varnish on tube head

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		6.4	11.7	26.6	36.3	42.2	48.8
O-79-20A	COBRA Reading	2	11	21	34	41	51	57
(a)	Total Acid No.	0.20	0.10	0.28	0.61	0.93	0.93	1.40
190°C	Viscosity @100°C,cs	3.47	3.64	3.75	3.97	4.12	4.36	4.24
	Viscosity Change, %		4.9	8.1	14.4	18.7	25.6	22.2
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	0.00	0.04
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	288	312	360	457	553	
	Weight Loss, %		49.0	53.5	58.3	69.8	76.6	
O-79-20Aex	COBRA Reading	2	65	75	63	74	84	
	Total Acid No.	0.20	1.24	1.37	1.33	1.41	1.99	
190°C	Viscosity @100°C,cs	3.47	4.45	4.58	4.75	5.37	6.20	
	Viscosity Change, %		28.2	32.0	36.9	54.8	78.7	
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	0.02	
	Visual Appearance of Deposits		Slight Varnish	Slight Varnish	Varnish	Varnish	Varnish	

(a) - See O-79-20Aex for extended testing results.

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		11.2	23.0	47.0	61.9	70.9	70.3
0-79-20A	COBRA Reading	2	26	45	57	59	54	62
	Total Acid No.	0.20	0.50	1.10	2.29	3.07	3.83	3.50
201°C	Viscosity @100°C,cs	3.47	3.72	3.92	4.55	5.14	6.09	5.87
	Viscosity Change, %		+7.2	+13.0	+31.1	+48.1	+75.5	+69.2
	Toluene Insol, % wt	0.00	0.01	0.00	0.00	0.00	0.00	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		12.8	24.9	51.1	61.4	72.5	80.7
0-79-20A	COBRA Reading	2	25	38	54	62	52	48
	Total Acid No.	0.20	0.55	0.75	2.08	4.38	4.65	5.88
205°C	Viscosity @100°C,cs	3.47	3.73	3.97	4.69	5.67	7.12	9.87
	Viscosity Change, %		7.5	14.4	35.2	63.4	105.2	184.4
	Toluene Insol, % wt	0.00	0.01	0.00	0.00	0.01	0.01	0.01
	Visual Appearance of Deposits		None	None	Slight Varnish	Slight Varnish	Slight Varnish	Slight Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		16.5	30.2				
0-79-20	COBRA Reading	2	37	74				
	Total Acid No.	0.20	1.01	8.92				
210°C	Viscosity @100°C,cs	3.47	3.83	4.85				
	Viscosity Change, %		10.4	39.8				
	Toluene Insol, % wt	0.00	ND	ND				
	Visual Appearance of Deposits		None	None				

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		20.1	52.3	45.1			
0-79-20	COBRA Reading	2	76	66	69			
	Total Acid No.	.20	6.35	11.46	12.80			
215°C	Viscosity @100°C,cs	3.47	4.42	15.52	22.20			
	Viscosity Change, %		27.4	360.5	539.8			
	Toluene Insol, % wt	0.00	ND	ND	0.09			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48	120	168	288		
	Weight Loss, %		4.5	12.8	17.6	33.9		
0-82-2	COBRA Reading	3	23	157	174	200		
	Total Acid No.	0.08	0.98	3.16	4.67	10.20		
175°C	Viscosity @100°C,cs	3.33	3.41	3.70	3.76	4.48		
	Viscosity Change, %		2.4	11.1	12.9	34.5		
	Toluene Insol, % wt	ND	ND	ND	ND	0.01		
	Visual Appearance of Deposits		None	Tacky dep on tube head	Tacky dep on tube head	Tacky dep on tube head		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		5.8	11.1	27.0	35.5	45.7	55.1
0-82-2	COBRA Reading	3	37	83	183	187	184	182
	Total Acid No.	0.08	0.73	1.63	4.27	6.36	8.32	10.11
190°C	Viscosity @100°C,cs	3.33	3.46	3.56	3.96	4.28	4.98	6.35
	Viscosity Change, %		3.9	6.9	18.9	28.5	49.5	90.7
	Toluene Insol, % wt	ND	ND	ND	ND	ND	0.02	0.04
	Visual Appearance of Deposits		None	None	None	None	None	None

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		13.1	24.4	55.4	72.3	79.1	84.4
O-82-2	COBRA Reading	3	99	148	130	89	32	4
	Total Acid No.	0.08	1.90	3.20	5.47	6.29	5.89	5.35
205°C	Viscosity @100°C,cs	3.33	3.59	3.82	5.22	10.02	27.63	ND
	Viscosity Change, %		7.8	14.7	56.8	201	730	N/A
	Toluene Insol, % wt	ND	ND	ND	ND	0.00	0.04	0.04
	Visual Appearance of Deposits		None	None	None	Light Thick Oil	Dark Thick Oil	Very Thick Oil

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	120	168	288	383	480	576
	Weight Loss, %		20.8	27.4	45.0	52.8	61.4	62.2
O-82-3	COBRA Reading	8	23	30	38	70	59	62
	Total Acid No.	0.12	0.22	0.29	0.40	0.53	0.55	0.54
175°C	Viscosity @100°C,cs	3.45	3.91	4.04	4.53	4.87	5.41	5.51
	Viscosity Change, %		13.3	17.1	31.3	41.2	56.8	59.7
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		8.9	17.6	40.8	54.4	60.1	67.0
O-82-3D	COBRA Reading	8	10	22	50	49	61	79
(a)	Total Acid No.	0.12	0.18	0.15	0.66	0.89	1.10	0.99
190°C	Viscosity @100°C,cs	3.45	3.71	3.86	4.43	4.90	5.49	6.23
	Viscosity Change, %		7.5	11.9	28.4	42.0	59.1	80.6
	Toluene Insol, % wt	ND	ND	ND	ND	ND	0.00	0.03
	Visual Appearance of Deposits		Clear Hard	Clear Hard	Clear Hard	Clear Hard	Clear Hard	Clear Hard

(a) - See O-82-3Dex for extended testing results.

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New C 1	288					
	Weight Loss, %		71.3					
O-82-3Dex	COBRA Reading	8	81					
	Total Acid No.	0.12	1.28					
190°C	Viscosity @100°C,cs	3.45	6.79					
	Viscosity Change, %		96.8					
	Toluene Insol, % wt	ND	0.03					
	Visual Appearance of Deposits		Hard Clear					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		19.1	36.9	71.5	78.4	83.3	ND
O-82-3	COBRA Reading	8	33	66	102	75	73	ND
	Total Acid No.	.12	.68	1.30	2.44	2.73	4.77	ND
205°C	Viscosity @100°C,cs	3.45	3.91	4.35	7.25	9.88	13.13	ND
	Viscosity Change, %		13.3	26.1	110	186	281	N/A
	Toluene Insol, % wt	ND	ND	ND	ND	0.00	0.00	ND
	Visual Appearance of Deposits		None	None	Sticky Varnish Deposits	Grainy Varnish Deposits	Sticky Varnish Deposits	N/A

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	96			
	Weight Loss, %		21.8	39.1	66.7			
O-82-3	COBRA Reading	8	37	21	80			
	Total Acid No.	0.12	0.87	1.72	3.46			
210°C	Viscosity @100°C,cs	3.45	3.67	4.48	6.96			
	Viscosity Change, %		15.4	29.8	101.7			
	Toluene Insol, % wt	ND	ND	ND	ND			
	Visual Appearance of Deposits		None	None	Tacky crystals sl. varn.			

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		25.8	54.4	37.9			
O-82-3	COBRA Reading	8	58	90	160			
	Total Acid No.	.12	1.72	8.76	19.29			
215°C	Viscosity @100°C,cs	3.45	4.14	8.01	11.30			
	Viscosity Change, %		20.0	132.2	227.5			
	Toluene Insol, % wt	ND	ND	ND	0.06			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	120	216	312	408		
	Weight Loss, %		12.0	22.1	32.5	38.9		
O-82-14D	COBRA Reading	3	200	200	200	200		
	Total Acid No.	0.15	1.04	2.08	3.47	4.18		
175°C	Viscosity @100°C,cs	3.40	3.75	3.94	4.35	4.82		
	Viscosity Change, %		10.3	15.9	27.9	41.8		
	Toluene Insol, % wt	ND	ND	ND	ND	0.01		
	Visual Appearance of Deposits		None	None	None	None		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		6.1	11.6	27.7	37.3	45.4	56.5
O-82-14D	COBRA Reading	3	185	200	200	200	200	200
	Total Acid No.	0.15	0.88	1.04	2.75	3.78	4.03	4.82
190°C	Viscosity @100°C,cs	3.40	3.66	3.74	4.10	4.47	5.00	6.36
	Viscosity Change, %		7.6	10.0	20.6	31.5	47.1	87.1
	Toluene Insol, % wt	ND	ND	ND	ND	ND	0.01	0.03
	Visual Appearance of Deposits		None	Varnish at Oil Level	Varnish at Oil Level	Varnish at Oil Level	Clear Hard	Clear Hard

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	
	Weight Loss, %		14.0	25.2	56.9	70.9	75.0	
0-82-14D	COBRA Reading	3	200	200	200	200	197	
	Total Acid No.	0.15	1.58	2.87	4.85	7.43	7.87	
205°C	Viscosity @100°C,cs	3.40	3.79	4.01	5.69	9.03	14.01	
	Viscosity Change, %		11.5	17.9	67.4	165.6	312.1	
	Toluene Insol, % wt	ND	0.00	0.01	0.01	0.01	0.03	
	Visual Appearance of Deposits		Light Crystals	Light Crystals	Light Crystals	Light Crystals Varnish	Light Crystals Varnish	

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil						
	Weight Loss, %							
	COBRA Reading							
	Total Acid No.							
	Viscosity @100°C,cs							
	Viscosity Change, %							
	Toluene Insol, % wt							
	Visual Appearance of Deposits							

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil						
	Weight Loss, %							
	COBRA Reading							
	Total Acid No.							
	Viscosity @100°C,cs							
	Viscosity Change, %							
	Toluene Insol, % wt							
	Visual Appearance of Deposits							

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		4.8	8.7	19.8	27.9		
0-71-6	COBRA Reading	2	20	30	39	53		
	Total Acid No.	0.06	0.35	0.74	1.21	7.84		
205°C	Viscosity @100°C,cs	4.95	5.46	5.65	6.07	7.80		
	Viscosity Change, %		10.3	14.1	22.6	57.6		
	Toluene Insol, % wt	ND	ND	ND	ND	0.03		
	Visual Appearance of Deposits		None	None	None	Varnish		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	168	216	264	
	Weight Loss, %		4.1	7.3	22.5	50.6	58.2	
0-77-15	COBRA Reading	2	32	59	63	54	27	
(a)	Total Acid No.	.43	0.74	0.52	2.21	5.96	6.29	
205°C	Viscosity @100°C,cs	4.95	5.32	5.50	6.46	20.64	ND	
	Viscosity Change, %		7.5	11.1	30.5	417.0	N/A	
	Toluene Insol, % wt	ND	ND	ND	ND	ND	0.06	
	Visual Appearance of Deposits		None	White Crystals	White + Brown Crystals	Slight Coke	Clear Deposit	

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	360
	Weight Loss, %		7.1	13.8	32.7	42.8	51.6	ND
0-85-1	COBRA Reading	8	36	63	109	119	110	110
	Total Acid No.	0.02	0.12	0.33	0.67	0.96	1.53	2.54
205°C	Viscosity @100°C,cs	4.04	4.14	4.23	4.55	5.03	5.22	9.94
	Viscosity Change, %		2.5	4.7	12.6	24.5	29.2	146.0
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	ND
	Visual Appearance of Deposits		None	Tacky Crystals	White Crystals	Crystals	Crystals	Crystals and Varnish

(a) - (TEL 6021)

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		10.4	22.1				
TEL 6031	COBRA Reading	14	200	200				
	Total Acid No.	0.06	1.37	4.99				
205°C	Viscosity @100°C, cs	7.53	8.45	9.33				
	Viscosity Change, %		12.2	23.9				
	Toluene Insol, % wt	ND	ND	0.00				
	Visual Appearance of Deposits		Slight Coke	Slight Coke				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		28.7					
TEL 6032	COBRA Reading	5	ND					
	Total Acid No.	0.02	12.85					
205°C	Viscosity @100°C, cs	7.50	6.42					
	Viscosity Change, %		-14.4					
	Toluene Insol, % wt	ND	0.05					
	Visual Appearance of Deposits		Coke					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil						
	Weight Loss, %							
	COBRA Reading							
	Total Acid No.							
	Viscosity @100°C, cs							
	Viscosity Change, %							
	Toluene Insol, % wt							
	Visual Appearance of Deposits							

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		22.6	37.1				
0-76-5A	COBRA Reading	1	20	25				
(a)	Total Acid No.	0.25	19.85	20.24				
175°C	Viscosity @100°C,cs	3.46	6.87	12.87				
	Viscosity Change, %		98.6	272.0				
	Toluene Insol, % wt	ND	ND	ND				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		29.8	52.8				
0-76-5A	COBRA Reading	1	31	24				
(b)	Total Acid No.	.25	13.99	16.52				
190°C	Viscosity @100°C,cs	3.46	8.36	19.25				
	Viscosity Change, %		141.6	456.4				
	Toluene Insol, % wt	ND	ND	0.05				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		42.8	61.4				
0-76-5A	COBRA Reading	1	21	7				
(c)	Total Acid No.	0.25	16.54	14.81				
205°C	Viscosity @100°C,cs	3.46	14.08	52.71				
	Viscosity Change, %		306.9	1568				
	Toluene Insol, % wt	ND	0.04	0.01				
	Visual Appearance of Deposits		None	None				

(a) - Basestock Oil
(b) - Basestock Oil
(c) - Basestock Oil

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		24.4	38.0				
0-76-8	COBRA Reading	6	200	200				
(a)	Total Acid No.	0.09	50.17	62.50				
175°C	Viscosity @100°C,cs	2.36	4.71	6.26				
	Viscosity Change, %		99.6	165.2				
	Toluene Insol, % wt	ND	ND	ND				
	Visual Appearance of Deposits		Clear Crystals	Clear Crystals				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		26.9	41.5				
0-76-8	COBRA Reading	6	200	200				
(b)	Total Acid No.	.09	44.07	48.22				
190°C	Viscosity @100°C,cs	2.36	4.68	6.42				
	Viscosity Change, %		98.3	172.0				
	Toluene Insol, % wt	ND	ND	0.12				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		18.7	37.4				
0-76-8	COBRA Reading	6	200	ND				
(c)	Total Acid No.	0.09	49.03	56.70				
205°C	Viscosity @100°C,cs	2.36	4.95	ND				
	Viscosity Change, %		109.7	N/A				
	Toluene Insol, % wt	ND	0.06	ND				
	Visual Appearance of Deposits		Thick dark oil	Black Deposits				

(a) - Basestock Oil
(b) - Basestock Oil
(c) - Basestock Oil

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		26.6					
O-77-1	COBRA Reading	4	182					
	Total Acid No.	0.03	58.82					
205°C	Viscosity @100°C,cs	2.33	7.04					
	Viscosity Change, %		202.1					
	Toluene Insol, % wt	ND	ND					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		25.5					
O-77-1+I	COBRA Reading	2	115					
(a)	Total Acid No.	0.03	54.20					
205°C	Viscosity @100°C,cs	2.34	6.85					
	Viscosity Change, %		192.7					
	Toluene Insol, % wt	ND	ND					
	Visual Appearance of Deposits		White Crystals on Tube					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		12.4					
O-77-1+P	COBRA Reading	9	48					
(b)	Total Acid No.	.03	1.18					
205°C	Viscosity @100°C,cs	2.44	2.42					
	Viscosity Change, %		-0.8					
	Toluene Insol, % wt	ND	ND					
	Visual Appearance of Deposits		None					

(a) - O-77-1 plus impurities
(b) - O-77-1 plus 1% PANA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		14.4					
O-77-1+IP	COBRA Reading	10	16					
(a)	Total Acid No.	.02	1.08					
205°C	Viscosity @100°C,cs	2.34	2.49					
	Viscosity Change, %		6.4					
	Toluene Insol, % wt	ND	ND					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120			
	Weight Loss, %		5.3	7.5	18.2			
UDRI 20	COBRA Reading	4	5	9	29			
(b)	Total Acid No.	.10	0.24	0.30	0.38			
190°C	Viscosity @100°C,cs	3.51	3.61	3.70	3.80			
	Viscosity Change, %		2.9	5.4	8.3			
	Toluene Insol, % wt		ND	ND	ND			
	Visual Appearance of Deposits		None	Tacky Deposit	Varnish			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		39.4	61.0				
UDRI 09	COBRA Reading	2	27	6				
(c)	Total Acid No.	0.14	14.93	15.66				
205°C	Viscosity @100°C,cs	3.47	11.76	ND				
	Viscosity Change, %		238.9	N/A				
	Toluene Insol, % wt	ND	0.01	0.05				
	Visual Appearance of Deposits		None	Slight Varnish				

(a) - O-77-1 plus impurities and 1% PANA
(b) - O-76-5A w/2% PANA
(c) - O-76-5 w/0.05% PANA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120			
	Weight Loss, %		36.7	61.8	73.4			
UDRI 10	COBRA Reading	2	33	10	ND			
(a)	Total Acid No.	0.06	14.42	16.09	ND			
205°C	Viscosity @100°C,cs	3.46	9.77	ND	ND			
	Viscosity Change, %		182.4	N/A	N/A			
	Toluene Insol, % wt	ND	0.00	ND	ND			
	Visual Appearance of Deposits		None	None	Rubbery			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		32.7	59.5	76.3	78.1		
UDRI 11	COBRA Reading	3	33	17	ND	ND		
(b)	Total Acid No.	0.15	13.4	15.9	ND	ND		
205°C	Viscosity @100°C,cs	3.46	8.31	ND	ND	ND		
	Viscosity Change, %		140.2	N/A	N/A	N/A		
	Toluene Insol, % wt	ND	0.00	ND	ND	ND		
	Visual Appearance of Deposits		None	Varnish at Oil Level	Rubbery	Rubbery		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		26.0	57.0	75.5	77.5		
UDRI 12	COBRA Reading	2	24	ND	ND	ND		
(c)	Total Acid No.	0.16	12.07	16.11	ND	ND		
205°C	Viscosity @100°C,cs	3.47	6.63	ND	ND	ND		
	Viscosity Change, %		91.1	N/A	N/A	N/A		
	Toluene Insol, % wt	ND	0.00	ND	ND	ND		
	Visual Appearance of Deposits		None	None	Rubbery	Rubbery		

(a) - O-76-5 w/0.1% PANA
(b) - O-76-5 w/0.15% PANA
(c) - O-76-5 w/0.2% PANA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	96	168		
	Weight Loss, %		8.6	16.4	64.1	79.5		
UDRI 17	COBRA Reading	3	6	12	48	ND		
(a)	Total Acid No.	.09	0.29	0.55	9.09	ND		
205°C	Viscosity @100°C, cs	3.46	3.56	3.60	30.52	ND		
	Viscosity Change, %		2.9	4.0	782.1	NA		
	Toluene Insol, % wt	ND	ND	0.02	ND	ND		
	Visual Appearance of Deposits		None	None	None	Solid Rubbery		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		9.8	15.8	35.7	81.5		
UDRI 18	COBRA Reading	3	6	15	32	ND		
(b)	Total Acid No.	0.10	0.31	0.41	0.66	13.52		
205°C	Viscosity @100°C, cs	3.47	3.61	3.65	3.79	ND		
	Viscosity Change, %		4.0	5.2	9.2	N/A		
	Toluene Insol, % wt	ND	ND	ND	.04	ND		
	Visual Appearance of Deposits		None	None	Slightly Sticky	Very Thick Oil		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168	216	264
	Weight Loss, %		8.6	17.5	35.1	52.6	64.5	79.9
UDRI 19	COBRA Reading	4	7	21	42	64	74	126
(c)	Total Acid No.	0.10	0.36	0.46	0.69	0.88	1.35	2.38
205°C	Viscosity @100°C, cs	3.50	3.66	3.73	3.88	4.11	4.52	5.68
	Viscosity Change, %		4.6	6.6	10.9	17.4	29.1	62.3
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	.03
	Visual Appearance of Deposits		None	Tacky Deposits	Tacky Deposits	Tacky Deposits	Tacky Deposits	Varnish

(a) - O-76-5A w/0.5% PANA
(b) - O-76-5A 1.0% PANA
(c) - O-76-5A 1.5% PANA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS					
		New Oil	24	48	168	216	264
UDRI 20 (a) 205°C	Weight Loss, %		8.4	16.1	28.8	66.6	80.2
	COBRA Reading	4	9	23	62	98	132
	Total Acid No.	.10	0.41	0.64	0.77	1.45	2.30
	Viscosity @100°C,cs	3.51	3.71	3.80	4.26	4.82	6.28
	Viscosity Change, %		5.7	8.3	21.4	37.3	78.9
	Toluene Insol, % wt	ND	ND	ND	ND	ND	0.05
	Visual Appearance of Deposits		None	Tacky Deposits	Tacky Deposits	Tacky Deposits	Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120			
UDRI 16 (b) 190°C	Weight Loss, %		3.9	8.1	19.7			
	COBRA Reading	0	12	14	23			
	Total Acid No.	.15	0.65	0.42	0.69			
	Viscosity @100°C,cs	3.61	3.66	3.72	3.86			
	Viscosity Change, %		1.4	3.0	6.9			
	Toluene Insol, % wt	ND	ND	ND	ND			
	Visual Appearance of Deposits		None	None	White Crystals			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
UDRI 13 (c) 205°C	Weight Loss, %		25.8	56.8				
	COBRA Reading	1	70	20				
	Total Acid No.	0.17	18.35	14.76				
	Viscosity @100°C,cs	3.48	8.06	36.38				
	Viscosity Change, %		131.6	945.4				
	Toluene Insol, % wt	ND	ND	0.03				
	Visual Appearance of Deposits		None	None				

(a) - O-76-5A w/2.0% PANA
(b) - O-76-5A w/2% DODPA
(c) - O-76-5A w/0.5% DODPA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	240					
	Weight Loss, %		92.4					
UDRI 16ex	COBRA Reading	0	ND					
(a)	Total Acid No.	0.15	ND					
205°C	Viscosity @100°C,cs	3.61	ND					
	Viscosity Change, %		ND					
	Toluene Insol, % wt	ND	ND					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		7.0	13.8				
UDRI 24	COBRA Reading	3	5	6				
(b)	Total Acid No.	0.13	0.47	0.53				
205°C	Viscosity @100°C,cs	3.52	3.67	3.72				
	Viscosity Change, %		4.3	5.7				
	Toluene Insol, % wt	0.01	0.06	0.01				
	Visual Appearance of Deposits		Heavy Coke, Oil Reddish	Heavy Coke, Oil Reddish				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		7.2	18.2				
UDRI 25	COBRA Reading	4	7	110				
(c)	Total Acid No.	0.10	0.38	5.43				
205°C	Viscosity @100°C,cs	3.59	3.67	5.43				
	Viscosity Change, %		2.2	32.3				
	Toluene Insol, % wt	0.03	0.05	0.01				
	Visual Appearance of Deposits		Heavy Coke Purple Stain	Slight Varnish				

(a) - O-76-5A w/2.0% DODPA
(b) - O-76-5 w/2% PTZ
(c) - O-76-5 w/2% DOPTZ

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		15.4	31.9				
UDRI 04	COBRA Reading	14	163	157				
(a)	Total Acid No.	.07	5.00	10.76				
190°C	Viscosity @100°C,cs	2.38	2.64	2.69				
	Viscosity Change, %		10.9	13.0				
	Toluene Insol, % wt	ND	ND	ND				
	Visual Appearance of Deposits		Black Coke Particles	Black Coke Particles				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		19.3	35.8				
UDRI 01	COBRA Reading	6	76	62				
(b)	Total Acid No.	0.05	11.89	16.93				
205°C	Viscosity @100°C,cs	2.37	2.63	2.84				
	Viscosity Change, %		11.0	19.8				
	Toluene Insol, % wt	ND	0.06	0.02				
	Visual Appearance of Deposits		Coke on samp. and air tubes	Coke on samp. and air tubes				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		23.4	41.0	89.4	95.3		
UDRI 02	COBRA Reading	6	78	55	42	ND		
(c)	Total Acid No.	0.06	9.55	9.75	7.82	ND		
205°C	Viscosity @100°C,cs	2.37	2.67	2.80	ND	ND		
	Viscosity Change, %		12.7	18.1	N/A	N/A		
	Toluene Insol, % wt	ND	0.01	0.05	ND	ND		
	Visual Appearance of Deposits		Coke on samp. and air tubes	Coke on samp. and air tubes	Much coke v. little oil left	Much coke no oil		

(a) - O-76-8 w/2% PANA
(b) - O-76-8 w/0.5% PANA
(c) - O-76-8 w/1.0% PANA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		18.8	36.9	91.7	92.7		
UDRI 03	COBRA Reading	10	80	65	53	57		
(a)	Total Acid No.	0.05	9.16	8.56	ND	ND		
205°C	Viscosity @100°C,cs	2.38	2.68	2.77	ND	ND		
	Viscosity Change, %		12.6	16.4	N/A	N/A		
	Toluene Insol, % wt	ND	0.10	0.08	ND	ND		
	Visual Appearance of Deposits		Coke on samp. and air tubes	Coke on samp. and air tubes	Coke on samp. and air tubes	Coke on samp. and air tubes		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120	168		
	Weight Loss, %		20.9	51.1	92.9	94.4		
UDRI 04	COBRA Reading	14	95	41	73	ND		
(b)	Total Acid No.	.07	5.74	4.06	ND	ND		
205°C	Viscosity @100°C,cs	2.38	2.63	2.81	ND	ND		
	Viscosity Change, %		10.5	18.1	N/A	N/A		
	Toluene Insol, % wt	ND	0.02	0.34	ND	ND		
	Visual Appearance of Deposits		Coke on samp. and air tubes	Coke on samp. and air tubes	Much Coke V. Little Oil Left	Much Coke No Oil		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		14.2	30.2				
UDRI 08	COBRA Reading	3	200	200				
(c)	Total Acid No.	.05	4.56	10.06				
190°C	Viscosity @100°C,cs	2.43	2.63	2.81				
	Viscosity Change, %		8.2	15.6				
	Toluene Insol, % wt	ND	ND	ND				
	Visual Appearance of Deposits		None	None				

(a) - O-76-8 W/ 1.5% PANA
(b) - O-76-8 W/ 2.0% PANA
(c) - O-76-8 w/2% DODPA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
UDRI 05 (a) 205°C	Weight Loss, %		16.1	37.0				
	COBRA Reading	2	200	200				
	Total Acid No.	.01	15.68	26.97				
	Viscosity @100°C,cs	2.37	2.69	3.02				
	Viscosity Change, %		13.5	27.4				
	Toluene Insol, % wt	ND	0.01	0.01				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
UDRI 06 (b) 205°C	Weight Loss, %		16.8	34.8				
	COBRA Reading	3	200	200				
	Total Acid No.	.02	13.46	22.12				
	Viscosity @100°C,cs	2.37	2.70	3.00				
	Viscosity Change, %		13.9	26.6				
	Toluene Insol, % wt	ND	0.00	0.00				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
UDRI 07 (c) 205°C	Weight Loss, %		16.8	26.9				
	COBRA Reading	5	200	200				
	Total Acid No.	.04	12.43	24.18				
	Viscosity @100°C,cs	2.41	2.74	3.01				
	Viscosity Change, %		13.7	24.9				
	Toluene Insol, % wt	ND	0.02	0.02				
	Visual Appearance of Deposits		None	None				

(a) - O-76-8 w/0.5 % DODPA
(b) - O-76-8 w/ 1.0% DODPA
(c) - O-76-8 w/1.5% DODPA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		19.4	29.7				
UDRI 08	COBRA Reading	3	200	200				
(a)	Total Acid No.	.05	11.58	17.72				
205°C	Viscosity @100°C, cs	2.43	2.78	3.00				
	Viscosity Change, %		14.4	23.5				
	Toluene Insol, % wt	ND	0.01	0.00				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		21.1					
UDRI 26	COBRA Reading	9	25					
(b)	Total Acid No.	0.04	11.92					
205°C	Viscosity @100°C, cs	2.39	2.73					
	Viscosity Change, %		14.2					
	Toluene Insol, % wt	0.01	0.04					
	Visual Appearance of Deposits		Heavy Coke, Oil Reddish					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24					
	Weight Loss, %		22.3					
UDRI 27	COBRA Reading	6	24					
(c)	Total Acid No.	0.03	9.93					
205°C	Viscosity @100°C, cs	2.45	2.75					
	Viscosity Change, %		12.2					
	Toluene Insol, % wt	0.02	0.05					
	Visual Appearance of Deposits		Heavy Coke, Oil Very Red					

(a) - O-76-8 w/2.0% DODPA
(b) - O-76-8 w/2% PTZ
(c) - O-76-8 w/2% DOPTZ

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		18.4	34.1				
UDRI 28	COBRA Reading	11	4	6				
(a)	Total Acid No.	0.04	0.78	0.94				
205°C	Viscosity @100°C,cs	2.37	2.42	2.44				
	Viscosity Change, %		2.1	3.0				
	Toluene Insol, % wt	0.02	0.02	0.02				
	Visual Appearance of Deposits		Heavy Coke, Oil Reddish	Heavy Coke, Oil Reddish				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		14.9	33.7				
UDRI 29	COBRA Reading	5	7	200				
(b)	Total Acid No.	0.03	0.85	9.13				
205°C	Viscosity @100°C,cs	2.42	2.42	2.66				
	Viscosity Change, %		0.0	9.9				
	Toluene Insol, % wt	0.01	0.02	0.01				
	Visual Appearance of Deposits		Heavy Coke, Oil Reddish	Slight Varnish				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120			
	Weight Loss, %		4.9	7.3	17.1			
UDRI 22	COBRA Reading	3	10	22	52			
(c)	Total Acid No.	.08	0.15	0.58	0.61			
190°C	Viscosity @100°C,cs	3.51	3.62	3.70	3.80			
	Viscosity Change, %		3.1	5.4	8.3			
	Toluene Insol, % wt	ND	ND	ND	ND			
	Visual Appearance of Deposits		None	Tacky Deposit	Slight Varnish			

(a) - O-77-1 w/2% PTZ
(b) - O-77-1 w/2% DOPTZ
(c) - O-76-5A w/ 1% PANA + 1% DODPA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	72	120	168	192	264
	Weight Loss, %		9.6	25.5	41.6	49.7	57.1	78.1
UDRI 22	COBRA Reading	3	20	59	74	104	87	123
(a)	Total Acid No.	.08	0.44	0.57	0.72	0.72	0.93	2.41
205°C	Viscosity @100°C,cs	3.51	3.75	3.90	4.10	4.25	4.43	5.96
	Viscosity Change, %		6.8	11.1	16.8	21.1	26.2	69.8
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.03
	Visual Appearance of Deposits		None	Slight Varnish	Slight Varnish	Slight Varnish	Slight Varnish	Slight Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		15.2	27.8				
UDRI 21	COBRA Reading	9	200	200				
(b)	Total Acid No.	0.02	4.95	6.26				
190°C	Viscosity @100°C,cs	2.41	2.65	2.76				
	Viscosity Change, %		9.9	14.5				
	Toluene Insol, % wt	ND	ND	ND				
	Visual Appearance of Deposits		Black Coke Particles	Black Coke Particles				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	96				
	Weight Loss, %		15.5	45.8				
UDRI 21	COBRA Reading	9	200	200				
(c)	Total Acid No.	.02	8.17	14.39				
205°C	Viscosity @100°C,cs	2.41	2.76	3.14				
	Viscosity Change, %		14.5	30.3				
	Toluene Insol, % wt	ND	ND	.07				
	Visual Appearance of Deposits		Black Coke	Black Coke				

(a) - O-76-5 w/1% PANA + 1% DODPA
(b) - O-76-8 w/1% PANA + 1% DODPA
(c) - O-76-8 w/1% PANA + 1% DODPA

SQUIRES OXIDATIVE TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48				
	Weight Loss, %		11.8	24.3				
UDRI 23	COBRA Reading	6	20	98				
(a)	Total Acid No.	.03	0.39	0.50				
190°C	Viscosity @100°C,cs	2.35	2.42	2.50				
	Viscosity Change, %		3.0	6.4				
	Toluene Insol, % wt	ND	ND	ND				
	Visual Appearance of Deposits		None	None				

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	120			
	Weight Loss, %		14.3	24.7	76.4			
UDRI 23	COBRA Reading	6	78	200	200			
(b)	Total Acid No.	.03	1.64	33.8	35.7			
205°C	Viscosity @100°C,cs	2.35	2.49	2.80	10.23			
	Viscosity Change, %		6.0	19.1	335.3			
	Toluene Insol, % wt	ND	ND	ND	0.10			
	Visual Appearance of Deposits		None	White dep on tube head	White dep on tube head			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil						
	Weight Loss, %							
	COBRA Reading							
	Total Acid No.							
	Viscosity @100°C,cs							
	Viscosity Change, %							
	Toluene Insol, % wt							
	Visual Appearance of Deposits							

(a) - O-77-1 w/1% PANA + 1% DODPA
(b) - O-77-1 w/1% PANA + 1% DODPA

TABLE A-2

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	96	192	288	384	480	577
	Weight Loss, %		0.0	0.1	0.0	0.0	1.8	1.0
O-79-16J	COBRA Reading	3	10	18	28	40	62	101
	Total Acid No.	0.20	0.34	0.51	2.28	2.03	4.18	4.58
175°C	Viscosity @100°C,cs	3.16	3.24	3.25	3.29	3.30	3.37	3.34
	Viscosity Change, %		2.5	2.8	4.1	4.4	6.6	5.7
	Toluene Insol, % wt	0.01	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	None	Slight Stain	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.9	0.1	0.4	0.1	0.0	0.7
O-79-16J	COBRA Reading	2	3	10	13	17	22	36
(a)	Total Acid No.	0.20	ND	0.40	1.13	1.83	3.52	5.34
190°C	Viscosity @100°C,cs	3.16	3.21	3.26	3.26	3.29	3.28	3.32
	Viscosity Change, %		1.6	3.2	3.2	4.1	3.8	5.1
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	240					
	Weight Loss, %		0.4					
O-79-16Jex	COBRA Reading	2	48					
	Total Acid No.	0.20	5.05					
190°C	Viscosity @100°C,cs	3.16	3.31					
	Viscosity Change, %		4.8					
	Toluene Insol, % wt	0.00	0.02					
	Visual Appearance of Deposits		None					

(a) - See O-79-16Jex for extended testing results.

