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## FLUIDS, LUBRICANTS, FUELS AND RELATED MATERIALS

*E. ERWIN KLAUS  
MERRELL R. FENSKE*

*PETROLEUM REFINING LABORATORY  
PENNSYLVANIA STATE UNIVERSITY*

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## ABSTRACT

This report describes work carried out on a continuing project directed toward the development of improved hydraulic fluids and jet engine lubricants for use in the high temperature range of 400° to 700°F. With few exceptions these studies are equally applicable to hydraulic fluids and jet engine lubricants.

Efforts have been concentrated on the study of mineral oils, hydrocarbons, and improved stability esters for application to high temperature systems. Super-refining of mineral oils and hydrocarbons by hydrogenation, acid extraction, and silica gel percolation has been shown to improve materially the overall quality of these materials as hydraulic fluids and lubricants. Selection of the molecular configuration of esters has been shown to affect their thermal stability and hence their overall utility as high temperature fluids.

A number of new and improved test techniques have been developed or applied to the high temperature evaluation of hydraulic fluids and jet engine lubricants. These tests include: (1) high temperature density measurement, (2) the controlled atmosphere panel coker test, (3) a modified thermal stability test including catalyst metals, (4) a high temperature PRL thin film oxidation test, and (5) a single pass high temperature lube rig.

A number of large scale blends designed for use in bench, mock-up, and actual service equipment (jet engines and high temperature hydraulic systems) have been formulated for evaluation by WADC and industry. Used samples of fluids from actual tests in jet engines and high temperature hydraulic systems have been evaluated. Results of these evaluations offer some insight into the actual behavior of fluids in service and the degree of realism in various laboratory tests in policing fluid quality necessary for adequate service behavior.

Some additional miscellaneous duties have been performed at the request of the Wright Air Development Center. These duties include: (1) distribution of Laboratory reports, (2) fluid formulation, (3) distribution of standard fluid samples, (4) preparation of WADC Technical Reports and papers on specific topics of interest, and (5) evaluation of specific fluid properties.

### PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



R. T. SCHWARTZ  
Chief, Organic Materials Branch  
Materials Laboratory  
Directorate of Research

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HIGH TEMPERATURE HYDRAULIC FLUID AND JET ENGINE LUBRICANT STUDIES. Studies for this period have been concentrated on the formulation of high temperature hydraulic fluids and jet engine lubricants for use at temperatures as high as 400° to 700°F. The same materials have been considered for both hydraulic fluid and jet engine lubricant applications. Hydraulic fluids are used in closed systems where air or oxygen is eliminated or severely limited. High temperature tests involving severe oxidation do not apply to the study of hydraulic fluids. The lubricity demands of the turbo-prop engine are more severe than those of the hydraulic system and the turbo jet engine. With these exceptions, the studies are equally applicable to both hydraulic systems and jet engines.

Paraffinic, naphthenic, and aromatic mineral oils, and paraffinic and aromatic hydrocarbons are discussed as base stocks for high temperature hydraulic fluids and lubricants. The beneficial effects of super-refining the mineral oils by hydrogenation, acid extraction, and silica gel percolation are demonstrated. Hydrogenation has also been applied to polyolefin and aromatic hydrocarbons. Improvements from super-refining by hydrogenation are brought about by saturation of unsaturated molecules and the removal of non-hydrocarbon impurities from the mineral oils and hydrocarbons. Hydrogenated condensed ring aromatic hydrocarbons (cycle stock extract) offer the optimum in thermal stability with good oxidation behavior for hydraulic fluid application. Hydrogenated paraffinic mineral oil and polyolefin hydrocarbon show excellent overall stability for use as high temperature jet engine oils.

A number of esters including: bis tridecyl sebacate, isosebacic acid esters, Hercules Synthetics J-20 (neopentyl glycol type ester), and bis (1-methyl cyclohexyl methyl) sebacate are discussed as base stock materials. Tridecyl sebacate and the isosebacic acid esters exhibit overall stability properties in the same class as di-2-ethylhexyl sebacate. The latter two neopentyl type esters show improved thermal and oxidative stability properties similar to those of Hercoflex 600.

Two chlorine-containing aryl phosphate esters have been evaluated. Esters of this type appear to be susceptible to catalytic deterioration in the presence of metals under high temperature oxidative and thermal conditions.

Two heavy mineral oil fractions (Kendall resins) have been evaluated in thermal and oxidative tests at high temperatures.

Oxidation and corrosion characteristics at 347°F. are compared for various classes of conventionally refined and super-refined mineral oils and hydrocarbons. Partial inhibitor response for the conventionally refined paraffinic and naphthenic mineral oils is compared with the improved inhibitor susceptibility of the super-refined mineral oils and hydrocarbons. The complete lack of inhibitor effectiveness in the

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polyolefin is shown. The polyolefins demonstrate excellent oxygen tolerance.

The effect of air rate as a variable in the 347°F. oxidation test has been evaluated. Increased oxidative deterioration with increased air rate is demonstrated for 347°F. evaluations in which the stable life is exceeded.

A number of oxidation inhibitors have been evaluated in conventionally refined and hydrogenated mineral oils. By the proper choice of additives (phenyl-alpha-naphthylamine and dithiocarbamate) in a hydrogenated stock, oxidative behavior at 347°F. superior to that of good quality Spec. MIL-L-7808 synthetic engine oil has been demonstrated. A number of additional experimental compounds have been studied as oxidation inhibitors and a number of specially treated oils have been evaluated for oxidation stability.

Oxidation and corrosion studies of mineral oils and hydrocarbons at 500° and 600°F. are discussed. The excellent high temperature behavior of mineral oils and hydrocarbons both before and after super-refining techniques is shown. The contribution of effective low temperature oxidation inhibitors to increased high temperature dirtiness is discussed. The superiority of polyolefins over hydrogenated polyolefins and mineral oils in high temperature oxygen tolerance is shown. The effectiveness of using polyolefin-mineral blends for increased oxygen tolerance is also illustrated.

The use of Acryloid dispersant additives to reduce dirt and sludge formation in esters and mineral oils is demonstrated. These dispersant additives are effective in all cases evaluated at 347° and 500°F. The Acryloid dispersants are shown to be ineffective in altering the panel coking tendencies of the base fluid. The effectiveness of the Acryloid dispersants after subjection to panel coker tests is demonstrated by 347°F. oxidation tests. The effect of thermal instability of the dispersants at 700°F. is illustrated.

The effect of tricresyl phosphate and diisopropyl acid phosphite as lubricity additives on the oxidation stability of a naphthenic mineral oil is discussed. These lubricity additives are shown to have much less effect on the oxidation stability of mineral oils than on esters under similar conditions.

Oxidation tests at 500°F. and thermal tests at 600°F. with esters are used to demonstrate the differences in steel corrosion as a function of alloy type. SAE 1010 and 52-100 bearing steels are readily corroded while stainless and M-10 tool steels resist corrosion by the esters under these test conditions.

The dirtiness behavior of some fluids and lubricants has been evaluated in a thin film oxidation procedure. The effect of such variables as gas rate, fluid circulation rate, test temperature, and type of metal surface on the dirtiness characteristics of some fluids and lubricants is discussed. The air rate studies and the tests conducted with nitrogen



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instead of air point out the advantage to be gained by eliminating or restricting the amount of air in contact with fluids and lubricants at these operating temperatures.

The development and use of a controlled atmosphere panel coker is described. Esters with and without additives and mineral oils are compared in this unit under essentially thermal as well as oxidative test conditions. In general, the esters show much less advantage over mineral oil compositions in this unit than in the Model C panel coker.

Panel coker studies indicate that phosphoric or phosphorous acid, in trace quantities, can materially affect the coking tendencies of Spec. MIL-L-7808 type compositions. It is also pointed out that oxidation inhibitor type can alter markedly the coking tendencies of esters containing acidic phosphorus additives. A comparison of Conradson carbon residues and panel coker values for various mineral oils is presented.

Increased coking values have been demonstrated for blends of bulk constituents in the panel coker. Additives, in addition to those of the phosphorus class, have also been shown to be effective in raising the panel coking value.

The effects of mineral oil volatility and panel temperature on coking values are also demonstrated. A sharp rise in coking values for Spec. MIL-L-7808 type fluids due to a change in panel temperature from 600° to 700°F. is compared with little or no change in coking values for several mineral oils under similar conditions.

Panel coker values at 600°F. panel temperature have been determined in both the Model C and controlled atmosphere cokers for 12 one-hundred gallon mineral oil and hydrocarbon blends.

The modification of the PRL fuel dirtiness rig to a single pass high temperature lube rig is described. The test fluid in this rig is heated to 700°F. on a metal surface in the presence of air. Mineral oils, hydrocarbons, dibasic acid esters, pentaerythritol esters, silicate esters, and silicones have been studied. Mineral oils, hydrocarbons, and pentaerythritol esters give substantially lower deposit and sludge values than the other three classes. The effect of mineral oil volatility on deposit formation is demonstrated in this unit. The effect of air rate on deposit has been demonstrated. Increased air rate has been shown to expand the differences in deposit formation noted at lower air rates.

A stainless steel pressure cylinder operated at 700°F. for six hours in the presence of a one-half inch diameter ball bearing each of 52-100 steel, M-10 tool steel, and Naval bronze has been chosen as a single test to evaluate the thermal stability and corrosion behavior of some mineral oil fractions. The effect of additional refining (hydrogenation) on the thermal stability of various mineral oil types has been determined. Hydrogenated mineral oils have been shown to have the best overall corrosion behavior in the 700°F. thermal test of any class of fluids evaluated.



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The thermal stability of several batches of polyolefins before and after hydrogenation are reported.

A satisfactory procedure for the determination of liquid density over a temperature range of 100° to 500°F. has been demonstrated for a variety of fluid types. Measured densities at 500°F. are in good agreement with density values obtained from extrapolated values using low temperature density values.

Vapor pressure measurements on five silane samples are reported. These materials appear to be thermally unstable at 650° to 750°F.

A discussion of procedures for the evaluation of foaming is presented. A series of tests in accordance with one of these procedures points out the effectiveness of a high molecular weight methyl silicone as an anti-foam agent in several classes on mineral oils and synthetic hydrocarbons.

Good lubricity characteristics of mineral oils and the continued effectiveness of diisopropyl acid phosphite in mineral oils is demonstrated in Shell four-ball wear tests at 700°F. using M-10 balls. A technique for the addition of test fluid during the 700°F. four-ball tests has been developed.

Oronite High Temperature Hydraulic Fluid 8515 has been evaluated in the PRL high temperature pump test unit at 400° and 500°F. Test results indicate the same behavior for 8515 as for silicate fluids MLO 8200 and O.S. 45.

The procedure used by this Laboratory for the preparation of diisopropyl acid phosphite is outlined. This material is used in the preparation of MLO 7010 and GTO 120 lubricants of the high gear loading type. The composition and properties of 12 one-hundred gallon blends of mineral oils and hydrocarbons are given. These blends have been prepared for mock-up and engine testing by the Power Plant Laboratory of WADC.

The properties of several used samples of Grade 1065 and ester-base jet engine oil from J-57 engine tests are discussed. Deterioration in the engine is shown to be more severe than that predicted by Spec. MIL-L-7808 bulk oil oxidation tests. A program designed to correlate deterioration in the engine with bench type oxidation and thermal stability tests is discussed. Thermal stability and panel coker tests are shown to have little effect on subsequent oxidation stability. High temperature (400° to 500°F.) bulk oil, thin film, and successive oxidation tests appear to be more closely related to the type of oxidative deterioration noted in the engine.

The properties of several used samples of Spec. MIL-L-6387 ester-base high temperature hydraulic fluid are presented. The fluid has been tested in a high temperature aircraft type hydraulic system. The similarity of used fluid property changes to changes noted in thermal stability tests is demonstrated. Thermal stability properties of Spec. MIL-L-6387 and Spec. MIL-L-7808 type fluids are compared.

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MISCELLANEOUS. Five papers have been prepared for presentation before technical meetings. Two of these papers have been issued as WADC Technical Reports. The other three have been published in the proceedings or minutes of the technical meeting.

The preparation of a number of experimental blends designed for hydraulic fluids, jet engine lubricants, and turbo-prop lubricants are discussed.

Results of cooperative wear tests in the Shell four-ball wear tester are shown.

The measurement of the effect of shearing stress on the non-Newtonian properties of four silicone fluids is described.

A sample of Acryloid HFS-5 proposed for use in the preparation of Spec. MIL-F-5602 shear stability reference fluid has been evaluated. It is recommended that Acryloid HF-5 be qualified for use in the preparation of this fluid.

Distribution of low temperature viscosity standards and wear tester standards is listed.

## I. HIGH TEMPERATURE HYDRAULIC FLUID AND JET ENGINE LUBRICANT STUDIES

A. GENERAL. High temperature studies for this period have been centered about fluid formulations for two types of hydraulic fluid and two classes of jet engine lubricant. The severity of operating conditions for these fluids has been tentatively defined by the Wright Air Development Center. Briefly, the two types of hydraulic fluid comprise a fluid suitable for extended use in hydraulic systems at 500° to 550°F. and a fluid suitable for use at 700°F. for relatively short periods. The two types of jet engine oil comprise an oil for use at 350°F. bulk oil temperature with improved lubricity over Spec. MIL-L-7808 fluid and an oil suitable for use at bulk oil temperatures of 500° and 600°F.

High temperature studies of fluids and lubricants have been conducted by this Laboratory for several years under Air Force Contracts AF33(038)18193 and AF33(616)2851. Several of the previous annual reports including WADC TR 55-30 parts 1, 2, 3, and 4 have dealt with the development of preliminary high temperature test procedures and techniques as well as a preliminary survey of high temperature behavior of a large number of chemical classes of fluids.

The properties studied at elevated temperatures in these reports include:

1. Viscosity
2. Volatility
3. Thermal stability
4. Lubricity
5. Corrosiveness to metal
6. Oxidation stability
7. Oxidation rate
8. Oxygen tolerance
9. Deposit formation

The chemical classes of fluids surveyed in these reports include:

1. Mineral oils
2. Hydrocarbons
3. Dibasic acid esters
4. Neopentyl type esters
5. Complex- and poly-esters
6. Chlorinated aromatic hydrocarbons
7. Halocarbons
8. Polyglycol ethers
9. Silicate esters
10. Phosphate esters
11. Silicones
12. Chlorine-containing silicones

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The preliminary evaluations indicated several promising fluid classes based on an overall high temperature evaluation. The more promising fluid types on this basis are mineral oils, synthetic hydrocarbons, esters of improved thermal stability (neopentyl type), improved lubricity silicones, chlorinated aromatic hydrocarbons, and silicates.

Emphasis during the period covered by this report has been placed on the more detailed evaluation of mineral oils, hydrocarbons, and esters of improved thermal stability. The improvement and simplification of preliminary high temperature test techniques as well as the development of new test techniques for the measurement of high temperature fluid stability have also been studied in some detail.

The same base stocks have been considered for both hydraulic fluid and jet engine lubricant applications. The hydraulic fluids are used in closed systems where air or oxygen is essentially eliminated or severely limited. High temperature tests involving severe oxidative conditions do not, therefore, apply to the study of hydraulic oils. The lubricity demands of the turbo-prop lubricant are more severe than the needs of the hydraulic fluid or jet engine oil. With these exceptions the studies reported are equally applicable to both hydraulic fluids and jet engine lubricants. Property evaluation is discussed in this report without particular reference to either program.

B. BASE STOCK EVALUATION. A number of base-stocks have been evaluated for high temperature application. These studies have been designed primarily to evaluate the high temperature stability properties. Exhaustive study of low temperature properties was not included in these preliminary evaluations. In several cases, specific base stocks were studied under conditions requested by the Materials Laboratory, WCRTRL, of the Wright Air Development Center.

1. Mineral Oils and Hydrocarbons. Mineral oils and hydrocarbons have shown very good overall high temperature stability in the preliminary evaluations. For a given mineral oil type, a wide range of viscosity, volatility, and molecular weight grades can be obtained. A typical series of paraffinic mineral oils, arranged in increasing viscosity grades are shown on Table 1.

The mineral oil fractions with viscosity properties approaching Spec. MIL-L-7808 and MIL-L-6387 requirements at low temperatures exhibit initial atmospheric boiling points below 700°F. The materials with satisfactory volatility characteristics at 700°F. exhibit 13,000 centistokes viscosity at temperatures of 27° to 105°F. These low volatility, high viscosity mineral oils also show advantages over low viscosity, high volatility oils in high temperature coking or deposit tests and high temperature oxidation characteristics. These data indicate the need for design changes in hydraulic and lubricant systems for use over the -65° to 700°F. temperature range.

The data suggest the use of a two oil system in which a light oil would be used for start up and a viscous, non-volatile oil used for

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the high temperature portion of the flight. On the basis of the mineral oil properties studied, a bulk oil temperature of 100° to 200°F. would be the point at which the high temperature oil would begin to function as a lubricant.

a. Super-Refined Mineral Oils and Hydrocarbons. A number of well refined mineral oils and hydrocarbons have been subjected to further refining by silica gel adsorption or hydrogenation over a nickel catalyst. The properties of various mineral oils and hydrocarbons before and after this additional treatment are shown on Table 2.

The silica gel treatment comprises percolation of approximately one gallon of each fluid through a column containing 28-200 mesh silica gel. The column is 84 inches high, 1.6 inches in diameter and holds 2000 grams of silica gel. The tower was maintained at a temperature of 150°F. by circulating hot water through a jacket surrounding the tower. The heating reduces the viscosity of the oils and consequently increases the rate of percolation through the silica gel. The heating method to reduce viscosity is preferred to dilution of the oils with a non-viscous solvent. The use of a solvent requires a prolonged stripping operation to recover the oil from the solvent. During the stripping operation the oil is susceptible to oxidation and contamination.

Approximately 70 per cent of the one gallon charge is recovered as product from the column. The remaining 30 per cent of the charge remains in the column as adsorbed material (aromatic and polar groups) and normal holdup. The fraction passing through the column has been evaluated as the silica gel treated fluid.

The mineral oils and hydrocarbons have been hydrogenated using 10 to 20 weight per cent U.O.P. nickel catalyst. The temperatures used range from 450° to 600°F. and the pressures from 600 to 1800 p.s.i. Hydrogenation time varies from 4 to 14 hours. Following the hydrogenation, the gaseous products from the hydrogenation of polar impurities as well as the excess hydrogen are vented from the system. The liquid product is removed from the catalyst by filtration.

Hydrogenation under these conditions tends to saturate olefinic and aromatic linkages and removes sulfur, oxygen, and nitrogen-containing impurities from the oil. The relative degree of severity of hydrogenation is indicated by the amount of hydrogen absorbed. This value is expressed in Table 2 as moles of hydrogen absorbed per 500 grams of oil. It can be seen that the amount of hydrogen absorbed by conventionally refined mineral oils of the naphthenic and paraffinic types is of the order of one to two moles of hydrogen per 500 grams of oil.

The catalytic cycle stock extract was hydrogenated to two degrees of saturation. In one case, a partial hydrogenation was carried to 2.5 moles of hydrogen per 500 grams of fluid. Catalyst poisoning stopped the hydrogenation at this point. The product from one of the partial hydrogenations was subjected to further hydrogenation over a new batch of U.O.P. nickel catalyst. The total hydrogen absorbed in the two

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step hydrogenation was 10.5 moles per 500 grams of oil.

The hydrogenation of the polyolefin hydrocarbons of the Indopol type proceeded smoothly under these conditions. In addition to the hydrogenated polyolefin prepared by this Laboratory, samples of the saturated Indopol have been obtained from the Standard Oil Company of Indiana.

Property evaluation after the super-refining treatments includes the measurement of viscosities, viscosity-temperature characteristics, pour points, thermal stability, oxygen tolerance and oxidation rate at 500° to 700°F., susceptibility to oxidation inhibitors at 347°F., corrosion characteristics at high temperatures, and deposit forming tendencies. For the conventionally refined naphthenic and paraffinic mineral oils, the data show little or no significant change in viscosity level or viscosity-temperature characteristics due to hydrogenation or silica gel percolation. The naphthenic mineral oil shows essentially no change in pour point due to these additional refining techniques. In general, the paraffinic mineral oils show a significant increase in pour point with hydrogenation. Hydrogenation of the polyolefin shows the same trends in viscosity level and viscosity-temperature characteristics as the conventionally refined mineral oil.

The hydrogenated cycle stock and lube oil extracts show a substantial lowering of viscosity level and pour point with hydrogenation. These changes are also accompanied by a substantial improvement in viscosity-temperature characteristics.

The changes in inhibitor susceptibility and high temperature behavior will be discussed in detail in subsequent sections. A brief summary of the effect of hydrogenation and silica gel adsorption on these properties is given here. Hydrogenation has shown little effect on the thermal stability, oxygen tolerance, and oxidation rate at or above 500°F. Preliminary results indicate that hydrogenation of mineral oils may reduce coking or deposit formation. This may be due primarily to the removal of polar impurities from the mineral oil.

Metal corrosion in high temperature thermal and oxidative tests is relatively very good for mineral oils compared with the other classes of fluids evaluated in this range. Hydrogenation tends to improve still further the corrosion characteristics of mineral oils at high temperatures. Hydrogenation and silica gel percolation improve substantially the susceptibility of mineral oils and hydrocarbons to oxidation and corrosion inhibitors at 347°F. or in the intermediate temperature range. It is in this range that Spec. MIL-L-7808 fluids currently enjoy the biggest quality margin over mineral oils. Hydrogenation is more effective than silica gel adsorption in improving both oxidation and corrosion characteristics in this intermediate temperature range.

A number of new conventionally refined, and several super refined, mineral oils have been obtained from commercial sources for inclusion in the high temperature program. These materials will be discussed in detail in the sections on inhibitor susceptibility and high temperature



stability properties. These properties are of primary interest for the evaluation of new high temperature lubricants and hydraulic fluids.

2. Hercules Synthetics J-20. A fluid (MLO 7047) designated as Synthetics J-20 has been received from the Hercules Powder Company. This material is believed to be a neopentyl glycol type ester. This fluid, MLO 7047, has been evaluated for thermal stability at 600° and 700°F., and for oxidation and corrosion stability at 500°F.

Thermal stability tests have been conducted at 600° and 700°F. in the glass thermal stability apparatus. These data are shown on Table 3. Similar data for Hercoflex 600, a pentaerythritol ester, are also included for purposes of comparison.

The results of these tests indicate that MLO 7047 has good thermal stability at both 600° and 700°F. Steel corrosion is obtained in the thermal stability tests at 600°F. with the metal catalysts, but no appreciable changes in viscosity or neutralization number are noted, and no volatility loss is incurred.

In the 700°F. thermal stability test, MLO 7047 shows some decrease in viscosity and an increase in neutralization number, but the loss due to volatility is substantially less than that incurred by Hercoflex 600 under comparable conditions.

MLO 7047 has also been evaluated for oxidation and corrosion stability at 500°F. An oxidation test has been conducted for a 40-hour test period with an air rate of five liters per hour using the conventional copper, steel, and aluminum metal catalysts. The results of this test are shown on Table 4. Comparable data for Hercoflex 600 have again been included for purposes of comparison.

The results of this oxidation test indicate that MLO 7047 has good oxidation and corrosion stability at 500°F. The rate of oxidation, that is, the amount of oxygen absorbed per unit time, for MLO 7047 appears to be comparable to that of dibasic acid esters and pentaerythritol esters evaluated previously. Evaluation of the fluid properties after completion of the test also indicates an oxygen tolerance of the same order of magnitude as that for a dibasic acid ester and a pentaerythritol ester. That is, the neutralization number increase, the formation of insolubles, and the volatility loss are about the same for the Synthetics J-20 and for Hercoflex 600. However, the viscosity increase for the Synthetics J-20 is somewhat less than for Hercoflex 600. A significant tendency to corrode steel is observed for both fluids. This steel corrosion in oxidation tests at 500°F. has been noted previously with dibasic acid esters and pentaerythritol esters.

3. Bis (1-Methyl Cyclohexyl Methyl) Sebacate. This ester (MLO 7106) has a neopentyl type configuration in the alcohol portion of the molecule. Previous studies have shown that esters containing this neopentyl configuration in the alcohol or polyhydroxy portion of the molecule exhibit improved thermal stability characteristics over simple dibasic

acid esters of the type used to prepare Spec. MIL-L-7808 jet engine oil. This sample of bis (1-methyl cyclohexyl methyl) sebacate has been supplied by the Materials Laboratory of the Wright Air Development Center under the sample designation of MLO 55-796, batch No. 2. Metal corrosion and thermal and oxidative stability of this ester have been measured and compared with a typical dibasic acid ester of the Spec. MIL-L-7808 type and several esters prepared from di- or tetrahydroxy alcohols and monobasic acids. These latter esters contain the neopentyl or substituted neopentyl configuration in the alcohol portion in the same fashion as the bis (1-methyl cyclohexyl methyl) sebacate.

Thermal stability data for these esters in the presence of metals are shown on Table 5 at 600°F. in the pressure cylinder and Table 6 at 600° to 700°F. in the glass apparatus. Data at 600°F. in the pressure cylinder in the presence of metals (bronze, 52-100 steel, and M-10 tool steel), indicate that the ester MLO 55-796 shows good thermal stability and metal corrosion characteristics. Data at 700°F. show the bis (1-methyl cyclohexyl methyl) sebacate ester to be about comparable in metal corrosiveness with the other improved stability esters. The mechanism of thermal deterioration differs for the two classes of improved stability esters evaluated. However, both the bis (1-methyl cyclohexyl methyl) sebacate and the other neopentyl type esters show thermal instability at 700°F. in the presence of metals. In all cases, stability at 700°F. is improved in the absence of metals.

It should be noted that the bis (1-methyl cyclohexyl methyl) sebacate, upon thermal decomposition at 700°F., produces a solid rubbery product with little volatility loss. Some of the other neopentyl type esters decompose thermally at 700°F. to form some volatile product with a substantial quantity of liquid products remaining. These residual liquid products show only moderate changes in physical properties. Synthetics J-20 is an example of the neopentyl diol and monobasic acid type ester which shows comparable or better thermal stability and corrosion characteristics than bis (1-methyl cyclohexyl methyl) sebacate in the 600° to 700°F. temperature range.

Oxidation and corrosion characteristics of the various esters are compared at 347°, 500°, and 600°F. on Tables 7, 8, and 9, respectively. A plot of the stable life at 347°F. is shown on Figure 1.

Oxidation stability of bis (1-methyl cyclohexyl methyl) sebacate and di-2-ethylhexyl sebacate are compared on Figure 1 and Table 7. These data indicate a good response to phenothiazine for the bis (1-methyl cyclohexyl methyl) sebacate at 347°F. The resultant stable life of 150 hours is typical of a high quality Spec. MIL-L-7808 ester of about two years ago. The di-2-ethylhexyl sebacate shown on Figure 1 is typical of the improved quality ester available currently.

Oxidation and corrosion characteristics at 500°F. for 20 hours are compared for three ester types on Table 8. Under these conditions, all of the esters have about the same rate of oxidation and oxygen tolerance. At 600°F. in the presence of metals, only neopentyl type esters



have been compared in a 20-hour oxidation and corrosion test. Under these conditions, as shown on Table 9, all of the esters show reduced oxygen tolerance, a high oxidation rate, and severe corrosive attack on steel. Again, as in the case of thermal stability, other neopentyl type esters show oxidation rates, oxygen tolerance, and metal corrosion properties which are at least as good as those of bis (1-methyl cyclohexyl methyl) sebacate.

4. Bis Tridecyl Sebacate. This ester (PRL 3600) is a typical dibasic acid ester of the type used in the preparation of Spec. MIL-L-7808 but has a somewhat higher molecular weight and viscosity level than di-2-ethylhexyl sebacate. The oxidation, corrosion, and thermal behavior of bis tridecyl sebacate have been determined and compared with that shown by di-2-ethylhexyl sebacate and pentaerythritol type esters. Thermal stability has been measured at 600° and 650°F. in the glass apparatus both with and without metals. Oxidation and corrosion characteristics have been determined at 347°, 500°, and 600°F. in a Spec. MIL-L-7808 type test. Thermal stability data are shown on Table 10. Oxidation and corrosion data are shown on Tables 11, 12, and 13.

In general, the bis tridecyl sebacate sample shows essentially the same thermal and oxidation characteristics as those shown by di-2-ethylhexyl sebacate under comparable test conditions. The higher viscosity level of the bis tridecyl sebacate does not measurably affect the basic stability pattern of the sebacate esters.

Some comparable data for pentaerythritol type esters are shown on Tables 10, 11, and 13. Hercoflex 600 and Synthetics J-7 have been used in this comparison. The pentaerythritol esters appear to be superior to the bis tridecyl sebacate in thermal stability and 600°F. oxidation studies.

5. Evaluation of Isosebacic Acid Esters. The Materials Laboratory, WCTRRL, of the Wright Air Development Center has requested that this Laboratory evaluate the thermal and oxidative stability of two isosebacic acid esters. The two esters are designated as MLO 7080 (di-2-ethylhexyl isosebacate) and MLO 7081 (diisooctyl isosebacate).

Thermal stability tests at 600°F. on these esters are shown on Table 14. Comparable data for di-2-ethylhexyl sebacate are also presented as a basis of comparison. These tests were conducted in the PRL glass thermal stability apparatus for a six-hour test period. Tests were conducted with and without metal catalysts for each of the fluids evaluated. Thermal stability of the isosebacate esters is in all cases essentially the same as for the esters of sebacic, azelaic, and adipic acids. That is, the chain branching in the dibasic acid does not, in this case, affect materially the thermal stability. The isosebacates, like the other diesters evaluated, do cause severe steel corrosion in the 600°F. thermal stability test. The phenomenon of high temperature steel corrosion by dibasic acid esters and pentaerythritol esters in thermal and oxidative tests is discussed in Annual Report WADC TR 55-30 Pt 4.