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	1.5	0.4	0.0	0.1	0.9
O-79-16J	COBRA Reading	3	14	13	23	31	70	108
	Total Acid No.	0.20	0.88	2.69	3.31	4.73	6.40	8.77
205°C	Viscosity @100°C,cs	3.16	3.23	3.23	3.27	3.29	3.35	3.38
	Viscosity Change, %		2.2	2.2	3.5	4.1	6.0	7.0
	Toluene Insol, % wt	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Visual Appearance of Deposits		None	None	None	None	Slight Varnish	Slight Varnish

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	96	192	288	384	480	577
	Weight Loss, %		0.5	0.1	0.0	0.1	0.5	0.5
O-79-17E	COBRA Reading	2	16	31	52	71	61	83
	Total Acid No.	0.08	1.36	2.19	2.04	4.16	5.28	5.55
175°C	Viscosity @100°C,cs	3.35	3.39	3.43	3.49	3.49	3.52	3.52
	Viscosity Change, %		1.2	2.4	4.2	4.2	5.1	5.1
	Toluene Insol, % wt	0.01	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.1	1.5	0.0	0.2	1.9	0.9
O-79-17E	COBRA Reading	2	10	18	20	23	31	35
(a)	Total Acid No.	0.08	0.82	1.17	2.08	3.02	4.62	5.71
190°C	Viscosity @100°C,cs	3.35	3.36	3.36	3.41	3.43	3.46	3.50
	Viscosity Change, %		0.3	0.3	1.8	2.4	3.3	4.5
	Toluene Insol, % wt	0.01	ND	ND	ND	ND	0.00	0.03
	Visual Appearance of Deposits		None	None	None	None	None	None

(a) - See O-79-17Eex for extended testing results.

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	240					
	Weight Loss, %		0.0					
O-79-17Eex	COBRA Reading	2	66					
	Total Acid No.	0.08	7.30					
190°C	Viscosity @100°C,cs	3.35	3.48					
	Viscosity Change, %		3.9					
	Toluene Insol, % wt	0.01	0.03					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.0	0.0	0.0	0.6
O-79-17E	COBRA Reading	2	9	28	23	39	60	86
	Total Acid No.	0.08	1.81	2.74	3.96	5.56	7.61	9.20
201°C	Viscosity @100°C,cs	3.35	3.35	3.43	3.43	3.46	3.44	3.54
	Viscosity Change, %		0.0	2.4	2.4	3.3	2.7	5.7
	Toluene Insol, % wt	0.01	0.00	0.01	0.00	0.00	0.01	0.01
	Visual Appearance of Deposits		None	None	None	Trace of Coke Particles	Stain Oil Level in Cup	Stain Oil Level in Cup

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	96	192	288	384	480	576
	Weight Loss, %		0.3	0.0	2.6	0.0	0.0	0.3
O-79-20C	COBRA Reading	2	5	13	21	32	35	41
	Total Acid No.	0.20	0.43	1.13	2.38	3.10	2.60	3.49
175°C	Viscosity @100°C,cs	3.47	3.54	3.57	3.62	3.63	3.65	3.65
	Viscosity Change, %		2.0	2.9	4.3	4.6	5.6	5.6
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	ND	ND
	Visual Appearance of Deposits		None	Clear, hard dep. on lid	None	None	Clear, hard dep on cup	None

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	696					
	Weight Loss, %		0.1					
O-79-20Cex	COBRA Reading	2	56					
	Total Acid No.	0.02	5.34					
175°C	Viscosity @100°C,cs	3.47	3.63					
	Viscosity Change, %		4.6					
	Toluene Insol, % wt	0.00	0.03					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.8	2.8	0.9	0.0	0.6
O-79-20C	COBRA Reading	2	3	10	13	22	39	43
(a)	Total Acid No.	0.20	0.31	0.45	1.11	1.05	2.00	2.59
190°C	Viscosity @100°C,cs	3.47	3.52	3.66	3.56	3.69	3.65	3.63
	Viscosity Change, %		1.4	5.5	2.6	6.3	5.2	4.6
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	240	360				
	Weight Loss, %		0.1	0.5				
O-79-20Cex	COBRA Reading	2	50	71				
	Total Acid No.	0.20	3.41	6.20				
190°C	Viscosity @100°C,cs	3.47	3.65	3.67				
	Viscosity Change, %		5.2	5.8				
	Toluene Insol, % wt	0.00	ND	0.00				
	Visual Appearance of Deposits		Faint Oil Ring	Faint Oil Ring				

(a) - See O-79-20Cex for extended testing results.

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.0	0.1	0.4	1.5
O-79-20A	COBRA Reading	2	7	27	21	25	45	44
	Total Acid No.	0.20	1.12	1.83	3.37	6.11	6.76	7.47
201°C	Viscosity @100°C,cs	3.47	3.51	3.58	3.59	3.57	3.65	3.68
	Viscosity Change, %		1.2	3.2	3.5	2.9	5.2	6.1
	Toluene Insol, % wt	0.00	ND	ND	ND	0.00	0.00	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	1.9	0.0	2.2	1.8	1.2
O-79-20A	COBRA Reading	2	6	21	20	23	26	48
	Total Acid No.	0.20	0.94	1.58	2.46	5.11	6.90	6.57
205°C	Viscosity @100°C,cs	3.47	3.52	3.61	3.42	3.59	3.64	3.69
	Viscosity Change, %		1.4	4.0	-1.4	3.5	4.9	6.3
	Toluene Insol, % wt	0.00	ND	ND	ND	ND	0.00	0.03
	Visual Appearance of Deposits		None	None	None	None	Faint Oil Ring	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	96	144	192	240	288	384
	Weight Loss, %		0.3	0.1	0.1	0.0	0.5	0.1
O-82-2	COBRA Reading	3	15	24	26	39	59	70
	Total Acid No.	0.08	1.31	1.42	2.36	3.74	3.90	4.21
175°C	Viscosity @100°C,cs	3.33	3.37	3.40	3.41	3.44	3.46	3.49
	Viscosity Change, %		1.2	2.1	2.4	3.3	3.9	4.8
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.0	0.0	4.3	0.1
O-82-2	COBRA Reading	3	12	13	20	33	47	87
(a)	Total Acid No.	0.08	0.99	1.74	2.39	2.24	4.56	4.79
190°C	Viscosity @100°C,cs	3.33	3.36	3.44	3.38	3.42	3.40	3.51
	Viscosity Change, %		0.9	3.3	1.5	2.7	2.1	5.4
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	240					
	Weight Loss, %		0.3					
O-82-2ex	COBRA Reading	3	156					
	Total Acid No.	0.08	6.22					
190°C	Viscosity @100°C,cs	3.33	3.58					
	Viscosity Change, %		7.5					
	Toluene Insol, % wt	ND	0.01					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.5	0.8	0.5	1.9
O-82-2	COBRA Reading	3	14	32	38	55	99	200
	Total Acid No.	0.08	1.66	3.53	5.30	6.14	7.92	9.13
205°C	Viscosity @100°C,cs	3.33	3.34	3.42	3.47	3.56	3.77	4.30
	Viscosity Change, %		0.3	2.7	4.2	6.9	13.2	29.1
	Toluene Insol, % wt	ND	ND	ND	ND	0.00	0.00	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

(a) - See O-82-2ex for extended testing results.

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	96	192	288	384	480	576
	Weight Loss, %		0.0	0.0	0.0	0.0	0.4	0.3
O-82-3	COBRA Reading	8	6	11	27	33	44	44
	Total Acid No.	0.12	0.19	0.45	0.90	1.45	1.74	1.70
175°C	Viscosity @100°C, cs	3.45	3.56	3.62	3.62	3.63	3.65	3.67
	Viscosity Change, %		3.2	4.9	4.9	5.2	5.8	6.4
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		3.6	0.4	0.0	0.1	0.0	0.1
O-82-3D	COBRA Reading	8	5	9	12	21	33	35
(a)	Total Acid No.	0.12	0.11	0.14	0.41	0.85	1.24	1.81
190°C	Viscosity @100°C, cs	3.45	3.55	3.60	3.62	3.61	3.64	3.64
	Viscosity Change, %		2.9	4.3	4.9	4.6	5.5	5.5
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	240	384				
	Weight Loss, %		0.4	1.0				
O-82-3ex	COBRA Reading	8	52	85				
	Total Acid No.	0.12	2.66	4.16				
190°C	Viscosity @100°C, cs	3.45	3.65	3.71				
	Viscosity Change, %		5.8	7.5				
	Toluene Insol, % wt	ND	ND	0.01				
	Visual Appearance of Deposits		None	Varnish in Vent Tube				

(a) - See O-82-3Dex for extended testing results.

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.3	0.9	0.5	0.0	0.4	0.3
O-82-3	COBRA Reading	8	12	10	13	17	31	55
	Total Acid No.	0.12	0.63	1.23	1.97	3.04	4.77	5.86
205°C	Viscosity @100°C, cs	3.45	3.57	3.57	3.60	3.63	3.66	3.70
	Viscosity Change, %		3.5	3.5	4.3	5.2	6.1	7.2
	Toluene Insol, % wt	ND	ND	ND	ND	0.00	0.05	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48	144	192			
	Weight Loss, %		0.0	0.1	0.3			
O-82-3	COBRA Reading	8	15	23	31			
	Total Acid No.	.12	1.08	4.23	5.82			
210°C	Viscosity @100°C, cs	3.45	3.64	3.67	ND			
	Viscosity Change, %		5.5	6.4	N/A			
	Toluene Insol, % wt	ND	ND	ND	ND			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	72	120	192	288	336
	Weight Loss, %		0.2	0.0	0.0	0.3	0.0	0.1
O-82-14D	COBRA Reading	3	30	119	187	200	200	200
	Total Acid No.	0.15	0.37	1.45	4.81	3.24	4.91	7.21
175°C	Viscosity @100°C, cs	3.40	3.52	3.54	3.55	3.72	3.77	3.74
	Viscosity Change, %		3.5	4.1	4.4	9.4	10.9	10.0
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		1.3	0.0	0.0	0.0	0.0	0.6
O-82-14D	COBRA Reading	3	65	163	200	200	200	200
	Total Acid No.	0.15	1.98	3.85	3.35	5.72	6.80	9.33
190°C	Viscosity @100°C,cs	3.40	3.45	3.51	3.59	3.61	3.69	3.86
	Viscosity Change, %		1.5	3.2	5.6	6.2	8.5	13.5
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	Oil Ring	Oil Ring	Oil Ring	Slight Oil Ring	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	216
	Weight Loss, %		0.1	0.9	0.0	2.2	1.7	8.9
O-82-14D	COBRA Reading	3	143	200	200	200	200	200
	Total Acid No.	0.15	3.40	6.20	6.81	7.80	12.10	12.71
205°C	Viscosity @100°C,cs	3.40	3.50	3.62	3.65	3.76	4.34	5.75
	Viscosity Change, %		2.9	6.5	7.4	10.6	27.6	144.7
	Toluene Insol, % wt	ND	0.01	0.00	0.00	0.01	0.01	0.01
	Visual Appearance of Deposits		Stain in cup	Stain Globs of oil	Stain Globs of Oil	Stain Globs of Oil	Stain Globs of Oil	Stain Globs of Oil

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil						
	Weight Loss, %							
	COBRA Reading							
	Total Acid No.							
	Viscosity @100°C,cs							
	Viscosity Change, %							
	Toluene Insol, % wt							
	Visual Appearance of Deposits							

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96		
	Weight Loss, %		0.0	0.0	0.0	0.0		
O-71-6	COBRA Reading	2	4	6	11	25		
	Total Acid No.	0.06	1.23	2.73	3.86	6.62		
205°C	Viscosity @100°C,cs	4.95	5.12	5.17	5.22	5.21		
	Viscosity Change, %		3.4	4.4	5.4	5.2		
	Toluene Insol, % wt	ND	ND	ND	ND	0.04		
	Visual Appearance of Deposits		None	Slight Sediment	None	None		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.0	0.0	0.0			
O-77-15	COBRA Reading	2	12	24	40			
(a)	Total Acid No.	0.43	1.63	3.42	5.72			
205°C	Viscosity @100°C,cs	4.95	5.22	5.15	5.32			
	Viscosity Change, %		5.4	4.0	5.6			
	Toluene Insol, % wt	ND	ND	ND	0.05			
	Visual Appearance of Deposits		None	None	Slight Stain			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	120		
	Weight Loss, %		0.0	0.1	0.4	0.2		
O-79-18	COBRA Reading	3	15	22	37	73		
(b)	Total Acid No.	.07	0.38	0.83	2.25	5.01		
205°C	Viscosity @100°C,cs	5.29	5.28	5.34	5.33	5.33		
	Viscosity Change, %		-0.2	0.9	0.7	0.7		
	Toluene Insol, % wt	ND	ND	ND	ND	0.00		
	Visual Appearance of Deposits		None	None	None	None		

(a) - (TEL 6021)
(b) - (TEL 6022)

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS					
		New Oil	24	48	96	192	288
	Weight Loss, %		0.0	0.0	0.8	0.5	1.0
O-85-1	COBRA Reading	8	32	31	53	76	140
	Total Acid No.	0.02	0.08	0.90	2.41	4.47	10.01
205°C	Viscosity @100°C,cs	4.04	4.04	3.87	4.04	4.10	4.08
	Viscosity Change, %		0.0	4.2	0.0	1.5	1.0
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND
	Visual Appearance of Depcsits		None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS					
		New Oil	24	48	96	192	
	Weight Loss, %		0.0	0.3	0.3	0.3	
TEL 6031	COBRA Reading	14	40	50	97	163	
	Total Acid No.	0.06	0.13	0.30	0.97	2.46	
205°C	Viscosity @100°C,cs	7.53	7.48	7.41	7.38	7.62	
	Viscosity Change, %		-0.7	-1.6	-2.0	1.2	
	Toluene Insol, % wt	ND	ND	ND	ND	0.01	
	Visual Appearance of Deposits		Slight Sediment	ND	Sediment	Sediment	

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS					
		New Oil	24	96	192		
	Weight Loss, %		0.0	0.7	0.6		
TEL 6032	COBRA Reading	5	14	23	23		
	Total Acid No.	0.02	0.00	0.62	1.32		
205°C	Viscosity @100°C,cs	7.50	7.48	7.49	7.47		
	Viscosity Change, %		0.3	0.1	0.4		
	Toluene Insol, % wt	ND	ND	0.02	ND		
	Visual Appearance of Deposits		Slight Sediment	Moderate Sediment	Moderate Sediment		

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.1	0.0	0.5			
O-76-5A	COBRA Reading	1	1	1	1			
(a)	Total Acid No.	0.25	2.07	4.43	6.62			
175°C	Viscosity @100°C,cs	3.46	3.48	3.57	3.59			
	Viscosity Change, %		0.6	3.2	3.7			
	Toluene Insol, % wt	ND	ND	ND	ND			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.1	0.1	0.6	0.5
O-76-5A	COBRA Reading	1	1	1	2	1	2	2
(b)	Total Acid No.	0.25	2.99	5.21	8.00	7.54	9.06	9.36
190°C	Viscosity @100°C,cs	3.46	3.55	3.64	3.71	3.64	3.80	3.72
	Viscosity Change, %		2.6	5.2	7.2	5.2	9.8	7.5
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.01
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.2	0.8	0.8	0.9	1.9
O-76-5A	COBRA Reading	1	2	2	4	2	2	2
(c)	Total Acid No.	0.25	2.44	5.68	6.91	8.85	8.68	8.82
205°C	Viscosity @100°C,cs	3.51	3.71	3.80	3.99	4.82	6.28	ND
	Viscosity Change, %		5.7	8.3	13.7	37.3	78.9	ND
	Toluene Insol, % wt	ND	ND	ND	ND	ND	.05	ND
	Visual Appearance of Deposits		None	Tacky Deposits	Tacky Deposits	Tacky Deposits	Varnish	ND

(a) - Basestock Oil
(b) - Basestock Oil
(c) - Basestock Oil

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.0	0.0	0.1			
O-76-8	COBRA Reading	6	16	34	56			
(a)	Total Acid No.	0.09	1.98	4.79	8.70			
175°C	Viscosity @100°C,cs	2.36	2.36	2.38	2.43			
	Viscosity Change, %		0.0	0.8	3.0			
	Toluene Insol, % wt	ND	ND	ND	ND			
	Visual Appearance of Deposits		Slight Brown Sediment	Slight Brown Sediment	Slight Brown Sediment			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.3	0.4	1.7	1.7
O-76-8	COBRA Reading	6	47	112	200	168	200	200
(b)	Total Acid No.	.09	4.19	6.77	10.16	8.78	13.71	15.74
190°C	Viscosity @100°C,cs	2.36	2.38	2.45	2.53	2.51	2.68	2.64
	Viscosity Change, %		0.8	3.8	7.2	6.4	13.6	11.9
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.02
	Visual Appearance of Deposits		Coke Particles	Coke Particles	Coke Particles	Coke Particles	Coke Particles	Coke Particles

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.3	0.0	1.2	2.6	3.4	0.5
O-76-8	COBRA Reading	2	84	168	200	177	157	124
(c)	Total Acid No.	0.09	4.98	5.92	8.66	9.14	12.48	13.86
205°C	Viscosity @100°C,cs	2.36	2.36	2.45	2.56	2.60	2.68	2.86
	Viscosity Change, %		0.0	3.8	8.5	10.2	13.6	21.2
	Toluene Insol, % wt	ND	0.01	0.01	0.01	0.01	0.01	0.03
	Visual Appearance of Deposits		Black particles in oil	Black particles in oil	Black particles in Oil	Coke in Vent Tube and Oil	Coke in Vent Tube and Oil	Coke in Vent Tube and Oil

(a) - Basestock Oil
(b) - Basestock Oil
(c) - Basestock Oil

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS	
		New Oil	72
O-77-1 205°C	Weight Loss, %		0.4
	COBRA Reading	4	1
	Total Acid No.	0.03	2.49
	Viscosity @100°C,cs	2.33	2.35
	Viscosity Change, %		0.8
	Toluene Insol, % wt	ND	ND
	Visual Appearance of Deposits		None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS	
		New Oil	72
O-77-1+I (a) 205°C	Weight Loss, %		0.3
	COBRA Reading	2	3
	Total Acid No.	0.03	2.44
	Viscosity @100°C,cs	2.34	2.37
	Viscosity Change, %		1.3
	Toluene Insol, % wt	ND	ND
	Visual Appearance of Deposits		None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS	
		New Oil	72
O-77-1+IW (b) 205°C	Weight Loss, %		1.8
	COBRA Reading	3	3
	Total Acid No.	0.13	5.33
	Viscosity @100°C,cs	2.33	2.42
	Viscosity Change, %		3.9
	Toluene Insol, % wt	ND	ND
	Visual Appearance of Deposits		None

(a) - O-77-1 plus impurities
(b) - O-77-1 plus impurities and water.

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS	
		New Oil	72
	Weight Loss, %		0.0
O-77-1+IP	COBRA Reading	10	16
(a)	Total Acid No.	0.02	1.20
205°C	Viscosity @100°C,cs	2.34	2.41
	Viscosity Change, %		3.0
	Toluene Insol, % wt	ND	ND
	Visual Appearance of Deposits		none

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS	
		New Oil	72
	Weight Loss, %		0.2
O-77-1+IPW	COBRA Reading	15	20
(b)	Total Acid No.	0.00	1.85
205°C	Viscosity @100°C,cs	2.46	2.39
	Viscosity Change, %		-2.8
	Toluene Insol, % wt	ND	ND
	Visual Appearance of Deposits		None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS	
		New Oil	72
	Weight Loss, %		0.3
O-77-1+P	COBRA Reading	9	27
(c)	Total Acid No.	0.03	1.53
205°C	Viscosity @100°C,cs	2.44	2.42
	Viscosity Change, %		-0.8
	Toluene Insol, % wt	ND	ND
	Visual Appearance of Deposits		None

(a) - O-77-1 plus impurities and 1% PANA
(b) - O-77-1 plus impurities, 1% PANA, and water.
(c) - O-77-1 plus 1% PANA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.4	0.0	0.6	0.0	0.5	1.1
UDRI 09	COBRA Reading	2	2	2	3	2	6	7
(a)	Total Acid No.	0.14	2.16	2.93	4.18	5.92	10.27	11.05
205°C	Viscosity @100°C,cs	3.47	3.55	3.50	3.51	3.56	3.78	3.81
	Viscosity Change, %		2.3	0.9	1.2	2.6	8.9	9.8
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	ND
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		2.4	0.0	0.0	1.1	0.2	1.3
UDRI 10	COBRA Reading	2	2	4	5	5	7	10
(b)	Total Acid No.	0.06	1.56	4.15	6.97	8.28	8.89	9.84
205°C	Viscosity @100°C,cs	3.46	3.49	3.56	3.65	3.78	3.66	3.81
	Viscosity Change, %		0.9	2.9	5.5	9.2	5.8	10.1
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.0	0.6	0.4	0.4	0.4
UDRI 11	COBRA Reading	3	3	5	6	8	10	26
(c)	Total Acid No.	0.15	1.02	2.06	4.48	6.95	9.40	10.20
205°C	Viscosity @100°C,cs	3.46	3.48	3.48	3.51	3.68	3.71	3.71
	Viscosity Change, %		0.6	0.6	1.4	6.3	7.2	7.2
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

(a) - O-76-5 w/0.05% PANA
(b) - O-76-5 w/0.1% PANA
(c) - O-76-5 w/0.15% PANA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
UDRI 12 (a) 205°C	Weight Loss, %		0.0	0.0	0.0	0.4	0.0	0.5
	COBRA Reading	2	3	5	5	5	7	16
	Total Acid No.	0.16	0.62	1.71	2.42	3.62	5.45	8.10
	Viscosity @100°C,cs	3.47	3.49	3.49	3.49	3.47	3.51	3.63
	Viscosity Change, %		3.6	3.6	3.6	3.0	4.2	4.6
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.00
	Visual Appearance of Deposits		None	None	None	None	None	None

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
UDRI 17 (b) 205°C	Weight Loss, %		0.0	0.0	0.0	0.1	0.4	1.1
	COBRA Reading	3	2	3	6	7	11	ND
	Total Acid No.	0.09	2.41	2.87	3.25	3.48	5.00	ND
	Viscosity @100°C,cs	3.46	3.50	3.50	3.53	3.53	3.55	ND
	Viscosity Change, %		1.2	1.2	2.0	2.0	2.6	ND
	Toluene Insol, % wt	ND	ND	ND	ND	ND	0.06	ND
	Visual Appearance of Deposits		None	None	None	None	None	ND

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96		
UDRI 18 (c) 205°C	Weight Loss, %		0.0	0.0	0.0	0.0		
	COBRA Reading	3	3	3	7	9		
	Total Acid No.	.10	1.41	3.21	4.28	5.38		
	Viscosity @100°C,cs	3.47	3.50	3.53	3.56	3.56		
	Viscosity Change, %		0.9	1.7	2.6	2.6		
	Toluene Insol, % wt	ND	ND	ND	ND	0.01		
	Visual Appearance of Deposits		None	None	None	None		

(a) - O-76-5 w/0.2% PANA
(b) - O-76-5A 0.5% PANA
(c) - O-76-5A 1.0% PANA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	144		
	Weight Loss, %		0.3	0.0	0.0	0.5		
UDRI 19	COBRA Reading	4	3	6	15	29		
(a)	Total Acid No.	.10	1.64	3.64	4.62	6.86		
205°C	Viscosity @100°C,cs	3.50	3.57	3.58	3.66	3.69		
	Viscosity Change, %		2.0	2.3	4.6	5.4		
	Toluene Insol, % wt	ND	ND	ND	ND	0.02		
	Visual Appearance of Deposits		None	None	Slight Coke	Coke Particles		

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.0	0.0	0.4			
UDRI 20	COBRA Reading	4	4	12	12			
(b)	Total Acid No.	.10	1.67	3.78	5.66			
205°C	Viscosity @100°C,cs	3.51	3.58	3.67	3.61			
	Viscosity Change, %		2.0	4.6	2.8			
	Toluene Insol, % wt	ND	ND	ND	0.02			
	Visual Appearance of Deposits		None	None	Slight Sediment			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	168		
	Weight Loss, %		0.0	0.4	0.2	0.0		
UDRI 13	COBRA Reading	1	5	5	6	11		
(c)	Total Acid No.	.17	2.09	3.23	4.07	7.09		
205°C	Viscosity @100°C,cs	3.48	3.53	3.54	3.51	3.51		
	Viscosity Change, %		1.4	1.7	0.9	0.9		
	Toluene Insol, % wt	ND	ND	ND	ND	ND		
	Visual Appearance of Deposits		None	None	None	None		

(a) - O-76-5A 1.5% PANA
(b) - O-76-5A 2.0% PANA
(c) - O-76-5A w/0.5% DODPA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		1.0	0.0	0.0			
UDRI 14	COBRA Reading	1	5	8	8			
(a)	Total Acid No.	0.18	1.60	3.29	4.67			
205°C	Viscosity @100°C,cs	3.51	3.52	3.53	3.55			
	Viscosity Change, %		0.3	0.6	1.1			
	Toluene Insol, % wt	ND	ND	ND	0.02			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.0	0.1	0.2			
UDRI 15	COBRA Reading	0	8	11	11			
(b)	Total Acid No.	0.23	1.84	3.39	4.76			
205°C	Viscosity @100°C,cs	3.55	3.59	3.61	3.60			
	Viscosity Change, %		1.1	1.7	1.4			
	Toluene Insol, % wt	ND	ND	ND	0.02			
	Visual Appearance of Deposits		None	None	None			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.0	0.0	0.1			
UDRI 16	COBRA Reading	0	8	9	14			
(c)	Total Acid No.	0.15	1.78	3.58	5.57			
205°C	Viscosity @100°C,cs	3.61	3.59	3.62	3.65			
	Viscosity Change, %		-0.6	0.3	1.1			
	Toluene Insol, % wt	ND	ND	ND	0.03			
	Visual Appearance of Deposits		None	None	None			

(a) - O-76-5A w/1% DODPA
(b) - O-76-5A w/1.5% DODPA
(c) - O-76-5A w/2% DODPA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48					
	Weight Loss, %		0.0					
UDRI 24	COBRA Reading	3	7					
(a)	Total Acid No.	0.13	4.02					
205°C	Viscosity @100°C,cs	3.52	3.62					
	Viscosity Change, %		2.8					
	Toluene Insol, % wt	0.01	0.66					
	Visual Appearance of Deposits		Heavy Coke, Stain					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48					
	Weight Loss, %		0.0					
UDRI 25	COBRA Reading	4	28					
(b)	Total Acid No.	0.10	4.90					
205°C	Viscosity @100°C,cs	3.59	3.69					
	Viscosity Change, %		2.8					
	Toluene Insol, % wt	0.03	0.06					
	Visual Appearance of Deposits		None					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
	Weight Loss, %		0.0	0.1	0.6	1.6	1.9	1.4
UDRI 01	COBRA Reading	6	79	95	85	88	87	76
(c)	Total Acid No.	0.05	2.95	4.79	5.61	6.02	7.05	10.06
205°C	Viscosity @100°C,cs	2.37	2.41	2.44	2.47	2.51	2.52	2.54
	Viscosity Change, %		1.7	2.9	4.2	5.9	6.3	7.2
	Toluene Insol, % wt	ND	0.05	0.08	0.17	0.29	0.41	0.32
	Visual Appearance of Deposits		Coke in cup and oil	Coke in cup and oil	Coke in Cup and Oil	Coke in Cup and Oil	Coke in Cup and Oil	Coke in Cup and Oil

(a) - O-76-5 w/2% PTZ
(b) - O-76-5 w/2% DOPTZ
(c) - O-76-8 W/0.5% PANA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
UDRI 02 (a) 205°C	Weight Loss, %		0.0	0.0	0.4	0.3	1.5	1.0
	COBRA Reading	6	112	108	143	140	107	76
	Total Acid No.	0.06	3.67	5.30	6.16	7.66	8.58	10.82
	Viscosity @100°C,cs	2.37	2.43	2.46	2.50	2.52	2.52	2.55
	Viscosity Change, %		2.5	3.8	5.5	6.3	6.3	7.6
	Toluene Insol, % wt	ND	0.09	0.17	0.34	0.48	0.56	0.21
	Visual Appearance of Deposits		Coke in cup and oil	Coke in cup and oil	Coke in Cup and Oil	Coke in Cup and Oil	Coke in Cup and Oil	Coke in Cup and Oil

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
UDRI 03 (b) 205°C	Weight Loss, %		0.0	0.9	0.3	0.8	0.9	2.0
	COBRA Reading	10	138	126	142	145	109	111
	Total Acid No.	0.05	3.64	5.52	5.51	7.78	7.90	11.28
	Viscosity @100°C,cs	2.38	2.45	2.48	2.51	2.55	2.53	2.56
	Viscosity Change, %		2.9	4.2	5.5	7.1	6.3	7.6
	Toluene Insol, % wt	ND	0.14	0.11	0.50	0.67	0.86	0.30
	Visual Appearance of Deposits		Coke in cup and oil	Coke in cup and oil	Coke in Cup and Oil	Coke in Cup and Oil	Coke in Cup and Oil	Coke in Cup and Oil

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72	96	144	192
UDRI 04 (c) 205°C	Weight Loss, %		0.0	0.0	0.8	1.2	1.0	3.2
	COBRA Reading	14	174	159	197	169	146	137
	Total Acid No.	0.07	3.86	5.68	6.38	8.27	7.71	9.11
	Viscosity @100°C,cs	2.38	2.45	2.50	2.52	2.54	2.58	2.61
	Viscosity Change, %		2.9	5.0	5.9	6.7	8.4	9.7
	Toluene Insol, % wt	ND	0.10	0.33	0.58	0.58	0.87	1.25
	Visual Appearance of Deposits		Dk. Brn. to Black Coke	Dk. Brn. to Black Coke	Dk. Brn. to Black Coke	Dk. Brn. to Black Coke	Dk. Brn. to Black Coke	Dk. Brn. to Black Coke

(a) - O-76-8 W/1% PANA
(b) - O-76-8 W/1.5% PANA
(c) - O-76-8 w/2.0% PANA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.3	0.3	0.4			
UDRI 05	COBRA Reading	2	200	200	200			
(a)	Total Acid No.	.01	3.95	5.24	7.08			
205°C	Viscosity @100°C,cs	2.37	2.42	2.45	2.51			
	Viscosity Change, %		2.1	3.4	5.9			
	Toluene Insol, % wt	ND	0.02	0.01	0.01			
	Visual Appearance of Deposits		Coke Particles	Coke Particles	Coke Particles			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.1	0.6	0.3			
UDRI 06	COBRA Reading	3	200	200	200			
(b)	Total Acid No.	.02	3.84	5.83	7.08			
205°C	Viscosity @100°C,cs	2.37	2.44	2.51	2.56			
	Viscosity Change, %		2.9	5.9	8.0			
	Toluene Insol, % wt	ND	0.00	0.16	0.00			
	Visual Appearance of Deposits		Coke Particles	Coke Particles	Coke Particles			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.0	0.8	0.4			
UDRI 07	COBRA Reading	5	200	200	200			
(c)	Total Acid No.	.04	4.03	5.55	7.38			
205°C	Viscosity @100°C,cs	2.41	2.45	2.53	2.56			
	Viscosity Change, %		1.6	5.0	6.2			
	Toluene Insol, % wt	ND	0.00	0.02	0.01			
	Visual Appearance of Deposits		None	Coke Particles	Coke Particles			

(a) - O-76-8 w/0.5% DODPA
(b) - O-76-8 w/1.0% DODPA
(c) - O-76-8 w/1.5% DODPA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
	Weight Loss, %		0.1	0.1	0.4			
UDRI 08	COBRA Reading	3	200	200	200			
(a)	Total Acid No.	.05	4.31	5.66	7.32			
205°C	Viscosity @100°C,cs	2.43	2.50	2.54	2.59			
	Viscosity Change, %		2.9	4.5	6.6			
	Toluene Insol, % wt	ND	0.00	0.00	0.00			
	Visual Appearance of Deposits		None	Coke Particles	Coke Particles			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48					
	Weight Loss, %		0.3					
UDRI 26	COBRA Reading	9	120					
(b)	Total Acid No.	0.04	5.75					
205°C	Viscosity @100°C,cs	2.39	2.47					
	Viscosity Change, %		3.3					
	Toluene Insol, % wt	0.01	0.10					
	Visual Appearance of Deposits		Purple Oil, Much Coke					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48					
	Weight Loss, %		0.4					
UDRI 27	COBRA Reading	6	200					
(c)	Total Acid No.	0.03	6.15					
205°C	Viscosity @100°C,cs	2.45	2.56					
	Viscosity Change, %		4.5					
	Toluene Insol, % wt	0.02	0.03					
	Visual Appearance of Deposits		Slight Sediment					

(a) - O-76-8 w/2.0% DODPA
(b) - O-76-8 w/2% PTZ
(c) - O-76-8 w/2% DOPTZ

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48					
	Weight Loss, %		0.0					
UDRI 28	COBRA Reading	11	100					
(a)	Total Acid No.	0.04	1.82					
205°C	Viscosity @100°C, cs	2.37	2.60					
	Viscosity Change, %		9.7					
	Toluene Insol, % wt	0.02	0.02					
	Visual Appearance of Deposits		Coke, Oil Brownish-Red					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	48					
	Weight Loss, %		0.6					
UDRI 29	COBRA Reading	5	46					
(b)	Total Acid No.	0.03	2.84					
205°C	Viscosity @100°C, cs	2.42	2.44					
	Viscosity Change, %		0.8					
	Toluene Insol, % wt	0.01	0.03					
	Visual Appearance of Deposits		Slight Sediment					

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	25	48	72	96		
	Weight Loss, %		0.9	0.0	0.0	0.0		
UDRI 22	COBRA Reading	3	4	10	11	15		
(c)	Total Acid No.	.08	2.42	3.96	5.09	6.28		
205°C	Viscosity @100°C, cs	3.51	3.57	3.64	3.64	3.57		
	Viscosity Change, %		1.7	3.7	3.7	1.7		
	Toluene Insol, % wt	ND	ND	ND	ND	0.03		
	Visual Appearance of Deposits		None	None	None	None		

(a) - O-77-1 w/2% PTZ
(b) - O-77-1 w/2% DOPTZ
(c) - O-76-5A w/ 1% PANA + 1% DODPA

SQUIRES CONFINED HEAT TEST DATA

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	24	48	72			
UDRI 21 (a) 205°C	Weight Loss, %		0.1	0.1	0.6			
	COBRA Reading	9	200	200	200			
	Total Acid No.	.02	4.40	5.54	6.56			
	Viscosity @100°C,cs	2.41	2.46	2.51	2.56			
	Viscosity Change, %		2.1	4.1	6.2			
	Toluene Insol, % wt	ND	ND	ND	0.13			
	Visual Appearance of Deposits		Black Coke	Black Coke	Black Coke			

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil	25	48	72	96	168	240
UDRI 23 (b) 205°C	Weight Loss, %		0.1	0.4	0.0	0.5	0.0	2.2
	COBRA Reading	6	13	21	83	51	80	146
	Total Acid No.	.03	0.30	0.78	2.24	3.11	4.19	8.63
	Viscosity @100°C,cs	2.35	2.40	2.40	2.50	2.45	2.46	2.51
	Viscosity Change, %		2.1	2.1	6.4	4.3	4.7	6.8
	Toluene Insol, % wt	ND	ND	ND	ND	ND	ND	0.02
	Visual Appearance of Deposits		None	None	None	None	Slight Stain	Slight Stain

LUBRICANT AND TEST TEMP.	LUBRICANT PROPERTY	TEST HOURS						
		New Oil						
	Weight Loss, %							
	COBRA Reading							
	Total Acid No.							
	Viscosity @100°C,cs							
	Viscosity Change, %							
	Toluene Insol, % wt							
	Visual Appearance of Deposits							

(a) - O-76-8 w/1% PANA + 1% DODPA
(b) - O-77-1 w/1% PANA + 1% DODPA

TABLE A-3

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
1	0-79-16J	1	315	180	Shim Stock	0.9698	11.9	Hard black smooth wavy deposit
2	0-79-16J	2	315	180	Shim Stock	0.9820	14.3	Hard black smooth wavy deposit. Small area of flaky residue
3	0-79-16J	3	315	180	Shim Stock	0.8399	13.0	Hard black smooth wavy deposit
4	0-79-16J	4	315	180	Shim Stock	0.9320	13.2	Hard black smooth wavy deposit
5	0-79-16J	1	315	180	Shim Stock	0.9514	13.6	Hard black smooth wavy deposit
6	0-79-16J	2	315	180	Shim Stock	0.9880	14.9	Hard black smooth wavy deposit
7	0-79-16J	3	315	180	Shim Stock	0.9784	11.7	Hard black smooth wavy deposit
8	0-79-16J	4	315	180	Shim Stock	0.9229	-	Seal leaked. Much less residue
9	0-79-16J	1	315	180	Shim Stock	0.9193	12.2	Hard black smooth wavy deposit
10	0-79-16J	2	315	180	Shim Stock	0.9326	13.3	Hard black smooth wavy deposit
11	0-79-16J	3	315	180	Shim Stock	0.9761	15.4	Hard black smooth wavy deposit
12	0-79-16J	4	315	180	Shim Stock	0.9517	15.0	Hard black smooth wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
13	0-79-16J	1	315	180	Shim Stock	0.9096	13.5	Hard glossy black smooth wavy deposit
14	0-79-16J	2	315	180	Shim Stock	0.9696	15.9	Hard glossy black smooth wavy deposit
15	0-79-16J	3	315	180	Shim Stock	0.9657	12.8	Hard glossy black smooth wavy deposit
16	0-79-16J	4	315	180	Shim Stock	0.9290	15.0	Hard glossy black smooth wavy deposit
17	0-79-16J	1	315	180	Shim Stock	0.4898	10.0	Hard glossy black smooth deposit uniform coverage
18	0-79-16J	2	315	180	Shim Stock	0.4415	9.7	Hard glossy black smooth deposit, not uniform coverage
19	0-79-16J	3	315	180	Shim Stock	0.4724	9.3	Hard glossy black smooth deposit
20	0-79-16J	4	315	180	Shim Stock	0.4969	15.3	Hard glossy black smooth deposit. Specimen weight suspect
21	0-79-16J	1	315	180	Shim Stock	0.7343	13.4	Hard glossy black smooth deposit
22	0-79-16J	2	315	180	Shim Stock	0.6916	11.7	Hard glossy black smooth deposit
23	0-79-16J	3	315	180	Shim Stock	0.7306	11.2	Hard glossy black smooth deposit
24	0-79-16J	4	315	180	Shim Stock	0.7046	12.5	Hard glossy black smooth deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
25	0-79-16J	1	315	180	Shim Stock	0.7385	15.7	Hard glossy black smooth wavy deposit. Seal not cleaned
26	0-79-16J	2	315	180	Shim Stock	0.7462	16.4	Hard glossy black smooth wavy deposit. Seal not cleaned
27	0-79-16J	3	315	180	Shim Stock	0.7453	16.9	Hard glossy black smooth wavy deposit. Seal not cleaned
28	0-79-16J	4	315	180	Shim Stock	0.7241	19.1	Hard glossy black smooth wavy deposit. Seal not cleaned
29	0-79-16J	1	315	180	Shim Stock	0.7424	13.3	Hard glossy black smooth wavy deposit
30	0-79-16J	2	315	180	Shim Stock	0.7178	15.3	Hard glossy black smooth wavy deposit
31	0-79-16J	3	315	180	Shim Stock	0.7501	14.5	Hard glossy black smooth wavy deposit
32	0-79-16J	4	315	180	Shim Stock	0.7425	14.5	Hard glossy black smooth wavy deposit
33	0-79-16J	1	315	180	Shim Stock	0.5099	10.6	Hard glossy black smooth wavy deposit
34	0-79-16J	2	315	180	Shim Stock	0.5025	12.5	Hard glossy black smooth wavy deposit
35	0-79-16J	3	315	180	Shim Stock	0.4579	12.2	Hard glossy black smooth wavy deposit
36	0-79-16J	4	315	180	Shim Stock	0.4568	14.2	Hard glossy black smooth wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
37	0-79-16J	1	315	180	Shim Stock	0.4662	13.7	Hard glossy black smooth wavy depsoit. Gum on seal
38	0-79-16J	2	315	180	Shim Stock	0.4632	11.2	Hard glossy black smooth wavy deposit. Gum on seal
39	0-79-16J	3	315	180	Shim Stock	0.4623	10.8	Hard glossy black smooth wavy deposit. Gum on seal
40	0-79-16J	4	315	180	Shim Stock	0.4333	13.2	Hard glossy black smooth wavy deposit. Gum on seal
41	0-79-16J	1	315	180	Shim Stock	1.4817	16.4	Hard glossy black smooth wavy deposit. Gum on seal
42	0-79-16J	2	315	180	Shim Stock	1.4772	18.6	Hard glossy black smooth wavy deposit. Gum on seal
43	0-79-16J	3	315	180	Shim Stock	1.4589	12.9	Hard glossy black smooth wavy deposit. Gum on seal
44	0-79-16J	4	315	180	Shim Stock	1.4135	17.3	Hard glossy black smooth wavy deposit. Gum on seal
45	0-79-16J	1	315	180	Shim Stock	1.4534	11.6	Hard glossy black smooth wavy deposit. Gum on seal
46	0-79-16J	2	315	180	Shim Stock	1.4492	17.5	Hard glossy black smooth wavy deposit. Gum on seal
47	0-79-16J	3	315	180	Shim Stock	1.4466	17.6	Hard glossy black smooth wavy deposit. Gum on seal
48	0-79-16J	4	315	180	Shim Stock	1.3722	17.7	Hard glossy black smooth wavy deposit. Seal had oil in vent holes

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
49	D-79-16J	1	315	180	Shim Stock	0.9818	9.7	Hard black glossy deposit. Seal "Carb-N-clean"
50	D-79-16J	2	315	180	Shim Stock	0.9851	16.1	Hard black glossy deposit. Seal "Carb-N-clean"
51	D-79-16J	3	315	180	Shim Stock	0.9685	13.2	Hard black glossy deposit. Seal "Carb-N-clean"
52	D-79-16J	4	315	180	Shim Stock	0.9431	15.2	Hard black glossy deposit. Seal "Carb-N-clean"
53	D-82-14	1	315	180	Shim Stock	0.9779	17.1	Hard black glossy wavy deposit
54	D-82-14	2	315	180	Shim Stock	0.9232	18.5	Hard black glossy wavy deposit
55	D-82-14	3	315	180	Shim Stock	0.9708	16.7	Hard black glossy wavy deposit
56	D-82-14	4	315	180	Shim Stock	0.9291	16.5	Hard black glossy wavy deposit
57	D-82-3	1	315	180	Shim Stock	0.9512	12.5	Hard glossy black wavy deposit
58	D-82-3	2	315	180	Shim Stock	0.9551	16.7	Hard glossy black wavy deposit
59	D-82-3	3	315	180	Shim Stock	0.9596	15.4	Hard glossy black wavy deposit
60	D-82-3	4	315	180	Shim Stock	0.9403	16.9	Hard glossy black wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
61	O-79-16J	1	315	180	Shim Stock	1.2227	9.7	Hard glossy black wavy deposit
62	O-79-16J	2	315	180	Shim Stock	1.2133	17.7	Hard glossy black wavy deposit
63	O-79-16J	3	315	180	Shim Stock	1.2096	14.9	Hard glossy black wavy deposit
64	O-79-16J	4	315	180	Shim Stock	1.1654	16.9	Hard glossy black wavy deposit
65	O-79-16J	1	315	180	Shim Stock	1.2699	16.1	Hard glossy black wavy deposit
66	O-79-16J	2	315	180	Shim Stock	1.2761	17.2	Hard glossy black wavy deposit
67	O-79-16J	3	315	180	Shim Stock	1.2681	14.5	Hard glossy black wavy deposit
68	O-79-16J	4	315	180	Shim Stock	1.2162	15.7	Hard glossy black wavy deposit
69	O-79-16J	1	315	120	Shim Stock	0.8415	9.5	Hard glossy black deposit. Seal cleaned with VM&P
70	O-79-16J	2	315	120	Shim Stock	0.7477	14.4	Hard glossy black deposit. Seal cleaned with VM&P
71	O-79-16J	3	315	120	Shim Stock	0.7688	13.0	Hard glossy black deposit. Seal cleaned with VM&P
72	O-79-16J	4	315	120	Shim Stock	0.7143	12.5	Hard glossy black deposit. Seal cleaned with VM&P

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
73	O-79-16J	1	315	120	Shim Stock	0.7761	12.5	Hard black smooth wavy deposit. Seal sticky
74	O-79-16J	2	315	120	Shim Stock	0.7957	13.4	Hard black smooth wavy deposit. Seal sticky
75	O-79-16J	3	315	120	Shim Stock	0.7890	14.1	Hard black smooth wavy deposit. Seal sticky
76	O-79-16J	4	315	120	Shim Stock	0.7168	-	Problem with seal
77	O-79-16J	1	315	60	Shim Stock	0.9814	12.5	Hard black smooth wavy deposit. Seal very sticky
78	O-79-16J	2	315	60	Shim Stock	0.9943	15.0	Hard black smooth wavy deposit. Seal very sticky
79	O-79-16J	3	315	60	Shim Stock	0.9878	12.6	Hard black smooth wavy deposit. Seal very sticky
80	O-79-16J	4	315	60	Shim Stock	0.9622	17.7	Hard black smooth wavy deposit. Seal very sticky
81	O-82-3	1	315	180	Shim Stock	0.9622	12.7	Hard Black smooth wavy deposit.
82	O-82-3	2	315	180	Shim Stock	0.9586	17.0	Hard black smooth wavy deposit.
83	O-82-3	3	315	180	Shim Stock	0.9570	13.3	Hard black smooth wavy deposit.
84	O-82-3	4	315	180	Shim Stock	0.9079	13.4	Hard black smooth wavy deposit.

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
85	O-82-2	1	315	180	Shim Stock	0.9613	11.4	Hard glossy black smooth wavy deposit.
86	O-82-2	2	315	180	Shim Stock	0.9715	14.0	Hard glossy black smooth wavy deposit.
87	O-82-2	3	315	180	Shim Stock	0.9750	11.0	Hard glossy black smooth wavy deposit.
88	O-82-2	4	315	180	Shim Stock	0.9221	12.3	Hard glossy black smooth wavy deposit.
89	O-79-16J	1	315	180	Aluminum	0.9828	9.5	Incomplete coverage. Hard black glossy deposit.
90	O-79-16J	2	315	180	Aluminum	0.9871	12.1	Incomplete coverage. Hard black glossy deposit.
91	O-79-16J	3	315	180	Aluminum	0.9900	7.0	Incomplete coverage. Hard black glossy deposit.
92	O-79-16J	4	315	180	Aluminum	0.9540	14.6	Incomplete coverage. Hard black glossy deposit.
93	O-79-16J	1	315	180	SS	0.9795	11.3	Hard semiglossy black wavy deposit.
94	O-79-16J	2	315	180	SS	0.9821	13.4	Hard semiglossy black wavy deposit.
95	O-79-16J	3	315	180	SS	0.9915	8.2	Hard semiglossy black wavy deposit.
96	O-79-16J	4	315	180	SS	0.9485	11.3	Hard semiglossy black wavy deposit.

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
97	O-79-16J	1	315	180	SS	0.9910	10.8	Hard semiglossy wavy deposit.
98	O-79-16J	2	315	180	SS	1.0067	14.0	Hard semiglossy wavy deposit.
99	O-79-16J	3	315	180	SS	1.0135	10.4	Hard semiglossy wavy deposit.
100	O-79-16J	4	315	180	SS	0.9772	10.2	Hard semiglossy wavy deposit.
101	O-79-16J	1	315	180	Aluminum	0.9993	5.7	Hard black non-uniform coverage deposit.
102	O-79-16J	2	315	180	Aluminum	0.9995	11.7	Hard black non-uniform coverage deposit.
103	O-79-16J	3	315	180	Aluminum	0.9950	8.3	Hard black non-uniform coverage deposit.
104	O-79-16J	4	315	180	Aluminum	0.9632	8.2	Hard black non-uniform coverage deposit.
105	O-79-17E	1	315	180	Shim Stock	1.0158	7.5	Hard black wavy glossy deposit.
106	O-79-17E	2	315	180	Shim Stock	1.0071	13.7	Hard black wavy glossy deposit.
107	O-79-17E	3	315	180	Shim Stock	1.0173	9.0	Hard black wavy glossy deposit.
108	O-79-17E	4	315	180	Shim Stock	0.9707	13.7	Hard black wavy glossy deposit.

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
109	O-79-20	1	315	180	Shim Stock	0.9853	7.8	Hard black wavy glossy deposit.
110	O-79-20	2	315	180	Shim Stock	0.9910	16.2	Hard black wavy glossy deposit.
111	O-79-20	3	315	180	Shim Stock	0.9371	12.3	Hard black wavy glossy deposit.
112	O-79-20	4	315	180	Shim Stock	0.9826	15.7	Hard black wavy glossy deposit.
113	O-82-2	1	315	180	Shim Stock	0.9875	14.0	Hard black wavy glossy deposit.
114	O-82-2	2	315	180	Shim Stock	0.9794	16.3	Hard black wavy glossy deposit.
115	O-82-2	3	315	180	Shim Stock	0.9793	14.3	Hard black wavy glossy deposit.
116	O-82-2	4	315	180	Shim Stock	0.9556	16.2	Hard black wavy glossy deposit.
117	O-82-2	1	315	180	Shim Stock	1.0092	13.8	Hard black wavy glossy deposit.
118	O-82-2	2	315	180	Shim Stock	1.0004	16.9	Hard black wavy glossy deposit.
119	O-82-2	3	315	180	Shim Stock	0.9872	14.0	Hard black wavy glossy deposit.
120	O-82-2	4	315	180	Shim Stock	1.0136	16.3	Hard black wavy glossy deposit.

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
121	0-79-20C	1	315	180	Shim Stock	1.0144	13.8	Hard black wavy glossy deposit.
122	0-79-20C	2	315	180	Shim Stock	0.9775	16.3	Hard black wavy glossy deposit.
123	0-79-20C	3	315	180	Shim Stock	0.9962	13.7	Hard black wavy glossy deposit.
124	0-79-20C	4	315	180	Shim Stock	0.9718	14.7	Hard black wavy glossy deposit.
125	0-79-17E	1	315	180	Shim Stock	1.0266	11.9	Hard black wavy glossy deposit.
126	0-79-17E	2	315	180	Shim Stock	0.9999	15.6	Hard black wavy glossy deposit.
127	0-79-17E	3	315	180	Shim Stock	0.9917	12.0	Hard black wavy glossy deposit.
128	0-79-17E	4	315	180	Shim Stock	0.9750	13.6	Hard black wavy glossy deposit.
129	0-79-16J	1	300	180	Shim Stock	0.9978	10.8	Hard black wavy glossy Deposit
130	0-79-16J	2	300	180	Shim Stock	0.9993	12.8	Hard black wavy glossy Deposit
131	0-79-16J	3	300	180	Shim Stock	0.9975	10.1	Hard black wavy glossy Deposit
132	0-79-16J	4	300	180	Shim Stock	0.9687	11.7	Hard black wavy glossy Deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
133	0-79-16J	1	300	180	Shim Stock	0.9923	11.8	Hard black wavy glossy Deposit
134	0-79-16J	2	300	180	Shim Stock	0.9805	13.3	Hard black wavy glossy Deposit
135	0-79-16J	3	300	180	Shim Stock	0.9881	12.3	Hard black wavy glossy Deposit
136	0-79-16J	4	300	180	Shim Stock	1.0039	13.8	Hard black wavy glossy Deposit
137	0-79-16J	1	300	180	Shim Stock	0.9786	13.1	Hard black wavy glossy Deposit
138	0-79-16J	2	300	180	Shim Stock	0.9852	15.9	Hard black wavy glossy Deposit
139	0-79-16J	3	300	180	Shim Stock	0.9885	13.6	Hard black wavy glossy Deposit
140	0-79-16J	4	300	180	Shim Stock	0.9855	13.5	Hard black wavy glossy Deposit
141	0-79-17E	1	300	180	Shim Stock	0.9986	13.0	Hard black wavy glossy Deposit
142	0-79-17E	2	300	180	Shim Stock	1.0330	14.0	Hard black wavy glossy Deposit
143	0-79-17E	3	300	180	Shim Stock	1.0034	10.7	Hard black wavy glossy Deposit
144	0-79-17E	4	300	180	Shim Stock	1.0065	13.4	Hard black wavy glossy Deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
145	0-79-20C	1	300	180	Shim Stock	0.9733	13.8	Hard black wavy glossy Deposit
146	0-79-20C	2	300	180	Shim Stock	0.9948	17.0	Hard black wavy glossy Deposit
147	0-79-20C	3	300	180	Shim Stock	0.9629	14.8	Hard black wavy glossy Deposit
148	0-79-20C	4	300	180	Shim Stock	1.0064	15.6	Hard black wavy glossy Deposit
149	0-82-2	1	300	180	Shim Stock	0.9974	14.4	Hard black wavy glossy Deposit
150	0-82-2	2	300	180	Shim Stock	0.9965	16.9	Hard black wavy glossy Deposit
151	0-82-3	3	300	180	Shim Stock	0.9644	13.3	Hard black wavy glossy Deposit
152	0-82-3	4	300	180	Shim Stock	0.9905	16.1	Hard black glossy wavy Deposit
153	0-82-14D	1	300	180	Shim Stock	0.9703	15.1	Hard black wavy glossy Deposit
154	0-82-14D	2	300	180	Shim Stock	0.9875	15.8	Hard black wavy glossy Deposit
155	0-82-14D	3	300	180	Shim Stock	0.9252	17.3	Hard black wavy glossy Deposit
156	0-82-14D	4	300	180	Shim Stock	0.9799	16.6	Hard black wavy glossy Deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
157	O-79-16J	1	260	180	Shim Stock	1.0350	16.6	Wavy glossy brown varnish
158	O-79-16J	2	260	180	Shim Stock	1.0109	16.2	Wavy glossy brown varnish
159	O-79-17E	3	260	180	Shim Stock	1.0183	14.4	Wavy glossy brown varnish
160	O-79-17E	4	260	180	Shim Stock	1.0483	12.4	Wavy glossy brown varnish
161	O-79-20	1	260	180	Shim Stock	1.0276	22.1	Wavy glossy brown varnish to black deposit
162	O-79-20	2	260	180	Shim Stock	1.0353	23.2	Wavy glossy brown varnish to black deposit
163	O-82-2	3	260	180	Shim Stock	1.0062	23.2	Wavy glossy brown varnish
164	O-82-2	4	260	180	Shim Stock	1.0352	24.8	Wavy glossy brown varnish
165	O-82-3	1	260	180	Shim Stock	0.9573	14.9	Smooth glossy dark brown deposit
166	O-82-3	2	260	180	Shim Stock	0.9685	20.5	Smooth glossy dark brown deposit
167	O-82-14	3	260	180	Shim Stock	0.9791	14.8	Flaky black coke to light dark brown varnish
168	O-82-14	4	260	180	Shim Stock	0.9158	15.1	Flaky black coke to light dark brown varnish

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
169	0-79-16 24 hrs. 0x 205 C	1	315	180	Shim Stock	0.9647	20.5	Hard black smooth wavy deposit
170	0-79-16 24 hrs. 0x 205 C	2	315	180	Shim Stock	0.9669	23.7	Hard black smooth wavy deposit
171	0-82-14 24 hrs. 0x 205 C	3	315	180	Shim Stock	0.9334	28.2	Hard black flaky deposit
172	0-82-14 24 hrs. 0x 205 C	4	315	180	Shim Stock	0.9014	29.1	Hard black flaky deposit
173	0-79-16 48 hrs. 0x 205 C	1	315	180	Shim Stock	1.0606	24.3	Hard black glossy deposit
174	0-79-16 48 hrs. 0x 205 C	2	315	180	Shim Stock	1.0572	30.9	Hard black glossy deposit
175	0-82-14 48 hrs. 0x 205 C	3	315	180	Shim Stock	0.9531	34.8	Hard black flaky deposit
176	0-82-14 48 hrs. 0x 205 C	4	315	180	Shim Stock	0.9642	35.7	Hard black flaky deposit
177	0-79-16 48 hrs. 0x 205 C	1	315	180	Shim Stock	0.9504	25.6	Hard black glossy deposit
178	0-79-16 48 hrs. 0x 205 C	2	315	180	Shim Stock	1.0512	25.0	Hard black glossy deposit
179	0-79-17 24 hrs. 0x 205 C	3	315	180	Shim Stock	1.0734	21.2	Hard black glossy wavy deposit
180	0-79-17 24 hrs. 0x 205 C	4	315	180	Shim Stock	0.9979	21.1	Hard black glossy wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
181	O-79-20 24 hrs. Ox 201 C	1	315	240	Shim Stock	0.9764	24.6	Hard black smooth glossy wavy deposit
182	O-79-20 24 hrs. Ox 201 C	2	315	240	Shim Stock	0.9495	28.7	Hard black smoothh glossy wavy deposit
183	O-82-2 24 hrs. Ox 205 C	3	315	240	Shim Stock	0.9644	19.6	Hard black smooth glossy wavy deposit
184	O-82-2 24 hrs. Ox 205 C	4	315	240	Shim Stock	0.9286	20.6	Hard black smooth glossy wavy deposit
185	O-79-20 24 hrs. Ox 201 C	1	315	180	Shim Stock	0.9534	23.9	Hard black smooth glossy wavy deposit
186	O-79-20 24 hrs. Ox 201 C	2	315	180	Shim Stock	0.9845	24.8	Hard black smooth glossy wavy deopsit
187	O-82-14 24 hrs. Ox 205 C	3	315	180	Shim Stock	0.8786	31.9	Hard black smooth glossy wavy deposit. Thinner at edges
188	O-82-14 24 hrs. Ox 205 C	4	315	180	Shim Stock	0.9180	33.0	Hard black smooth glossy wavy deposit. Thinner at edges
189	O-82-2 24 hrs. Ox 205 C	1	315	180	Shim Stock	0.9544	18.3	Hard black smooth glossy wavy deposit
190	O-82-2 24 hrs. Ox 205 C	2	315	180	Shim Stock	0.9558	22.0	Hard black smooth glossy wavy deposit
191	O-82-3 24 hrs. Ox 205 C	3	315	180	Shim Stock	0.9638	25.2	Hard black smooth glossy wavy deposit
192	O-82-3 24 hrs. Ox 205 C	4	315	180	Shim Stock	0.9152	27.6	Hard black smooth glossy wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
193	D-79-20 24 hrs. Ox 205 C	1	315	180	Shim Stock	0.9690	24.2	Hard black glossy wavy deposit
194	D-79-20 24 hrs. Ox 205 C	2	315	180	Shim Stock	0.9524	28.4	Hard black glossy wavy deposit
195	D-79-20 48 hrs. Ox 205 C	3	315	180	Shim Stock	0.9770	27.1	Hard black glossy wavy deposit
196	D-79-20 48 hrs. Ox 205 C	4	315	180	Shim Stock	0.9349	30.8	Hard black glossy wavy deposit
197	D-82-2 48 hrs. Ox 205 C	1	315	180	Shim Stock	1.0504	23.8	Hard black smooth glossy wavy deposit
198	D-82-2 48 hrs. Ox 205 C	2	315	180	Shim Stock	1.0478	28.7	Hard black smooth glossy wavy deposit
199	D-79-17 48 hrs. Ox 205 C	3	315	180	Shim Stock	1.0331	23.8	Hard black smooth glossy wavy deposit
200	D-79-17 48 hrs. Ox 205 C	4	315	180	Shim Stock	0.8506	23.0	Hard black smooth glossy wavy deposit
201	D-82-2 48 hrs. Ox 205 C	1	315	180	Shim Stock	0.9780	24.8	Hard black smooth glossy wavy deposit
202	D-82-2 48 hrs. Ox 205 C	2	315	180	Shim Stock	0.9350	27.5	Hard black smooth glossy wavy deposit
203	D-82-3 48 hrs. Ox 205 C	3	315	180	Shim Stock	0.9442	31.7	Hard black smooth wavy deposit. Flaky on one edge.
204	D-82-3 48 hrs. Ox 205 C	4	315	180	Shim Stock	0.9595	35.1	Hard black smooth wavy deposit. Flaky on one edge.