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Oxidation and corrosion tests with the isosebacates are shown on Tables 15, 16, and 17, and Figure 2. Conventional Spec. MIL-L-7808 oxidation and corrosion tests for di-2-ethylhexyl sebacate and the two isosebacate esters are shown on Table 15. All of the esters contain 0.5 weight per cent phenothiazine as the oxidation inhibitor. These data indicate that the two isosebacate esters fail the specification test in neutralization number increase. The neutralization number increase of the isosebacate is about twice that of the di-2-ethylhexyl sebacate. All other property changes are within the specification limits.

Stable life test data at 347°F. for these esters are shown on Table 16 and Figure 2. In the case of the isosebacate esters, no sharp break in the curve of neutralization number increase with time was noted. The tests were terminated after 210 hours. At this point the neutralization numbers of the two isosebacates were 6.5 and 7.2. Values of this order of magnitude are obtained after the induction period for the conventional dibasic acid esters studied. Di-2-ethylhexyl sebacate is typical of this behavior. The data indicate that there is a sharp rise in neutralization number and other property changes following the induction period. This period of rapid property change for di-2-ethylhexyl sebacate, as shown on Figure 2, is between 200 to 214 hours. At the end of 210 hours the isosebacate esters do not show evidence of exceeding the stable life in the changes in physical properties noted. The high neutralization number noted does not result in metal corrosion in this test. It appears that neutralization number increase is a severe limitation in the use of phenothiazine-inhibited isosebacic acid esters as base materials for the preparation of Spec. MIL-L-7808 lubricants.

Oxidation and corrosion data for the isosebacic acid esters at 500°F. are shown on Table 17. There are no 500°F. oxidation and corrosion requirements in Spec. MIL-L-7808. Since oil film temperatures of this order of magnitude in the presence of air are probably encountered in the use of this lubricant, comparative oxidation and corrosion data at 500°F. are considered pertinent.

The 500°F. oxidation and corrosion tests were conducted for a period of 20 hours using the test procedures and techniques of the Spec. MIL-L-7808 oxidation and corrosion test. The rate of oxidation of all three esters is essentially the same under these test conditions. The oxygen tolerance of the isosebacates appears to be somewhat poorer than that shown by di-2-ethylhexyl sebacate. That is, for approximately the same oxygen absorption, property changes such as insolubles formation and metal corrosion are more severe in the case of the isosebacates.

On the basis of this evaluation, it would appear that these isosebacate esters offer no particular advantage and may have some disadvantages relative to esters of the di-2-ethylhexyl sebacate type for use in Spec. MIL-L-7808 fluids.

6. Phosphate Esters. Two phosphate type esters have been received from the Wright Air Development Center (WADC) for high temperature evaluation. These fluids are designated by the WADC as MLO 9574

and MLO 9553. MLO 9574 is diphenyl (o-chlorophenyl) phosphate, and MLO 9553 is tris (meta-chlorophenyl) phosphate.

These two phosphate esters have been evaluated for oxidation and corrosion stability at 500° and 600°F., and for thermal stability at 600°F. In addition, the high temperature viscosity properties for these esters have also been determined. This is essentially the same procedure used to evaluate earlier samples of phosphate type esters. These earlier evaluations are discussed in Annual Report WADC TR 55-30 Pt 4.

The composition and viscosity properties of these materials are shown on Table 18. The viscosity levels of these two materials are somewhat lower for a given temperature than the phosphate esters evaluated previously. The viscosity-temperature characteristics of all of the chlorine-containing phosphate esters evaluated are of about the same order of magnitude. In all cases the viscosity levels at both 500° and 700°F. are quite low. In addition, the ASTM slopes are relatively poor for these fluids when compared to some of the silicates and esters made from dibasic acids.

The oxidation and corrosion tests conducted at 500° and 600°F. are summarized on Tables 19 and 20. The 500°F. tests were conducted for a 60-hour test period, and the 600°F. tests were conducted for a 20-hour test period. In all cases, the air rate was five liters per hour, and the conventional copper, steel, and aluminum metal catalysts were used.

The results of the tests at 500°F. indicate that both phosphate fluids show severe oxidative deterioration and cause excessive metal corrosion. In the case of MLO 9533, the aluminum test specimen was completely removed by corrosion. The tests conducted at 600°F. also show severe oxidative deterioration and metal corrosion. In both the 500° and 600°F. oxidation tests, MLO 9553 and MLO 9574 show the same general trends as the phosphate esters evaluated previously. That is, these phosphate esters show a relatively low rate of oxidation, but a very poor oxygen tolerance as compared with diesters, mineral oils, and silicates.

The thermal stability tests conducted at 600°F. are shown on Table 21. These tests were conducted in the conventional glass thermal stability apparatus for a six-hour test period using copper, steel, and aluminum catalysts.

The results of these thermal stability tests indicate that both phosphates show evidence of incipient metal corrosion and thermal instability under these test conditions. These results are also in good agreement with thermal stability tests conducted previously on chlorine-containing phosphate esters. That is, previous thermal stability tests show that esters of this type have relatively good thermal stability at both 600° and 700°F. when the tests are conducted in an all glass system. However, in the presence of metals these esters show some fluid deterioration and metal corrosion at both 600° and 700°F.

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7. Evaluation of Two Kendall Resins. Two high molecular weight resins (MLO 7072 and MLO 7073) obtained from Pennsylvania crude oil and supplied by the Kendall Refining Company have been evaluated for thermal and oxidation stability. MLO 7072 is designated as a light resin and MLO 7073 is a heavy resin. Resins of this type are of interest as mineral oil thickening agents for high temperature applications because they appear to be more stable thermally than the synthetic polymers (polybutenes, polyesters, and Acryloids) used conventionally as thickening agents and viscosity-index improvers.

The blending efficiency and thermal stability for a petroleum resin have been discussed and compared with conventional polymer thickeners previously. These data are found in Annual Report WADC TR 55-30 Pt 4.

The oxidation and corrosion characteristics for the two Kendall resins have been determined at 347° and 500°F. The 347°F. tests were conducted for the standard 72-hour test period using Spec. MIL-L-7808 test procedure and techniques. These tests were conducted using an air rate of five liters per hour and copper, steel, aluminum, and magnesium catalysts. The results of these tests are summarized of Table 22.

Both of the resins show reasonably good oxidation stability under these conditions. A substantial increase in viscosity (69 per cent for MLO 7072 and 137 per cent for MLO 7073) is obtained, but the volatility loss, neutralization number increase, and the amount of sludge formed are low for both fluids. No significant metal corrosion is obtained in these tests.

The 500°F. tests were conducted for 20 hours at an air rate of five liters per hour using copper, steel, and aluminum catalysts. These results are found on Table 23. Again, both resins show good oxidation stability under these test conditions. The rate of oxidation is low for both MLO 7072 and MLO 7073 and the fluid deterioration is not severe in either case. No metal corrosion is obtained for either fluid under these conditions at 500°F.

Thermal stability tests have been conducted at 600° and 700°F. with MLO 7072 and MLO 7073. The thermal stability tests at 600°F. were conducted in the glass apparatus. The tests at 700°F. were conducted in both the glass apparatus and in the stainless steel pressure cylinder. In the pressure cylinder tests at 700°F., metal catalysts which consisted of a one-half inch diameter ball bearing each of 52-100 bearing steel, M-10 tool steel, and Naval bronze were used. The thermal stability tests were conducted for a six-hour test period. The results of these tests are found on Table 24.

The thermal stability tests at 600°F. show that the two Kendall resins have reasonably good thermal stability at this temperature. In each case no loss due to the formation of volatile decomposition products is incurred and the viscosity decrease is moderate. MLO 7072 shows a viscosity decrease of 16 per cent and MLO 7073 a decrease of 14 per cent during the six-hour test.



The thermal stability of the resins is somewhat poorer at 700°F. In the glass thermal stability tests, both resins have about eight per cent loss due to the formation of volatile decomposition products, and the decrease in viscosity was greater than 90 per cent for both materials. Similarly, in the pressure cylinder MLO 7072 could not be run for the six-hour test period because the gas pressure became too high. This test had to be terminated after a two hour test period. At this time the system pressure was 280 p.s.i. The safe limit for the stainless steel pressure cylinder is considered to be 300 p.s.i.

The six-hour test period in the pressure cylinder was completed with the heavy resin (MLO 7073). Both fluids show the same large decrease in viscosity (greater than 90 per cent) in the pressure cylinder that was obtained in the glass thermal stability apparatus at 700°F. No serious amount of catalyst corrosion is obtained for the metals in the thermal stability tests at 700°F.

The results of these thermal stability tests for the two Kendall resins indicate that these materials are not as stable thermally at 700°F. as lower molecular weight paraffinic oils. The relative thermal stability of a series of paraffinic mineral oils refined from a Pennsylvania crude oil are listed in order of decreasing thermal stability which also lines up the oils in the order of increasing molecular weight and boiling point: light neutral > heavy neutral > bright stock > resin fractions. However, these resins are somewhat more stable thermally than the synthetic polymers used as thickening agents and viscosity-index improvers. These materials are reasonably stable at 600°F., whereas polyesters and Acryloids exhibit incipient thermal instability at about 500°F. Polyisobutylene becomes unstable at about 600°F., and is extremely unstable at 700°F.

It is of interest to note that, in general, with naphthenic or paraffinic mineral oils, an increase in molecular weight or viscosity grade will result in a decrease in dirt-forming tendencies under severe high temperature oxidation conditions. There is some evidence available in previous reports to indicate that the same generality may be applied to simple, complex, and polyesters of increasing molecular weight.

C. OXIDATION AND CORROSION STUDIES AT 347°F. A large number of oxidation and corrosion tests have been conducted under essentially Spec. MIL-L-7808 test conditions with conventionally refined mineral oils. Under these test conditions, the esters have shown a marked superiority over conventionally refined mineral oils for use in Spec. MIL-L-7808 jet engine oil and Spec. MIL-L-6387 hydraulic fluid. The use of super-refined (hydrogenated) mineral oils and hydrocarbons has improved additive susceptibility substantially. Oxidation inhibitors have, therefore, been re-evaluated in hydrogenated mineral oils and hydrocarbons. The more effective additives have also been evaluated in mineral oils refined by other special techniques.

1. Effect of Air Rate on 347°F. Oxidation. All tests reported in previous annual reports have been conducted at an air rate of  $10 \pm 1$

*Contrails*

Table 1  
VISCOSITY PROPERTIES OF A SERIES OF PARAFFINIC MINERAL OILS

Paraffinic Mineral Oil Fraction	Centistoke Viscosity at				Temperature in °F. at which Viscosity equals 13,000 Centistokes	ASTM Slope 100° to 210°F.	Viscosity Index
	100°F.	210°F.	490°F.	707°F.			
Light Neutral	12.7	3.0	0.7	-	-57	0.776	100
Medium Neutral	36.3	5.64	0.9	0.5	-32	0.749	103
Heavy Neutral	107	11.2	1.3	0.6	0	0.706	98
Bright Stock	467	31.5	2.5	1.0	27	0.632	104
Light Resin	5,860	173	6.4	2.3	84	0.573	-
Heavy Resin	16,180	332	9.3	3.2	105	0.563	-

Table 2

PROPERTIES OF SEVERAL FLUIDS BEFORE AND AFTER SUPER-REFINING PROCEDURES

TEST FLUID	HYDROGENATION CONDITIONS			CENTISTOKE VISCOSITY AT		ASTM SLOPE	VISCOSITY INDEX	POUR POINT, °F.
	MAX. TEMP., °F.	MAX. PRESSURE, P.S.I.G.	MOLS H <sub>2</sub> PER 500 GMS. OIL	210°F.	100°F.			
PRL 2782	-	-	-	5.73	44.7	0.802	65	-25
-	-	-	-	5.30	36.7	0.787	78	-20
PRL 2052	-	-	-	7.19	65.2	0.787	68	-15
MLO 7066	450	600	0.45	7.15	64.3	0.786	69	-15
MLO 7097	500	630	0.86	6.99	61.6	0.785	70	-
MLO 7096	470	610	1.32	7.15	63.2	0.781	72	-
MLO 7027	-	-	-	4.94	27.6	0.736	116	+30
-	-	-	-	-	-	-	-	+35
MLO 7026	-	-	-	8.27	65.2	0.718	104	+5
-	-	-	-	7.53	53.7	0.711	111	+10
MLO 7026	-	-	-	12.2	113	0.682	106	+5
MLO 7077	500	500	1.13	11.2	97.2	0.683	108	+35
MLO 7076	-	-	-	5.56	33.6	0.733	113	+15
MLO 7093	514	1590	1.56	5.44	32.0	0.730	117	-
MLO 7113	-	-	-	31.5	467	0.632	104	-
MLO 7094	514	1520	1.77	28.6	397	0.632	105	-
MLO 7118	-	-	-	11.8	113	0.693	101	-40
MLO 7014	479	1300	1.90	11.9	106	0.675	109	-30
MLO 7101	-	-	-	-	-	-	-	-
MLO 7089	-	-	-	7.54	178	0.998	-206	+25
MLO 7103	613	2010	1.84	5.74	88.5	0.981	-160	+15
MLO 7079	615	1450	2.52	5.78	89.9	0.981	-162	-
MLO 7087	650	2170	10.52(2)	4.51	44.0	0.932	-116	-5
MLO 7092	-	-	-	15.6	578	0.910	-124	+30
MLO 7091	606	2290	9.37(2)	9.31	151	0.864	13	-5

(1) OIL PASSED THROUGH A COLUMN 1.6 INCHES IN DIAMETER AND 7 FEET HIGH CONTAINING ACTIVATED SILICA GEL AT APPROX. 150°F. (OIL TO SILICA GEL RATIO APPROX. 2 TO 1).

(2) HYDROGENATION CARRIED OUT IN TWO STAGES. THAT IS, AFTER PARTIAL HYDROGENATION CATALYST WAS REPLACED BY FRESH CATALYST AND HYDROGENATION RESUMED. VALUE INDICATES TOTAL HYDROGEN ABSORPTION.

Table 3

## THERMAL STABILITY OF HERCULES SYNTHETICS J-20 AT HIGH TEMPERATURES

TESTS CONDUCTED IN GLASS TEST TUBES FOR A 6 HOUR TEST PERIOD USING A TWENTY-FIVE GRAM CHARGE OF TEST FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 0.25 LB./SQ. INCH. ONE INCH SQUARE SPECIMENS OF METAL CATALYSTS HAVE BEEN USED AS INDICATED.

TEST FLUID	TEST TEMP., °F.	CENTISTOKE VISC. AT 100°F.			% LOSS IN WT. (1)	NEUT. NO. (MG. KOH/GM. OIL)		CATALYST WT. LOSS (MG./SQ. CM.)	
		ORIG.	FINAL	% CHANGE		ORIG.	FINAL	COPPER	STEEL
HERCULES SYNTHETICS J-20	600	9.57	9.40	-2	0.0	0.1	3.0	-	-
	600	9.57	9.44	-2	0.0	0.1	4.5	0.02	0.29
HERCOFLEX 600	700	9.57	7.92	-17	4.0	0.1	10.6	-	-
	600	22.1	22.4	+1	0.0	0.1	2.1	-	-
	600	24.5	23.5	-4	2.0	0.1	15.6	0.00	1.92
	700	22.1	27.1	+23	16.0	0.1	6.8	-	-

(1) PER CENT LOSS IN WEIGHT IS GOOD TO  $\pm 2.0$  % OR 0.5 GRAMS.