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
205	O-76-5A Basestock Oil	1	315	180	Shim Stock	0.9977	21.6	Hard black glossy wavy deposit
206	O-76-5A Basestock Oil	2	315	180	Shim Stock	0.9927	26.7	Hard black glossy wavy deposit
207	O-76-8 Basestock Oil	3	315	180	Shim Stock	0.9566	5.3	Hard dull black thin smooth deposit
208	O-76-8 Basestock Oil	4	315	180	Shim Stock	0.9723	6.5	Hard dull black thin smooth deposit
209	O-76-5A 0.05% PANA	1	315	180	Shim Stock	0.9853	20.4	Hard black glossy wavy "orange peel" deposit
210	O-76-5A 0.05% PANA	2	315	180	Shim Stock	1.0013	17.4	Hard black glossy wavy "orange peel" deposit
211	O-76-8 0.5% PANA	1	315	180	Shim Stock	0.9663	6.3	Hard black glossy smooth deposit
212	O-76-8 0.5% PANA	2	315	180	Shim Stock	0.9685	6.2	Hard black glossy smooth deposit
213	O-76-5A 0.2% PANA	1	315	180	Shim Stock	0.9780	18.9	Hard black shiny wavy "orange peel" deposit
214	O-76-5A 0.2% PANA	2	315	180	Shim Stock	0.9790	15.9	Hard black shiny wavy orange peel" deposit
215	O-76-8 2.0% PANA	3	315	180	Shim Stock	0.9678	11.9	Slight shine matte grainy toward edges non-wavy deposit
216	O-76-8 2.0% PANA	4	315	180	Shim Stock	0.9700	12.2	Slight Shine matte grainy toward edges non-wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
217	0-76-5A 0.1% PANA	1	315	180	Shim Stock	0.9890	19.3	Hard black shiny wavy "orange peel" deposit
218	0-76-5A 0.1% PANA	2	315	180	Shim Stock	0.9915	16.8	Hard black shiny wavy "orange peel" deposit
219	0-76-8 1.0% PANA	3	315	180	Shim Stock	0.9561	8.6	Slight shine matte grainy toward edges smooth black deposit
220	0-76-8 1.0% PANA	4	315	180	Shim Stock	0.9909	8.9	Slight shine matte grainy toward edges smooth black deposit
221	0-76-5A 0.5% PANA	1	315	180	Shim Stock	1.0172	15.5	Hard black wavy semi-glossy deposit
222	0-76-5A 0.5% PANA	2	315	180	Shim Stock	1.0072	18.4	Hard black wavy semi-glossy deposit
223	0-76-5A 1.0% PANA	3	315	180	Shim Stock	1.0306	13.9	Hard black wavy semi-glossy deposit
224	0-76-5A 1.0% PANA	4	315	180	Shim Stock	1.0548	19.7	Hard black wavy semi-glossy deposit
225	0-76-5A 1.5% PANA	1	315	180	Shim Stock	1.0401	15.4	Hard black glossy wavy deposit
226	0-76-5A 1.5% PANA	2	315	180	Shim Stock	1.0516	19.1	Hard black glossy wavy deposit
227	0-76-5A 2.0% PANA	3	315	180	Shim Stock	1.0404	15.3	Hard black glossy wavy deposit
228	0-76-5A 2.0% PANA	4	315	180	Shim Stock	1.0896	17.2	Hard black glossy wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
229	O-79-16J	1	260	180	Aluminum	0.9775	14.7	Non-uniform light brown stain to hard sticky dark deposit
230	O-79-16J	2	260	180	Aluminum	0.9885	17.6	Non-uniform light brown stain to hard sticky dark brown deposit
231	O-79-16J	3	260	80	SS	0.9874	11.1	Non-uniform hard dark brown and black coke deposits
232	O-79-16J	4	260	180	SS	1.0108	13.8	Non-uniform hard dark brown and black coke deposits
233	O-79-16J 24 hrs. Ox 205 C	1	315	180	Aluminum	0.9876	21.4	Hard black coke brown stain non-uniform semi-glossy deposit
234	O-79-16J 24 hrs. Ox 205 C	2	315	180	Aluminum	1.0014	23.5	Hard black coke brown stain non-uniform semiglossy deposit
235	O-79-16J 24 hrs. Ox 205 C	3	315	180	SS	0.9968	17.4	Hard8 black glossy and semiglossy deposits No stained areas
236	O-79-16J 24 hrs. Ox 205 C	4	315	180	SS	1.0946	23.2	Hard black glossy spotty deposit "rainbow" stain areas
237	O-79-16J 48 hrs. CH 205 C	1	315	180	Shim Stock	0.9844	16.4	Hard black glossy wavy deposit
238	O-79-16J 48 hrs. CH 205 C	2	315	180	Shim Stock	0.9951	20.0	Hard black glossy wavy deposit
239	O-79-17E 48 hrs. CH 201 C	3	315	180	Shim Stock	0.9631	19.4	Hard black glossy wavy deposit
24[illegible]	O-79-17E 48 hrs. CH 201 C	4	315	180	Shim Stock	1.0161	19.1	Hard black glossy wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
241	O-76-B 1.5% PANA	1	315	180	Shim Stock	0.9608	12.6	Hard black grainy non-wavy glossy middle matte edges
242	O-76-B 1.5% PANA	2	315	180	Shim Stock	0.9574	11.4	Hard black grainy non-wavy glossy middle matte edges
243	O-79-20C 48 hrs. CH 205 C	3	315	180	Shim Stock	0.9922	23.7	Hard black shiny wavy deposit
244	O-79-20C 48 hrs. CH 205 C	4	315	180	Shim Stock	1.0135	25.6	Hard black shiny wavy deposit
245	O-82-2 48 hrs. CH 205 C	1	315	180	Shim Stock	0.9586	17.5	Hard black shiny wavy deposit
246	O-82-2 48 hrs. CH 205 C	2	315	180	Shim Stock	0.9238	19.8	Hard black shiny wavy deposit
247	O-82-2 48 hrs. CH 205 C	3	315	180	Shim Stock	0.9679	16.5	Hard black shiny wavy deposit
248	O-82-2 48 hrs. CH 205 C	4	315	180	Shim Stock	0.9843	19.9	Hard black shiny wavy deposit
249	O-82-3 48 hrs. CH 205 C	1	315	180	Shim Stock	0.9750	16.4	Hard black shiny wavy deposit
250	O-82-3 48 hrs. CH 205 C	2	315	180	Shim Stock	0.9o96	19.4	Hard black shiny wavy deposit
251	O-82-14D 48 hrs. CH 205 C	3	315	180	Shim Stock	0.9454	25.6	Hard black shiny wavy deposit
252	O-82-14D 48 hrs. CH 205 C	4	315	180	Shim Stock	0.9859	27.2	Hard black shiny wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
253	O-77-1 1.0% PANA	1	315	180	Shim Stock	0.9543	6.9	Thin hard black smooth glossy deposit
254	O-77-1 1.0 % PANA	2	315	180	Shim Stock	0.9616	6.0	Thin hard black smooth glossy deposit
255	O-77-1 2.0% PANA	3	315	180	Shim Stock	0.9721	6.4	Thin hard black smooth glossy deposit
256	O-77-1 2.0% PANA	4	315	180	Shim Stock	0.9800	8.9	Thin hard black smooth glossy deposit
257	O-77-1 Basestock Oil	1	315	180	Shim Stock	0.9591	5.3	Thin hard black smooth glossy deposit concentrated in center
258	O-77-1 Basestock Oil	2	315	180	Shim Stock	0.9627	3.7	Thin hard black smooth glossy deposit concentrated in center
259	O-77-1 0.5% PANA	3	315	180	Shim Stock	0.9655	6.1	Thin hard black smooth glossy deposit
260	O-77-1 0.5% PANA	4	315	180	Shim Stock	0.9762	6.9	Thin hard black smooth glossy deposit
261	O-76-8 Basestock Oil	1	260	180	Shim Stock	0.9809	11.7	Hard brown uneven deposit
262	O-76-8 Basestock Oil	2	260	180	Shim Stock	0.9975	13.2	Hard black wavy thin deposit in center
263	O-77-1 Basestock Oil	3	260	180	Shim Stock	0.9894	6.5	Hard black smooth thin deposit
264	O-77-1 Basestock Oil	4	260	180	Shim Stock	1.0019	7.4	Hard black smooth thin deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
265	O-76-8 1.0% DODPA	1	315	180	Shim Stock	0.9641	16.1	Hard black wavy glossy deposit
266	O-76-8 1.0% DODPA	2	315	180	Shim Stock	1.0001	7.1	Hard black smooth deposit
267	O-76-8 2.0% DODPA	3	315	180	Shim Stock	0.9954	10.6	Hard black wavy deposit
268	O-76-8 2.0% DODPA	4	315	180	Shim Stock	1.0079	14.9	Hard black wavy deposit
269	O-79-16J	1	245	180	Shim Stock	0.9783	12.2	Hard dark brown spotty smooth non-uniform deposit
270	O-79-16J	2	245	180	Shim Stock	0.9847	15.5	Hard dark brown smooth non-uniform deposit
271	O-79-17E	3	245	180	Shim Stock	1.0030	10.3	Light brown non-uniform sticky with hard black coke on edges
272	O-79-17E	4	245	180	Shim Stock	1.0202	11.6	Brown sticky smooth non-uniform varnish-like deposit
273	O-79-20C	1	245	180	Shim Stock	0.9883	20.9	Hard dark brown smooth wavy glossy deposit
274	O-79-20C	2	245	180	Shim Stock	0.9800	12.9*	Hard dark brown smooth wavy glossy deposit
275	O-82-2	3	245	180	Shim Stock	0.9873	22.6	Sticky smeary brown varnish with shiny non-uniform deposit
276	O-82-2	4	245	180	Shim Stock	0.9873	22.6	Hard dark brown smooth wavy glossy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
277	O-82-3	1	245	180	Shim Stock	0.9643	24.7	Soft semi-solid dark brown spotty shiny tar-like deposits
278	O-82-3	2	245	180	Shim Stock	0.9701	22.9	Soft semi-solid dark brown spotty shiny tar-like deposits
279	O-82-14D	3	245	180	Shim Stock	0.9745	12.7	Hard dark brown flaky smooth shiny deposit concentrated in center
280	O-82-14D	4	245	180	Shim Stock	1.0004	13.5	Hard dark brown flaky smooth shiny deposit concentrated in center
281	O-79-20C	1	245	180	Shim Stock	0.9744	22.2	Hard shiny wavy dark brown deposit
282	O-79-20C	2	245	180	Shim Stock	0.9917	23.2	Hard shiny wavy dark brown deposit
283	O-82-14D	3	330	180	Shim Stock	0.9834	14.9	Hard shiny wavy black deposit
284	O-82-14D	4	330	180	Shim Stock	1.0102	15.9	Hard shiny wavy black deposit
285	O-76-5A	1	260	180	Shim Stock	0.9901	25.2	Hard black coke on edges, shiny wavy varnish in middle
286	O-76-5A	2	260	180	Shim Stock	0.9935	21.6	Hard black coke on edges, shiny wavy varnish in middle
287	O-76-8 1.0% DODPA	3	315	180	Shim Stock	0.9617	8.3	Hard black shiny smooth deposit
288	O-76-8 1.0% DODPA	4	315	180	Shim Stock	0.9828	6.9	Hard black shiny smooth deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
289	O-77-1 1.5% PANA	1	315	180	Shim Stock	0.9623	8.6	Hard black smooth shiny wavy deposit, concentrated in middle.
290	O-77-1 1.5% PANA	2	315	180	Shim Stock	0.9408	7.0	Hard black smooth shiny wavy deposit, concentrated in middle.
291	O-76-8 1.5% DODPA	3	315	180	Shim Stock	0.9521	12.3	Hard black smooth slightly wavy shiny deposit
292	O-76-8 1.5% DODPA	4	315	180	Shim Stock	0.9759	10.2	Hard black smooth slightly wavy shiny deposit
293	O-76-5A	1	315	180	Shim Stock	0.9930	19.6	Hard black shiny wavy smooth deposit
294	O-76-5A	2	315	180	Shim Stock	0.9844	22.9	Hard black thin very smooth deposit
295	O-76-5A 0.5% DODPA	3	315	180	Shim Stock	1.0005	5.8	Hard Black thin very smooth deposit
296	O-76-5A 0.5% DODPA	4	315	180	Shim Stock	0.9646	7.0	Hard black thin very smooth deposit
297	O-76-8 0.5% DODPA	1	315	180	Shim Stock	0.9594	*16.3	Hard black glossy thick slightly wavy deposit
298	O-76-8 0.5% DODPA	2	215	180	Shim Stock	0.9508	7.0	Hard black low gloss thin flat non-wavy deposit
299	O-76-5A 1.0% DODPA	3	315	180	Shim Stock	1.0105	12.6	Hard black glossy thick areas on top of flat thin areas
300	O-76-5A 1.0% DODPA	4	315	180	Shim Stock	0.9945	15.6	Hard black glossy thick areas on top of flat thin areas

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
301	O-76-8 0.5% DODPA	1	315	180	Shim Stock	0.9581	6.8	Hard black shiny smooth deposit
302	O-76-8 0.5% DODPA	2	315	180	Shim Stock	0.9563	6.0	Hard black shiny smooth deposit
303	O-76-5A 1.0% DODPA	3	315	180	Shim Stock	0.9962	13.9	Hard black shiny wavy deposit
304	O-76-5A 1.0% DODPA	4	315	180	Shim Stock	1.0009	15.6	Hard black shiny wavy deposit
305	O-76-5A 0.5% DODPA	1	315	180	Shim Stock	0.9931	15.2	Hard black shiny wavy deposit
306	O-76-5A 0.5% DODPA	2	315	180	Shim Stock	0.9897	16.7	Hard black shiny wavy deposit
307	O-76-5A 1.5% DODPA	3	315	180	Shim Stock	0.9868	12.5	Hard black shiny wavy deposit
308	O-76-5A 1.5% DODPA	4	315	180	Shim Stock	1.0061	14.6	Hard black shiny wavy deposit
309	O-76-5A 2.0% DODPA	1	315	180	Shim Stock	0.9876	14.1	Hard black shiny wavy deposit
31	O-76-5A 2.0% DODPA	2	315	180	Shim Stock	0.9817	14.4	Hard black shiny wavy deposit
311	O-71-6	3	315	180	Shim Stock	1.0024	32.4	Hard black shiny wavy deposit
312	O-71-6	4	315	180	Shim Stock	1.0356	36.5	Hard black shiny wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
313	O-77-15A	1	315	180	Shim Stock	1.0081	33.6	Hard black shiny wavy deposit
314	O-77-15A	2	315	180	Shim Stock	0.9770	33.1	Hard black shiny wavy deposit
315	O-79-18C	3	315	180	Shim Stock	1.0279	38.0	Hard black shiny smooth non-wavy deposit
316	O-79-18	4	315	180	Shim Stock	0.9812	41.8	Hard black shiny smooth non-wavy deposit
317	E-109	1	315	180	Shim Stock	0.9420	5.5	Hard black smooth thin deposit - Contaminated Sample
318	E-109	2	315	180	Shim Stock	0.9396	6.7	Hard black smooth thin deposit - Contaminated Sample
319	E-120	3	315	180	Shim Stock	0.9198	4.9	Hard black smooth thin deposit
320	E-120	4	315	180	Shim Stock	0.9421	6.4	Hard black smooth thin deposit
321	E-129	1	315	180	Shim Stock	0.9941	20.1	Hard black shiny wavy deposit
322	E-129	2	315	180	Shim Stock	0.9785	21.5	Hard black shiny wavy deposit
323	E-139	3	315	180	Shim Stock	0.9179	13.5	Hard black shiny wavy deposit with cracks in edges
324	E-139	4	315	180	Shim Stock	0.9494	18.4	Hard black shiny wavy deposit cracked flaky loose brittle edges

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
325	E-149	1	315	180	Shim Stock	1.0142	73.6	Hard black shiny wavy cracked brittle thick deposit
326	E-149	2	315	180	Shim Stock	1.0068	77.3	Hard black shiny wavy, cracked brittle thick deposit
327	O-71-6 24 Ox 205 C	3	315	180	Shim Stock	0.9958	40.4	Hard black shiny wavy thick deposit
328	O-71-6 24 Ox 205 C	4	315	180	Shim Stock	1.0227	40.7	Hard black shiny wavy thick deposit
329	O-71-6 24 CH 205 C	1	315	180	Shim Stock	1.0123	33.3	Hard black shiny wavy deposit
330	O-71-6 24 CH 205 C	2	315	180	Shim Stock	1.0259	37.1	Hard black shiny wavy deposit
331	O-77-15 24 Ox 205 C	3	315	180	Shim Stock	0.9659	42.6	Hard black shiny wavy deposit
332	O-77-15 24 Ox 205 C	4	315	180	Shim Stock	1.0250	43.3	Hard black shiny wavy deposit
333	E-139	1	315	180	Shim Stock	.9327	21.7	Hard black shiny brittle flaky cracked deposit
334	E-139	2	315	180	Shim Stock	.9445	16.5	Hard black shiny brittle flaky cracked deposit
335	O-77-15 24 CH 205 C	3	315	180	Shim Stock	.9775	35.3	Hard black shiny wavy deposit
336	O-77-15 24 CH 205 C	4	315	180	Shim Stock	1.0042	39.8	Hard black shiny wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
337	0-79-18 24 Ox 205 C	1	315	180	Shia Stock	1.0952	44.6	Hard black shiny wavy deposit
338	0-79-18 24 Ox 205 C	2	315	180	Shia Stock	1.0793	43.8	Hard black shiny wavy deposit
339	0-79-18 24 CH 205 C	3	315	180	Shia Stock	1.0287	37.0	Hard black shiny wavy deposit
340	0-79-18 24 CH 205 C	4	315	180	Shia Stock	1.0299	44.6	Hard black shiny wavy deposit
341	0-77-15 48 Ox 205 C	1	315	180	Shia Stock	0.9920	46.9	Hard black shiny wavy deposit
342	0-77-15 48 Ox 205 C	2	315	180	Shia Stock	0.9899	48.7	Hard black shiny wavy deposit
343	0-77-15 48 CH 205 C	3	315	180	Shia Stock	0.9691	39.2	Hard black shiny wavy deposit
344	0-77-15 48 CH 205 C	4	315	180	Shia Stock	1.0000	38.7	Hard black shiny wavy deposit
345	0-71-6 48 Ox 205 C	1	315	180	Shia Stock	1.0003	44.9	Hard black shiny wavy deposit
346	0-71-6 48 Ox 205 C	2	315	180	Shia Stock	0.9845	46.3	Hard black shiny wavy deposit
347	0-71-6 48 CH 205 C	3	315	180	Shia Stock	1.0289	33.3	Hard black shiny wavy deposit
348	0-71-6 48 CH 205 C	4	315	180	Shia Stock	1.0165	38.1	Hard black shiny wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
349	O-79-18 48 Ox 205 C	1	315	180	Shia Stock	1.0289	42.6	Hard black shiny wavy deposit
350	O-79-18 48 Ox 205 C	2	315	180	Shia Stock	1.0030	46.0	Hard black shiny wavy deposit
351	O-79-18 48 CH 205 C	3	315	180	Shia Stock	0.0781	34.6	Hard black shiny non-wavy deposit
352	O-79-18 48 CH 205 C	4	315	180	Shia Stock	0.9874	37.2	Hard black shiny non-wavy deposit
353	O-77-1+I	1	315	180	Shia Stock	0.9128	3.4	Hard black thin dull smooth deposit
354	O-77-1+I	2	315	180	Shia Stock	0.9557	4.6	Hard black thin dull smooth deposit
355	O-77-1+IP	3	315	180	Shia Stock	0.9699	5.6	Hard black thin dull smooth deposit
356	O-77-1+IP	4	315	180	Shia Stock	0.9651	7.9	Hard black thin dull smooth deposit
357	O-79-17E	1	315	180	Aluminum	0.9841	9.8	Hard black smooth dull deposit
358	O-79-17E	2	315	180	Aluminum	0.9979	3.9	Seal leaked
359	O-79-20C	3	315	180	Aluminum	0.9869	2.2	Seal leaked
360	O-79-20C	4	315	180	Aluminum	0.9983	15.0	Hard black smooth dull deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
361	O-79-17E	1	315	180	Aluminum	0.9809	8.4	Hard black smooth dull deposit
362	O-79-17E	2	315	180	Aluminum	0.9907	15.5	Hard black smooth dull deposit
363	O-79-20C	3	315	180	Aluminum	0.9923	12.9	Hard black smooth dull deposit
364	O-79-20C	4	315	180	Aluminum	0.9912	14.1	Hard black smooth dull deposit
365	Sim Test 48	1	315	180	Shim Stock	1.0497	14.2	Hard black smooth glossy deposit
366	Sim Test 48	2	315	180	Shim Stock	1.0385	16.9	Hard black smooth glossy deposit
367	Sim Test 36	3	315	180	Shim Stock	1.0340	19.8	Hard black smooth glossy deposit
368	Sim Test 36	4	315	180	Shim Stock	1.0574	21.9	Hard black smooth glossy deposit
369	O-79-17E	1	315	180	Aluminum	1.0057	15.8	Hard brown to black smooth deposit
370	O-79-17E	2	315	180	Aluminum	0.9758	9.7	Hard brown to black wavy deposit
371	O-82-2	3	315	180	Aluminum	0.9731	13.9	Hard brown to black deposit. Non-uniform coverage
372	O-82-2	4	315	180	Aluminum	0.9757	22.1	Hard brown to black deposit. Non-uniform coverage

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
373	O-82-3	1	315	180	Aluminum	0.9651	8.7	Hard brown to black smooth low gloss deposit
374	O-82-3	2	315	180	Aluminum	0.9852	10.8	Hard brown to black smooth low gloss deposit
375	O-82-14	3	315	180	Aluminum	0.9840	12.5	Hard brown to black glossy wavy deposit
376	O-82-14	4	315	180	Aluminum	0.9710	11.3	Hard brown to black glossy wavy deposit
377	O-77-15	1	315	180	Aluminum	1.0313	40.2	Hard black glossy wavy deposit
378	O-77-15	2	315	180	Aluminum	1.0373	38.5	Hard black glossy wavy deposit
379	O-79-18	3	315	180	Aluminum	1.0710	38.1	Hard black glossy wavy deposit
380	O-79-18	4	315	180	Aluminum	1.0413	34.5	Hard black glossy wavy deposit
381	E-109	1	315	180	Shim Stock	0.9219	4.5	Hard black smooth semi-glossy deposit
382	E-109	2	315	180	Shim Stock	0.9898	6.0	Hard black smooth semi-glossy deposit
383	E-139	3	315	180	Shim Stock	0.8543	13.9	Hard black wavy glossy deposit
384	E-139	4	315	180	Shim Stock	0.9869	16.6	Hard black wavy glossy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
385	O-71-6	1	315	180	Aluminum	1.0432	10.3	Oil seeped under seal around entire seal cir.
386	O-71-6	2	315	180	Aluminum	1.0732	5.8	Oil seeped under seal around entire seal cir.
387	O-79-17	3	315	180	Aluminum	1.0298	12.2	Hard black semiglossy wavy deposit
388	O-79-17	4	315	180	Aluminum	1.0482	13.5	Hard black semiglossy wavy deposit
389	E-139	1	315	180	Shim Stock	0.9857	10.9	Flaky wavy black deposits
390	E-139	2	315	180	Shim Stock	0.9655	18.3	Flaky wavy black deposits
391	E-139	3	315	180	Shim Stock	0.9486	10.9	Flaky wavy black deposits
392	E-139	4	315	180	Shim Stock	0.9778	12.4	Flaky wavy black deposits
393	TEL 6031	1	315	180	Shim Stock	0.9884	28.4	Hard black wavy semi-glossy deposit
394	TEL 6031	2	315	180	Shim Stock	1.0062	32.7	Hard black wavy semi-glossy deposit
395	TEL 6032	3	315	180	Shim Stock	1.0137	10.3	Hard black smooth glossy deposit
396	TEL 6032	4	315	180	Shim Stock	1.0077	10.5	Hard black smooth glossy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
397	E-139	1	315	180	Shia Stock	0.9278	10.0*	Hard black wavy glossy deposit
398	E-139	2	315	180	Shia Stock	0.9835	12.7*	Hard black wavy glossy deposit
399	E-139	3	315	180	Shia Stock	0.9602	9.4*	Hard black wavy glossy deposit
400	E-139	4	315	180	Shia Stock	0.9592	8.5	Hard black wavy glossy deposit
401	O-77-1+I	1	315	180	Shia Stock	0.9734	2.2*	Hard black smooth deposit
402	O-77-1+I	2	315	180	Shia Stock	0.9786	1.8*	Hard black smooth deposit
403	O-77-1+IP	3	315	180	Shia Stock	1.0073	5.3*	Hard black smooth deposit
404	O-77-1+IP	4	315	180	Shia Stock	1.0015	5.2*	Hard black smooth deposit
405	E-139	1	315	180	Shia Stock	0.8065	13.1	Hard black cracked non-uniform deposit
406	E-139	2	315	180	Shia Stock	0.9398	15.6	Hard black cracked flaky non-uniform deposit
407	E-139	3	315	180	Shia Stock	0.9365	16.1	Hard black cracked flaky non-uniform deposit
408	E-139	4	315	180	Shia Stock	0.8598	20.5	Hard black cracked flaky non-uniform deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
409	O-77-1+I	3	315	180	Shim Stock	0.9358	5.3	Hard black wavy smooth low gloss deposit
410	O-77-1+I	1	315	180	Shim Stock	0.9366	8.7	Hard black very smooth low gloss deposit raised edges
411	O-77-1+IP	3	315	180	Shim Stock	0.9414	5.3	Hard black smooth low gloss slightly grainy deposit
412	O-77-1+IP	4	315	180	Shim Stock	0.9340	7.2	Hard black smooth low gloss slightly grainy deposit
413	O-79-16J	1	315	180	Shim Stock	0.9981	13.0	Hard black wavy glossy uniform coverage
414	O-79-16J	2	315	180	Shim Stock	0.9912	14.7	Hard black wavy glossy uniform coverage
415	O-79-16J	3	315	180	Shim Stock	0.9376	10.4	Hard black wavy glossy uniform coverage
416	O-79-16J	4	315	180	Shim Stock	0.9270	15.3	Hard black wavy glossy uniform coverage
417	O-79-17E	1	315	180	Shim Stock	1.0162	7.7	Hard black low gloss slightly wavy deposit
418	O-79-17E	2	315	180	Shim Stock	1.0231	12.0	Hard black low gloss wavy deposit
419	O-79-20C	3	315	180	Shim Stock	0.9991	9.7	Hard black low gloss wavy deposit thick edges
420	O-79-20C	4	315	180	Shim Stock	1.0117	13.4	Hard black low gloss wavy deposit thick edges

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
421	O-79-18 48 CH 205 C	1	315	180	Shim Stock	1.0168	37.2	Hard black shiny wavy deposit
422	O-79-182 48 CH 205 C	2	315	180	Shim Stock	1.0116	40.8	Hard black shiny wavy deposit
423	O-82-2	3	315	180	Shim Stock	0.9734	9.0	Hard black low gloss slightly wavy deposit thick edges
424	O-82-2	4	315	180	Shim Stock	0.9436	13.2	Hard black low gloss slightly wavy deposit thick edges
425	O-79-16J Pin-on-disk	1	315	180	Shim Stock	1.0134	5.4e	Hard black low gloss slightly wavy deposit thick edges
426	O-79-16J Pin-on-disk	2	315	180	Shim Stock	1.0088	16.4	Hard black low gloss slightly wavy deposit thick edges
427	O-79-16J Gear. Sim.	3	315	180	Shim Stock	0.7686	12.1	Hard black low gloss slightly wavy deposit thick edges
428	O-79-16J Gear. Sim.	4	315	180	Shim Stock	0.8642	15.8	Hard black low gloss slightly wavy deposit thick edges
429	O-85-1	1	315	180	Shim Stock	0.9976	26.1	Glossy hard black wavy deposit thin in center
430	O-85-1	2	315	180	Shim Stock	1.0048	29.3	Glossy hard black wavy deposit
431	O-82-3	3	315	180	Shim Stock	0.9759	7.8	Hard black flat deposit with glossy areas non-uniform cov.
432	O-82-3	4	315	180	Shim Stock	0.9606	9.5	Hard black flat deposit with glossy areas non-uniform cov.

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
433	0-82-14	1	315	180	Shim Stock	1.0299	11.6	Hard black shiny wavy dep. incomplete coverage
434	0-82-14	2	315	180	Shim Stock	1.0207	15.9	Hard black shiny wavy dep. incomplete coverage
435	0-77-15	3	315	180	Shim Stock	1.0603	31.4	Hard black flat deposit with glossy waves
436	0-77-15	4	315	180	Shim Stock	1.0599	32.6	Hard black flat deposit with glossy waves
437	0-79-18	1	315	180	SS	0.9969	34.8	Black wavy glossy hard deposit
438	0-79-18	2	315	180	SS	1.0447	40.5	Black wavy glossy hard deposit
439	0-76-6	3	315	180	SS	1.0534	27.2	Black wavy glossy hard deposit
440	0-71-6	4	315	180	SS	1.0405	31.2	Black wavy glossy hard deposit
441	0-79-16J Pin-on-disk	1	315	180	Shim Stock	1.0210	12.5	Hard black semiglossy slightly grainy deposit
442	0-79-16J Pin-on-disk	2	315	180	Shim Stock	1,0199	17.1	Hard black semiglossy slightly grainy deposit
443	0-82-3 Gear Sim	3	315	180	Shim Stock	1.0053	13.2	Hard gray to black semi-glossy wavy deposit
444	0-82-3 Gear Sim	4	315	180	Shim Stock	1.0199	16.6	Hard gray to black semi-glossy wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
445	O-77-1 0.5% DODPA	1	315	180	Shim Stock	0.9578	4.1	Hard black semiglossy deposit
446	O-77-1 0.5% DODPA	2	315	180	Shim Stock	0.9680	3.5	Hard black semiglossy deposit
447	O-77-1 1.0% DODPA	3	315	180	Shim Stock	0.9734	4.2	Hard black semiglossy deposit
448	O-77-1 1.0% DODPA	4	315	180	Shim Stock	0.9758	4.2	Hard black semiglossy deposit
449	O-77-1 1.5% DODPA	1	315	180	Shim Stock	0.9563	4.2	Smooth hard black semi-glossy deposit
450	O-77-1 1.5% DODPA	2	315	180	Shim Stock	0.9801	4.5	Smooth hard black semi-glossy deposit
451	O-77-1 2.0% DODPA	3	315	180	Shim Stock	0.9556	4.5	Smooth hard black semi-glossy deposit
452	O-77-1 2.0% DODPA	4	315	180	Shim Stock	0.9612	8.4	Smooth hard black semi-glossy deposit
453	O-76-8 Gear Sim	1	315	180	Shim Stock	0.9569	4.6	Hard flat black deposit
454	O-76-8 Gear Sim	2	315	180	Shim Stock	0.9752	4.7	Hard flat black deposit
455	O-76-5 Gear Sim	3	315	180	Shim Stock	1.0038	20.5	Hard black wavy deposit
456	O-76-5 Gear Sim	4	315	180	Shim Stock	0.9858	22.2	Hard black wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
457	0-79-16J	1	315	180	Quartz	0.9947	10.9	Hard dark brown glossy wavy deposit
458	0-79-16J	2	315	180	Quartz	0.9873	13.8	Hard dark brown glossy wavy deposit
459	0-77-1 2.0% DODPA	3	315	180	Shim Stock	0.9529	4.3	Hard black semi-glossy grainy deposit
460	0-77-1 2.0% DODPA	4	315	180	Shim Stock	0.9632	4.7	Hard black semi-glossy grainy deposit
461	0-77-15	1	300	180	Shim Stock	1.0063	35.8	Hard black glossy wavy deposit
462	0-77-15	2	300	180	Shim Stock	1.0124	35.6	Hard black glossy wavy deposit
463	0-79-18	3	300	180	Shim Stock	1.0231	35.6	Hard black glossy wavy deposit
464	0-79-18	4	300	180	Shim Stock	0.9912	36.6	Hard black glossy wavy deposit
465	0-71-6	1	260	180	Shim Stock	1.0228	59.9	Hard smooth glossy black deposit
466	0-71-6	2	260	180	Shim Stock	1.0323	56.5	Hard smooth glossy black deposit
467	0-71-6	3	300	180	Shim Stock	1.0678	36.2	Hard smooth glossy black deposit
468	0-71-6	4	300	180	Shim Stock	1.0012	32.8	Hard smooth glossy black deposit

AFAPL STATIC COKER TEST DATA

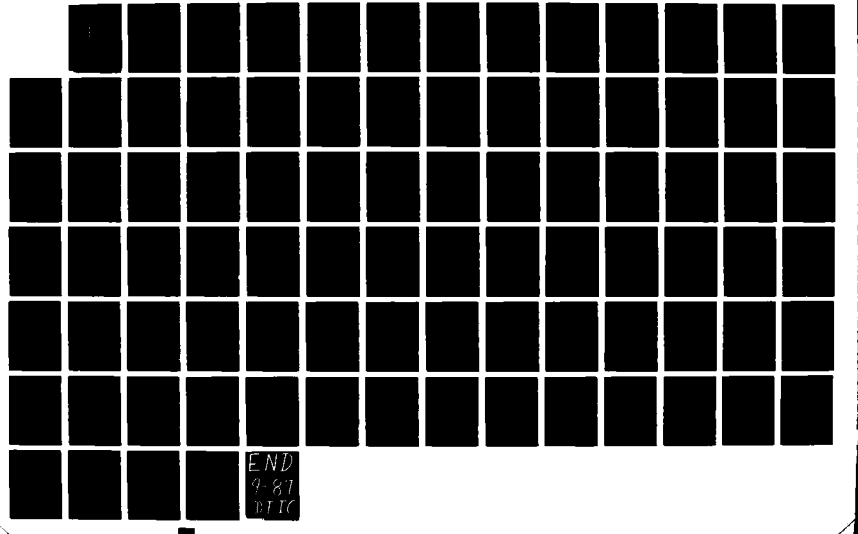
Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
469	0-77-15	1	260	180	Shim Stock	1.0034	72.1	Hard wavy black deposit
470	0-77-15	2	260	180	Shim Stock	1.0040	64.1	Hard wavy black deposit
471	0-79-18	3	260	180	Shim Stock	1.0303	61.9	Sticky deposit. Glossy black in center. Varnish towards edges
472	0-79-18	4	260	180	Shim Stock	1.0111	67.1	Sticky deposit. Glossy black in center. Varnish towards edges
473	0-79-16J Pin-on-disk	1	315	180	Quartz	1.012[illegible]	13.1	Glossy black deposit in center. Varnish towards edges
474	0-79-16J Pin-on-disk	2	315	180	Quartz	1.0439	15.1	Glossy black deposit in center. Varnish towards edges
475	TEL 6034	3	315	180	Shim Stock	1.0076	[illegible]	Hard semi-glossy black deposit
476	TEL 6034	4	315	180	Shim Stock	0.9407	9.[illegible]	Hard semi-glossy black deposit
477	0-79-16J	1	245	180	Shim Stock	0.9889	15.[illegible]	Hard brown to black smooth wavy deposit
478	0-79-16J	2	245	180	Shim Stock	1.0301	[illegible]	Hard brown to black smooth wavy deposit
479	0-79-17E	3	245	180	Shim Stock	1.0383	[illegible]	Hard brown to black smooth wavy deposit
480	0-79-17E	4	245	180	Shim Stock	1.[illegible]29[illegible]	[illegible]	Hard brown to black smooth wavy deposit

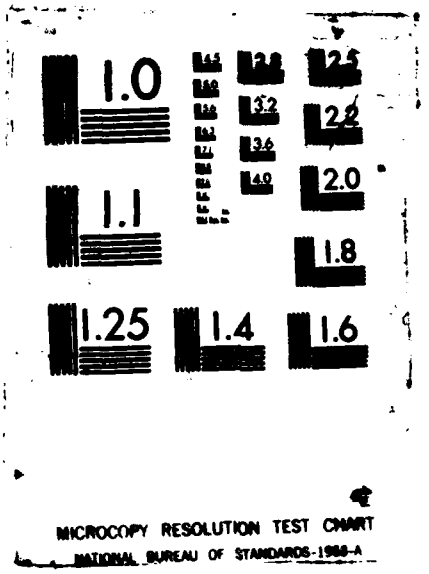
AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
481	E-120	1	315	180	Shim Stock	0.9536	9.7	Hard black smooth deposit
482	E-120	2	315	180	Shim Stock	0.9500	10.7	Hard black smooth deposit
483	E-120	3	315	180	Shim Stock	0.9551	9.5	Hard black smooth deposit
484	E-120	4	315	180	Shim Stock	0.9567	12.1	Hard black smooth deposit
485	E-109	1	315	180	Shim Stock	0.9759	4.5	Hard black smooth deposit
486	E-109	2	315	180	Shim Stock	0.9714	5.5	Hard black smooth deposit
487	E-1090	3	315	180	Shim Stock	0.9774	13.7	Hard black flaky deposit
488	E-1090	4	315	180	Shim Stock	1.0022	18.6	Hard black flaky deposit
489	E-120	1	315	180	Shim Stock	0.9479	4.9	Hard smooth glossy black deposit
490	E-120	2	315	180	Shim Stock	0.9589	4.9	Hard smooth glossy black deposit
491	E-120	3	315	180	Shim Stock	0.9388	5.2	Hard smooth glossy black deposit
492	E-120	4	315	180	Shim Stock	0.9655	4.5	Hard smooth glossy black deposit

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MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
493	E-105	1	315	180	Shim Stock	0.9953	2.0	Hard smooth glossy black deposit
494	E-105	2	315	180	Shim Stock	0.9850	1.7	Hard smooth glossy black deposit
495	E-105	3	315	180	Shim Stock	1.0002	1.6	Hard smooth glossy black deposit
496	E-105	4	315	180	Shim Stock	0.9615	1.6	Hard smooth glossy black deposit
497	D-77-1	1	315	180	Shim Stock	0.8950	3.5	Hard black smooth deposit
498	D-77-1	2	315	180	Shim Stock	0.9693	3.1	Hard black smooth deposit
499	D-77-1	3	315	180	Shim Stock	0.9587	3.1	Hard black smooth deposit
500	D-77-1	4	315	180	Shim Stock	0.9545	3.2	Hard black smooth deposit
501	D-79-17	1	245	180	Shim Stock	1.0054	12.0	Slightly sticky dark brown deposit
502	D-79-17	2	245	180	Shim Stock	1.0069	12.7	Dark brown shiny deposit
503	D-82-14	3	245	180	Shim Stock	0.9776	12.1	Hard black deposit and brown stain
504	D-82-14	4	245	180	Shim Stock	0.9600	13.2	Hard black deposit and brown stain

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
505	TEL-6034	1	245	180	Shim Stock	0.8679	5.1	Hard brown smooth varnish
506	TEL-6034	2	245	180	Shim Stock	0.9774	5.5	Hard brown smooth varnish
507	O-85-1	3	245	180	Shim Stock	0.9868	32.3	Sticky dark brown deposit
508	O-85-1	4	245	180	Shim Stock	1.0105	34.7	Sticky dark brown deposit
509	O-77-15	1	245	180	Shim Stock	1.0419	104.5	Brown to black tacky deposits
510	O-77-15	2	245	180	Shim Stock	1.0476	91.9	Brown to black tacky deposits
511	O-79-18	3	245	180	Shim Stock	1.0694	114.6	Oil and sludge residue
512	O-79-18	4	245	180	Shim Stock	1.0869	118.7	Oil and sludge residue
513	O-71-6	1	245	180	Shim Stock	1.0978	80.3	Brownish black deposit
514	O-71-6	2	245	180	Shim Stock	1.0962	49.5	Seal Leaked
515	TEL-6031	3	245	180	Shim Stock	1.0237	62.9	Hard glossy black deposit
516	TEL-6031	4	245	180	Shim Stock	0.9991	52.6	Hard glossy black deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
517	O-76-8	1	315	180	Quartz	0.9763	4.2	Hard brown to black coke and varnish
518	O-76-8	2	315	180	Quartz	0.9710	6.4	Hard brown to black coke and varnish
519	TEL-6032	3	245	180	Shim Stock	1.0236	23.5	Smooth hard black coke
520	TEL-6032	4	245	180	Shim Stock	1.0138	24.7	Smooth hard black coke
521	TEL-6034	1	260	180	Shim Stock	1.0011	4.3	Brown tacky wavy deposits
522	TEL-6034	2	260	180	Shim Stock	0.9739	6.9	Brown tacky wavy deposits
523	O-76-5	3	245	180	Shim Stock	1.0146	28.3	Hard brown wavy deposit
524	O-76-5	4	245	180	Shim Stock	1.0164	28.1	Hard brown wavy deposit
525	O-76-5	1	315	180	Quartz	1.0224	14.5	Hard brown wavy deposit
526	O-76-5	2	315	180	Quartz	1.0358	19.2	Hard brown wavy deposit
527	TEL-6031	3	260	180	Shim Stock	0.9695	41.7	Hard black glossy smooth deposit
528	TEL-6031	4	260	180	Shim Stock	1.0329	41.1	Hard black glossy smooth deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
529	O-71-6	1	245	180	Shim Stock	1.0753	84.4	Hard black glossy deposit
530	O-71-6	2	245	180	Shim Stock	1.0628	83.9	Hard black glossy deposit
531	TEL-6032	3	260	180	Shim Stock	1.0271	19.9	Hard black glossy wavy deposit
532	TEL-6032	4	260	180	Shim Stock	0.9879	21.2	Hard black glossy wavy deposit
533	O-82-3	1	315	180	Quartz	0.9805	9.4	Hard black wavy coke and varnish
534	O-82-3	2	315	180	Quartz	0.9994	7.9	Hard black wavy coke and varnish
535	TEL-6031	3	300	180	Shim Stock	1.0171	30.3	Hard black wavy deposit
536	TEL-6031	4	300	180	Shim Stock	1.0119	29.9	Hard black wavy deposit
537	O-79-16	1	315	180	Quartz	1.2223	12.3	Hard dark brown wavy deposit
538	O-79-16	2	315	180	Quartz	1.0062	19.6	Hard dark brown wavy deposit
539	TEL-6032	3	300	180	Shim Stock	0.9937	12.2	Hard black smooth deposit
540	TEL-6032	4	300	180	Shim Stock	1.0143	13.3	Hard black smooth deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
541	TEL-6034	1	315	180	Shim Stock	0.9822	6.2	Hard black smooth glossy deposit
542	TEL-6034	2	315	180	Shim Stock	1.0105	8.5	Hard black smooth glossy deposit
543	O-85-1	3	300	180	Shim Stock	1.0510	25.7	Hard black glossy wavy deposit
544	O-85-1	4	300	180	Shim Stock	1.0536	29.4	Hard black glossy wavy deposit
545	O-82-3 Gear Sim	1	315	180	Quartz	1.0132	12.1	Hard black flat wavy flaky deposit
546	O-82-3 Gear Sim	2	315	180	Quartz	0.9728	16.2	Hard black flat wavy flaky deposit
547	O-76-5 Gear Sim	3	315	180	Quartz	1.0148	19.3	Hard black glossy wavy deposit
548	O-76-5 Gear Sim	4	315	180	Quartz	1.0390	22.1	Hard black glossy wavy deposit
549	O-76-5	1	300	180	Shim Stock	1.0399	16.5	Hood airflow and temperature variable
550	O-76-5	2	300	180	Shim Stock	1.0314	19.2	Hood airflow and temperature variable
551	O-76-8	3	300	180	Shim Stock	0.9998	13.5	Hood airflow and temperature variable
552	O-76-8	4	300	180	Shim Stock	0.9979	18.9	Hood airflow and temperature variable

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
553	O-79-16 Gear Sim	1	315	180	Quartz	0.8792	9.8	Hood airflow and temperature variable
554	O-79-16 Gear Sim	2	315	180	Quartz	0.7352	14.8	Hood airflow and temperature variable
555	O-76-8 Gear Sim	3	315	180	Quartz	0.9830	10.0	Hood airflow and temperature variable
556	O-76-8 Gear Sim	4	315	180	Quartz	1.0082	15.0	Hood airflow and temperature variable
557	TEL-6034	1	300	180	Shim Stock	1.0024	8.8	Smooth hard black glossy deposit
558	TEL-6034	2	300	180	Shim Stock	0.9395	10.2	Smooth hard black glossy deposit
559	O-77-1	3	300	180	Shim Stock	1.0021	26.1	Sample suspect
560	O-77-1	4	300	180	Shim Stock	1.0202	21.9	Sample suspect
561	O-79-17	1	315	180	Quartz	1.0472	11.4	Hard dark brown flaky deposit
562	O-79-17	2	315	180	Quartz	1.0533	19.2	Hard dark brown flaky deposit
563	O-79-20	3	315	180	Quartz	1.0134	16.8	Hard dark brown glossy wavy deposit
564	O-79-20	4	315	180	Quartz	1.0186	14.9	Hard dark brown glossy wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
565	O-76-5	1	300	180	Shim Stock	0.9944	16.0	Hard black glossy wavy deposit
566	O-76-5	2	300	180	Shim Stock	1.0092	22.5	Hard black glossy wavy deposit
567	O-76-8	3	300	180	Shim Stock	0.9735	10.5	Hard black glossy slightly wavy deposit
568	O-76-8	4	300	180	Shim Stock	0.9691	15.0	Hard black glossy slightly wavy deposit
569	O-79-16	1	315	180	Shim Stock	1.0456	13.3	Hard black glossy deposit
570	O-79-16	2	315	180	Shim Stock	1.0195	16.7	Contained external contamination
571	O-79-16	3	315	180	Shim Stock	1.0072	12.5	Hard black glossy deposit
572	O-79-16	4	315	180	Shim Stock	1.0057	14.5	Hard black glossy deposit
573	O-77-1	1	300	180	Shim Stock	0.9903	7.2	Hard smooth glossy black deposit
574	O-77-1	2	300	180	Shim Stock	0.9845	15.6	Controller problem
575	O-76-8	3	300	180	Shim Stock	0.9871	10.6	Hard smooth glossy black wavy deposit
576	O-76-8	4	300	180	Shim Stock	0.9903	16.3	Hard smooth glossy black wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
577	O-76-8 Gear Sim	1	315	180	Quartz	0.9795	9.7	Hard black glossy flaky deposits
578	O-76-8 Gear Sim	2	315	180	Quartz	0.9636	16.7	Hard black glossy flaky deposits
579	O-82-14	3	315	180	Quartz	0.9877	12.8	Hard black glossy wavy deposit
580	O-82-14	4	315	180	Quartz	0.9985	19.4	Hard black glossy wavy deposit
581	O-76-5	1	300	180	Shim Stock	1.0214	17.6	Hard black glossy wavy deposit
582	O-76-5	2	300	180	Shim Stock	1.0326	23.4	Hard black glossy wavy deposit
583	O-76-8	3	300	180	Shim Stock	0.9992	11.4	Hard black semiglossy deposit
584	O-76-8	4	300	180	Shim Stock	0.9985	15.0	Hard black semiglossy deposit
585	O-85-1	1	260	180	Shim Stock	0.9732	37.0	Hard black shiny wavy deposit
586	O-85-1	2	260	180	Shim Stock	1.0131	29.2	Seal leaked
587	TEL-6034	3	300	180	Shim Stock	0.9500	7.4	Hard black smooth shiny deposit
588	TEL-6034	4	300	180	Shim Stock	0.9594	9.4	Hard black smooth shiny deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
589	O-71-6	1	315	180	Aluminum	1.0452	28.9	Hard black glossy wavy deposit
590	O-71-6	2	315	180	Aluminum	1.0196	35.4	Hard black glossy wavy deposit
591	O-77-1	3	300	180	Shia Stock	0.9494	8.7	Hard black glossy wavy deposit
592	O-77-1	4	300	180	Shia Stock	0.9548	16.1	Suspect test data
593	O-79-17	1	315	180	Quartz	0.9655	7.2	Hard dark brown to black wavy deposit
594	O-79-17	2	315	180	Quartz	0.9896	11.3	Hard dark brown to black wavy deposit
595	O-79-20	3	315	180	Quartz	0.9842	10.3	Hard dark brown to black wavy deposit
596	O-79-20	4	315	180	Quartz	0.9789	14.3	Hard dark brown to black wavy deposit
597	O-77-1	1	245	180	Shia Stock	0.8959	9.5	Hard black smooth deposit
598	O-77-1	4	245	180	Shia Stock	0.9748	9.9	Hard black smooth deposit
599	O-77-15	3	315	180	Aluminum	1.0058	29.5	Hard black smooth deposit
600	O-77-15	2	315	180	Aluminum	1.0249	34.7	Hard black smooth deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
601	O-76-8 1.0% PANA 1.0% DODPA	1	315	180	Shim Stock	0.9656	15.4	Hard black shiny wavy deposit
602	O-76-8 1.0% PANA 1.0% DODPA	3	315	180	Shim Stock	0.9621	11.9	Hard black shiny wavy deposit
603	O-77-1 1.0% PANA 1.0% DODPA	2	315	180	Shim Stock	0.9637	9.9	Hard black low gloss smooth thin depost
604	O-77-1 1.0% PANA 1.0% DODPA	4	315	180	Shim Stock	0.9662	10.4	Hard black low gloss smooth thin deposit
605	O-76-5 1.0% PANA 1.0% DODPA	1	315	180	Shim Stock	1.2139	14.3	Hard black shiny wavy dull in spots
606	O-76-5 1.0% PANA 1.0% DODPA	3	315	180	Shim Stock	1.0109	12.6	Hard black shiny wavy dull in spots
607	O-76-5 0.5% PTZ	2	315	180	Shim Stock	0.9994	24.4	Hard black shiny wavy deposit
608	O-76-5 0.5% PTZ	4	315	180	Shim Stock	1.0055	21.6	Hard black shiny wavy deposit
609	O-76-5 1.0% PTZ	1	315	180	Shim Stock	1.0116	17.9	Hard black shiny wavy deposit. Dull in spots
610	O-76-5 1.0% PTZ	3	315	180	Shim Stock	1.0018	17.2	Hard black shiny wavy deposit. Dull in spots
611	O-76-5 1.5% PTZ	2	315	180	Shim Stock	1.0098	21.7	Hard black shiny wavy deposit. Dull in spots
612	O-76-5 1.5% PTZ	4	315	180	Shim Stock	1.0064	18.3	Hard black shiny wavy deposit. Dull in spots

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
613	O-76-5 2.0% PTZ	1	315	180	Shim Stock	0.9610	5.2	Hard black very smooth dull deposit. Conc. in center
614	O-76-5 2.0% PTZ	3	315	180	Shim Stock	0.9689	3.8	Hard black very smooth dull deposit. Conc. in center
615	O-77-1 0.5% PTZ	2	315	180	Shim Stock	0.9925	22.0	Hard black shiny wavy deposit
616	O-77-1 0.5% PTZ	4	315	180	Shim Stock	1.0035	20.0	Hard black shiny wavy deposit
617	O-77-1 1.0% PTZ	1	315	180	Shim Stock	0.9821	5.7	Hard black low gloss very smooth deposit
618	O-77-1 1.0% PTZ	3	315	180	Shim Stock	0.9321	5.0	Hard black low gloss very smooth deposit
619	O-77-1 1.5% PTZ	2	315	180	Shim Stock	0.9719	6.2	Hard black low gloss very smooth deposit
620	O-77-1 1.5% PTZ	4	315	180	Shim Stock	0.9603	5.9	Hard black low gloss very smooth deposit
621	O-77-1 2.0% PTZ	1	315	180	Shim Stock	0.9641	7.0	Hard black glossy smooth deposit
622	O-77-1 2.0% PTZ	3	315	180	Shim Stock	0.9763	6.2	Hard black glossy smooth deposit
623	O-76-8 2.0% PTZ	2	315	180	Shim Stock	0.9565	10.9	Hard black flat deposit
624	O-76-8 2.0% PTZ	4	315	180	Shim Stock	0.9757	10.6	Hard black flat deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
625	O-76-8 0.5% PTZ	1	315	180	Shim Stock	0.9142	6.9	Hard black partially glossy flat deposit
626	O-76-8 0.5% PTZ	3	315	180	Shim Stock	0.9296	5.5	Hard black partially glossy flat deposit
627	O-76-8 1.0% PTZ	2	315	180	Shim Stock	0.9103	7.1	Hard grayish black non-glossy smooth grainy deposit
628	O-76-8 1.0% PTZ	4	315	180	Shim Stock	0.9462	10.6	Hard grayish black non-glossy smooth grainy deposit
629	O-76-8 1.5% PTZ	1	315	180	Shim Stock	0.9914	10.0	Hard grayish black non-glossy smooth grainy deposit
630	O-76-8 1.5% PTZ	3	315	180	Shim Stock	0.9878	8.7	Hard grayish black non-glossy smooth grainy deposit
631	O-76-5 2.0% DOPTZ	2	315	180	Shim Stock	1.0556	21.7	Hard black glossy wavy deposit
632	O-76-5 2.0% DOPTZ	4	315	180	Shim Stock	1.0371	22.0	Hard black glossy wavy deposit
633	O-77-1 2% PTZ 24 Ox 205	1	315	180	Shim Stock	0.9203	8.0	Hard black non-glossy grainy deposit
634	O-77-1 2% PTZ 24 Ox 205	3	315	180	Shim Stock	0.9513	7.9	Hard black non-glossy deposit
635	O-77-1 2% PTZ 24 Ox 205 Fil	2	315	180	Shim Stock	0.7490	7.6	Hard black partially glossy smooth deposit
636	O-77-1 2% PTZ 24 Ox 205 Fil	4	315	180	Shim Stock	0.8700	6.7	Hard black partially glossy smooth deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
637	D-77-1 2% PTZ 48 CH 205	1	315	180	Shim Stock	0.9612	8.0	Hard black shiny smooth deposit. Conc. in center
638	D-77-1 2% PTZ 48 CH 205	3	315	180	Shim Stock	10153	7.6	Hard black shiny smooth deposit. Conc. in center
639	D-77-1 2% PTZ 48 CH 205 Fil	2	315	180	Shim Stock	0.9472	8.1	Hard black shiny smooth deposit
640	D-77-1 2% PTZ 48 CH 205 Fil	4	315	180	Shim Stock	0.9515	11.1	Hard black shiny smooth deposit
641	D-77-1 2.0% DOPTZ	1	315	180	Shim Stock	0.9654	14.8	Hard black shiny smooth deposit
642	D-77-1 2.0% DOPTZ	2	315	180	Shim Stock	0.9638	15.5	Hard black shiny smooth deposit
643	D-76-8 2.0% DOPTZ	3	315	180	Shim Stock	0.9628	15.7	Hard black shiny smooth deposit
644	D-76-8 2.0% DOPTZ	4	315	180	Shim Stock	0.9628	16.2	Hard black shiny smooth deposit
645	D-76-5 2% PTZ 24 Ox 205	1	315	180	Shim Stock	0.9774	26.4	Hard black shiny wavy grainy deposit
646	D-76-5 2% PTZ 24 Ox 205	2	315	180	Shim Stock	0.9640	27.3	Hard black shiny wavy grainy deposit
647	D-76-8 2% PTZ 24 Ox 205	3	315	180	Shim Stock	0.9462	17.3	Hard grayish black flat dull grainy deposit
648	D-76-8 2% PTZ 24 Ox 205	4	315	180	Shim Stock	0.9100	20.3	Hard grayish black flat dull grainy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
649	O-76-5 2% PTZ 24 Ox 205 Fil	1	315	180	Shim Stock	0.9600	20.2	Hard black shiny very smooth deposit
650	O-76-5 2% PTZ 24 Ox 205 Fil	2	315	180	Shim Stock	1.0040	22.4	Hard black shiny very smooth deposit
651	O-76-8 2% PTZ 24 Ox 205 Fil	3	315	180	Shim Stock	0.9642	15.3	Hard black flat slightly grainy deposit
652	O-76-8 2% PTZ 24 Ox 205 Fil	4	315	180	Shim Stock	0.9552	14.0	Hard black flat slightly grainy deposit
653	O-76-8 2% DOPTZ 24 Ox 205	1	315	180	Shim Stock	0.9507	18.2	Hard grayish black flat grainy deposit
654	O-76-8 2% DOPTZ 24 Ox 205	2	315	180	Shim Stock	0.9615	18.2	Hard grayish black flat grainy deposit
655	O-76-5 2% DOPTZ 24 Ox 205	3	315	180	Shim Stock	0.9904	24.4	Hard black shiny wavy grainy deposit
656	O-76-5 2% DOPTZ 24 Ox 205	4	315	180	Shim Stock	0.9916	25.2	Hard black shiny wavy grainy deposit
657	O-76-8 2% DOPTZ 24 Ox 205 Fil	1	315	180	Shim Stock	0.9531	16.0	Hard black flat grainy deposit
658	O-76-8 2% DOPTZ 24 Ox 205 Fil	2	315	180	Shim Stock	0.9552	15.1	Hard black flat slightly grainy deposit
659	O-76-5 2% DOPTZ 24 Ox 205 Fil	3	315	180	Shim Stock	0.9759	20.1	Hard black shiny wavy deposit
660	O-76-5 2% DOPTZ 24 Ox 205 Fil	4	315	180	Shim Stock	0.9430	20.8	Hard black shiny wavy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
661	O-77-1 2% DOPTZ 24 Ox 205	1	315	180	Shim Stock	0.9435	11.8	Hard black shiny grainy deposit
662	O-77-1 2% DOPTZ 24 Ox 205	2	315	180	Shim Stock	0.9462	8.0	Hard black shiny grainy deposit
663	O-77-1 2% DOPTZ 24 Ox 205 Fil	3	315	180	Shim Stock	0.9328	9.4	Hard black shiny smooth deposit
664	O-77-1 2% DOPTZ 24 Ox 205 Fil	4	315	180	Shim Stock	0.9496	9.1	Hard black shiny smooth deposit
665	O-76-5 2% PTZ 48 CH 205	1	315	180	Shim Stock	0.9764	28.4	Hard black shiny very grainy deposit
666	O-76-5 2% PTZ 48 CH 205	2	315	180	Shim Stock	0.9772	31.3	Hard black shiny very grainy deposit
667	O-77-1 2% DOPTZ 48 CH 205	3	315	180	Shim Stock	0.9710	16.0	Hard gray-black partially shiny grainy deposit
668	O-77-1 2% DOPTZ 48 CH 205	4	315	180	Shim Stock	0.9626	16.7	Hard gray-black partially shiny grainy flaky deposit
669	O-76-8 2% DOPTZ 48 CH 205	1	315	180	Shim Stock	0.9671	22.1	Hard black shiny wavy deposit
670	O-76-8 2% DOPTZ 48 CH 205	2	315	180	Shim Stock	0.9567	25.4	Hard black shiny wavy deposit
671	O-76-8 2% PTZ 48 CH 205	3	315	180	Shim Stock	0.9783	15.0	Hard grayish black flat very grainy deposit
672	O-76-8 2% PTZ 48 CH 205	4	315	180	Shim Stock	0.8939	13.4	Hard grayish black flat very grainy deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
673	O-76-5 0.5% DOPTZ	1	315	180	Shim Stock	0.9922	19.0	Hard black shiny wavy deposit. Controller problem
674	O-76-5 0.5% DOPTZ	2	315	180	Shim Stock	0.9822	11.0	Hard black shiny wavy deposit. Controller problem
675	O-76-5 1.0% DOPTZ	3	315	180	Shim Stock	1.0811	14.3	Hard black shiny wavy deposit. Controller problem
676	O-76-5 1.0% DOPTZ	4	315	180	Shim Stock	0.9843	22.4	Hard black shiny wavy deposit. Controller problem
677	O-76-5 0.5% DOPTZ	1	315	180	Shim Stock	1.0106	20.7	Hard black shiny wavy deposit
678	O-76-5 0.5% DOPTZ	2	315	180	Shim Stock	1.0196	19.4	Hard black shiny wavy deposit
679	O-76-5 1.0% DOPTZ	3	315	180	Shim Stock	1.0186	17.2	Hard black shiny wavy deposit
680	O-76-5 1.0% DOPTZ	4	315	180	Shim Stock	1.0235	20.4	Hard black shiny wavy deposit
681	O-76-5 1.5% DOPTZ	1	315	180	Shim Stock	0.9785	16.4	Hard black wavy glossy deposit
682	O-76-5 1.5% DOPTZ	2	315	180	Shim Stock	0.9701	22.0	Hard black wavy glossy deposit
683	O-77-1 0.5% DOPTZ	3	315	180	Shim Stock	0.9259	3.7	Hard black thin deposit
684	O-77-1 0.5% DOPTZ	4	315	180	Shim Stock	0.9352	6.8	Hard black thin deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
685	O-77-1 1.0% DOPTZ	1	315	180	Shim Stock	0.9488	5.2	Hard black wavy glossy deposit
686	O-77-1 1.0% DOPTZ	2	315	180	Shim Stock	0.9733	7.7	Hard black wavy glossy deposit
687	O-77-1 1.5% DOPTZ	3	315	180	Shim Stock	0.9446	6.3	Hard black wavy glossy deposit
688	O-77-1 1.5% DOPTZ	4	315	180	Shim Stock	0.9468	9.9	Hard black wavy glossy deposit
689	O-76-8 0.5% DOPTZ	1	315	180	Shim Stock	0.9116	7.3	Hard black shiny slightly wavy deposit
690	O-76-8 0.5% DOPTZ	2	315	180	Shim Stock	0.9658	6.9	Hard black shiny smooth deposit
691	O-76-8 1.0% DOPTZ	3	315	180	Shim Stock	0.9130	5.7	Hard black shiny smooth deposit
692	O-76-8 1.0% DOPTZ	4	315	180	Shim Stock	0.9174	9.6	Hard black shiny slightly wavy deposit
693	O-85-1	1	260	180	Shim Stock	0.9184	44.4	Hard black shiny wavy deposit. Seal seepage
694	O-85-1	2	260	180	Shim Stock	1.0153	36.4	Hard black shiny wavy deposit
695	O-76-8 1.5% DOPTZ	3	315	180	Shim Stock	0.9644	9.2	Hard black smooth shiny deposit
696	O-76-8 1.5% DOPTZ	4	315	180	Shim Stock	0.8922	11.4	Hard black smooth shiny deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
697	O-76-5 2% DOPTZ 48 CH 205	1	315	180	Shim Stock	0.9790	26.5	Hard black shiny wavy deposit
698	O-76-5 2% DOPTZ 48 CH 205	2	315	180	Shim Stock	0.9669	25.1	Hard black shiny wavy deposit
699	O-77-1 2% DOPTZ 48 CH 205 Fil	3	315	180	Shim Stock	0.9295	13.0	Hard black shiny wavy grainy deposit
700	O-77-1 2% DOPTZ 48 CH 205 Fil	4	315	180	Shim Stock	0.9545	14.5	Hard black shiny flaky wavy grainy deposit
701	O-76-5 2% PTZ 48 CH 205 Fil	1	315	180	Shim Stock	0.9934	26.0	Hard black shiny wavy deposit
702	O-76-5 2% PTZ 48 CH 205 Fil	2	315	180	Shim Stock	0.9621	26.4	Hard black shiny wavy deposit
703	O-76-5 2% DOPTZ 48 CH 205 Fil	3	315	180	Shim Stock	0.9657	18.3	Hard black shiny wavy deposit
704	O-76-5 2% DOPTZ 48 CH 205 Fil	4	315	180	Shim Stock	0.9286	24.2	Hard black shiny wavy deposit
705	O-77-1 2% PTZ 48 Ox 205	1	315	180	Shim Stock	0.9864	10.5	Hard black shiny flaky deposit
706	O-77-1 2% PTZ 48 Ox 205	2	315	180	Shim Stock	0.9526	13.8	Hard black shiny slightly grainy deposit
707	O-76-5 2% PTZ 48 Ox 205	3	315	180	Shim Stock	0.9753	24.0	Hard black shiny wavy grainy deposit
708	O-76-5 2% PTZ 48 Ox 205	4	315	180	Shim Stock	0.9548	17.9	Hard black shiny wavy deposit (seal leak)

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
709	O-77-1 2% DOPTZ 48 Ox 205	1	315	180	Shim Stock	0.8791	17.2	Hard black very flaky deposit
710	O-77-1 2% DOPTZ 48 Ox 205	2	315	180	Shim Stock	0.9720	17.3	Hard black very flaky deposit
711	O-76-5 2% DOPTZ 48 Ox 205	3	315	180	Shim Stock	1.0001	65.3	Hard black shiny wavy very thick deposit
712	O-76-5 2% DOPTZ 48 Ox 205	4	315	180	Shim Stock	1.0196	72.7	Hard black shiny wavy very thick deposit
713	O-77-1 2% PTZ 48 OX 205 Fil	1	315	180	Shim Stock	0.9191	8.5	Hard black thin slightly wavy deposit
714	O-77-1 2% PTZ 48 OX 205 Fil	2	315	180	Shim Stock	0.8884	10.1	Hard black thin slightly wavy deposit
715	O-76-5 2% PTZ 48 OX 205 Fil	3	315	180	Shim Stock	0.9995	25.2	Hard black shiny wavy deposit
716	O-76-5 2% PTZ 48 OX 205 Fil	4	315	180	Shim Stock	0.9496	30.4	Hard black shiny wavy deposit
717	O-77-1 2% DOPTZ 48 Ox 205 Fil	1	315	180	Shim Stock	0.9439	17.4	Hard black shiny very flaky deposit
718	O-77-1 2% DOPTZ 48 Ox 205 Fil	2	315	180	Shim Stock	0.9330	16.2	Hard black shiny very flaky deposit
719	O-76-5 2% DOPTZ 48 Ox 205 Fil	3	315	180	Shim Stock	0.9800	68.6	Hard black shiny wavy very thick deposit
720	O-76-5 2% DOPTZ 48 205 Fil	4	315	180	Shim Stock	1.0096	71.5	Hard black shiny wavy very thick deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
721	O-76-5 2% PTZ 48 Ox 205	1	315	180	Shim Stock	0.9906	27.8	Hard black shiny wavy grainy deposit
722	O-76-5 2% PTZ 48 Ox 205	2	315	180	Shim Stock	0.9544	34.0	Hard black shiny wavy grainy deposit
723	O-76-5 2% PTZ 48 Ox 205 Fil	3	315	180	Shim Stock	0.9734	22.5	Hard black shiny wavy deposit
724	O-76-5 2% PTZ 48 Ox 205 Fil	4	315	180	Shim Stock	0.9420	28.8	Hard black shiny wavy deposit
725	O-77-1 2% PTZ 48 CH 205	1	315	180	Shim Stock	0.9660	9.4	Hard black shiny thin slightly grainy deposit
726	O-77-1 2% PTZ 48 CH 205	2	315	180	Shim Stock	0.9601	8.3	Hard black shiny thin slightly grainy deposit
727	O-77-1 2% PTZ 48 CH 205 Fil	3	315	180	Shim Stock	0.9593	7.8	Hard black shiny thin slightly grainy deposit
728	O-77-1 2% PTZ 48 CH 205 Fil	4	315	180	Shim Stock	0.9519	9.1	Hard black shiny thin slightly grainy deposit
729	O-76-8 2% PTZ 48 CH 205 Fil	1	315	180	Shim Stock	0.9184	15.4	Hard black dull grainy flat deposit
730	O-76-8 2% PTZ 48 CH 205 Fil	2	315	180	Shim Stock	0.9316	15.2	Hard black dull grainy flat deposit
731	O-76-8 2% DOPTZ 48 CH 205 Fil	3	315	180	Shim Stock	0.9190	15.8	Hard black shiny flaky deposit
732	O-76-8 2% DOPTZ 48 CH 205 Fil	4	315	180	Shim Stock	0.8820	14.2	Hard black shiny flaky deposit