Table 4

## OXIDATION AND CORROSION CHARACTERISTICS OF HERCULES SYNTHETICS J-20 AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 40 HOURS; AIR RATE = 5 + 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

NOTE: ALL FLUIDS CONTAIN 0.5 WT.% PHENOTHIAZINE.

TEST FLUID (COMPOSITION IN WT.%)	DI-2-ETHYLHEXYL SEBACATE	SYNTHETICS J-20	HERCOFLEX 600
LIQUID CHARGED, GRAMS	90	90	97
LIQUID LOSS, WT.%	5	13	15
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS.(1)	52.4	52.4	52.4
APPROX. AMOUNT O <sub>2</sub> USED, GMS.(1)	11.6	12.9	13.7
MOLS O <sub>2</sub> USED/426 GRAMS OF FLUID(2)	1.72	1.92	1.95
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL(3)			
AT 130°F.	+164	+272	+4550
AT 0°F.	+328	+900	SOLID
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.1	0.1	0.2
FINAL	17.1	15.4	17.7
WT.% OIL INSOLUBLE MATERIAL(3)	0.9	0.7	1.7
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	CORRODED	DULL	DULL
STEEL	DULL	CORRODED	CORRODED
ALUMINUM	BRIGHT	DULL	DULL
WT. LOSS (MG./SQ. CM.)			
COPPER	0.75	0.16	0.13
STEEL	0.09	1.17	12.05
ALUMINUM	0.00	+0.05	0.03

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 5

## THERMAL STABILITY OF SEVERAL ESTERS IN THE PRESSURE CYLINDER AT 600°F.

ALL TESTS CONDUCTED IN THE STAINLESS STEEL PRESSURE CYLINDER AT  $600 \pm 10^\circ\text{F}$ ; TEST TIME = 6 HOURS; TEST FLUID CHARGED = 20 ML.; TOTAL VOLUME OF CYLINDER IS 46 ML.; CATALYST = A 0.5 INCH DIAMETER BALL BEARING EACH OF M-10 TOOL STEEL, 52-100 STEEL, AND NAVAL BRONZE. PRIOR TO THE START OF THE TEST THE SYSTEM IS PURGED WITH NITROGEN. THE SYSTEM IS THEN SEALED AND THE TEST STARTED. THE NITROGEN PRESSURE IN THE PRESSURE CYLINDER IS ABOUT 20 P.S.I. AT 600°F.

TEST FLUID	CENTISTOKE VISCOSITY AT 100°F.		CATALYST WT. LOSS, MG./SQ. CM.		NEUT. NO. (MG. KOH/GM. OIL)		MAX. SYSTEM PRESSURE, P.S.I.	FINAL PRESSURE AT ROOM TEMP., P.S.I.
	ORIG.	FINAL	% CHANGE	M-10 STEEL	52-100 STEEL	BRONZE	ORIGINAL	FINAL
BIS (1-METHYL CYCLOHEXYL METHYL) SEBACATE (MLO 55-796, BATCH NO. 2)	47.6	45.5	-4	0.00	0.46	0.20	0.1	2.1
DI-2-ETHYLHEXYL SEBACATE	12.7	12.4	-2	+2.48	7.76	+3.46	0.1	19.5
HERCOFLEX 600 (PRL 3542)	18.9	31.7	+68	13.72	18.66	0.42	0.1	62.5
							22	5
							156	36
							265	87

Table 6

## THERMAL STABILITY OF SEVERAL ESTERS IN THE GLASS APPARATUS AT HIGH TEMPERATURES

TESTS CONDUCTED IN GLASS THERMAL STABILITY APPARATUS. GLASS TUBES ARE SEALED WITH A U TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF AIR IN OR GAS OUT WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID -- APPROXIMATELY 0.25 LB./SQ. INCH. FLUID CHARGED IS 25 GRAMS FOR TESTS WITHOUT CATALYSTS AND 50 GRAMS FOR TESTS WITH CATALYSTS.

TEST FLUID	TEST TEMP., OF.	TEST TIME, HRS.	CENTISTOKE VISCOSITY AT 100°F.	% LOSS IN WEIGHT (1)	NEUT. NUMBER (MG. KOH/GM. OIL)	CATALYST WT. LOSS (MG./SQ. CM.)	WT. % INSOL. MATERIAL
BIS(1-METHYL CYCLOHEXYL METHYL) SEBACATE (MLO 55-796, BATCH #2)	600	6	47.6	0	0.1	0.02	NONE
	650	6	47.6	1	0.1	0.02	TRACE
	700	6	47.6	0	0.1	-	NONE
	700	6	47.6	66	0.1	0.01	-
	700	6	47.6	50	0.1	+0.03	-
DI-2-ETHYLHEXYL SEBACATE	600	6	12.7	13	0.1	-	NONE
	600	6	12.6	2	0.1	0.02	1.1
	650	6	12.7	6	0.1	7.00	-
HERCOFLEX 600	600	6	22.1	0	0.1	-	-
	600	6	24.5	2	0.1	-	-
	650	6	22.1	0	0.1	0.00	-
	650	6	22.1	16	0.1	-	-
	700	6	27.1	16	0.1	-	-
HERCULES SYNTHETICS J-20	600	6	9.57	0	0.1	-	-
	600	6	9.57	0	0.1	0.02	-
	700	6	9.57	4	0.1	0.29	-
	700	6	8.28	30	0.1	0.05	-

(1) LOSS IN WEIGHT IS GOOD TO  $\pm 2$  PER CENT OR 0.5 GRAMS

(2) RESIDUE AFTER TEST IS SOLID. NEUTRALIZATION NUMBER COULD NOT BE DETERMINED.

Table 7

OXIDATION STABILITY OF BIS (1-METHYL CYCLOHEXYL METHYL) SEBACATE AT 347°F.

Test procedure and techniques in accordance with Spec. MIL-L-7808.

Test conditions include: Test temperature =  $347 \pm 3^\circ\text{F}$ .; Test time as indicated; Air rate =  $10 \pm 1$  liters per hour; Test fluid charged = 100 ml.; Catalyst = a 1 inch square each of copper, steel, aluminum, and magnesium.

Test Fluids: MLO 7106 = Bis (1-Methyl Cyclohexyl Methyl) Sebacate  
(MLO 55-796, Batch No. 2).

MLO 7038 = Di-2-Ethylhexyl Sebacate

Both fluids contain 0.5 wt. per cent phenothiazine.

Test Fluid	MLO 7106	MLO 7038
Test time, hours	192	280
Approx. stable life, hours(1)	160	270
% Change in Centistoke Viscosity		
At 130°F.	+10	+11
At 0°F.	+14	-
ASTM Union Color		
Original	1	2
Final	> 8	> 8
Neut. No. (mg. KOH/gm. oil)		
Original	0.1	0.2
Final	4.2	8.5
Wt.% Insoluble Material	0.4	1.2
Final Catalyst Condition		
Appearance		
Copper	corroded	dull
Steel	dull	dull
Aluminum	dull	coated
Magnesium	dull	dull
Wt. Loss (mg./sq. cm.)		
Copper	0.34	0.03
Steel	+0.08	+0.04
Aluminum	+0.08	+0.11
Magnesium	+0.08	+0.03

(1) The stable life of a fluid is taken as the point at which there is a rapid increase in neutralization number with test time.

Table 8

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL ESTERS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7106 = BIS (1-METHYL CYCLOHEXYL METHYL) SEBACATE (MLO 55-796, BATCH NO. 2).  
 PRL 3483 = DI-2-ETHYLHEXYL SEBACATE.  
 MLO 7018 = HERCULES SYNTHETICS J-7.

ALL FLUIDS CONTAIN 0.5 WT. PER CENT PHENOTHIOZINE.

TEST FLUID	MLO 7106	PRL 3483	MLO 7018
LIQUID CHARGED, GRAMS	95	91	98
LIQUID LOSS, WT. %	1	7	4
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	3.8	5.3	4.7
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	0.53	0.86	0.65
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3)			
AT 130°F.	+39	+44	+51
AT 0°F.	+90	+48	+126
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.0	0.1	0.1
FINAL	8.3	15.4	13.5
WT. % OIL INSOLUBLE MATERIAL (3)	0.2	0.1	TRACE
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	CORRODED	DULL	DULL
STEEL	DULL	DULL	COATED
ALUMINUM	BRIGHT	BRIGHT	DULL
WT. LOSS (MG./SQ. CM.)			
COPPER	0.24	0.10	0.12
STEEL	0.00	0.07	+0.57
ALUMINUM	+0.02	0.00	+0.02

- (1) AMOUNT O<sub>2</sub> SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) x TIME (HR.) x O<sub>2</sub> CONTENT (FRACTION) x 1.43 (GM./LITER). AMOUNT O<sub>2</sub> CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS DETERMINED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 9

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL ESTERS AT 600°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7106 = BJS (1-METHYL CYCLOHEXYL METHYL) SEBACATE (MLO 55-796, BATCH #2)

PRL 3542 = HERCOFLEX 600

MLO 7018 = HERCULES SYNTHETICS J-7

MLO 7047 = HERCULES SYNTHETICS J-20

ALL FLUIDS CONTAIN 0.5 WT. PER CENT PHENOTHIAZINE.

TEST FLUID	MLO 7106	PRL 3542	MLO 7018	MLO 7047
LIQUID CHARGED, GRAMS	96	98	98	90
LIQUID LOSS, WT. %	18	20	6	25
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	9.9	8.1	9.4	-
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	1.38	1.09	1.28	-
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	-(4)	+2320	+87	+4900
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL	0.0	0.1	0.1	0.1
FINAL	14.9	20.9	22.2	8.4
WT. % OIL INSOLUBLE MATERIAL (3)	-(4)	3.5	2.4	3.6
FINAL CATALYST CONDITION				
APPEARANCE	CORRODED	DULL	DULL	DULL
COPPER	CORRODED	CORRODED	CORRODED	CORRODED
STEEL	BRIGHT	DULL	DULL	DULL
ALUMINUM				
WT. LOSS (MG./SQ. CM.)				
COPPER	0.23	0.10	0.02	0.03
STEEL	13.40	5.78	5.23	5.57
ALUMINUM	0.02	0.01	0.00	0.02

(1) AMOUNT O<sub>2</sub> SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT O<sub>2</sub> CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS DETERMINED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF OIL INSOLUBLE MATERIAL AND OXIDIZED OIL. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.

Figure 1

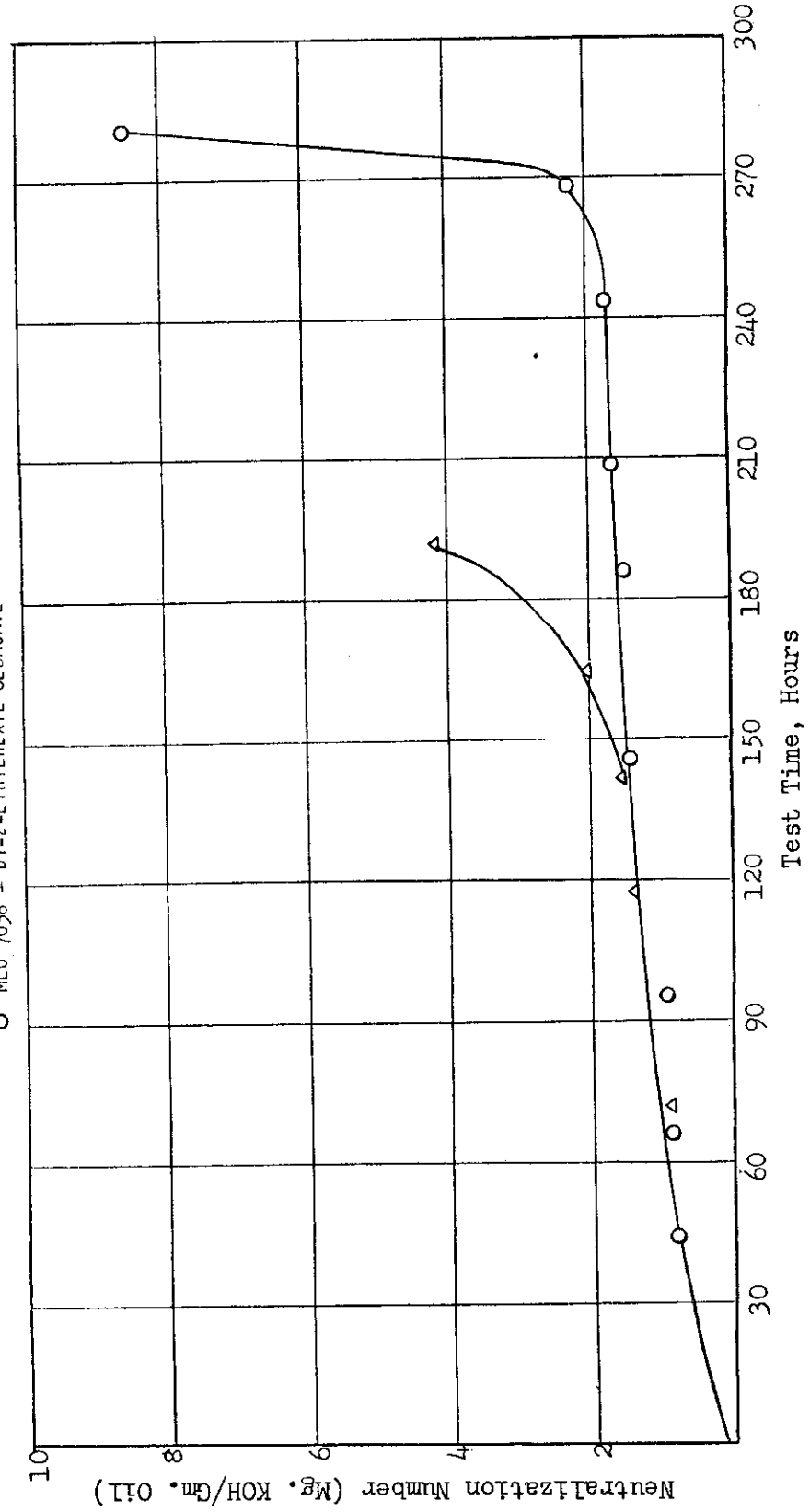
OXIDATION STABILITY OF BIS (1-METHYL CYCLOHEXYL METHYL) SEBACATE AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME AS INDICATED; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A ONE-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

ALL FLUIDS CONTAIN 0.5 WEIGHT PER CENT PHENOTHIAZINE.