AFAPL STATIC COKER TEST DATA

Test No.	Sample	Coker No.	Test Temp. °C	Test Time min.	Type Test Specimen	Sample Size gm	Deposit mg/gm Oil	Description of Deposit
733	O-76-8 2% DOPTZ 48 CM 205	1	315	180	Shim Stock	0.9441	14.5	Hard black shiny flaky deposit
734	O-76-8 2% DOPTZ 48 CM 205	2	315	180	Shim Stock	0.9452	12.7	Hard black shiny flaky deposit
735	O-77-1 2% DOPTZ	1	315	180	Shim Stock	0.9674	6.4	Hard black shiny smooth deposit
736	O-77-1 2% DOPTZ	2	315	180	Shim Stock	0.9645	7.2	Hard black shiny smooth deposit
737	O-77-1 2% DOPTZ 24 Ox 205	3	315	180	Shim Stock	0.9405	6.8	Hard black semi-shiny wavy grainy deposit
738	O-77-1 2% DOPTZ 24 Ox 205	4	315	180	Shim Stock	0.9560	10.4	Hard black semi-shiny wavy grainy deposit

TABLE A-4

LUBRICANT COKING PROPENSITY TEST DATA
210°C TEST TEMPERATURE

Test Lubricant	Type Test Dish	Avg/Test Cycle (mg)	DEPOSIT MEASUREMENT Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] mg/gm oil	Residue[d] mg/gm oil
0-79-16	Polished Aluminum	0.3	2.7	2.7	5.4	5.4	0.7	0.7
0-79-17	"	Lubricant crept out of test dish onto dish holder.						
0-79-20	"	0.9	8.6	9.2	17.2	18.4	2.1	2.2
0-82-2	"	Lubricant crept out of test dish onto dish holder.						
0-82-3	"	Lubricant crept out of test dish onto dish holder.						
0-82-14	"	1.3	12.9	13.5	25.8	27.0	3.1	3.2
0-79-16	Stainless Steel	0.4	3.8	2.9	7.6	5.8	0.9	0.7
0-79-17	"	Lubricant crept out of test dish onto dish holder.						
0-79-20	"	0.9	8.6	9.9	17.2	19.8	2.1	2.4
0-82-2	"	Lubricant crept out of test dish onto dish holder.						
0-83-3	"	Lubricant crept out of test dish onto dish holder.						
0-82-14	"	1.5	15.4	16.5	30.8	33.0	3.7	4.0

[a]Mean Value

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
235°C TEST TEMPERATURE

Test Lubricant	Test Test Dish	Avg /Test Cycle (mg)	DEPOSIT MEASUREMENTS Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] mg/gm oil	Residue[d] mg/gm oil
0-79-16	Sandblasted Aluminum	0.3	2.5	0.0	5.0	0.0	0.6	0.0
0-79-17	"	0.4	3.6	1.5	7.2	3.0	0.8	0.3
0-79-20	"	Oil not tested.						
0-82-2	"	Oil crept from test dish. Test discontinued						
0-82-3	"	Oil crept from test dish. Test discontinued						
0-82-14	"	0.3	3.1	0.8	6.2	1.6	0.7	0.2

[a]Mean Value X 10

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
235°C TEST TEMPERATURE, TEST NO. 1

Test Lubricant	Test Test Dish	Avg/Test Cycle (mg)	DEPOSIT MEASUREMENTS Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] mg/gm oil	Residue[d] mg/gm oil
0-79-16	Polished Aluminum	0.8	8.3	7.4	16.6	14.8	1.9	1.7
0-79-17	"	0.7	6.6	5.7	13.2	11.4	1.6	1.4
0-79-20	"	0.5	4.6	4.0	9.2	8.0	1.1	0.9
0-82-2	"	1.0	10.3	10.6	20.6	21.2	2.4	2.4
0-82-3	"	0.9	9.4	9.7	18.8	19.4	2.2	2.2
0-82-14	"	0.6	6.4	6.1	12.8	12.2	1.6	1.5
0-79-16	Stainless Steel	0.9	9.1	8.1	18.2	16.2	2.2	2.0
0-79-17	"	0.8	8.3	8.2	16.6	16.4	2.0	2.0
0-79-20	"	0.8	7.6	6.9	15.2	13.8	1.8	1.6
0-82-2	"	1.5	14.7	14.6	29.4	29.2	3.5	3.5
0-82-3	"	0.8	8.3	8.4	16.6	16.8	2.0	2.1
0-82-14	"	0.7	7.3	7.7	14.6	15.4	1.8	1.9

[a]Mean Value X 10

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
235°C TEST TEMPERATURE, TEST NO. 2

Test Lubricant	Type Test Dish	Avg/Test Cycle (mg)	DEPOSIT MEASUREMENTS Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] mg/gm oil	Residue[d] mg/gm oil
0-79-16	Polished Aluminum	0.4	3.9	3.4	7.8	6.8	0.9	0.8
0-79-17	"	0.5	5.0	5.9	10.0	11.2	1.2	1.4
0-79-20	"	0.4	3.7	2.9	7.4	5.8	0.9	0.7
0-82-2	"	1.0	10.2	10.1	20.4	20.2	2.5	2.4
0-82-3	"	0.9	8.5	8.6	17.0	17.2	2.0	2.0
0-82-14	"	0.5	5.3	5.2	10.6	10.4	1.3	1.3
0-79-16	Stainless Steel	0.5	5.3	1.4	10.6	2.8	1.2	0.3
0-79-17	"	0.7	6.9	5.2	13.8	10.4	1.7	1.2
0-79-20	"	0.4	3.6	2.4	7.2	4.8	0.9	0.6
0-82-2	"	1.1	11.1	11.6	22.2	23.2	2.8	2.9
0-82-3	"	0.7	7.4	8.3	14.8	16.6	1.9	2.1
0-82-14	"	0.6	6.3	6.6	12.6	13.2	1.5	1.6

[a]Mean Value X 10

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
235°C TEST TEMPERATURE

Lubricant	Type Test Dish	Avg Test Cycle (mg)	DEPOSIT MEASUREMENTS Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] (mg/g oil)	Residue[d] (mg/g oil)
O-71-6	Stainless Steel	7.2	72.2	72.2	144.4	144.4	15.6	15.6
O-77-15	"	5.7	56.6	56.6	113.2	113.2	12.2	12.2
O-79-18	"	8.1	80.6	80.6	161.2	161.2	16.7	16.7
O-85-1	"	5.8	58.5	58.5	117.0	117.0	12.6	12.6
TEL 6031	"	4.7	47.0	47.0	94.0	94.0	9.4	9.4
TEL 6032	"	1.7	16.6	16.5	33.2	33.0	3.5	3.4

[a]Mean Value

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
260°C TEST TEMPERATURE, TEST NO. 1

DEPOSIT MEASUREMENTS

Test Lubricant	Type Test Dish	Avg/Test Cycle (mg)	Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] (mg/gm oil)	Residue[d] (mg/gm oil)
0-79-16	Polished Aluminum	0.7	7.2	7.1	14.4	14.2	1.7	1.7
0-79-17	"	0.3	3.2	3.0	6.4	6.0	0.8	0.7
0-79-20	"	0.7	6.6	6.1	13.2	12.2	1.5	1.4
0-82-2	"	0.7	7.1	7.2	14.2	14.4	1.7	1.7
0-82-3	"	0.2	2.2	1.5	4.4	3.0	0.5	0.4
0-82-14	"	0.2	2.4	1.9	4.8	3.8	0.6	0.5
0-79-16	Sand Blasted Aluminum	0.4	3.8	2.6	7.6	5.2	0.9	0.6
0-79-17	"	0.1	1.0	1.0	2.0	2.0	0.3	0.3
0-79-20	"	0.1	1.0	0.2	1.0	0.4	0.1	0.1
0-82-2	"	0.2	1.7	1.7	3.4	3.4	0.4	0.4
0-82-14	"	0.2	1.8	0.7	3.6	1.4	0.4	0.2

[a] Mean Value X 10

[b] Final test dish weight minus initial weight for 10 cycles

[c] Calculated from the mean for 10 cycles

[d] Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
260°C TEST TEMPERATURE, TEST NO. 2

Test Lubricant	Type Test Dish	Avg/Test Cycle (mg)	Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] (mg/gm oil)	Residue[d] (mg/gm oil)
			DEPOSIT MEASUREMENTS					
0-79-16	Polished Aluminum	0.7	6.9	5.2	13.8	10.2	1.6	1.2
0-79-17	"	0.4	4.5	2.1	9.0	4.2	1.1	0.5
0-79-20	"	0.7	6.5	5.8	13.0	11.6	1.6	1.4
0-82-2	"	0.6	6.3	4.8	12.6	9.6	1.5	1.2
0-82-3	"	0.3	3.1	0.0	6.2	0.0	0.7	0.0
0-82-14	"	0.3	3.3	1.0	6.6	2.0	0.8	0.2
0-79-16	Stainless Steel	0.5	4.8	3.6	9.6	7.2	1.2	0.9
0-79-17	"	0.4	3.9	(-0.1)	7.8	(-0.2)	0.9	0.0
0-79-20	"	0.7	7.0	5.8	14.0	11.6	1.7	1.4
0-82-2	"	0.5	5.4	1.8	10.8	3.6	1.4	0.5
0-83-3	"	0.4	4.4	(-0.4)	8.8	(-0.8)	1.0	0.0
0-82-14	"	0.5	4.5	0.7	9.0	1.4	1.0	0.2

[a] Mean Value X 10

[b] Final test dish weight minus initial weight for 10 cycles

[c] Calculated from the mean for 10 cycles

[d] Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
260°C TEST TEMPERATURE

Test Lubricant	Type Test Dish	Avg/Test Cycle (mg)	DEPOSIT MEASUREMENTS Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] (mg/gm oil)	Residue[d] (mg/gm oil)
O-71-6	Aluminum	5.0	50.4	48.1	100.8	96.2	12.4	11.8
O-77-15 (TEL 6021)	"	4.2	42.3	42.3	84.6	84.6	10.3	10.3
O-79-18 (TEL 6022)	"	6.2	62.3	62.3	124.6	124.6	14.9	14.9
TEL-6032	"	1.9	19.1	19.0	38.2	38.0	4.4	4.4
TEL-6031	"	4.7	46.6	46.6	93.2	93.2	10.5	10.5
O-71-6	Stainless Steel	5.7	56.7	56.6	113.4	113.2	14.0	13.9
O-77-15 (TEL 6021)	"	6.3	63.3	63.3	126.6	126.6	15.2	15.2
O-79-18 (TEL 6022)	"	6.8	68.1	68.1	136.2	136.2	15.8	15.8
TEL-6032	"	2.5	25.2	22.7	50.4	45.4	5.9	5.3
TEL-6031	"	4.8	48.5	48.1	97.0	96.2	10.2	10.1
O-85-1		4.1	40.8	41.1	81.6	82.2	8.6	8.7

[a]Mean Value

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
300°C TEST TEMPERATURE

Test Lubricant	Type Test Dish	DEPOSIT MEASUREMENTS Avg/Test Cycle (mg)	Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] (mg/g oil)	Residue[d] (mg/g oil)
0-71-6	Aluminum	4.1	40.7	40.7	81.4	81.4	9.6	9.6
0-85-1	"	2.5	25.1	25.1	50.2	50.2	5.9	5.9
0-77-15	"	3.2	31.9	33.7	63.8	67.4	7.5	7.9
0-79-18	"	4.1	40.8	37.8	81.6	75.6	9.7	9.0
TEL 6031	"	2.7	27.3	27.3	54.6	54.6	6.2	6.2
TEL 6032	"	1.3	12.5	12.1	25.0	24.2	2.9	2.8
0-71-6	Stainless Steel	4.8	47.6	47.6	95.2	95.2	11.1	11.1
0-85-1	"	2.7	27.4	27.4	54.8	54.8	6.2	6.2
0-77-15	"	3.9	39.2	39.2	78.4	78.4	9.1	9.1
0-79-18	"	4.5	44.7	44.7	89.4	89.4	10.1	10.1
TEL 6031	"	2.9	29.4	29.4	58.8	58.8	6.3	6.3
TEL-6032	"	1.4	14.0	13.3	28.0	26.6	3.2	3.0

[a]Mean Value

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

LUBRICANT COKING PROPENSITY TEST DATA
300°C TEST TEMPERATURE

Test Lubricant	Type Test Dish	Avg Test Cycle (mg)	DEPOSIT MEASUREMENTS Total/10[a] Cycles (mg)	Total/10[b] Cycles (mg)	Total/20[c] Cycles (mg)	Total/20[d] Cycles (mg)	Residue[c] (mg/g oil)	Residue[d] (mg/g oil)
0-71-6	Sandblasted Aluminum	0.5	5.1	4.8	10.2	9.6	0.9	0.9
0-77-15	"	0.5	4.8	3.6	9.6	7.2	1.0	0.8
0-79-18	"	1.5	15.2	15.2	30.4	30.4	3.1	3.1
0-85-1	"	0.9	9.0	9.0	18.0	18.0	1.8	1.8
TEL-6031	"	1.1	10.7	10.3	21.4	20.6	2.0	2.0
TEL-6032	"	0.4	4.1	2.8	8.2	5.6	0.8	0.6

[a]Mean Value

[b]Final test dish weight minus initial weight for 10 cycles

[c]Calculated from the mean for 10 cycles

[d]Calculated from final & initial weight for 10 cycles

TABLE A-5

DESCRIPTION OF COKING PROPENSITY TEST DEPOSITS

ALUMINUM TEST DISH

MIL-L-7808 Type Fluids

O-79-16	(210°C Test)	Hard dark brown deposit on rim of test dish
	(235°C Test)	Hard dark brown deposit, nonuniform coverage
	(260°C Test)	Hard dark brown to black deposit, nonuniform coverage
O-79-17	(210°C Test)	Oil crept from dish. Test discontinued
	(235°C Test)	Hard brown to black deposits, nonuniform coverage
	(260°C Test)	Hard brown to black deposits, nonuniform coverage
O-79-20	(210°C Test)	Spots of sticky brown varnish
	(235°C Test)	Light brown uniform varnish
	(260°C Test)	Hard brown to black deposits, nonuniform coverage
O-82-2	(210°C Test)	Oil crept from dish. Test discontinued
	(235°C Test)	Sticky light to dark brown varnish, nonuniform coverage
	(260°C Test)	Few areas of hard dark brown deposits, slight varnish over remaining test area
O-82-3	(210°C Test)	Oil crept from dish. Test discontinued
	(235°C Test)	Sticky light to dark brown varnish, nonuniform coverage
	(260°C Test)	Light brown uniform stain, and a few black deposits
O-82-14	(210°C Test)	Hard dark brown deposit and brown stain
	(235°C Test)	Hard dark brown uniform varnish
	(260°C Test)	Uniform brown varnish deposit

MIL-L-23699 Type Fluids

O-71-6	(260°C Test)	Hard, glossy, very smooth, dark brown uniform deposit
	(300°C Test)	Hard black smooth deposit, peeling in center and slight amount of hard dark brown deposit creeping up test cup lip
O-77-15	(260°C Test)	Hard, glossy, very smooth uniform black deposit with brownish coke on lip
	(300°C Test)	Hard, black, cracked brittle deposit with hard brown flaky deposit on cup lip

O-79-18	(260°C Test)	Hard, glossy, flaky, reddish brown uniform deposit
	(300°C Test)	Hard, brown to black, wrinkled, flaky deposit

4 cSt Fluid

O-85-1	(300°C Test)	Hard, brittle, flaky brown to black deposit, slight stain on cup lip

7.5 cSt Fluids

TEL-6031	(260°C Test)	Hard, flaky, dark brown deposit with light brown stain
	(300°C Test)	Hard, dark brown, very flaky cracked deposit
TEL-6032	(260°C Test)	Hard, dark brown, slightly flaky uniform deposit.
	(300°C Test)	Hard, dark brown, semi-smooth, slightly flaky deposit

STAINLESS STEEL DISH

MIL-L-7808 Fluids

O-79-16	(210°C Test)	Light brown nonuniform varnish
	(235°C Test)	Hard dark brown varnish and deposit, nonuniform coverage
	(260°C Test)	Hard, flaky, dark brown deposit, nonuniform coverage
O-79-17	(210°C Test)	Oil crept from dish. Test discontinued
	(235°C Test)	Hard dark brown varnish, uniform light brown stain
	(260°C Test)	Brown stain, dark brown deposits in edge of test dish
O-79-20	(210°C Test)	Spots of sticky brown varnish
	(235°C Test)	Hard dark brown varnish, uniform light brown stain
	(260°C Test)	Hard, dark brown, nonuniform deposit
O-82-2	(210°C Test)	Oil crept from dish. Test discontinued
	(235°C Test)	Sticky, light to dark brown varnish, nonuniform coverage
	(260°C Test)	Hard, dark brown, flaky nonuniform deposit

O-82-3	(210°C Test)	Oil crept from dish. Test discontinued
	(235°C Test)	Sticky, dark brown varnish, light brown stain, nonuniform coverage
	(260°C Test)	Uniform light brown varnish deposit
O-82-14	(210°C Test)	Heavy hard brown flaky deposit and light brown stain
	(235°C Test)	Hard dark brown stain/varnish, uniform coverage
	(260°C Test)	Uniform brown varnish deposit

MIL-L-23699 Type Fluids

O-71-6	(235°C Test)	Hard brown to black, flaky, nonuniform deposit
	(260°C Test)	Hard, black, very flaky deposit with lighter brown smooth deposits
	(300°C Test)	Hard, black deposit, very flaky and cracked, with slight hard deposit on cup lip
O-77-15	(235°C Test)	Hard brown to black, flaky, nonuniform deposit
	(260°C Test)	Hard, flaky, black deposit with smooth dark brown deposit extending over lip of test cup
	(300°C Test)	Hard, black, very flaky and cracked deposit with some hard deposit on cup lip
O-79-18	(235°C Test)	Hard brown to black uniform deposit
	(260°C Test)	Hard, dark brown, flaky uniform deposit
	(300°C Test)	Hard, brown to black deposit, with areas of thin, flexible, film-like peeling deposits

4 cSt Fluid

O-85-1	(235°C Test)	Hard brown to black uniform deposit extending over lip of test dish
	(260°C Test)	Hard, dark brown, flaky uniform deposit
	(300°C Test)	Hard light to dark brown deposit, very flaky with areas of flexible, thin, film-like deposits peeling up and away from test cup surface

7.5 cSt Fluids

TEL-6031	(235°C Test)	Hard brown to black, uniform, slightly flaky deposit
	(260°C Test)	Hard, black, uniform deposit with soft flaky areas

TEL-6032	(235°C Test)	Hard brown to black uniform slightly flaky deposit.
	(260°C Test)	Hard, black, slightly flaky uniform deposit
	(300°C Test)	Hard dark brown, semi-smooth flaky deposit

SANDBLASTED ALUMINUM TEST DISH

MIL-L-7808 Type Fluids

O-79-16	(235°C Test)	No visible deposits
	(260°C Test)	Small amount of brown to black residue over test area
O-79-17	(235°C Test)	No visible deposits
	(260°C Test)	Light to dark brown stain over part of test area
O-79-20	(235°C Test)	Not tested
	(260°C Test)	Hard brown to black deposit, nonuniform coverage
O-82-2	(235°C Test)	Oil crept from dish. Test discontinued
	(260°C Test)	No visual deposits on staining
O-82-3	(235°C Test)	Oil crept from dish. Test discontinued
	(260°C Test)	Not tested
O-82-14	(235°C Test)	No visible deposits. Stain on lip of test dish
	(260°C Test)	Light brown stain over test area

MIL-L-23699

O-71-6	(300°C Test)	Dark brown to black nonuniform deposit on test surface and cup rim
O-77-15	(300°C Test)	Slight coke on test surface with hard black heavier deposit on rim of cup
O-79-18	(300°C Test)	Dark brown uniform deposit with brown deposit on rim of cup

4 cSt Fluid

O-85-1	(300°C Test)	Brown uniform deposit with heavier coke on test cup rim

7.5 cSt Fluids

TEL-6031 (300°C Test)	Uniform dark brown deposit
TEL-6032 (300°C Test)	Slight brown deposit

TABLE A-6

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
1	O-67-21D	ASTM # 1A	500	200	25[a] (245)[b]	525[a] (40)[b]	37[a] (44)[b]
2	O-71-10	ASTM # 1A	500	200	-	overfm 1 min	-
3	QUAL 5K3-L6	ASTM # 1A	500	200	25 (245)	450 (25)	52 (43)
4	QUAL 5K3-L6	ASTM # 1A	500	134	20 (160)	295 (30)	21 (20)
5	QUAL 5K3-L6	ASTM # 1A	500	67	20 (80)	180 (35)	24 (30)
6	QUAL 5K3-L6	ASTM # 1A	500	36	15 (35)	120 (40)	11 (12)
7	QUAL 5K3-L6	ASTM # 1A	500	20	15 (20)	75 (35)	13 (16)
8	QUAL 5K3-L6	ASTM # 6A	500	200	30 (240)	470 (30)	31 (39)
9	QUAL 5K3-L6	1/4" Sparger (2 micron)	500	200	220 (225)	85 (25)	37 (32)
10	QUAL 5K3-L6	3/8" Sparger #1 (10 micron)	500	200	225 (225)	85 (15)	11 (8)
11	QUAL 5K3-L6	3/8" Sparger #2 (10 micron)	500	200	225 (225)	85 (20)	33 (24)
12	QUAL 5K3-L6	1/2" Sparger #1 (40 micron)	500	200	225 (220)	80 (20)	36 (26)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
13	QUAL 5K3-L6	1/2 " Sparger, #2 (40 micron)	500	200	220[a] (220)[b]	80[a] (15)[b]	36[a] (36)[b]
14	QUAL 5K3-L6	13/16 " Sparger (5 micron)	500	200	35 (245)	455 (25)	25 (26)
15	QUAL 5K3-L6	Glass Frit #1 (Cylinder) (40 to 60 micron nominal)	500	200	230 (230)	130 (30)	32 (20)
16	QUAL 5K3-L6	Glass Frit #2 (Cylinder) (40 to 60 micron nominal)	500	200	225 (230)	105 (25)	46 (22)
17	QUAL 5K3-L6	Glass Frit Sphere (1" Diam.) (Nominal Porosity unknown)	500	200	235 (230)	65 (15)	39 (28)
18	QUAL 5K3-L6	Two 1/2 Spargers (40 micron) (500 cc/min airflow/sparger)	500	200	235	95	31
19	QUAL 5K3-L6	2 Glass Frits (Cylinder) (500 cc/min airflow/sparger)	500	200	40	435	37
20	QUAL 5K3-L6	Two 3/8 Spargers (500 cc/min airflow/sparger) (10 micron)	500	200	235	135	38
21	QUAL 5K3-L6	ASTM #1A	1000	200	230	70	15
22	QUAL 5K3-L6	2 ASTM Stones (1A&2A) (500 cc/min airflow/stone)	1000	200	240	100	12
23	QUAL 5K3-L6	1/2" Sparger #3 (2 micron)	500	200	40	450 (30)	45 (28)
24	QUAL 5K3-L6	1/2" Sparger #3 (2 micron)	250	25	12 (20)	115 (50)	25 (40)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
25	O-79-17E	ASTM #1A	500	200	255[a] (235)[b]	15[a] (5)[b]	5[a] (4)[b]
26	O-79-17E	ASTM #1A	250	25	40 (40)	10 (5)	1 (2)
27	O-79-17E	1/2" Sparger (2 micron)	250	25	25 (25)	20 (10)	1 (1)
28	O-79-17E	1/4 " Sparger (2 micron)	250	25	25 (25)	10 (5)	1 (1)
29	5K3-L6	1/4" Sparger (2 micron)	250	25	15 (20)	75 (40)	7 (25)
30	5K3-L6	ASTM #1A	250	25	8 (10)	104 (64)	10 (15)
31	5K3-L6	Glass Frit #2 (40-60 microns)	500	200	235 (235)	100 (25)	25 (25)
32	5K3-L6	13/16" Sparger (5 micron)	250	25	8 (12)	120 (72)	14 (10)
33	5K3-L6	3/8" Sparger #1 (10 micron)	250	25	12 (18)	78 (40)	20 (8)
34	O-79-17 (3 ppm DC-200)	ASTM #1A	500	200	215 (225)	195 (80)	24 (18)
35	O-79-17 (3 ppm DC-200)	ASTM #1A	500	100	105 (115)	105 (35)	11 (11)
36	O-79-17 (3 ppm DC-200)	ASTM #1A	500	50	55 (65)	55 (15)	7 (2)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
37	O-79-17 (3 ppm DC-200)	ASTM #1A	250	25	10[a] (34)[b]	80[a] (12)[b]	8[a] (7)[b]
38	O-79-17 (3 ppm DC-200)	1/2" Sparger #3 (2 micron)	250	25	18 (24)	24 (10)	5 (4)
39	O-79-17 (3 ppm DC-200)	13/16" Sparger #1 (5 micron)	250	25	10 (22)	94 (38)	40 (19)
40	5K3-L6	Glass Frit #2 (40-60 microns)	250	25	16 (20)	72 (38)	10 (20)
41	O-79-17 (3 ppm DC-200)	1/2" Sparger #3 (2 micron)	250	25	20 (26)	30 (15)	18 (25)
42	O-79-17 (3 ppm DC-200)	ASTM #1A	250	25	10 (34)	74 (18)	25 (6)
43	O-79-17 (3 ppm DC-200)	1/2" Sparger #3 (2 micron)	500	200	200 (215)	200 (40)	22 (14)
44	O-79-17E (3 ppm DC-200)	ASTM 1A	500	200	215	225	49
45	O-79-17E (3 ppm DC-200)	ASTM 1A	500	200	220	225	30
46	O-79-17 (3 ppm DC-200)	ASTM 1A	500	200	210	215	43
47	5K3-L6	ASTM 1A	500	200	20	425	21
48	O-79-17 (3 ppm DC-200)	ASTM 1A	500	200	220	195	20

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
49	O-79-17 (3 ppm DC-200)	ASTM 1A	500	200	220[a] (-)[b]	230[a] (-)[b]	29[a] (-)[b]
50	5K3-L6	1/2" Sparger #15 (2 micron)	250	25	12 (22)	102 (36)	17 (12)
51	5K3-L6	1/2" Sparger #14 (5 micron)	250	25	12 (20)	104 (40)	21 (30)
52	5K3-L6	1/2" Sparger #13 (10 micron)	250	25	10 (16)	108 (52)	17 (18)
53	5K3-L6	1/2" Sparger #12 (20 micron)	250	25	16 (22)	66 (26)	21 (27)
54	5K3-L6	1/2" Sparger #1 (40 micron)	250	25	14 (24)	56 (24)	41 (15)
55	O-79-17 (3 ppm DC-200)	1/2"Sparger # 15 (2 micron)	250	25	16 (26)	46 (14)	49 (12)
56	O-79-17 (3 ppm DC-200)	1/2" Sparger # 14 (5 micron)	250	25	24 (28)	16 (12)	10 (8)
57	O-79-17 (3ppm DC-200)	1/2" Sparger #13 (10 micron)	250	25	22 (30)	20 (8)	29 (8)
58	O-79-17 (3ppm DC-200)	1/2" Sparger # 12 (20 micron)	250	25	22 (29)	16 (7)	11 (8)
59	O-79-17 (3ppm DC-200)	1/2" Sparger # 1 (40 micron)	250	25	18 (30)	20 (6)	36 (26)
60	5K3L6	1/2" Sparger #14 (5 micron)	TUBE A	25	5	105	28

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
61	5K3L6	1/2" Sparger #14 (5 micron)	TUBE B	25	Excess aerat.[a] [b]	-[a] -[b]	-[a] -[b]
62	5K3L6	1/2" Sparger #15 (2 micron)	500	200	45 (230)	400 35	41 (43)
63	5K3L6	1/2" Sparger # 14 (5 micron)	500	200	55 (225)	330 (35)	49 (55)
64	5K3L6	1/2" Sparger #13 (10 micron)	500	200	225 (225)	105 (35)	38 (37)
65	5K3L6	1/2" Sparger #12 (20 micron)	500	200	230 (225)	60 (20)	33 (33)
66	5K3L6	1/2" Sparger # 12 (20 micron)	500	200	230 (225)	75 (25)	42 (35)
67	0-76-5 2% TCP 2% PANA	ASTM Stone 1A	500	200	25 (245)	460 (30)	19 (12)
68	0-76-5 2% TCP 2% PANA	1/2" Sparger # 15 (2 micron)	500	200	30 (195)	570+ (75)	35 (25)
69	0-76-5 2% TCP 2% PANA	1/2" Sparger # 14 (5 micron)	500	200	45 (235)	360 (35)	16 (15)
70	0-76-5 2% TCP 2% PANA	1/2" Sparger # 13 (10 micron)	500	200	220 (225)	105 (30)	12 (12)
71	0-76-5 2% TCP 2% PANA	1/2" Sparger #12 (20 micron)	500	200	230 (225)	45 15	10 (9)
72	0-76-5 2% TCP 2% PANA	1/2" Sparger (40 micron)	500	200	230 (220)	45 (20)	8 (6)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
73	0-76-5 2% TCP 2% PANA	ASTM Stone 1A	250	25	6[a] (12)[b]	140[a] (74)[b]	16[a] (15)[b]
74	0-76-5 2% TCP 2% PANA	1/2" Sparger #15 (2 micron)	250	25	12 (18)	92 (50)	11 (12)
75	0-76-5 2% TCP 2% PANA	1/2" Sparger #14 (5 micron)	250	25	14 (20)	80 (44)	11 (9)
76	0-76-5 2% TCP 2% PANA	1/2" Sparger #13 (10 micron)	250	25	10 (14)	88 (62)	11 (14)
77	0-76-5 2% TCP 2% PANA	1/2" Sparger # 12 (20 Micron)	250	25	18 (22)	58 (36)	11 (10)
78	5K3L6	1/2" Sparger (40 micron)	500	200	225 (220)	75 (20)	36 (42)
79	0-79-17 (3 ppm DC-200)	1/2" Sparger #15 (2 micron)	500	200	205 (215)	160 (40)	24 (19)
80	0-79-17 (3ppm DC-200)	1/2" Sparger #15 (5 micron)	500	200	220 (215)	65 (30)	50 (18)
81	0-79-17 (3ppm DC-200)	1/2" Sparger # 13 (10 micron)	500	200	225 (220)	60 (25)	20 (17)
82	0-79-17 (3ppm DC-200)	1/2" Sparger # 12 (20 micron)	500	200	225 (220)	25 (20)	23 (15)
83	0-79-17 (3ppm DC-200)	1/2 Sparger # 1 (40 micron)	500	200	225 (215)	20 (20)	10 (13)
84	0-76-5 2% TCP 2% PANA	1/2" Sparger # 1 (40 micron)	200	25	18 (26)	78 (30)	10 (9)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
85	O-79-17 (3ppm DC-200)	1/2" Sparger (5 micron)	200	25	22[a] (26)[b]	20[a] (10)[b]	10[a] (10)[b]
86	O-76-5 2% TCP 2% PANA	1/2" Sparger (20 Micron)	200	25	16 (27)	78 (40)	15 (13)
87	O-76-8 (3ppm DC-200-20)	ASTM 1A	500	200	225 (215)	10 (10)	-
88	O-76-8 (3ppm DC 200-20)	1/2" Sparger # 14 (5 micron)	250	25	26	12 (6)	-
89	O-76-8 (3ppm DC-200-20)	1/2" Sparger #14 (5 micron)	500	200	215	5 (5)	-
90	O-76-8 (6ppm DC-200-20)	ASTM 1A	500	200	230	10 (10)	-
91	O-76-1 (3ppm DC-200-20)	ASTM 1A	500	200	220	135 (60)	52 (50)
92	O-76-1 (3ppm DC-200-20)	1/2" Sparger # 14 (5 micron)	500	200	225	15 (10)	29 (34)
93	O-76-1 (3ppm DC-200-20)	ASTM 1A	250	25	38	12 (8)	35 (33)
94	O-76-1 (3ppm DC-200-20)	1/2" Sparger # 14 (5 micron)	250	25	24	16 (10)	6 (8)
95	O-76-1 (3ppm DC-200-20)	13/16" Sparger (5 micron)	500	200	235	20 (15)	27 (40)
96	O-76-1 (6ppm DC-200-20)	ASTM 1A	500	200	185	330 (115)	50 (50)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
97	O-76-1 (6ppm DC-200-20)	13/16" Sparger (5 micron)	500	200	190[a] (200)[b]	245[a] (125)[b]	60[a] (60)[b]
98	O-76-1 (3ppm DC-200-20)	13/16" Sparger (5 micron)	500	200	230	75 (25)	45 (48)
99	O-76-1 (3ppm DC-200-20)	13/16" Sparger (5 micron)	250	25	49 (48)	31 (12)	- (-)
100	O-79-17	Sealed Frit Tube A (4 to 8 microns)	250	25	4 (10)	68 (32)	17 (-)
101	O-79-17 3ppm DC-200-500cs	Sealed Frit Tube A (4 to 8 microns)	250	25	2 (24)	94 (26)	24 (27)
102	5K3L6	Sealed Frit Tube A (4 to 8 microns)	250	25	2 (4)	148 (76)	33 (-)
103	O-76-5 2% TCP 2% PANA	Sealed Frit Tube A (4 to 8 microns)	250	25	2 (4)	122 (76)	20 (22)
104	O-76-1 (3ppm DC-200-20cs)	13/16" Sparger (5 micron)	500	200	225 (220)	95 (28)	55 (63)
105	O-76-1 (6ppm DC-200-20cs)	13/16" Sparger (5 micron)	250	25	16 (28)	42 (16)	33 (66)
106	O-76-1 (6ppm DC-200-20cs)	ASTM 1A	250	25	10 (32)	52 (12)	37 (37)
107	O-76-1 (6ppm DC-200-20cs)	1/2 Sparger # 14 (5 micron)	250	25	24 (26)	18 (12)	49 (57)
108	O-76-1 (6ppm DC-200-20cs)	1/2" Sparger #14 (5 micron)	500	200	205 (205)	165 (80)	57 (62)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
109	O-79-17	Sealed Frit Tube B (4 to 8 micron)	250	25	8[a] (12)[b]	38[a] (26)[b]	18[a] (18)[b]
110	O-79-17 3 ppm DC-200-500	Sealed Frit Tube B (4 to 8 micron)	250	25	24	14	21
111	O-76-5 2% TCP 2% PANA	Sealed Frit Tube B (4 to 8 micron)	250	25	2 (4)	146 (82)	23 (27)
112	O-79-17	Sealed Firt Tube B (4 to 8 micron)	250	50	60 (54)	14 (8)	13 (15)
113	O-76-1 3 ppm DC-200-20 cs	ASTM 1A	500	200	225 (225)	160 (40)	56 (54)
114	O-76-1 3 ppm DC-200-20cs	13/16" Sparger (5 micron)	500	200	210 (220)	130 (25)	64 (61)
115	O-76-1 3 ppm DC-200-20cs	Two 1/2" Spargers (Each 5 micron)	500	200	- (220)	- (75)	- (49)
116	O-76-1 3 ppm DC-200-20cs	1/2" Sparger (5 micron)	500	200	225 (215)	15 (15)	47 (47)
117	O-76-8 3 ppm DC-200-20cs	Sealed Frit Tube A (4 to 8 micron)	250	25	30 (28)	10 (6)	31 (28)
118	O-76-1 6 ppm DC-200-20cs	Sealed Frit Tube A (4 to 8 micron)	250	25	10 (22)	88 (52)	74 (82)
119	O-76-1 3 ppm DC-200-20cs	Sealed Frit Tube A 4 to 8 micron)	250	25	30 (28)	22 (14)	60 (100)
120	O-76-8 3 ppm DC-200-20cs	ASTM 1A	250	25	36 (34)	10 (5)	- -