Δ MLO 7106 = BIS (1-METHYL CYCLOHEXYL METHYL) SEBACATE  
○ MLO 7038 = DI-2-ETHYLHEXYL SEBACATE





Contrails

Table 10  
THERMAL STABILITY OF SEVERAL ESTERS AT HIGH TEMPERATURES

ALL TESTS ARE CONDUCTED IN GLASS TEST TUBES. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF AIR IN OR GAS OUT WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 0.25 LB./SQ. IN. TEST CONDITIONS INCLUDE: TEST TEMPERATURE AS INDICATED; TEST TIME = 6 HOURS; CATALYSTS = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM WHERE INDICATED.

TEST FLUID	TEST TEMP., °F.	CENTISTOKE VISC. AT 100°F.		% LOSS IN WEIGHT	CATALYST WT. LOSS (MG./SQ. CM.)			NEUT. NO. (MG. KOH/GM. OIL)		WT. % INSOLUBLE MATERIAL
		ORIG.	FINAL		COPPER	STEEL	ALUMINUM	ORIG.	FINAL	
BIS TRIDECYL SEBACATE	600	39.0	27.2	0	-	-	-	0.1	32.2	-
	600	39.0	29.4	0	0.01	8.07	+1.22	0.1	20.7	0.7
	650	39.0	SOLID	30	-	-	-	0.1	133	-
DI-2-ETHYLHEXYL SEBACATE	600	12.6	11.3	2	-	-	-	0.1	25.3	-
	600	12.6	10.7	2	0.02	7.00	+2.08	0.1	11.4	1.1
	650	12.6	SOLID	6	-	-	-	0.1	126	-
HERCOFLEX 600	600	24.5	23.4	0	-	-	-	0.1	7.7	NONE
	600	24.5	23.5	2	0.00	1.92	0.00	0.1	15.6	TRACE
	650	22.1	23.1	0	-	-	-	0.1	14.2	-

Table 11

OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL ESTERS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID (COMPOSITION IN WT.%)	BIS TRIDECYL SEBACATE + 0.5 PHENOTHIAZINE	D1-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE	HERCOFLEX 600 +0.5 PHENOTHIAZINE	HERCULES SYNTHETICS J-7 + 0.5 PHENOTHIAZINE
LIQUID LOSS, WT.%	2	3	3	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F. AT 0°F.	+5 +5	+3 +7	+5 +12	+8 +15
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 1.5	0.1 1.5	0.1 0.3	0.1 0.4
WT. % OIL INSOLUBLE MATERIAL	0.3	0.3	0.4	0.3
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM MAGNESIUM	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL BRIGHT BRIGHT
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM MAGNESIUM	+0.05 +0.03 +0.03 +0.03	+0.02 +0.03 +0.02 +0.01	+0.04 +0.09 +0.09 +0.09	0.01 0.01 +0.01 +0.08

Table 12

OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL ESTERS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

ALL FLUIDS CONTAIN 0.5 WT.% PHENOTHIAZINE.

TEST FLUIDS: PRL 3103 = DI-2-ETHYLHEXYL SEBACATE

PRL 3600 = BIS TRIDECYL SEBACATE

TEST FLUID	PRL 3103	PRL 3600
LIQUID CHARGED, GRAMS	91	88
LIQUID LOSS, WT.%	7	2
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS.(1)	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS.(1)	5.3	4.6
MOLS O <sub>2</sub> USED/426 GRAMS FLUID(2)	0.86	0.69
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL(3)		
AT 130°F.	+44	+57
AT 0°F.	+88	+174
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	0.1	0.1
FINAL	15.4	13.1
WT.% OIL INSOLUBLE MATERIAL(3)	0.1	0.2
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	DULL	DULL
STEEL	DULL	COATED
ALUMINUM	BRIGHT	DULL
WT. LOSS (MG./SQ. CM.)		
COPPER	0.10	0.15
STEEL	0.07	+2.44
ALUMINUM	0.00	0.02

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING THE OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 13

OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL ESTERS AT 600°F.

TEST PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600 + 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 + 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM WHERE INDICATED.

TEST FLUID (COMPOSITION IN WT.%)	BIS TRIDECYL SEBACATE + 0.5 PHENOTHIAZINE	DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE	HERCOFLEX 600 + 0.5 PHENOTHIAZINE	HERCULES SYNTHETICS J-7 + 0.5 PHENOTHIAZINE
LIQUID CHARGED, GRAMS	87	91	98	98
LIQUID LOSS, WT.%	32	33	20	6
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	13.2	12.9	8.1	9.4
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (4)	2.02	1.93	1.09	1.28
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (2) AT 130°F.	SOLID	+612	+2320	+87
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.2 10.8 -(3)	0.1 82.0 3.2	0.1 20.9 3.5	0.1 22.2 2.4
WT. % OIL INSOLUBLE MATERIAL (2)				
FINAL CATALYST CONDITION				
APPEARANCE	CORRODED CORRODED BRIGHT	- - -	DULL CORRODED DULL	DULL CORRODED DULL
WT. LOSS (MG./SQ. CM.)				
COPPER	0.23	-	0.10	0.02
STEEL	26.90	-	5.78	5.23
ALUMINUM	0.02	-	0.01	0.00

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.
- (3) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF INSOLUBLE MATERIAL AND OXIDIZED FLUID. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.
- (4) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

Table 14

## THERMAL STABILITY OF SEVERAL DIBASIC ACID ESTERS AT 600°

ALL TESTS WERE CONDUCTED IN GLASS TEST TUBES. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID -- APPROXIMATELY 0.25 LB./SQUARE INCH. TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600 ± 5°F.; TEST TIME = 6 HOURS; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM AS INDICATED.

TEST FLUID	CENTISTOKE VISCOSITY AT 100°F.			% LOSS IN WEIGHT (1)	CATALYST WT. LOSS (MG./SQ. CM.)			NEUT. NO. (MG. KOH/GM. OIL)		WT. % INSOLUBLE MATERIAL
	ORIGINAL	FINAL	% CHANGE		COPPER	STEEL	ALUMINUM	ORIGINAL	FINAL	
MLO 7080 MLO 7080	12.2 12.2	9.69 12.0	-22 -2	2 8	0.05 0.05	17.85 17.85	- +0.05	0.1 0.1	53.0 35.6	- 0.4
MLO 7081 MLO 7081	13.7 13.7	12.1 15.3	-11 +12	4 6	0.07 0.07	24.13 24.13	- +0.43	0.1 0.1	75.8 42.4	- 1.1
PRL 3483 PRL 3483	12.6 12.6	11.3 10.7	-10 -15	2 2	0.02 0.02	7.00 7.00	- +2.08	0.1 0.1	25.3 11.4	- 1.1

(1) PER CENT LOSS IN WEIGHT IS GOOD TO ±2.0% OR 0.5 GRAMS.

Table 15

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL DIBASIC ACID ESTERS AT 347°F.

Test Procedure and Techniques in Accordance with Spec. MIL-L-7808.  
 Test Conditions Include: Test Temperature = 347±3°F.; Test Time = 72  
 Hours; Air Rate = 10±1 Liters per Hour; Test Fluid Charged = 100  
 ml.; Catalyst = a 1 Inch Square Each of Copper, Steel, Aluminum,  
 and Magnesium.  
 Test Fluids: MLO 7080 = Di-2-Ethylhexyl Isebacate  
                   MLO 7081 = Diisooctyl Isebacate  
                   PRL 3207 = Di-2-Ethylhexyl Sebacate  
 All Fluids Contain 0.5 wt.% Phenothiazine

Test Fluid	MLO 7080	MLO 7081	PRL 3207
Overall Liquid Loss, wt.%	1	1	3
% Change in Centistoke Viscosity			
at 130°F.	+3	+3	+3
at 0°F.	+6	+7	+7
Neut. No. (mg. KOH/gm. oil)			
Original	0.1	0.1	0.1
Final	2.7	3.0	1.5
A.S.T.M. Union Color			
Original	1	1	2
Final	>8	>8	>8
Wt.% Insoluble Material	0.3	0.4	0.3
Final Catalyst Condition			
Appearance			
Copper	Coated	Dull	Dull
Steel	Coated	Coated	Dull
Aluminum	Coated	Dull	Dull
Magnesium	Coated	Dull	Dull
Wt. Loss (mg./sq. cm.)			
Copper	+0.09	+0.05	+0.02
Steel	+0.17	+0.09	+0.03
Aluminum	+0.11	+0.07	+0.02
Magnesium	+0.16	+0.07	+0.01

OXIDATION CHARACTERISTICS OF SEVERAL ESTERS AT 347°F.

Test Procedure and Techniques in Accordance with Spec. MIL-L-7808.

Test Conditions Include: Test Temperature =  $347 \pm 3^\circ\text{F}.$ ; Test Time = as Indicated; Air Rate =  $10 \pm 1$  Liters per Hour; Test Fluid Charged = 100 ml.; Catalyst = a 1 inch Square Each of Copper, Steel, Aluminum, and Magnesium.

Test Fluids: MLO 7080 = Di-2-Ethylhexyl Isebacate

MLO 7081 = Diisooctyl Isebacate

PRL 3483 = Di-2-Ethylhexyl Sebacate

All Fluids Contain 0.5 wt.% Phenothiazine as an Oxidation Inhibitor.

Test Fluid	MLO 7080	MLO 7081	PRL 3483
Test time, hours	210	210	214
% Change in Centistoke Viscosity			
at 130°F.	+6	+6	+15
at 0°F.	+42	+42	+37
Neut. No. (mg. KOH/gm. oil)			
Original	0.1	0.1	0.1
Final	7.2	6.5	11.3
A.S.T.M. Union Color			
Original	1	1	2
Final	>8	>8	>8
Wt.% Insoluble Material	0.3	0.2	0.6
Final Catalyst Condition			
Appearance			
Copper	Coated	Coated	Dull
Steel	Coated	Coated	Coated
Aluminum	Coated	Coated	Coated
Magnesium	Coated	Coated	Coated
Wt. Loss (mg./sq. cm.)			
Copper	+0.24	+0.12	+0.03
Steel	+0.14	+0.16	+0.18
Aluminum	+0.10	+0.21	+0.33
Magnesium	+0.12	+0.14	+0.26



Table 17

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL DIBASIC ACID ESTERS AT 500°F.

TEST PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7080 = DI-2-ETHYLHEXYL ISOSEBACATE

MLO 7081 = DIISOOCTYL ISOSEBACATE

PRL 3207 = DI-2-ETHYLHEXYL SEBACATE

ALL FLUIDS CONTAIN 0.5 WT.% PHENOTHIAZINE AS AN OXIDATION INHIBITOR.

TEST FLUID	PRL 3207	MLO 7080	MLO 7081
LIQUID CHARGED, GRAMS	91	89	89
LIQUID LOSS, WT.%	7	8	10
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	5.8	5.5	5.3
MOLS O <sub>2</sub> USED/426 GMS. FLUID (3)	0.86	0.82	0.79
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (2)			
AT 130°F.	+44	+51	+52
AT 0°F.	+48	+153	+162
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.1	0.1	0.1
FINAL	15.4	20.0	28.6
WT.% OIL INSOLUBLE MATERIAL (2)	0.1	3.8	11.4
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	DULL	DULL	CORRODED
STEEL	DULL	CORRODED	CORRODED
ALUMINUM	BRIGHT	DULL	DULL
WT. LOSS (MG./SQ. CM.)			
COPPER	0.10	0.19	0.42
STEEL	0.07	0.98	3.21
ALUMINUM	0.00	+0.06	+0.02

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(3) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

Figure 2

# OXIDATION STABILITY OF SEVERAL PHENOTHIAZINE-INHIBITED ESTERS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME AS INDICATED; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

ALL FLUIDS CONTAIN 0.5 WT.-% PHENOTHIAZINE.

○ MLO 7080 = DI-2-ETHYLHEXYL ISOSEBACATE

△ MLO 7081 = DIISOOCTYL ISOSEBACATE

□ PRL 3483 = DI-2-ETHYLHEXYL SEBACATE

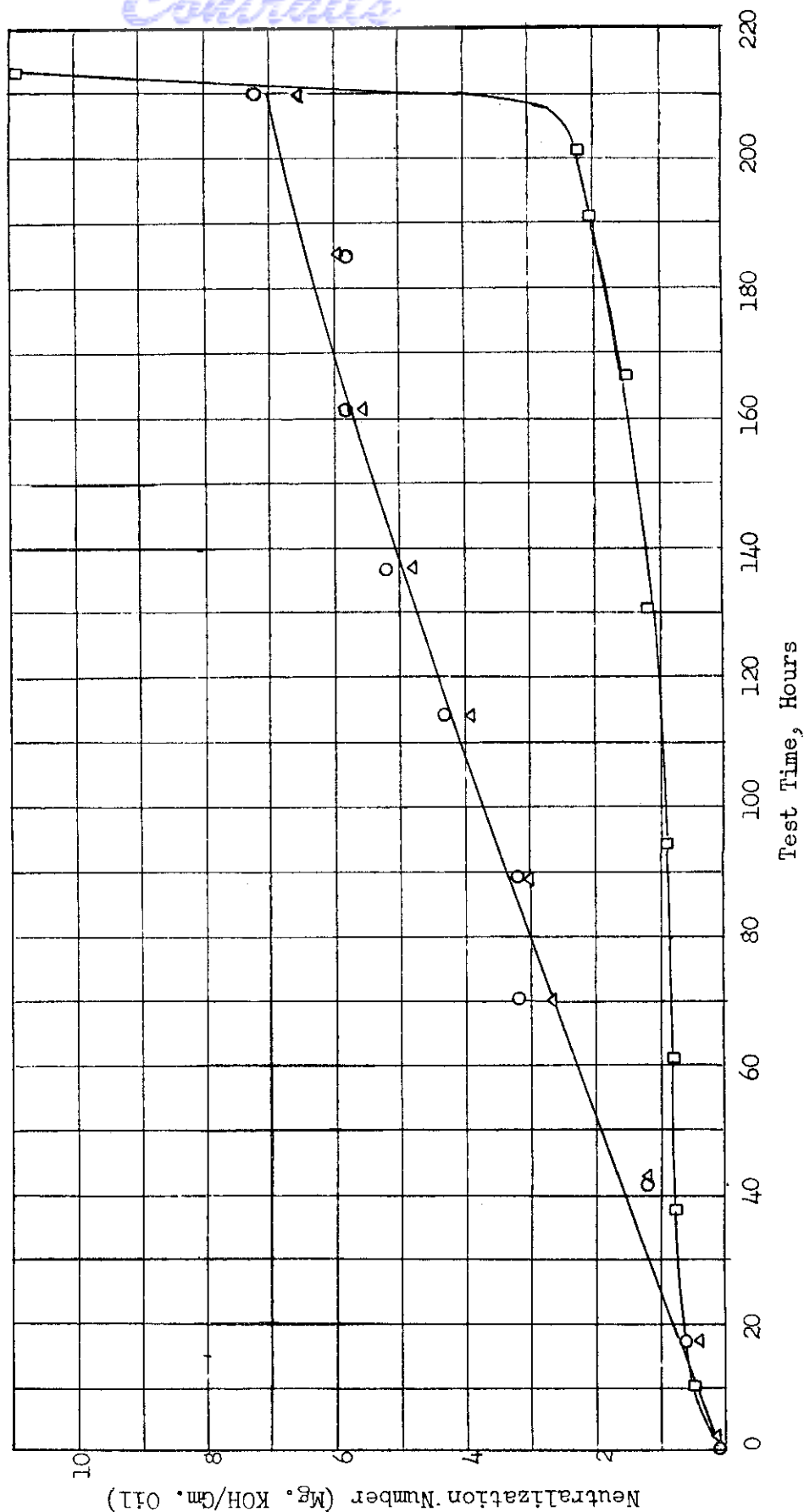


Table 18

VISCOSITIES OF TWO PHOSPHATE ESTERS AT VARIOUS TEMPERATURES

Fluid	Tris (meta-chlorophenyl) Phosphate (MLO 9574)	Diphenyl (o-chlorophenyl) Phosphate (MLO 9553)
Centistoke Viscosity at 100°F.	18.9	27.5
130°F.	13.3	10.2
210°F.	3.45	3.92
490°F.	0.69	0.72
707°F.	0.39	0.41
ASTM Slope (100° to 210°F.)	0.832	0.875