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
121	O-76-5 2% TCP 2% PANA	13/16" Sparger #1 (5 micron)	500	200	10[a] (250)[b]	> 500[a] (45)[b]	24[a] (20)[b]
122	O-76-5 2% TCP 2% PANA	13/16" Sparger #1 (5 micron)	250	25	16 (10)	116 (85)	13 (10)
123	5K3L6	3/8" Sparger #18 (2 micron)	500	200	50 (230)	385 (35)	38 (34)
124	5K3L6	11/16" Sparger #21 (2 micron)	500	200	45 (240)	430 (30)	34 (25)
125	5K3L6	3/8" Sparger #18 (2 micron)	250	25	108 (68)	96 (50)	14 (10)
126	5K3L6	11/16" Sparger #21 (2 micron)	250	25	126 (84)	118 (72)	25 (13)
127	O-76-8 3 ppm DC-200-20cs	13/16" Sparger #1 (5 micron	500	200	215 (215)	0 (0)	- -
128	O-76-8 3 ppm DC-200-20cs	13/16" Sparger #1 (5 micron)	250	25	34 (34)	0 (0)	- (-)
129	O-76-5 2% TCP 2% PANA	11/16" Sparger #23 (5 micron)	250	25	10 (12)	82 (64)	10 (10)
130	O-76-5 2% TCP 2% PANA	11/16" Sparger #23 (5 micron)	500	200	220 (235)	145 (30)	25 (13)
131	O-79-17E	Sealed Glass Frit (A)	250	25	5 (10)	45 (30)	10 (7)
132	O-79-17E 3ppm DC-200-500cs	Sealed Glass Frit (A)	250	25	120 (28)	118 (42)	28 (32)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
133	O-79-17E 3 ppm DC-200-500cs	1/4" Sparger #1 (2 micron)	250	25	20[a] (26)[b]	20[a] (10)[b]	54[a] (18)[b]
134	O-79-17E 3 ppm DC-200-500cs	1/4" Sparger #1 (2 micron)	500	200	220 (215)	55 (25)	35 (22)
135	O-79-17E 3 ppm DC-200-500cs	13/16" Sparger #1 (5 micron)	500	200	220 (225)	240 (80)	47 (41)
136	O-76-5 2% TCP 2% PANA	1/2" Sparger #14 (5 micron) 1/2" Sparger #20 (5 micron)	500	200	40 (250)	445 (40)	15 (12)
137	O-76-1 3 ppm DC-200-20cs	1/2" Sparger #14 (5 micron) 1/2" Sparger #20(5 micron)	500	200	- -	- -	- -
138	O-79-17 3 ppm DC-200-500cs	1/2" Sparger #14 (5 micron) 1/2" Sparger #20 (5 micron)	500	200	225 (225)	190 (110)	60 (58)
139	O-76-1 6 ppm DC-200-20cs	1/2" Sparger #14 (5 micron) 1/2" Sparger #20 (5 micron)	500	200	225 (205)	230 (150)	56 (64)
140	O-76-1 3 ppm DC-200-500cs	1/2" Sparger #21 (2 micron)	500	200	230 (220)	290 (135)	72 (77)
141	O-79-17 3 ppm DC-200-500cs	3/8" Sparger #18 (2 micron)	250	25	18 (24)	26 (16)	66 (90)
142	O-79-17 3 ppm DC-200-500cs	11/16" Sparger #21 (2 micron)	250	25	10 (28)	74 (18)	40 (24)
143	O-76-5 2% TCP 2% PANA	11\16" Sparger #21 (2 micron)	500	200	500 (245)	500 (35)	- (17)
144	O-76-5 2% TCP 2% PANA	11/16" Sparger # 21 (2 micron)	250	25	6 (10)	166 (100)	10 (11)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
145	O-76-5 2% TCP 2% PANA	1/4" Sparger # 1 (2 micron)	500	200	225[a] (220)[b]	65[a] (35)[b]	12[a] (11)[b]
146	O-76-5 2% TCP 2% PANA	1/4" Sparger # 1 (2 micron)	250	25	14 (16)	88 (58)	12 (11)
147	O-76-5 2% TCP 2% PANA	3/8" Sparger # 18 (2 (micron)	500	200	150 (230)	310 (35)	11 (15)
148	O-76-5 2% TCP 2% PANA	3/8" Sparger # 18 (2 micron)	250	25	10 (16)	116 (72)	14 (12)
149	O-79-17 3 ppm DC-200-500cs	13/16" Sparger # 1 (5 micron)	250	25	14 (26)	78 (40)	34 (9)
150	O-76-1 3 ppm DC-200-20cs	ASTM Stone 2A-1	250	25	36 (44)	14 (8)	41 (44)
151	O-76-1 6 ppm DC-200-20cs	ASTM Stone 2A-1	500	200	205 (210)	250 (140)	49 (47)
152	O-76-5 2% TCP 2% PANA	1/2" Sparger # 14 (5 micron) 1/2" Sparger # 20 (5 micron)	500	200	- (-)	overfm (-)	- (-)
153	O-76-1 3 ppm DC-200-20cs	1/2" Sparger # 14 (5 micron) 1/2" Sparger # 20 (5 micron)	500	200	250 (-)	105 (-)	45 (-)
154	O-79-17 3 ppm DC-200-500cs	11/16" Sparger #23 (5 micron)	250	25	34 (34)	22 (8)	23 (21)
155	5K3L6	11/16" Sparger #23 (5 micron)	250	25	8 (12)	146 (78)	20 (17)
156	5K3L6	11/16" Sparger #23 (5 micron)	500	200	140 (235)	345 (40)	68 (70)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
157	0-76-1 6 ppm DC-200-20cs	11/16" Sparger # 23 (5 micron)	250	25	34[a] (30)[b]	10[a] (8)[b]	38[a] (34)[b]
158	0-79-17 3 ppm DC-200-500cs	11/16" Sparger # 23 (5 micron)	500	200	220 (225)	130 (30)	70 (63)
159	5K3L6	3/8" Sparger # 19 (5 micron)	250	25	14 (20)	106 (52)	13 (11)
160	0-76-5 2% TCP 2% PANA	3/8" Sparger #1 (10 micron)	500	200	230 (225)	40 (20)	14 (14)
161	0-76-5 2% TCP 2% PANA	3/8" Sparger # 19 (5 micron)	250	25	14 (18)	94 (58)	12 (12)
162	0-76-5 2% TCP 2% PANA	3/8" Sparger # 19 (5 micron)	500	200	240 (230)	40 (20)	15 (14)
163	0-76-5 2% TCP 2% PANA	3/8" Sparger # 1 (10 micron)	250	25	12 (16)	96 (56)	13 (11)
164	0-79-17 3 ppm DC-200-500cs	3/8" Sparger # 19 (5 micron)	250	25	24 (26)	16 (12)	11 (11)
165	0-79-17 3 ppm DC-200-500cs	3/8" Sparger # 19 (5 micron)	500	200	230 (220)	70 (20)	67 (48)
166	0-76-1 6 ppm DC-200-20cs	1/2" Sparger # 14 (5 micron)	250	25	25 (26)	16 (12)	58 (46)
167	0-76-1 6 ppm DC-200-20cs	11/16" Sparger # 23 (5 micron)	500	200	230 (205)	270 (135)	60 (60)
168	5K3L6	3/8" Sparger # 19 (5 micron)	500	200	230 (225)	70 (35)	25 (22)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
169	0-82-2	ASTM 1A	500	200	245[a] (230)[b]	10[a] (10)[b]	5[a] (4)[b]
170	0-82-2	Sealed Frit Tube A	250	25	6 (28)	50 (14)	9 (7)
171	0-79-20A	ASTM 1A	500	200	250 (232)	15 (10)	6 (6)
172	0-79-20	Sealed Frit Tube A	250	25	6 (28)	70 (14)	11 (7)
173	0-79-16F	ASTM 1A	500	200	260 (235)	45 (20)	8 (7)
174	0-79-16F	Sealed Frit Tube A	250	25	2 (6)	142 (54)	15 (10)
175	0-82-3	ASTM 1A	500	200	240 (230)	10 (10)	4 (3)
176	0-82-3	Sealed Frit Tube A	250	25	35 (28)	15 (10)	10 (4)
177	0-79-17	ASTM 1A	500	200	255 (235)	10 (5)	5 (5)
178	0-79-17	Sealed Frit Tube A	250	25	6 (30)	46 (12)	9 (7)
179	0-76-8 6 ppm DC-200-20cs	Sealed Frit Tube A	250	25	28 (26)	16 (10)	7 (7)
180	0-82-14D	ASTM 1A	500	200	250 (235)	75 (20)	11 (11)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
181	O-82-14D	Sealed Frit Tube A	250	25	2[a] (6)[b]	188[a] (64)[b]	44[a] (42)[b]
182	5K3L6	11/16" Sparger # 23 (5 micron)	250	25	8 (16)	108 (52)	8 (5)
183	O-76-5 2% TCP 2% PANA	11/16" Sparger # 23 (5 micron)	250	25	6 (10)	124 (80)	14 (12)
184	O-76-1 6 ppm DC-200-500cs	11/16" Sparger # 23 (5 micron)	250	25	30 (30)	20 (10)	50 (49)
185	O-79-17E 3 ppm DC-200-500cs	11/16" Sparger # 23 (5 micron)	250	25	32 (32)	18 (8)	25 (19)
186	O-76-1 3 ppm DC-200-20cs	ASTM Stone 1A	250	25	38 (36)	14 (8)	35 (38)
187	O-76-1 3 ppm DC-200-20 cs	11/16" Sparger # 23 (5 micron)	500	200	235 (225)	15 (15)	39 (41)
188	O-76-5 2% TCP 2% PANA	11/16" Sparger #23 (5 micron)	500	200	220 (235)	155 (40)	20 (40)
189	O-76-1 6 ppm DC-200-20cs	13/16" Sparger #1 (5 micron)	500	200	185 (200)	340 (200)	52 (58)
190	O-76-1 6 ppm DC-200-20 cs	11/16" Sparger #23 (5 micron)	500	200	200 (205)	230 (115)	47 (53)
191	O-76-5 2% TCP 2% PANA	13/16" Sparger #1 (5 micron)	250	25	8 (12)	130 (88)	11 (11)
192	O-76-5 2% TCP 2% PANA	13/16" Sparger #1 (5 micron)	500	200	30 (250)	500 (35)	22 (15)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

LUBRICANT FOAMING TEST DATA

Test No	Test Lubricant	Type Air Diffuser	Test Cylinder Size (ml)	Sample Volume (ml)	Oil Volume at Maximum Foam Height (ml)	Foam Volume (ml)	Foam Collapse Time (sec)
193	O-76-1 6 ppm DC-200-20 cs	11/16" Sparger #24 (5 micron)	500	200	200[a] (205)[b]	225[a] (110)[b]	40[a] (50)[b]
194	O-76-1 6 ppm DC-200-20 cs	13/16" Sparger #1 (5 micron)	250	25	28 (32)	40 (18)	35 (39)
195	O-76-5 2% TCP 2% PANA	11/16" Sparger #23 (5 micron)	500	200	190 (230)	230 (35)	12 (8)
196	O-76-5 2% TCP 2% PANA	13/16" Sparger #1 (5 micron)	500	200	ovrfm (240)	ovrfm (40)	18 (9)
197	O-76-5 2% TCP 2% PANA	ASTM Stone 1A	500	200	20 (245)	500 (40)	17 (19)
198	O-76-5 2% TCP 2% PANA	11/16" Sparger #24 (5 micron)	500	200	180 (230)	250 (40)	13 (9)
199	O-76-1 3 ppm DC-200-20 cs	13/16" Sparger #1 (5 micron)	500	200	205 (-)	165 (-)	31 (-)
200	O-76-1 3 ppm DC-200-20 cs	11/16" Sparger #24 (5 micron)	500	200	235 (-)	15 (-)	40 (-)
201	O-76-5 2% TCP 2% PANA	11/16" Sparger #24 (5 micron)	200	25	8 (12)	124 (84)	7 (8)

a - Airflow of 1000 cc/minute
b - Airflow of 500 cc/minute

APPENDIX B

LUBRICANT ADDITIVE ANALYSIS BY GAS CHROMOTOGRAPHY WITH THERMIONIC SPECIFIC DETECTION

1. INTRODUCTION

Gas chromatography with thermionic specific detection is a sensitive and selective method for the detection of volatile nitrogen and phosphorous containing compounds. Specificities of 7×10^4 g C/g N and 10^5 g C/g P, linear responses of 10^4 to 10^5, and sensitivities at the picogram level have been reported [78,79]. This combination of selectivity and sensitivity makes this technique a very useful method for the analysis of nitrogen and phosphorous containing additives in lubricants while avoiding the basestock interferences that may plague other methods. A method is then described here for analysis of three antioxidants and tricresyl phosphate (TCP) in MIL-L-7808 lubricants using an internal standard method. The method can be used to quantitatively analyze these antioxidants in the range of 0.05 to 2.5% (and lower with minor modifications). Since TCP is a mixture of isomers and can vary in composition considerably from one supplier to the next, it is reported as weight fraction with a fresh sample containing, by definition, 1.00.

2. MATERIALS AND METHODS

a. Apparatus

A Varian 6000 gas chromatograph, fitted with a large bore capillary column adaptor kit (capillary mode) and a thermionic specific detector accessory, and a Vista 402 chromatography data system were used. The column was a 25 meter x 0.53 mm ID fused silica glass capillary column with a 0.25 micron film thickness of methylphenyl silicone (Quadrex Corp.). Injections were made on-column with a Hamilton #7001 1 μL syringe with a 26S gauge

needle.

b. Reagents

Three antioxidants, phenyl-1-naphthylamine (PANA), p-octylphenyl-1-naphthylamine (Octyl-PANA), and di(p-octylphenyl)amine (DODPA) were recrystallized in 70/30, 80/20, and 95/5 methanol/water, respectively, (the latter 3 times) and dried under 22 mm Hg vacuum at 50°C for 48 hours. Reagent grade diphenyl amine (Aldrich) was used without purification. Four tricresyl phosphate samples were obtained from the following suppliers: two from Stauffer Chemicals, one from FMC and one from Mobil. Di-2-ethylhexyl azelate was used as an unformulated ester basestock. Toluene was certified ACS from Fisher Scientific.

c. Calibration Solutions

The internal standard solution consisted of a 1% solution of diphenyl amine in the di-2-ethylhexyl azelate basestock, although any unformulated basestock could be used. Calibration solutions containing about 50 ng/0.1 µL and 200 ng/0.1 µL of each recrystallized antioxidant in toluene were made, and the former was used to calculate response factors for this method. Diluting the above solutions gave standard solutions of 200, 100, 5, and 1 ng/0.1 µL which were used for checking linearity, accuracy, and precision.

d. Gas Chromatographic Conditions

Flow Rates (ml/min)		Temperatures (^{o}C)	
Carrier Gas:	Helium	Injector :	170
Column :	3.0	Detector :	325
Makeup Gas :	27.0	Oven (Initial):	150
Hydrogen :	4.0 (20 mm Hg)	Oven (Final) :	300
Air :	175	Heating Rate :	$6^{o}C$/min
Detector :	Thermionic specific	Final Hold :	5.0 minutes
Attenuation:	64×10^{-12}		
Injection Volume :	0.1 µL		

The bead current to the detector should be adjusted to give a stable background level of about 5 mV.

e. Method Calibratrion

Calibration for antioxidant determination is made by analysis of a sample prepared from one drop of the internal standard solution (analytically weighed) and 1.0 mL of the 50 ng/0.1 µL calibration solution. Response factors for each antioxidant are calculated usirg the standard formula:

$$\text{Response Factor} = \frac{\text{(Area Int. Std. Peak)}}{\text{(Area Antioxidant Peak)}} X \frac{\text{(Wt. Antioxidant)}}{\text{(Wt. Int. Std. Soln.)}}$$

The results of the average of three runs are shown in the peak table of method TSD (Figure B-1). Also analyzed in this way were the 200, 100, 5, and 1 ng/0.1 µL solutions. Percent antioxidant can be determined by:

$$\% \text{ Antioxidant} = \frac{\text{(Resp Factor) X (Peak Area Antiox)}}{\text{(Peak Area Int. Std.)}} X \frac{\text{(Int Std Wt)}}{\text{(Antioxidant Wt.)}} X 100$$

The results of these analyses are shown in Table B-1.

Tricresyl phosphate (TCP) is not calibrated. However, a range of retention times (12 to 20 minutes) were obtained by analyzing the four different samples which were obtained from three different suppliers.

f. Sample Preparation

One drop each of the internal standard solution and lubricant sample are weighed analytically into a small vial and about 0.25 ml of toluene is added.

g. Sample Analysis

The Vista 402 data system is prepared using method TSD (Figure B-1). Also, method TSD1 (Figure B-2), which is linked to method TSD and is used for analysis of TCP, must exist in the system. The weight of the internal standard solution is entered at AMT STD and the weight of the lubricant is entered at DIVISOR. 100 is placed in the MULTIPLIER. Once the gas chromatograph has equillibrated (about 30 minutes after reaching temperature), a 0.1 μL injection is made.

h. Calculations

The TSD method will calculate and report the antioxidant concentration. The content and weight fraction of TCP must be calculated by method TSD1 which reports the total summed peak area for TCP from 12 to 20 minutes. But since the retention times of both DODPA and O-PANA fall in this range, their peak areas (from TSD report) must be subtracted from the summed TCP area of if these compounds are present. Once the adjusted summed peak area TCP is known, TCP content is calculated by:

$$\text{Content} = \frac{(\text{adjusted summed area TCP})}{(\text{peak area of Int. Std.})} \times \frac{(\text{Int. Std. Wt})}{(\text{Sample Wt.})}$$

and the weight fraction is then calculated by:

$$\text{Weight Fraction} = \frac{\text{(Content of Sample)}}{\text{(Content of Fresh Sample)}}$$

3. RESULTS AND DISCUSSION

The results of the calibration check are in Table B-1. The data shows that accuracy and precision are fairly good until the 1 ng sample. Using this method as it exists, the 1 ng sample represents about 0.01% antioxidant in the lubricant. So, values below about 0.05% may not be accurate. The lower range of course can be extended by increasing the total sample load on the column by either using more sample in the sample preparation, less toluene diluent, or a higher injection volume up to the point preceding column overload. In this way, percent antioxidant levels much less than 0.05 can be accurately determined if required.

There is some overlap of TCP with Octyl-PANA and DODPA, the degree of which is determined by the source of the TCP. A gas chromatograph capable of more complex thermal gradients, which the present system cannot do, would allow better separation of these compounds. A chromatogram of a typical analysis for O-79-16J lubricant is shown in Figure B-3. The TCP content from this sample together with two other replicates are shown in Table B-2. Precision is very good.

```
SINGLE CHANNEL METHOD: TSD


SECTION 1: BASIC
PAGE 1
  ANALYSIS PARAMETERS
  CHANNEL: 2
  CALCULATION: IS
  AREA/HT: A
  STOP TIME:  22.00
  NUMB EXPECTED PKS:   60
  EQUILIBRATION TIME:  0
  UNRETAINED PK TIME:   0.00
  UNIDENT PK FACTOR: 0.000000
  SLICE WIDTH:  10

PAGE 2
  SAMPLE PARAMETERS
  RUN TYPE: A
  SAMPLE ID: 0-79-16J
  DIVISOR: 1.000000
  AMT STD: 1.000000
  MLTPLR: 100.0000

PAGE 3
  REPORT INSTRUCTIONS
  WHERE TO REPORT: L
  COPIES: 1
  TITLE:
  FORMAT: N
  DECIMAL PLACE: 4
  RESULT UNITS:
  REPORT UNIDENT PKS: N
  REPORT INSTRUMENT CONDITIONS: N

PAGE 4
  PLOT INSTRUCTIONS
  PLOT: Y
  ZERO OFFSET:  5
  ANNOTATION
    RETENTION TIME: Y
    PLOT CONTROL: Y
    TIME TICKS: Y
    TIME EVENTS: Y
    PK START/END: Y

PAGE 5
  CHART SPEED
    PAGES OR CM/MIN: C
    INIT VALUE:  0.5

PAGE 6
  PLOT ATTEN
    INIT PLOT ATTEN: 64


SECTION 2: TIME EVENTS
PAGE 1
   LINE#  TIME  EVENT   VALUE
    1      0.00  PR       1000
    2      0.00  SN          5
    3      0.00  T%        5.0
    4      0.00  WI          3
    5      0.01  II       3.00
    6      0.10  WI          3
    7      4.00  II       8.50
    8      9.50  II      12.00
```

Figure B-1

Vista 402 Method TSD

```
SECTION 3: PEAK TABLE
PAGE 1
  STD PK#:  1
  RELATIVE RETEN PK#:  0
  RESOLUTION PK#:  0
  RESOLUTION MINIMUM:   0.0
  FACT%:   5.0
  IDENTIFICATION TIME WINDOWS +/-
  REF
    %: 10
    MIN:   0.00
  NON REF
    %:  5
    MIN:   0.00

PAGE 2
   PK# TIME  NAME         FACTOR   AMOUNT REF GR# MUST LO   MUST HI
    1    3.43 INT STD    1.000000 1.000000 R      0.000000 0.000000
    2    9.00 PANA       0.015470 1.000000        0.000000 0.000000
    3   17.22 O-PANA     0.026670 1.000000        0.000000 0.000000
    4   18.56 DODPA      0.029960 1.000000        0.000000 0.000000


SECTION 7: POST RUN
PAGE 1
  FILE NAME:
  SAVE INSTRUCTIONS
    TYPE:
    WHERE TO SAVE: M
  TRANSMIT/REPLOT INSTRUCTIONS
    TRANSMIT RAW DATA: N
    REPLOT WITH BASELINES: N
    RAW DATA LOCATION: U
    TRANSMIT REPORT: N

PAGE 2
  METHOD LINKING INSTRUCTIONS
    METHOD: TSD1
    LINK CALC RESULTS: N
  PROGRAM EXECUTION
    PROGRAM:
    PARAMETERS:
    RESERVE PRINTER: Y
```

```
SINGLE CHANNEL METHOD: TSD1


SECTION 1: BASIC
PAGE 1
  ANALYSIS PARAMETERS
  CHANNEL: 2
  CALCULATION: A%
  AREA/HT: A
  STOP TIME:  20.00
  NUMB EXPECTED PKS:   60
  EQUILIBRATION TIME:  0
  UNRETAINED PK TIME:   0.00
  UNIDENT PK FACTOR: 0.000000
  SLICE WIDTH:  10

PAGE 2
  SAMPLE PARAMETERS
  RUN TYPE: A
  SAMPLE ID:
  DIVISOR: 1.000000
  AMT STD: 1.000000
  MLTPLR: 1.000000

PAGE 3
  REPORT INSTRUCTIONS
  WHERE TO REPORT: L
  COPIES: 1
  TITLE:
  FORMAT: N
  DECIMAL PLACE: 4
  RESULT UNITS:
  REPORT UNIDENT PKS: N
  REPORT INSTRUMENT CONDITIONS: N

PAGE 4
  PLOT INSTRUCTIONS
  PLOT: Y
  ZERO OFFSET:  5
  ANNOTATION
    RETENTION TIME: Y
    PLOT CONTROL: Y
    TIME TICKS: Y
    TIME EVENTS: N
    PK START/END: Y

PAGE 5
  CHART SPEED
    PAGES OR CM/MIN: C
    INIT VALUE:  0.5

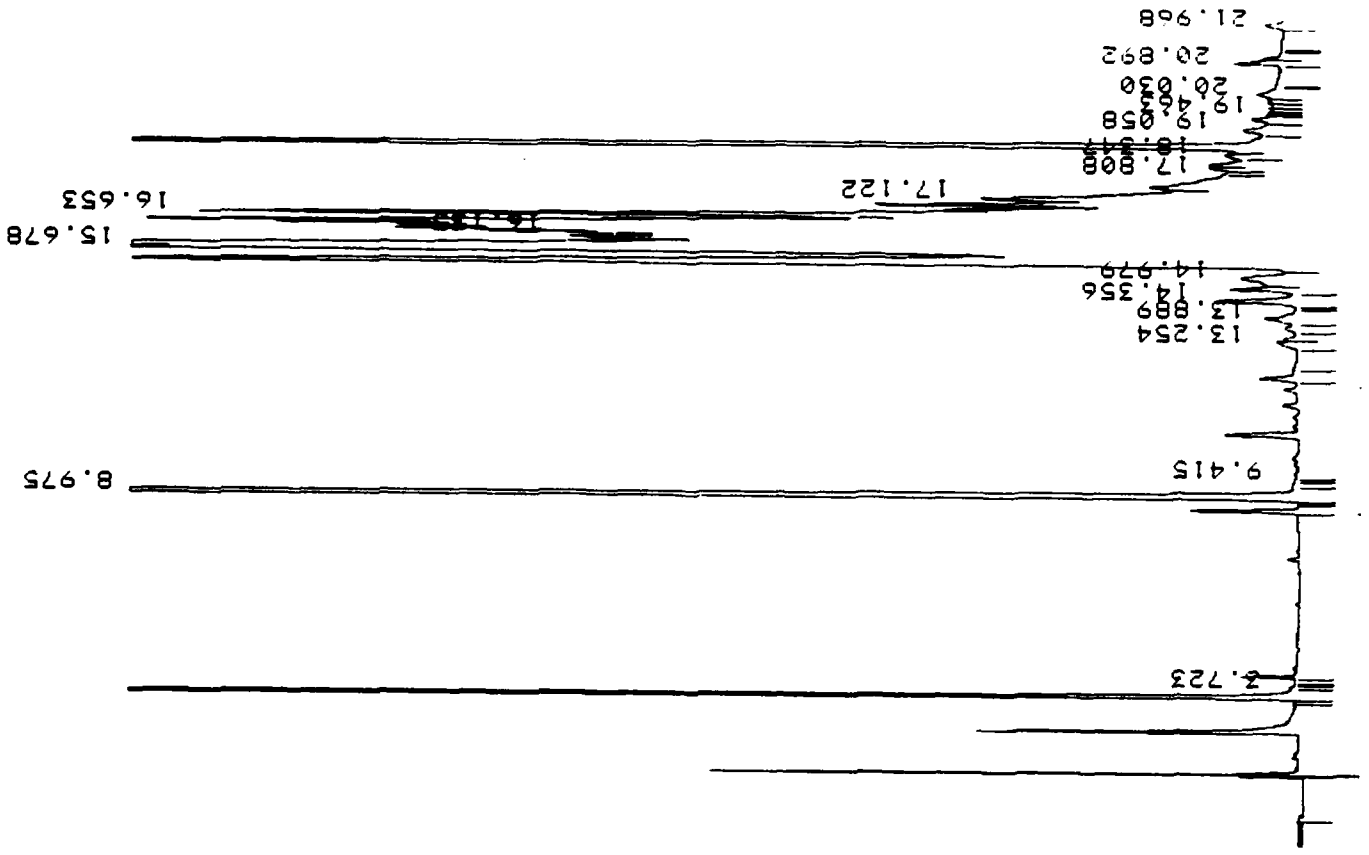
PAGE 6
  PLOT ATTEN
    INIT PLOT ATTEN: 64
```

Figure B-2
Vista 402 Method TSD1

```
SECTION 2: TIME EVENTS
PAGE 1
   LINE#  TIME  EVENT   VALUE
   1       0.00  PR        100
   2       0.00  SN          5
   3       0.00  T%        5.0
   4       0.00  WI          3
   5       0.01  WI          3
   6       0.01  II      12.00
   7      12.00  GR      20.00


SECTION 3: PEAK TABLE
PAGE 1
  STD PK#:  0
  RELATIVE RETEN PK#:  0
  RESOLUTION PK#:  0
  RESOLUTION MINIMUM:   0.0
  FACT%:   5.0
  IDENTIFICATION TIME WINDOWS +/-
  REF
    %: 10
    MIN:   0.00
  NON REF
    %:  5
    MIN:   0.00

PAGE 2
   PK# TIME  NAME        FACTOR   AMOUNT REF GR# MUST LO   MUST HI
   1    3.42 IS       1.000000 1.000000 R      0.000000 0.000000
   2   16.00 TCP      1.000000 1.000000        0.000000 0.000000
```



TITLE: 13:58 3 FEB 86

CHANNEL NO: 2 SAMPLE: 0-79-16J METHOD: TSD

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	AREA COUNTS	SEP CODE
1	INT STD	INT STD	3.427R	1524360	VV
3	PANA	0.0102	8.975	1054530	BV
29	O-PANA	0.0036	17.122	217665	VV
35	DODPA	0.0113	18.598	601397	VV
TOTALS:		0.0251		3397950	

UNIDENT AREA: 4508530

DIVISOR: 22.5000 AMT STD: 21.5000 MULTIPLIER: 1.00000

CHANNEL NO: 2 SAMPLE: 0-79-16J METHOD: TSD1

PEAK NO	PEAK NAME	RESULT	TIME (MIN)	AREA COUNTS	SEP CODE
2	IS	19.2758	3.427R	1524360	VV
7	TCP	66.6290	16.000	5269130	G
TOTALS:		85.9048		6793490	

UNIDENT AREA: 1114670

MULTIPLIER: 1.00000

Figure 8-3
Gas Chromatographic Analysis of 0-79-16J

TABLE B-1

CALIBRATION OF ANTIOXIDANTS

Antioxidant	ng/0.1 µL	Accuracy		Precision		Representative % in Lubricant Sample**
		Found	% Deviation	No. Runs	RSD (%)	
PANA	216	229	+6.0	3	2.0	2.5
	108	111	+3.2	3	2.4	1.2
	50.4	*	*	3	0.5	0.6
	5.04	5.11	+1.4	3	0.5	0.06
	1.008	1.087	+7.8	3	10.2	0.01
O-PANA	234	236	+0.9	3	3.0	2.7
	117	115	-1.7	3	4.1	1.3
	56.8	*	*	3	1.7	0.6
	5.68	5.45	-4.0	3	0.6	0.06
	1.136	1.155	+1.7	3	5.8	0.01
DODPA	224	225	+0.4	3	3.2	2.5
	112	111	-0.9	3	4.1	1.2
	50.8	*	*	3	2.0	0.6
	5.08	4.67	-8.1	3	0.3	0.06
	1.016	0.909	-10.5	3	3.0	0.01

*Calibration solution

**Based on 0.022 g oil diluted to 0.25 mL, 0.1 µL injection

TABLE B-2

TCP CONTENT OF 0-79-16J

Run #	Area Int. Std.	Adjusted Area TCP*	Content	Average	RSD (%)
1	1,524,360	4,667,733	2.93		
2	1,529,460	4,769,225	2.98	2.98	1.7
3	1,641,530	5,200,241	3.03		

*(Summed Area TCP - Area DODPA)

APPENDIX C

LUBRICANT QUALIFICATION DATA FORM

Table C-1 shows the format of the oil qualification data form obtained from the Lube Data Storage and Retrieval System. Qualification data for the six lubricants having qualification test data entered into the Lube Data Storage and Retrieval System are not shown due to the proprietary nature of the test data.

TABLE C-1

LUBRICANT QUALIFICATION DATA SHEET

CODE							
AIR FORCE CODE							
DATE OF SAMPLING							
TYPE OF SAMPLE							
SOURCE OF SAMPLE							
SOURCE DESIGNATION							
QUALIFICATION NUMBER							
LOT NUMBER							
PACKAGE DATE							
TYPE AIRCRAFT							
AIRCRAFT SERIAL NUMBER							
TYPE ENGINE							
ENGINE SERIAL NUMBER							
TIME SINCE OVERHAUL							
TIME SINCE OIL CHANGE							
	LIMITS						
TRACE ELEMENTS: ppm							
Al; Fe; Cr; Ag							
Cu; Sn; Mg; Ni							
Ti; Si; ;							
PARTICULATE CONTAMINATION							
Contiminant, mg/L							
Filtering Time, min/qt							
Filtering Time, min/7.5 cz							
COBRA READING							
TAN, mg KOH/g oil							
VISCOSITY: At C (F), cSt							
At C (F), cSt							
At C (F), cSt							
At C (F), cSt							
After 35 minutes, max							
After 3 hours, max							
After 72 hours, max							
Change at 3 hours, %							
Change at 72 hours, %							
FLASH POINT, deg C (deg F)							
EVAPORATION LOSS AT 205 C (401 F), % wt							
GEAR LOAD CARRYING CAPACITY							
Capacity, ppi/determinations							

AIR FORCE CODE .	LIMITS	NEW OIL					
FOAMING CHARACTERISTICS (STATIC)							
Foam Volume at 1000 cc/min air, ml							
Collapse Time, sec							
Foam Volume at cc/min air, ml							
Collapse Time, sec							
FOAMING CHARACTERISTICS (DYNAMIC)							
Foam Volume, 80 C, 1000 cc/min air, ml							
Collapse Time, sec							
Foam Volume, 80 C, 1500 cc/min air, ml							
Collapse Time, sec							
Foam Volume, 80 C, 2000 cc/min air, ml							
Collapse Time, sec							
Foam Volume, 110 C, 1000 cc/min air, ml							
Collapse Time, sec							
Foam Volume, 110 C, 1500 cc/min air, ml							
Collapse Time, sec							
Foam Volume, 110 C, 2000 cc/min air, ml							
Collapse Time, sec							
DEPOSITION TEST (WADC)							
Deposit Rating							
TAN change							
Viscosity at 40 C, % change							
Oil Consumption, ml							
Filter Screen Deposits, grams							
Coking Tube Deposits, grams							
LEAD CORROSION, g/m2 (mg/in2)							
CORROSION TEST AT 232 C (450 F)							
Silver Weight change, g/m2 (mg/in2)							
Weight change, g/m2 (mg/in2)							
COMPATABILITY (TURBIDITY)							
COMPATABILITY (INTERMIXING)							
AFAPL ENGINE SIMULATOR							
Test Time, Hours							
Test Temperature, deg C							
Deposit Rating							
COBRA Reading at End of Test							
TAN change							
Viscosity at 40 C, % Change							
Iron Content (final), ppm							
Content, ppm							

LUBRICANT QUALIFICATION DATA SHEET

AIR FORCE CODE .	LIMITS	NEW OIL					
ELASTOMER COMPATABILITY							
NRB-H, Swell, % Vol							
F-A, Swell, % Vol							
T. Strength, % Change							
Elongation, % Change							
Hardness Change							
FS, Swell, % Vol							
T. Strength, % Change							
Elongation, % Change							
Hardness Change							
QVI, Swell, % Vol							
T. Strength, % Change							
Elongation, % Change							
Hardness Change							
BEARING DEPOSITION TEST							
Depcsit Rating							
Number of Tests							
TAN Change							
Viscosity at 40 C, % Change							
Filter Deposits, g							
Oil Consumption, ml							
Aluminum Wt. Change, mg/cm2							
Silver Wt. Change, mg/cm2							
Bronze Wt. Change, mg/cm2							
Iron Wt. Change, mg/cm2							
M-50 Steel Wt. Change, mg/cm2							
Waspaloy Wt. Change, mg/cm2							
Titanium Wt. Change, mg/cm2							
C&O, C (F), HOURS (ALTERNATE)							
TAN Change							
Viscosity at 40 C, % Change							
Viscosity at 100 C, % Change							
Sludge, % Volume							
Oil Loss, % Wt.							
Aluminum Wt. Change, mg/cm2							
Silver Wt. Change, mg/cm2							
Bronze Wt. Change, mg/cm2							
Iron Wt. Change, mg/cm2							
M-50 Wt. Change, mg/cm2							
Magnesium Wt. Change, mg/cm2							
Titanium Wt. Change, mg/cm2							

LUBRICANT QUALIFICATION DATA SHEET

AIR FORCE CODE .	LIMITS	NEW OIL					
C&O, C (F), HOURS (BASIC)							
TAN Change							
Viscosity at 40 C, % Change							
Viscosity at 100 C, % Change							
Sludge, % Volume							
Oil Loss, % Wt.							
COBRA Reading (Final)							
Breakpoint to TAN of 4.0, Hours							
Breakpoint to Viscosity Change 25%, Hrs							
Aluminum Wt. Change, mg/cm2							
Silver Wt. Change, mg/cm2							
Bronze Wt. Change, mg/cm2							
Iron Wt. Change, mg/cm2							
M-50 Wt. Change, mg/cm2							
Magnesium Wt. Change, mg/cm2							
Titanium Wt. Change, mg/cm2							
C&O, C (F), HOURS (ALTERNATE)							
TAN Change							
Viscosity at 40 C, % Change							
Viscosity at 100 C, % Change							
Sludge, % Volume							
Oil Loss, % Wt.							
COBRA Reading							
Aluminum Wt. Change, mg/cm2							
Silver Wt. Change, mg/cm2							
Bronze Wt. Change, mg/cm2							
Iron Wt. Change, mg/cm2							
M-50 Wt. Change, mg/cm2							
Magnesium Wt. Change, mg/cm2							
Titanium Wt. Change, mg/cm2							
ACCELERATED STORAGE STABILITY							
Lead Wt Change (48 hrs), g/m2 (mg/in2)							
Lead Wt Change (168 hrs), g/m2 (mg/in2)							
ENGINE ENDURANCE TEST							
EXTENDED STORAGE STABILITY							

NOTES:

APPENDIX D

COMPUTER PROGRAMS TO OPERATE THE CV-1B AND SINGLE BOARD VOLTAMMOGRAPH BASED RULLER CANDIDATES

Basic Language Program Used to Operate the CV-1B Based RULLER Candidate

```
10  POKE 232,253: POKE 233,39
20  POKE 7676,1: POKE 7677,0: POKE 7678,4: POKE 7679,0: POKE 7680,34: PO-E
    7681,36: POKE 7682,0
100  REM  3/22/85
110  LOMEM: 24576
130 IMAX = 2550
160 D$ =  CHR$ (4):I$ =  CHR$ (9)
170  DIM NP(9)
175 SA = 16 * 1024
180  ONERR  GOTO 2060
190  PRINT D$;"BLOAD CYCLO.OBJ"
200  REM  COMMAND *******
220  PRINT : GET A$
225  IF A$ = "A" THEN  GOSUB 1310
230  IF A$ = "C" THEN  PRINT : PRINT D$;"CATALOG"
240  IF A$ = "L" THEN  GOSUB 1130
245  IF A$ = "Q" THEN  END
250  IF A$ = "S" THEN  GOSUB 1000
260  IF A$ = "D" THEN  GOSUB 1860
270  IF A$ = "H" THEN  GOSUB 320
310  IF A$ = "V" THEN  GOSUB 500
315  GOTO 220
320  PRINT "A- AQUIRE DATA"
330  PRINT "C- CATALOG"
335  PRINT "D- DERIVATIVE "
336  PRINT "H- HELP"
340  PRINT "L- LOAD FROM DISK"
350  PRINT "Q- QUIT"
370  PRINT "S- SAVE TO DISK"
430  PRINT "V- VIEW GRAPHICS"
450  PRINT "CTRL S- STOP START"
460  PRINT "CTRL X- ABORT ROUTINE"
490  RETURN
500  REM    VIEW GRAPHICS *******
505 T0 = S0
510  VTAB 24
515 I = 0: HGR :X = 0
520 C2 = 1:ADD = SA: HCOLOR= 3
530 SF = 191 / (DMAX - D0)
535  INPUT "DO YOU WISH TO CHANGE THE SCALE FACTOR? ";A$
540  IF  LEFT$ (A$,1) = "Y" THEN  INPUT "INPUT THE NEW SCALE FACTOR. ";SX
    :SF = SX * SF
545  INPUT "DO YOU WANT A PRINT OUT? ";A$
550  IF  LEFT$ (A$,1) = "Y" THEN  PRINT D$;"PR# 1": PRINT ID$: PRINT "LE..
    REE/DIV ";SS / 2: PRINT "RADIATION ";R$: PRINT DT$: PRINT  TAB( 22/2
    0
552  POKE 49234,0
555  FOR N = 0 TO 191 STEP 9.54: HPLOT X,N: NEXT N: HPLOT X,191 - Y
560  FOR J = 1 TO 20
565 Y =  PEEK (ADD + 1) * 256 +  PEEK (ADD)
570 ADD = ADD + 2
575 Y = (Y - D0) * SF
580  IF Y ) 191 THEN Y = 191
585  IF Y ( 0 THEN Y = 0
590  HPLOT  TO X,191 - Y
595 CNT = CNT - 1
600 X = X + 1
605 I = I + 1
```

```
    IF . = IMAX THEN 665
    NEXT J
520 P =  PEEK (49152): POKE -49168,0
530  IF P = 152 GOTO 700
    IF P = 147 THEN  GET A$
536  IF P = 155 THEN  GOSUB 704
    IF X ( 280 THEN  GOTO 700
550 I$ =  CHR$ (3)
    PRINT I$;"X"
570  PRINT I$;"BH"
    T0 = T0 + (7 * SS)
600  PRINT  TAB( 26)T0
    HGR : POKE 49234,0
635 X = 0
    IF I ( IMAX THEN 555
700  PRINT I$;"IK": PRINT D$;"PR#0": TEXT : RETURN
704  SCALE= 1:XP = X - 1:P = 0
710  IF P = 202 THEN XP = XP - 1: FOR D = 1 TO 50: NEXT D
713  IF P = 203 THEN XP = XP + 1: FOR D = 1 TO 50: NEXT D
713  IF P = 141 THEN 800
714  IF P = 155 THEN  RETURN
730  IF P = 70 THEN XP = XP + 3
731  IF P = 68 THEN XP = XP - 3
730  IF XP ( 0 OR XP ) 279 THEN 765
750 YP =  PEEK (SA + 1 + (XP + I - X) * 2) + 256 *  PEEK (SA + (XP + I -
    X) * 2)
755 YP = 191 - (YP - D0) * SF
757  IF YP ( 0 THEN YP = 0
760  XDRAW 1 AT XP,YP
765  IF XP ( 0 THEN XP = 0
770  IF XP ) 279 THEN XP = 279
772 P =  PEEK (49152): POKE 49168,0
775  XDRAW 1 AT XP,YP
780  GOTO 710
820 F = F + 1
830 T3 = 50 + (XP + I - X) * SS / 40
832  IF F = 1 THEN HT = YP:P = 0: PRINT  CHR$ (7): GOTO 710
840  PRINT D$;"PR#1": PRINT HT - YP: PRINT D$;"PR#0":P = 0:F = 0: GOTO 71
    0
1000  REM   SAVE **********
1010  INPUT "FILE NAME ? ";N$
1020  PRINT D$;"OPEN ";N$
1030  PRINT D$;"WRITE ";N$
1050  PRINT ID$: PRINT DT$
1070  PRINT IMAX: PRINT DMAX
1080  PRINT D0
1090  PRINT D$;"CLOSE ";N$
1100 L = (IMAX) * 2
1110  PRINT D$;"BSAVE ";N$;".DAT,A16384,L";L
1120  RETURN
1130  REM   LOAD **********
1140 ADD = SA:I = 0:DMAX = 0
1150  INPUT "FILE NAME ";N$
1160  PRINT D$;"OPEN ";N$
1170  PRINT D$;"READ ";N$
1200  INPUT ID$: INPUT DT$
1210  INPUT IMAX: INPUT DMAX
1220  INPUT D0
1230  PRINT D$;"CLOSE ";N$
1240  PRINT D$;"BLOAD ";N$;".DAT"
1260  PRINT "SAMPLE ID ";ID$
1290  PRINT "DATE ";DT$
1300  RETURN
1310  REM    AQUIRE DATA ********
1320  VTAB 24
1330 SF = 279 / 1200
```

```
1340 SUM = 0:X = 0:ADD = SA
1400 CNT = 1:I = 0
1460  PRINT "SAMPLE ID ";ID$;" ";: INPUT A$: IF A$ ( ) "" THEN ID$ = A$
1470  PRINT "DATE ";DT$;" ";: INPUT A$: IF A$ ( ) "" THEN DT$ = A$
1480  INPUT "ARE YOU READY TO START ? ";A$: PRINT
1490  IF  LEFT$ (A$,1) ( )  "Y" THEN  RETURN
1500  CALL 768
1510 IMAX = (256 * ( PEEK (255) - 4 * 16) +  PEEK (254)) / 2
1520 DMAX = 0:D0 = 12020
1600  FOR ADD = SA TO SA + (IMAX * 2) - 2 STEP 2
1610 S =  PEEK (ADD) * 256 +  PEEK (ADD + 1)
1680  IF S ) DMAX THEN DMAX = S
1690  IF S ( D0 THEN D0 = S
1700  NEXT ADD
1850  TEXT : RETURN
1860  REM     DERVATIVE ******
1870 M = IMAX - 6
1880  FOR I = 2 TO 7
1890 J = I - 1
1900 NP(I) =  PEEK (SA - 1 + 2 * J) + 256 *  PEEK (SA - 2 + 2 * J)
1910  NEXT I
1920  FOR I = 1 TO M
1930  VTAB 23: PRINT I
1940 J = I + 6
1950  FOR K = 1 TO 6
1960 KA = K + 1
1970 NP(K) = NP(KA)
1980  NEXT K
1990 NP(7) =  PEEK (SA - 1 + 2 * J) + 256 *  PEEK (SA - 2 + 2 * J)
2000 NSUM =  - 1 * (NP(3) - NP(5)) - 2 * (NP(2) - NP(6)) - 3 * (NP(1) - N
     P(7))
2010 YS = NSUM / 28 * 10 + 5 * 10
2020  IF YS ( 0 THEN 2050
2030  POKE SA - 1 + 2 * (I + 3),YS - 256 *  INT (YS / 256)
2040  POKE SA - 2 + 2 * (I + 3), INT (YS / 256)
2050  NEXT I:D0 = 0:DMAX = 45 * 10: RETURN
2060  REM     ERROR*********
2070  PRINT "ERROR": PRINT  PEEK (222)
2080  GOTO 220
```

Machine Code Program Used to Operate the Single Board Voltammograph

```
SOURCE FILE: CYCLO.ASM
0000:              1 *CYCLOVOLTAMETRY CODE FOR APPLE
0000:              2 *A/D IN SLOT 5
0000:              3 *D/A IN SLOT 6
----- NEXT OBJECT FILE NAME IS CYCLO.ASM.OBJ0
0300:              4          ORG   $300       ;ORIGIN OF PROGRAM
000F:              5 RMPSTHI  EQU   $0F        ;RAMP START HIGH  BYTE
00FF:              6 RMPSTLO  EQU   $FF        ;RAMP START LOW BYTE
00FD:              7 RMPADHI  EQU   $FD        ;RAMP ADD HIGH BYTE
00FC:              8 RMPADLO  EQU   $FC        ;RAMP ADD LOW BYTE
0040:              9 DATSTHI  EQU   $40        ;DATA START HIGH BYTE
0000:             10 DATSTLO  EQU   $00        ;DATA START LOW BYTE
00FF:             11 DATADHI  EQU   $FF        ;DATA ADD HIGH BYTE
00FE:             12 DATADLO  EQU   $FE        ;DATA ADD LOW BYTE
C0F0:             13 DAHI     EQU   $C0F0      ;D/A HIGH BYTE
C0F1:             14 DALO     EQU   $C0F1      ;D/A LOW BYTE
C0D1:             15 ADHI     EQU   $C0D1      ;A/D HIGH BYTE
C0D0:             16 ADLO     EQU   $C0D0      ;A/D LOW BYTE
0054:             17 DATENDHI EQU   $54        ;DATA END HIGH BYTE
0022:             18 SPEED    EQU   $22        ;USED IN DELAY ROUTINE
0010:             19 GAIN     EQU   $10        ;GAIN
0300:             20 *PROGRAM START
0300:A9 0F        21 INIT     LDA   #RMPSTHI   ;BEGIN RAMP START
0302:85 FD        22          STA   RMPADHI
0304:A9 FF        23          LDA   #RMPSTLO
0306:85 FC        24          STA   RMPADLO
0308:A9 40        25          LDA   #DATSTHI   ;BEGIN DATA START
030A:85 FF        26          STA   DATADHI
030C:A9 00        27          LDA   #DATSTLO
030E:85 FE        28          STA   DATADLO
0310:A0 00        29          LDY   #$00       ;SET Y=0, RAMP DOWN (NO DATA TAKEN)
0312:A5 FD        30 RAMPDOWN LDA   RMPADHI    ;SET RAMP
0314:8D F0 C0     31          STA   DAHI
0317:A5 FC        32          LDA   RMPADLO
0319:8D F1 C0     33          STA   DALO
031C:C6 FC        34          DEC   RMPADLO    ;DECREMENT RAMP
031E:A9 22        35          LDA   #SPEED     ;DELAY
0320:20 63 03     36          JSR   DELAY
0323:A5 FC        37          LDA   RMPADLO
0325:D0 EB        38          BNE   RAMPDOWN
0327:F0 02        39          BEQ   NOINC
0329:E6 FC        40 RAMPUP   INC   RMPADLO    ;INCREASE RAMP
032B:A5 FD        41 NOINC    LDA   RMPADHI
032D:8D F0 C0     42          STA   DAHI
0330:A5 FC        43          LDA   RMPADLO
0332:8D F1 C0     44          STA   DALO
0335:A9 10        45          LDA   #GAIN      ;START SAMPLE
0337:8D D0 C0     46          STA   ADLO
033A:A9 22        47          LDA   #SPEED     ;DELAY
033C:20 63 03     48          JSR   DELAY
033F:AD D1 C0     49          LDA   ADHI       ;BEGIN TO STORE DATA
0342:29 0F        50          AND   #$0F
0344:91 FE        51          STA   (DATADLO),Y
0346:E6 FE        52          INC   DATADLO
0348:AD D0 C0     53          LDA   ADLO
034B:91 FE        54          STA   (DATADLO),Y
034D:E6 FE        55          INC   DATADLO    ;BEGIN TO CHECK FOR LAST DATA POINT
034F:D0 09        56          BNE   RAMPCONT
0351:E6 FF        57          INC   DATADHI
```

```
0353:A9 54      58          LDA  #DATENDHI
0355:C5 FF      59          CMP  DATADHI
0357:D0 01      60          BNE  RAMPCONT
0359:60         61          RTS
035A:A5 FC      62 RAMPCONT LDA  RMPADLO    ;BEGIN DELAY SUBROUTINE
035C:C9 FF      63          CMP  #RMPSTLO
035E:D0 C9      64          BNE  RAMPUP
0360:4C 12 03   65          JMP  RAMPDOWN
0363:38         66 DELAY    SEC
0364:48         67 BRANCH2  PHA
0365:E9 01      68 BRANCH1  SBC  #$01
0367:D0 FC      69          BNE  BRANCH1
0369:68         70          PLA
036A:E9 01      71          SBC  #$01
036C:D0 F6      72          BNE  BRANCH2
036E:60         73          RTS

*** SUCCESSFUL ASSEMBLY: NO ERRORS
```

Basic Language Program to Operate the Single Board Voltammograph Based RULLER Candidate

```
10  POKE 232,252: POKE 233,29
20  POKE 7676,1: POKE 7677,0: POKE
    7678,4: POKE 7679,0: POKE 76
    80,34: POKE 7681,36: POKE 76
    82,0
30  POKE  - 16296.1: REM WORKING
    ELECTRODE OFF
100  REM  2/22/85
110  LOMEM: 24576
130 IMAX = 2560
160 D$ =  CHR$ (4):I$ =  CHR$ (9)

170  DIM NP(9)
175 SA = 16 * 1024
180  ONERR  GOTO 2060
190  PRINT D$;"BLOAD CYCLO.OBJ"
200  REM  COMMAND *******
220  PRINT : GET A$
225  IF A$ = "A" THEN  GOSUB 1310

230  IF A$ = "C" THEN  PRINT : PRINT
    D$;"CATALOG"
240  IF A$ = "L" THEN  GOSUB 1130

245  IF A$ = "Q" THEN  END
250  IF A$ = "S" THEN  GOSUB 1000

260  IF A$ = "D" THEN  GOSUB 1860

270  IF A$ = "H" THEN  GOSUB 320
310  IF A$ = "V" THEN  GOSUB 500
315  GOTO 220
320  PRINT "A- AQUIRE DATA"
330  PRINT "C- CATALOG"
335  PRINT "D- DERIVATIVE "
336  PRINT "H- HELP"
340  PRINT "L- LOAD FROM DISK"
350  PRINT "Q- QUIT"
370  PRINT "S- SAVE TO DISK"
430  PRINT "V- VIEW GRAPHICS"
450  PRINT "CTRL S- STOP START"
460  PRINT "CTRL X- ABORT ROUTINE
    "
490  RETURN
500  REM     VIEW GRAPHICS *******

505 T0 = S0
510  VTAB 24
515 I = 0: HGR :X = 0
520 C2 = 1:ADD = SA: HCOLOR= 3
530 SF = 191 / (DMAX - D0)
535  INPUT "DO YOU WISH TO CHANGE
     THE SCALE FACTOR? ";A$
540  IF  LEFT$ (A$,1) = "Y" THEN
     INPUT "INPUT THE NEW SCALE
    FACTOR. ";SX:SF = SX * SF
545  INPUT "DO YOU WANT A PRINT O
    UT? ";A$
550  IF  LEFT$ (A$,1) = "Y" THEN
     PRINT D$;"PR# 1": PRINT ID$
    : PRINT "DEGREE/DIV ";S$ / 2
    : PRINT "RADIATION ";R$: PRINT
    DT$: PRINT  TAB( 26)S0
552  POKE 49234,0
555  FOR N = 0 TO 191 STEP 9.54: HPLOT
    X,N: NEXT N: HPLOT X,191 - Y

560  FOR J = 1 TO 20
565 Y =  PEEK (ADD + 1) * 256 *  PEEK
    (ADD)
570 ADD = ADD + 2
575 Y = (Y - D0) * SF
580  IF Y ) 191 THEN Y = 191
585  IF Y ( 0 THEN Y = 0
590  HPLOT  TO X,191 - Y
595 CNT = CNT + 1
600 X = X + 1
605 I = I + 1
610  IF I = IMAX THEN 665
615  NEXT J
620 P =  PEEK (49152): POKE 49168
    ,0
630  IF P = 152 GOTO 702
635  IF P = 147 THEN  GET A$
636  IF P = 155 THEN  GOSUB 704
640  IF X ( 280 THEN  GOTO 700
660 I$ =  CHR$ (9)
665  PRINT I$;"K"
670  PRINT I$;"8H"
675 T0 = T0 + (7 * S5)
680  PRINT  TAB( 26)T0
685  HGR : POKE 49234,0
695 X = 0
700  IF I ( IMAX THEN 555
702  PRINT I$;"1K": PRINT D$;"PR#
    0": TEXT : RETURN
704  SCALE= 1:XP = X - 1:P = 0
710  IF P = 202 THEN XP = XP - 1:
     FOR D = 1 TO 50: NEXT D
712  IF P = 203 THEN XP = XP + 1:
     FOR D = 1 TO 50: NEXT D
713  IF P = 141 THEN 800
714  IF P = 155 THEN  RETURN
720  IF P = 70 THEN XP = XP + 3
721  IF P = 68 THEN XP = XP - 3
730  IF XP ( 0 OR XP ) 279 THEN 7
    65
750 YP =  PEEK (SA + 1 + (XP + I -
    X) * 2) + 256 *  PEEK (SA +
    (XP + I - X) * 2)
755 YP = 191 - (YP - D0) * SF
757  IF YP ( 0 THEN YP = 0
760  XDRAW 1 AT XP,YP
765  IF XP ( 0 THEN XP = 0
770  IF XP ) 279 THEN XP = 279
772 P =  PEEK (49152): POKE 49168
    ,0
775  XDRAW 1 AT XP,YP
780  GOTO 710
800 F = F - 1
810 T2 = S0 + (XP + I - X) * S8 /
    40
830  IF F = 1 THEN HT = YP:P = 0:
     PRINT  CHR$ (7): GOTO 710
```

```
840  PRINT D$;"PR#1": PRINT MT +
     YP: PRINT D$;"PR#0":P = 0:F =
     0: GOTO 710
1000  REM   SAVE **********
1010  INPUT "FILE NAME ? ";N$
1020  PRINT D$;"OPEN ";N$
1030  PRINT D$;"WRITE ";N$
1060  PRINT ID$: PRINT DT$
1070  PRINT IMAX: PRINT DMAX
1080  PRINT D0
1090  PRINT D$;"CLOSE ";N$
1100 L = (IMAX) * 2
1110  PRINT D$;"BSAVE ";N$;".DAT,
     A16384,L";L
1120  RETURN
1130  REM   LOAD **********
1140 ADD = SA:I = 0:DMAX = 0
1150  INPUT "FILE NAME ";N$
1160  PRINT D$;"OPEN ";N$
1170  PRINT D$;"READ ";N$
1200  INPUT ID$: INPUT DT$
1210  INPUT IMAX: INPUT DMAX
1220  INPUT D0
1230  PRINT D$;"CLOSE ";N$
1240  PRINT D$;"BLOAD ";N$;".DAT"

1280  PRINT "SAMPLE ID ";ID$
1290  PRINT "DATE ";DT$
1300  RETURN
1310  REM    AQUIRE DATA ********

1320  VTAB 24
1340 SUM = 0:X = 0:ADD = SA
1400 CNT = 1:I = 0
1460  PRINT "SAMPLE ID ";ID$;" ";
     : INPUT A$: IF A$ (  ) "" THEN
     ID$ = A$
1470  PRINT "DATE ";DT$;" ";: INPUT
     A$: IF A$ (  ) "" THEN DT$ =
     A$
1480  INPUT "ARE YOU READY TO STA
     RT ? ";A$: PRINT
1490  IF  LEFT$ (A$,1) (  ) "Y" THEN
      RETURN
1495  POKE  - 16295,0
1500  CALL 768
1505  POKE  - 16296,1
1510 IMAX = (256 * ( PEEK (255) -
     4 * 16) +  PEEK (254)) / 2
1520 DMAX = 4096:D0 = 0
1850  TEXT : RETURN
1860  REM    DERVATIVE ******
1870 M = IMAX - 6
1880  FOR I = 2 TO 7
1890 J = 1 - 1
1900 NP(I) =  PEEK (SA - 1 + 2 *
     J) + 256 *  PEEK (SA - 2 + 2
      * J)
1910  NEXT I
1920  FOR I = 1 TO M
1930  VTAB 23: PRINT I
1940 J = I + 6
1950  FOR K = 1 TO 6
1960 KA = K + 1
1970 NP(K) = NP(KA)
1980  NEXT K
1990 NP(7) =  PEEK (SA - 1 + 2 *
     J) + 256 *  PEEK (SA - 2 + 2
      * J)
2000 NSUM =  - 1 * (NP(3) - NP(5)
     ) - 2 * (NP(2) - NP(6)) - 3 *
     (NP(1) - NP(7))
2010 YS = NSUM / 28 * 10 + 5 * 10

2020  IF YS ( 0 THEN 2050
2030  POKE SA - 1 + 2 * (I + 3),Y
     S - 256 *  INT (YS / 256)
2040  POKE SA - 2 + 2 * (I + 3), INT
     (YS / 256)
2050  NEXT I:D0 = 0:DMAX = 45 * 1
     0: RETURN
2060  REM     ERROR*********
2070  PRINT "ERROR": PRINT  PEEK
     (232)
2080  GOTO 220
4096 :D0 = 0
```

Apple IIe Microcomputer Basic Variables and Locations

```
A$     220, 225, 230, 240, 245, 250, 260, 270, 310, 535, 540, 545, 550, 635, 1460
, 1470, 1480, 1490
AD     520, 565, 570, 1140, 1340
C2     520
CN     595, 1400
D      710, 712
D$     160, 190, 230, 550, 702, 840, 1020, 1030, 1090, 1110, 1160, 1170, 1230, 12
40
D0     530, 575, 755, 1080, 1220, 1520, 2050, 4096
DM     530, 1070, 1140, 1210, 1520, 2050
DT$    550, 1060, 1200, 1290, 1470
F      800, 830, 840
HT     830, 840
I      515, 605, 610, 700, 750, 810, 1140, 1400, 1880, 1890, 1900, 1910, 1920, 19
30, 1940, 2030, 2040, 2050
I$     160, 660, 665, 670, 702
ID$    550, 1060, 1200, 1280, 1460
IM     130, 610, 700, 1070, 1100, 1210, 1510, 1870
J      560, 615, 1890, 1900, 1940, 1990
K      1950, 1960, 1970, 1980
KA     1960, 1970
L      1120, 1110
M      1970, 1920
N      555
N$     1010, 1020, 1030, 1090, 1110, 1150, 1160, 1170, 1230, 1240
NP(    170, 1900, 1970, 1990, 2000
P$     2000, 2010
R      620, 630, 635, 636, 704, 710, 712, 713, 714, 720, 721, 772, 830, 840
R$     550
S0     505, 550, 810
SA     175, 520, 750, 1140, 1340, 1900, 1990, 2030, 2040
SF     530, 540, 575, 755
SS     550, 675, 810
SU     1340
SX     540
T0     505, 675, 680
TE     810
X      515, 555, 590, 600, 640, 695, 704, 750, 810, 1340
XP     704, 710, 712, 720, 721, 730, 750, 760, 765, 770, 775, 810
Y      555, 565, 575, 580, 585, 590
YD     750, 755, 757, 760, 775, 830, 840
Y$     2010, 2020, 2030, 2040
```

Apple IIe Microcomputer Memory Map

Location Hex	Location Decimal	Description
$4000		1st Cycle Data
$4200		2nd Cycle Data
$4400		3rd Cycle Data
$4600		4th Cycle Data
$4800		5th Cycle Data
$4A00		6th Cycle Data
$4C00	19456	7th Cycle Data (1/2 way point 197/2)
$4E00	19968	8th Cycle Data
$5000		9th Cycle Data
$5200		10th Cycle Data
$		A/D
$C0D0		Start A/D Poke Gain Code $10 0-1V f.s SLOT=5
$C0D1		READ MSB (AND $0F)
$C0D0		READ LSB Data=MSB*256+LSB
$C0F0	-16144	MSB D/A SLOT=7
$C0F1	-16143	LSB D/A Write Data to Port
	-16295	1 bit TTL ON
	-16296	1 bit TTL OFF

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