OXIDATION AND CORROSION CHARACTERISTICS OF TWO PHOSPHATE  
TYPE ESTERS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 60 HRS.; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 9574 = TRIS (META-CHLOROPHENYL) PHOSPHATE  
MLO 9533 = DIPHENYL (O-CHLOROPHENYL) PHOSPHATE

Test Fluid	MLO 9553	MLO 9574
Liquid Charged, grams	127	137
Liquid Loss, Wt. %	2	0
Approx. Amount O <sub>2</sub> supplied, grams <sup>(1)</sup>	78.6	78.6
Approx. Amount O <sub>2</sub> used, grams <sup>(1)</sup>	6.5	6.6
Mols O <sub>2</sub> used/426 grams of fluid <sup>(2)</sup>	0.68	0.64
% Change in Centistoke Visc. at 130°F.	-(3)	-(3)
Neut. No. (mg. KOH/gm. oil)		
Original	1.3	0.3
Final	42.2	34.5
Wt. % Oil Insoluble Material <sup>(4)</sup>	-(4)	-(4)
Final Catalyst Condition		
Appearance		
Copper	Corroded	Corroded
Steel	Corroded	Corroded
Aluminum	Corroded	Corroded
Wt. Loss (mg./sq. cm.)		
Copper	48.41	50.47
Steel	37.02	48.16
Aluminum	105.23(5)	55.22

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSES FOR O<sub>2</sub>.
- (2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF OIL INSOLUBLE MATERIAL AND OXIDIZED FLUID. THE VISCOSITY OF THIS MIXTURE COULD NOT BE DETERMINED.
- (4) OIL INSOLUBLES WERE NOT DETERMINED FOR THESE TESTS SINCE THE TEST FLUID WAS INSOLUBLE IN THE LOW BOILING PETROLEUM NAPHTHA USED TO SEPARATE THE INSOLUBLE MATERIAL FROM THE OXIDIZED LIQUID.
- (5) THE ALUMINUM CATALYST WAS COMPLETELY CORRODED DURING THIS TEST. THE WEIGHT LOSS RECORDED IS THE TOTAL WEIGHT OF THE CATALYST USED FOR THE TEST.

# OXIDATION AND CORROSION CHARACTERISTICS OF TWO PHOSPHATE TYPE ESTERS AT 600°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808  
TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $600 \pm 10^\circ\text{F}$ .; TEST TIME = 20  
HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;  
CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL AND ALUMINUM.  
TEST FLUIDS: MLO 9574 = TRIS (META-CHLOROPHENYL) PHOSPHATE  
MLO 9553 = DIPHENYL (O-CHLOROPHENYL) PHOSPHATE

Test Fluid	MLO 9553	MLO 9574
Liquid Charged, grams	138	128
Liquid Loss, Wt.%	0	0
Approx. Amount O <sub>2</sub> supplied, grams <sup>(1)</sup>	26.2	26.2
Approx. Amount O <sub>2</sub> used, grams <sup>(1)</sup>	2.8	6.8
Mols O <sub>2</sub> used/426 grams of fluid <sup>(2)</sup>	0.27	0.70
% Change in Centistoke Visc. at 130°F.	-(3)	+890
Neut. No. (mg. KOH/gm. oil)		
Original	0.3	1.3
Final	47.7	41.2
Wt.% Oil Insoluble Material <sup>(4)</sup>	-(4)	-(4)
Final Catalyst Condition		
Appearance		
Copper	Corroded	Corroded
Steel	Corroded	Corroded
Aluminum	Corroded	Corroded
Wt. Loss (mg./sq. cm.)		
Copper	0.31	19.75
Steel	48.60	49.45
Aluminum	101.10(5)	57.65

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF OXYGEN FOR 426 GRAMS HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF OIL INSOLUBLE MATERIAL AND OXIDIZED FLUID. THE VISCOSITY OF THIS MIXTURE COULD NOT BE DETERMINED.
- (4) OIL INSOLUBLES WERE NOT DETERMINED FOR THESE TESTS SINCE THE TEST FLUID WAS INSOLUBLE IN THE LOW BOILING PETROLEUM NAPHTHA USED TO SEPARATE THE INSOLUBLE MATERIAL FROM THE OXIDIZED LIQUID.
- (5) THE ALUMINUM CATALYST WAS COMPLETELY CORRODED DURING THIS TEST. THE WEIGHT LOSS RECORDED IS THE TOTAL WEIGHT OF THE CATALYST USED FOR THE TEST.

Table 21

## THERMAL STABILITY OF TWO PHOSPHATE TYPE ESTERS AT 600°F.

Tests were Conducted in Glass Test Tubes Using a 25 Gram Charge of Test Fluid. The Test Tubes are Sealed with a U Tube Containing Approximately 3 ml. of Test Fluid. The Seal Allows Passage of Gas Out or Air in with Pressure Changes Greater than 4 Inches of Fluid -- Approximately 0.25 lb/sq. in. Test Time = 6 Hours. One Inch Squares of Copper, Steel, and Aluminum were used as Catalysts.

Test Fluid	Cs. Visc. at 100°F.		% Loss in Weight (1)	Neut. No. (mg. KOH/gm. Oil)		Wt. Loss of Catalysts (mg./sq. cm.)		Oil Insol. Material, Wt. %
	Orig.	Final		Orig.	Final	Copper	Steel	
Tris (m-chlorophenyl) Phosphate (MLO 9574)	18.9	20.0	0.0	1.3	2.3	0.36	1.63	1.0
Diphenyl (o-chlorophenyl) Phosphate (MLO 9553)	27.5	28.2	0.0	0.3	0.7	0.07	1.27	0.4

(1) Per cent loss in weight is good to  $\pm 2.0\%$  or 0.5 grams.

Table 22

OXIDATION AND CORROSION CHARACTERISTICS OF TWO KENDALL RESINS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: MLO 7072 = A LIGHT RESIN OBTAINED FROM THE KENDALL REFINING COMPANY.  
MLO 7073 = A HEAVY RESIN OBTAINED FROM THE KENDALL REFINING COMPANY.

TEST FLUID	MLO 7072	MLO 7073
OVERALL LIQUID LOSS, WT.%	1	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+69	+137
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	1.1	0.9
FINAL	3.0	2.2
ASTM UNION COLOR		
ORIGINAL	>8	>8
FINAL	>8	>8
WT.% INSOLUBLE MATERIAL	TRACE	TRACE
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	DULL	DULL
STEEL	DULL	DULL
ALUMINUM	DULL	BRIGHT
MAGNESIUM	DULL	DULL
WT. LOSS (MG./SQ. CM.)		
COPPER	0.08	0.14
STEEL	0.00	0.02
ALUMINUM	+0.01	0.00
MAGNESIUM	0.00	+0.02



Table 23

OXIDATION AND CORROSION CHARACTERISTICS OF TWO KENDALL RESINS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7072 = A LIGHT RESIN OBTAINED FROM THE KENDALL REFINING COMPANY.

MLO 7073 = A HEAVY RESIN OBTAINED FROM THE KENDALL REFINING COMPANY.

TEST FLUID	MLO 7072	MLO 7073
LIQUID CHARGED, GRAMS	87	85
LIQUID LOSS, WT. %	0	2
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	3.0	2.4
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	0.47	0.38
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+61	+89
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	1.1	0.9
FINAL	1.1	0.6
WT. % OIL INSOLUBLE MATERIAL (3)	TRACE	TRACE
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	DULL	DULL
STEEL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)		
COPPER	+0.02	+0.05
STEEL	0.00	0.00
ALUMINUM	0.01	+0.01

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 24

## THERMAL STABILITY OF TWO KENDALL RESINS

TESTS CONDUCTED IN GLASS TEST TUBES CONSIST OF A TWENTY-FIVE GRAM CHARGE OF TEST FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 0.25 LB./SQ. IN.

TESTS CONDUCTED IN THE STAINLESS STEEL PRESSURE CYLINDER CONSIST OF A 20 ML. CHARGE OF TEST FLUID. THE TOTAL VOLUME OF THE CYLINDER IS 426 ML. PRIOR TO THE START OF THE TEST THE SYSTEM IS PURGED WITH NITROGEN. THE SYSTEM IS THEN SEALED AND THE TEST STARTED. IN THE STAINLESS STEEL PRESSURE CYLINDER THE NITROGEN PRESSURE AT 700°F. IS ABOUT 20 P.S.I. GAGE.

TEST FLUIDS: MLO 7072 = A LIGHT RESIN OBTAINED FROM THE KENDALL REFINING COMPANY.

MLO 7073 = A HEAVY RESIN OBTAINED FROM THE KENDALL REFINING COMPANY.

TEST FLUID	TEST TEMP., °F.	TEST TIME, HRS.	TYPE OF APPARATUS (1)	CENTISTOKE VISCOSITY AT 100°F.		% LOSS IN WT. (2)	CATALYST WT. LOSS, MG./SQ. CM.			NEUT. NO. MG. KOH/GM. OIL		MAX. SYSTEM PRESSURE, P.S.I.	FINAL PRESSURE AT ROOM TEMP., P.S.I.
				ORIG.	FINAL		M-10 STEEL	52-100 STEEL	BRONZE	ORIG.	FINAL		
MLO 7072	600	6	P.C.	5,860	4,580	-22	-	-	-	0.2	0.0	25	-
	600	6	GLASS	5,860	4,930	-16	-	-	-	0.2	0.0	-	-
	700	6	GLASS	5,860	464	-92	-	-	-	0.2	0.1	-	-
	700	2	P.C.	5,860	143	-98	0.06	0.28	+0.08	0.2	0.1	290	55
MLO 7073	600	6	GLASS	16,200	13,900	-14	-	-	-	0.1	0.0	-	-
	700	6	GLASS	16,200	1,010	-94	-	-	-	0.1	0.2	-	-
	700	6	P.C.	16,200	542	-97	0.14	0.23	0.00	0.1	0.1	182	40

liters of air per hour in the 347°F. oxidation and corrosion test. This air rate (10 l./hr.) has been standard for oxidations of this type for the past 15 years. Studies of varying air rates in oxidation and corrosion tests where stable life is the critical measurement indicate that test results are essentially independent of air rate over the range of 5 to 15 liters per hour.

On the basis of these studies, 10 liters per hour air rate was maintained for intra-laboratory standards when a value of five liters per hour was chosen for inclusion in the Spec. MIL-L-7808 oxidation and corrosion test at 347°F. These conditions are satisfactory for the ester-base fluids where stable life exceeds the 72 hours test time. However, preliminary tests with conventionally refined mineral oils, as shown in Tables 25 through 27, indicate that the 72-hour tests at 347°F. are exceeding the stable life of the fluid. This is evident for all of the oils and additive combinations tested at the 10 liters per hour air rate. All future tests at 347°F. will be conducted at five liters of air per hour.

The effect of differences in air rate with one conventionally refined naphthenic and two paraffinic mineral oils is shown on Table 28. Each of the test fluids contains 1.0 weight per cent phenyl-alpha-naphthylamine as the oxidation inhibitor. These data indicate that, while the volatility loss, viscosity change, and neutralization number change are somewhat greater for the ten liter per hour tests, the formation of insolubles is slightly less in each case where the air rate is ten liters per hour. There is no appreciable catalyst corrosion in any of the tests. It should be emphasized that the relationship between the test results at five and ten liters of air per hour shown in Table 28 can not be used to apply to other fluids. The remaining 347°F. oxidation and corrosion tests with mineral oil fluids discussed in this report, unless specifically noted, have been conducted at an air rate of five liters per hour.

## 2. Oxidation Characteristics of Polyolefin Hydrocarbons.

Oxidation and corrosion studies at 347°F. have been conducted with polyolefin hydrocarbons before and after hydrogenation. All of the polyolefins used in this study were obtained from the Standard Oil Company of Indiana. Three fractions were used in this study.

One is the commercially available Indopol L-50 and the second is a specially prepared narrow boiling fraction of polyolefin (MLO 7123) to match the 100°F. viscosity properties of L-50. The third is a low viscosity fraction of about 15 centistokes at 100°F. (PRL 3473). The hydrogenated L-50 was prepared by this Laboratory in accordance with the hydrogenation conditions described in the preceding section. The hydrogenated materials from MLO 7123 and PRL 3473 were prepared by the Standard Oil Company of Indiana and are designated in this report as MLO 7124 and PRL 3474, respectively. These materials are assumed to be pure hydrocarbons. In this respect they differ from conventionally refined mineral oils which may contain small quantities of polar non-hydrocarbon impurities. These impurities contain, in addition to carbon

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and hydrogen, one or more of such elements as sulfur, nitrogen, and oxygen.

The polyolefins and saturated polyolefins have been evaluated in the 347°F. oxidation and corrosion test with various inhibitors as shown on Tables 29 through 32. Inhibitors used in this study include: a combination of a hindered phenol (Antioxidant 2246) and a dialkyl acid phosphite, phenyl-alpha-naphthylamine, and two dithiocarbamates. It should be noted that the tests shown on Tables 29 and 30 were conducted at an air rate of 10 liters per hour.

The unsaturated polyolefins without additives show evidence of oxidative deterioration in the form of viscosity and neutralization number increases. The oxidized polyolefin after 72 hours at 347°F. shows little or no evidence of dirt or sludge and metal corrosion. The additive studies with the polyolefin indicate that the additives evaluated are completely ineffective in preventing oxidative deterioration. The additives do appear to contribute materially to the fluid degradation and metal corrosion. The phenyl-alpha-naphthylamine shows only a slight increase in sludge or dirtiness and no metal corrosion. The combination of hindered phenol and dialkyl acid phosphite and the dithiocarbamates cause a substantial increase in sludge or dirtiness. The dithiocarbamates cause substantial copper corrosion while the hindered phenol-dialkyl acid phosphite combinations produce a protective coating on the metals.

The hydrogenated polyolefins show excellent inhibitor susceptibility with all of the additives tested. Tests conducted on the hydrogenated polyolefin without additives show excellent property values after the oxidation test. The only substantial property change noted for the non-additive hydrogenated polyolefin is that of neutralization number increase. All three additive types adequately control oxidation and metal corrosion for the 72-hour test period at 347°F. The resultant property changes are well within the Spec. MIL-L-7808 oxidation and corrosion test limits, with the exception of metal coatings in the test involving the additive combination of the hindered phenol and dialkyl acid phosphite.

Tests with phenyl-alpha-naphthylamine in the hydrogenated polyolefin have been conducted for 144 hours at 347°F. This test severity is about equivalent to the stable life of a high quality ester-base Spec. MIL-L-7808 fluid under these conditions. The properties from this extended test indicate that the fluid is still within the stable life period after 144 hours. This means that oxidation-wise the hydrogenated polyolefin can be prepared to be on a par with the ester-base fluid in the intermediate temperature range. The data from the hydrogenated polyolefin may also be of value in pointing up the optimum properties to be obtained by super-refining of certain mineral oil fractions.

3. Oxidation Characteristics of Paraffinic Mineral Oils. A series of conventionally refined paraffinic stocks have been evaluated in the 347°F. oxidation and corrosion test. These oils include Barosa 43

# Contrails

and Aviation Bright Stock obtained from the Esso Standard Oil Company, a heavy neutral prepared by the Kendall Refining Company, and a 300 neutral, a 180 neutral and an SAE 30 (neutral-bright stock blend) prepared from Pennsylvania type crude oil.

The effects of additives on these stocks before and after hydrogenation are shown on Tables 33 through 38. Silica gel treatment has been used as an additional refining step with the Pennsylvania extracted 180 and 300 neutrals. Data for these materials are shown on Table 39.

The additives or oxidation inhibitors evaluated in these paraffinic stocks include phenyl-alpha-naphthylamine (PAN), three dithiocarbamate additives from the R. T. Vanderbilt Company, a thiophosphate additive from the Lubrizol Corporation, and a thiophosphate and a hindered phenol from the American Cyanamid Company.

Several of the paraffinic mineral oils have been evaluated without additives in the 347°F. oxidation test. Severe oxidative deterioration has been obtained for these evaluations. The use of the various additives in the conventionally refined paraffinic stocks results in an improvement over the non-additive fluid in the magnitude of the property changes. In all cases, however, there is evidence that the stable life of the fluid had been exceeded in the 72-hour test period. Oxidative deterioration is apparent in most cases from the change in neutralization number, viscosity increase, and sludge formation. Metal corrosion is also noted in many cases. Substantial differences are apparent in the effectiveness of additives and the type of deterioration noted in the various conventionally refined stocks.

The several neutrals evaluated show relatively high sludge values and low values of viscosity change. The bright stock (MLO 7094) on the other hand shows a high viscosity change value and low sludge formation. This trend toward decreasing sludge formation with increasing molecular weight has previously been noted with conventionally refined mineral oils. The bright stock (MLO 7094) shows considerably more metal corrosion than do the neutrals.

The relative effectiveness of the additives in the conventionally refined stocks appears to vary. For example, PRL 3605, the dithiocarbamate, is more effective than PAN in Barosa 43 but about the same or less effective in Kendall heavy neutral. In general, PAN and the hindered phenol type additive are more effective in controlling metal corrosion than are the thiophosphates and dithiocarbamates at 347°F. There is some evidence that these latter types may contribute to increased copper corrosion.

In all of the cases where hydrogenated paraffinic mineral oil fractions have been evaluated, substantially improved oxidative and corrosion behavior are obtained. The improved properties include a lowering of the neutralization number change, viscosity increase, sludge formation, and metal corrosion. The hydrogenated stocks evaluated with either 1.0 weight per cent phenyl-alpha-naphthylamine or 2.0 weight per

cent of a dithiocarbamate solution meet the property requirements of the Spec. MIL-L-7808 oxidation and corrosion test.

These data indicate that the stable life of these hydrogenated paraffinic oils with additives is now greater than 72 hours at 347°F. The hindered phenol type additive is less effective than PAN and the dithiocarbamate in the hydrogenated stocks at 347°F. In general, hindered phenol type inhibitors also fail to protect Spec. MIL-L-7808 type fluids for the 72-hour test period at 347°F.

The silica gel treated Pennsylvania 180 and 300 neutrals have been evaluated with PAN or a dithiocarbamate (PRL 3605) additive. The silica gel treated stock shows a substantial reduction in sludge formation. However, viscosity change, neutralization number increase, and corrosion of copper remain unchanged from the unhydrogenated stock. That is, silica gel percolation is not particularly effective in improving additive susceptibility.

There are two important types of molecules present in the conventionally refined paraffinic mineral oil which are affected by hydrogenation and silica gel percolation. These two groups are the unsaturated (olefinic and aromatic) molecules and the polar molecules (containing sulfur, nitrogen, and oxygen). The silica gel tends to remove both types of compounds by physical adsorption, while hydrogenation converts the unsaturates to saturates and removes some of the non-hydrocarbon components as volatile reaction products such as hydrogen disulfide, ammonia, and water.

On the basis of the polyolefin hydrocarbon and conventionally refined mineral oil oxidation data at 347°F. obtained thus far, the polar impurities may contribute more substantially to sludge and metal corrosion while the unsaturates may act to reduce inhibitor effectiveness and cause viscosity and neutralization number increase. Hydrogenation appears to be more effective in producing an overall improvement in the paraffinic stocks than silica gel treatment.

4. Oxidation Characteristics of Naphthenic Mineral Oils. Two highly refined naphthenic mineral oils, Necton 45 and Necton 50, have been evaluated in the 347°F. oxidation and corrosion test before and after hydrogenation or silica gel adsorption. Primol D which is an acid extracted white oil has also been evaluated in this program. The Nectons and Primol D were obtained from the Esso Research and Engineering Company.

Oxidation and corrosion test results with the Necton samples are shown on Tables 40 and 41. The dithiocarbamate and phenyl-alpha-naphthylamine additives are not particularly effective in the Necton 45 and 50 stocks. In general, all property changes and metal corrosion are sufficiently high to indicate oxidative deterioration beyond the stable life.

Silica gel treatment of the Nectons does not significantly change the inhibitor effectiveness. The amount of sludge formed is



substantially lower following silica gel treatment. Other property changes on oxidation have not been significantly altered by the silica gel treatment. This is the same behavior noted with the paraffinic mineral oil on silica gel treatment.

Oxidation and corrosion data are also shown for the hydrogenated Necton 50. The dithiocarbamate additives appear to be very effective in controlling corrosion and all property changes. The thiophosphate type additive shows a relatively low order of effectiveness in both metal corrosion and property change in this type of stock. The effect of PAN and the hindered phenol are intermediate between the dithiocarbamate and the thiophosphate.

The PAN-inhibited hydrogenated or silica gel treated Necton 50 shows the same order of protection in the 347°F. oxidation test. The additive effectiveness in the hydrogenated Necton is lower than in the hydrogenated paraffinic stocks evaluated. This low order of inhibitor effectiveness may again be due to the incomplete hydrogenation of the unsaturated linkages in the Necton.

Primol D is a naphthenic base oil which has been acid treated to produce a white oil. The 347°F. oxidation and corrosion data shown on Table 42 indicate good inhibitor response with phenyl-alpha-naphthylamine or dithiocarbamate. The behavior of Primol D in the 347°F. oxidation and corrosion test compares favorably with that of the hydrogenated polyolefin.

5. Oxidation Characteristics of Aromatic Mineral Oils and Hydrocarbons. Several alkylated aromatic hydrocarbons and aromatic concentrates from mineral oils have been evaluated in the 347°F. oxidation test. These data are shown on Tables 43 and 44. The butylated naphthalene, diamyl naphthalene, and tetraamyl benzene are essentially pure hydrocarbons prepared synthetically by alkylation. The mineral oil fraction is an aromatic type heat transfer oil marketed by Socony Mobil Oil Company. These oxidation data are characterized by high viscosity increase, moderate sludge, and copper corrosion. The synthetic hydrocarbons appear to be slightly better than the heat transfer oil under comparable test and additive conditions.

Some 347°F. oxidation tests have been made on hydrogenated aromatic fractions of mineral oil. In general, these stocks are highly aromatic concentrates prepared by liquid extraction. These fractions exhibit excellent thermal stability in the 700°F. temperature range. Hydrogenation is a useful step in the refining of these extracts, which contain the non-hydrocarbon impurities that tend to be concentrated with the aromatic fractions by liquid extraction. Partial hydrogenation reduces or eliminates these non-hydrocarbon impurities.

MLO 7079 is an example of a partially hydrogenated stock. MLO 7087 and MLO 7091 are more completely hydrogenated materials in which the aromatic rings are also saturated. Data on Table 44 show that these hydrogenated aromatic fractions show relatively good overall behavior in



OXIDATION AND CORROSION CHARACTERISTICS OF A NAPHTHENIC  
MINERAL OIL AT 347°F.

Test Procedure and Techniques in Accordance with Spec. MIL-L-7808.  
Test Conditions Include: Test Temperature =  $347 \pm 3^\circ\text{F}$ .; Test Time = 72  
Hours; Air Rate =  $10 \pm 1$  Liters per Hour; Test Fluid Charged = 100  
ml.; Catalyst = a 1 Inch Square each of Copper, Steel, Aluminum, and  
Magnesium.  
Test Fluid: PRL 2052 = Necton 50, a Well Refined Naphthenic Mineral Oil  
of 65.2 Centistokes Viscosity at 100°F.

Test Fluid (Composition in Wt.%)	PRL 2052	PRL 2052 + 1.0 Phenyl-alpha- naphthylamine
Overall Liquid Loss, Wt.%	1	5
% Change in Centistoke Viscosity at 130°F.	+103	+12
Neut. No. (mg. KOH/gm. Oil)		
Original	0.1	0.1
Final	8.7	2.3
ASTM Union Color		
Original	2	5
Final	>8	>8
Wt.% Insoluble Material	5.3	2.2
Final Catalyst Condition		
Appearance		
Copper	Corroded	Dull
Steel	Corroded	Dull
Aluminum	Dull	Dull
Magnesium	Dull	Dull
Wt. Loss (mg./sq. cm.)		
Copper	1.28	0.09
Steel	0.49	+0.06
Aluminum	+0.06	+0.04
Magnesium	+0.06	+0.06

Table 26

## OXIDATION AND CORROSION CHARACTERISTICS OF A PARAFFINIC MINERAL OIL AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: MLO 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC MINERAL OIL).

TEST FLUID	NONE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 ANTIOXIDANT 2246	1.0 PARANOX 441	1.0 PARANOX 441 + 0.5 DIISOPROPYL ACID PHOSPHITE
ANTI-OXIDANT, WT. %	6	5	4	2	4
OVERALL LIQUID LOSS, WT. %	SOLID	+81	+157	+110	+131
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	0.1 8.7	0.1 6.2	0.4 6.9	0.1 7.2	0.4 8.1
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	4	5	4	4	4
FINAL	>8	>8	>8	>8	>8
ASTM UNION COLOR					
ORIGINAL	4	5	4	4	4
FINAL	>8	>8	>8	>8	>8
WT. % INSOLUBLE MATERIAL	-(1)	2.0	1.9	1.2	1.0
FINAL CATALYST CONDITION					
APPEARANCE	CORRODED	DULL	DULL	CORRODED	CORRODED
COPPER	DULL	DULL	DULL	DULL	COATED
STEEL	BRIGHT	DULL	DULL	BRIGHT	COATED
ALUMINUM	BRIGHT	DULL	BRIGHT	BRIGHT	COATED
MAGNESIUM					
WT. LOSS (MG./SQ. CM.)					
COPPER	2.87	0.02	0.16	0.54	0.78
STEEL	+0.01	+0.02	0.00	+0.03	+0.27
ALUMINUM	+0.05	+0.04	0.00	+0.03	+0.33
MAGNESIUM	+0.04	+0.04	0.00	0.01	+0.37

(1) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF INSOLUBLE MATERIAL AND OXIDIZED LIQUID. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.

Table 27

## OXIDATION AND CORROSION CHARACTERISTICS OF A PARAFFINIC MINERAL OIL AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL).

TEST FLUID ANTI-OXIDANT, WT.-%	NONE	1.0 PHENYL-ALPHA- NAPHTHYLAMINE	MLO 7027 1.0 ANTIOXIDANT 2246	1.0 PARANOX 441	1.0 PARANOX 441 + 0.5 DIISOPROPYL ACID PHOSPHITE
OVERALL LIQUID LOSS, WT.-%	3	4	3	2	3
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	SOLID	+36	+46	+36	+46
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.1	0.1	0.3	0.1	0.3
FINAL	9.1	5.7	7.0	7.2	8.7
ASTM UNION COLOR					
ORIGINAL	2	5	2	2	2
FINAL	>8	>8	>8	>8	>8
WT.-% INSOLUBLE MATERIAL	(1)	1.4	3.4	4.2	4.1
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	CORRODED	DULL	DULL	CORRODED	CORRODED
STEEL	DULL	DULL	DULL	DULL	COATED
ALUMINUM	BRIGHT	DULL	BRIGHT	DULL	COATED
MAGNESIUM	BRIGHT	DULL	BRIGHT	DULL	COATED
WT. LOSS (MG./SQ. CM.)					
COPPER	3.48	0.06	0.14	0.35	3.67
STEEL	+0.02	+0.02	+0.06	0.04	+0.24
ALUMINUM	+0.01	+0.02	+0.01	+0.01	+0.19
MAGNESIUM	+0.01	+0.05	0.00	+0.03	+0.41

(1) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF OXIDIZED LIQUID AND INSOLUBLE MATERIAL. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.

Table 28

## EFFECT OF AIR RATE ON THE OXIDATION BEHAVIOR OF SOME MINERAL OILS

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^{\circ}\text{F.}$ ; TEST TIME = 72 HOURS; AIR RATE AS INDICATED; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A ONE-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: PRL 2052 = NECTON 50 (A HIGHLY REFINED NAPHTHENIC MINERAL OIL).

ML0 7026 = A PENNSYLVANIA 300 NEUTRAL MINERAL OIL.

ML0 7027 = A PENNSYLVANIA 180 NEUTRAL MINERAL OIL.

NOTE: ALL TEST FLUIDS CONTAIN 1.0 WEIGHT PER CENT PHENYL-ALPHA-NAPHTHYLAMINE.

TEST FLUID AIR RATE, L./HR.	PRL 2052		ML0 7026		ML0 7027	
	5	10	5	10	5	10
OVERALL LIQUID LOSS, WT. %	0	5	0	5	1	4
% CHANGE IN CENTISTOKE VISCOSITY AT $130^{\circ}\text{F.}$	+9	+12	+33	+81	+15	+36
NEUT. NO. (MG. KOH/GM. OIL)						
ORIGINAL	0.1	0.1	0.1	0.1	0.1	0.1
FINAL	1.4	2.3	4.6	6.2	3.0	5.7
WT. % INSOLUBLE MATERIAL	2.9	2.2	2.3	2.0	1.9	1.4
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL
MAGNESIUM	DULL	DULL	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)						
COPPER	0.05	0.09	0.11	0.02	0.11	0.06
STEEL	+0.03	+0.06	+0.02	+0.02	+0.01	+0.02
ALUMINUM	+0.05	+0.04	+0.01	+0.04	+0.01	+0.02
MAGNESIUM	+0.02	+0.06	+0.02	+0.04	+0.04	+0.05

Table 29

## OXIDATION AND CORROSION CHARACTERISTICS OF INDOPOL L-50 AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $10 \pm 1$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.TEST FLUID: MLO 7014 = INDOPOL L-50 (POLYBUTENE POLYMER OF 110 CENTISTOKES VISCOSITY AT  $100^\circ\text{F}$ .).

TEST FLUID ANTI-OXIDANT, WT.-%	<-----> NONE	1.0 PHENYL-ALPHA- NAPHTHYLAMINE	MLO 7014 1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246 + 0.5 DIISOPROPYL ACID PHOSPHITE
OVERALL LIQUID LOSS, WT.-%	9	7	6	7
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F}$ .	+484	+383	+247	+154
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 9.4	0.1 6.1	0.1 6.5	0.4 6.4
ASTM UNION COLOR ORIGINAL FINAL	2 >8	5 >8	5 >8	2 >8
WT.-% INSOLUBLE MATERIAL	0.6	0.6	0.4	0.4
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM MAGNESIUM	DULL DULL BRIGHT BRIGHT	DULL DULL BRIGHT BRIGHT	DULL DULL BRIGHT BRIGHT	DULL COATED COATED COATED
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM MAGNESIUM	0.03 0.00 +0.04 +0.06	0.02 +0.02 0.00 0.01	0.09 0.02 0.03 0.03	+0.05 +0.26 +0.28 +0.27

Table 30

OXIDATION AND CORROSION CHARACTERISTICS OF A POLYBUTENE AND  
A HYDROGENATED POLYBUTENE AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F.}$ ; TEST TIME = 72 HOURS; AIR RATE =  $10 \pm 1$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: PRL 3473 = SAMPLE LF-0741 (POLYBUTENE POLYMER OF 15 CENTISTOKES VISCOSITY AT  $100^\circ\text{F.}$ ).

PRL 3474 = SAMPLE LF-0742 POLYBUTENE POLYMER (PRODUCT OF HYDROGENATION OF PRL 3473).

TEST FLUID (COMPOSITION IN WT.%)	PRL 3473 + 1.0 ANTIOXIDANT 2246 + 0.5 DIISOPROPYL ACID PHOSPHITE	PRL 3474 + 1.0 ANTIOXIDANT 2246 + 0.5 DIISOPROPYL ACID PHOSPHITE
OVERALL LIQUID LOSS, WT.%	8	2
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F.}$ AT $0^\circ\text{F.}$	+100 +368	+3 +8
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.5 9.8	0.5 0.3
ASTM UNION COLOR ORIGINAL FINAL	1 >8	1 >8
WT.% INSOLUBLE MATERIAL	3.5	0.5
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM MAGNESIUM	COATED COATED COATED COATED	COATED COATED COATED DULL
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM MAGNESIUM	+0.30 +0.30 +0.27 +0.20	+0.51 +0.17 +0.33 0.00

Table 31

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL FLUIDS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: MLO 7102 = INDOPOL L-50 (POLYOLEFIN) OBTAINED FROM THE INDOIL CHEMICAL COMPANY.

MLO 7101 = HYDROGENATED MLO 7102.

ADDITIVE: PRL 3605 AND PRL 3606 = DITHIOCARBAMATE TYPE ADDITIVES OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID	<-----MLO 7101----->		<-----MLO 7102----->		<-----MLO 7102----->	
	NONE	1.0 PHENYL-ALPHA-NAPHTH/LAMINE	2.0 PRL 3605	NONE	1.0 PHENYL-ALPHA-NAPHTH/LAMINE	2.0 PRL 3606
OXIDATION INHIBITOR, WT. %						
OVERALL LIQUID LOSS, WT. %	6	3	3	5	4	4
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	-14	+17	+14	+124	+120	+106
NEUT. NO. (MG. KOH/GM. OIL)						
ORIGINAL	0.1	0.0	0.0	0.1	0.0	0.0
FINAL	5.1	0.0	0.1	4.9	5.2	6.3
ASTM UNION COLOR						
ORIGINAL	1	4	1	1	4	1
FINAL	5	5	3	>8	>8	>8
WT. % INSOLUBLE MATERIAL	TRACE	0.2	0.1	TRACE	0.1	1.1
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	DULL	DULL	DULL	BRIGHT	BRIGHT	CORRODED
STEEL	DULL	DULL	DULL	DULL	COATED	DULL
ALUMINUM	BRIGHT	DULL	DULL	DULL	DULL	DULL
MAGNESIUM	BRIGHT	DULL	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)						
COPPER	0.09	+0.08	0.08	0.02	+0.02	2.33
STEEL	0.07	+0.02	0.00	0.00	+0.09	0.01
ALUMINUM	0.08	0.00	0.00	0.00	+0.02	0.02
MAGNESIUM	0.00	+0.04	+0.01	0.06	+0.04	+0.03

Table 32

OXIDATION AND CORROSION CHARACTERISTICS OF A HYDROGENATED  
POLYOLEFIN AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME AS INDICATED; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: MLO 7124 = HYDROGENATED POLYOLEFIN OBTAINED FROM THE INDOIL CHEMICAL COMPANY.

ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM THE R. T. VANDERBILT COMPANY.

TEST FLUID	<----- MLO 7124 ----->					
OXIDATION INHIBITOR, WT.%	-		1.0 PHENYL-ALPHA-NAPHTHYLAMINE		2.0 PRL 3605	
TEST TIME, HOURS	72	144	72	144	72	144
OVERALL LIQUID LOSS, WT.%	4	-	3	-	2	-
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	-4	+13	+7	+8	+6	+3
NEUT. NO. (MG. KOH/GM. OIL)						
ORIGINAL	0.1	0.1	0.1	0.1	0.1	0.1
FINAL	5.3	4.2	0.1	0.2	0.2	0.2
ASTM UNION COLOR						
ORIGINAL	1	1	3	3	1	1
FINAL	5	>8	5	>8	3	5
WT.% INSOLUBLE MATERIAL	TRACE	1.3	TRACE	0.2	0.1	0.1
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	BRIGHT	CORRODED	DULL	DULL	DULL	CORRODED
STEEL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	BRIGHT	DULL	DULL	DULL	DULL
MAGNESIUM	COATED	COATED	DULL	BRIGHT	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)						
COPPER	0.05	0.61	0.04	+0.03	0.17	0.21
STEEL	0.02	0.15	0.02	0.02	+0.03	0.00
ALUMINUM	0.02	0.06	0.04	0.05	+0.04	+0.03
MAGNESIUM	+0.10	+0.10	0.00	+0.02	0.00	+0.03



Table 33

## OXIDATION AND CORROSION CHARACTERISTICS OF BAROSA 43 AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;  
 CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
 TEST FLUID: MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.  
 ADDITIVE: PRL 3605, 3606, AND 3607 = DITHIOCARBAMATE TYPE ADDITIVES OBTAINED FROM R. T. VANDERBILT COMPANY.  
 PRL 3615 = A THIOPHOSPHATE ADDITIVE OBTAINED FROM THE LUBRIZOL CORPORATION.

TEST FLUID	<- - - - - MLO 7093 - - - - - >				
OXIDATION INHIBITOR, WT. %	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605	1.0 PRL 3607	2.0 PRL 3606	1.0 PRL 3615
OVERALL LIQUID LOSS, WT. %	1	2	2	2	2
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+9		+8	+14	+11
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.1	0.1	0.1	0.2	1.0
FINAL	2.6	0.8	1.8	2.6	3.0
ASTM UNION COLOR					
ORIGINAL	4	2	2	2	2
FINAL	>8	>8	>8	>8	>8
WT. % INSOLUBLE MATERIAL	3.4	0.8	1.2	0.5	1.0
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	DULL	CORRODED	CORRODED	CORRODED	CORRODED
STEEL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	BRIGHT	BRIGHT	DULL	DULL
MAGNESIUM	DULL	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)					
COPPER	0.03	1.23	0.95	0.24	2.05
STEEL	+0.05	+0.02	0.00	0.02	0.04
ALUMINUM	+0.05	0.01	0.00	0.02	0.00
MAGNESIUM	+0.05	+0.02	+0.04	0.00	0.03

Table 34

OXIDATION AND CORROSION CHARACTERISTICS OF AN AVIATION BRIGHT STOCK AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: MLO 7094 = AVIATION BRIGHT STOCK OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.

ADDITIVE: PRL 3605, 3606, AND 3607 = DITHIOCARBAMATE ADDITIVES OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID	MLO 7094				
OXIDATION INHIBITOR, WT. %	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605	1.0 ANTIOXIDANT 2246	1.0 PRL 3607	2.0 PRL 3606
OVERALL LIQUID LOSS, WT. %	2	2	1	1	3
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+172	+52	+68	+49	+39
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.2	0.4	0.2	0.2	0.2
FINAL	5.2	5.8	4.5	3.5	3.7
ASTM UNION COLOR					
ORIGINAL	6	6	6	6	6
FINAL	>8	>8	>8	>8	>8
WT. % INSOLUBLE MATERIAL	TRACE	0.1	TRACE	0.8	0.2
FINAL CATALYST CONDITION					
APPEARANCE	CORRODED DULL BRIGHT BRIGHT	CORRODED DULL BRIGHT DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL
WT. LOSS (MG./SQ. CM.)					
COPPER	2.16	1.36	0.66	1.12	1.12
STEEL	0.00	0.00	0.01	0.02	0.02
ALUMINUM	0.00	+0.01	0.03	0.01	0.01
MAGNESIUM	+0.02	+0.07	0.05	0.01	0.01

Table 35

OXIDATION AND CORROSION CHARACTERISTICS OF A MINERAL OIL AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST  
 FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
 TEST FLUID: MLO 7070 = A PENNSYLVANIA NEUTRAL (PARAFFINIC MINERAL OIL) OBTAINED FROM THE KENDALL REFINING COMPANY.  
 ADDITIVE: PRL 3605, 3606, AND 3607 = DITHIOCARBAMATE TYPE ADDITIVES OBTAINED FROM R. T. VANDERBILT CO.  
 PRL 3615 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM THE LUBRIZOL CORP.  
 PRL 3616 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM AMERICAN CYANAMID CO.

TEST FLUID	<-----< 1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605	2.0 PRL 3606	1.0 PRL 3607	1.0 PRL 3616	1.0 PRL 3615	>----->
OXIDATION INHIBITOR, WT.-%							
OVERALL LIQUID LOSS, WT.-%	2	0	2	2	2	2	2
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+17	+16	+13	+13	+17	+15	+15
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.0 3.0	0.1 3.2	0.1 3.2	0.1 4.1	1.7 3.6	0.9 3.8	0.9 3.8
ASTM UNION COLOR ORIGINAL FINAL	5 > 8	4 > 8	4 > 8	4 > 8	4 > 8	4 > 8	4 > 8
WT.-% INSOLUBLE MATERIAL	4.8	6.2	5.7	4.3	3.4	3.7	3.7
FINAL CATALYST CONDITION APPEARANCE							
COPPER	DULL	CORRODED	CORRODED	DULL	CORRODED	CORRODED	CORRODED
STEEL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL	DULL
MAGNESIUM	DULL	DULL	DULL	COATED	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)							
COPPER	0.12	1.09	0.70	+0.05	1.57	1.72	1.72
STEEL	0.00	0.01	+0.05	+0.05	+0.05	+0.06	+0.06
ALUMINUM	0.00	0.04	+0.03	+0.07	0.07	+0.15	+0.15
MAGNESIUM	+0.01	0.01	+0.02	+0.13	0.04	+0.09	+0.09

Table 36

EFFECT OF HYDROGENATION ON THE OXIDATION AND CORROSION CHARACTERISTICS OF BAROSA 43 AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;  
 CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
 TEST FLUIDS: MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.  
 MLO 7113 = HYDROGENATED MLO 7093.  
 ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID	<----- MLO 7093 ----->		<----- MLO 7113 ----->	
OXIDATION INHIBITOR, WT. %	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605
OVERALL LIQUID LOSS, WT. %	1	1	1	2
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.				
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL				
FINAL				
ASTM UNION COLOR				
ORIGINAL				
FINAL				
WT. % INSOLUBLE MATERIAL				
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER				
STEEL				
ALUMINUM				
MAGNESIUM				
WT. LOSS (MG./SQ. CM.)				
COPPER				
STEEL				
ALUMINUM				
MAGNESIUM				

Table 37

## EFFECT OF HYDROGENATION ON THE OXIDATION AND CORROSION CHARACTERISTICS OF AN AVIATION BRIGHT STOCK AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: MLO 7094 = AVIATION BRIGHT STOCK OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.  
MLO 7118 = HYDROGENATED MLO 7094.

ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID	<- - - - - MLO 7094 - - - - ->	<- - - - - MLO 7118 - - - - ->
OXIDATION INHIBITOR, WT. %	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605
OVERALL LIQUID LOSS, WT. %	2	3
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.		
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	+105	+32
FINAL	-0.2	0.1
ASTM UNION COLOR		
ORIGINAL	4.7	1.7
FINAL	6	4
WT. % INSOLUBLE MATERIAL	>8	>8
FINAL CATALYST CONDITION	TRACE	TRACE
APPEARANCE		
COPPER	CORRODED	CORRODED
STEEL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT
MAGNESIUM	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)		
COPPER	1.75	+0.02
STEEL	+0.01	+0.02
ALUMINUM	+0.01	+0.02
MAGNESIUM	0.00	+0.02

Contrails

Table 38

COMPARISON OF THE OXIDATION AND CORROSION CHARACTERISTICS OF A PENNSYLVANIA SAE 30 MINERAL OIL  
BEFORE AND AFTER HYDROGENATION AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
TEST FLUIDS: MLO 7077 = A PENNSYLVANIA SAE 30 (PARAFFINIC MINERAL OIL).  
MLO 7076 = HYDROGENATED MLO 7077.  
ADDITIVE: PRL 3605 AND PRL 3606 = DITHIOCARBAMATE TYPE ADDITIVES OBTAINED FROM R. T. VANDERBILT COMPANY.  
PRL 3615 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM THE LUBRIZOL CORPORATION.

TEST FLUID	<----- MLO 7077 ----->				<----- MLO 7076 ----->					
OXIDATION INHIBITOR, WT. %	1.0 PHENYL-ALPHA- NAPHTHYLAMINE	2.0 PRL 3605	2.0 PRL 3606	1.0 ANTI- OXIDANT 2246	1.0 PRL 3615	1.0 PHENYL-ALPHA- NAPHTHYLAMINE	2.0 PRL 3605	1.0 ANTI- OXIDANT 2246	1.0 PRL 3615	2.0 PRL 3606
OVERALL LIQUID LOSS, WT. %	2	5	2	1	0	1	1	1	1	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+42	+59	+27	+84	+74	+9	+2	+14	+12	+17
NEUT. NO. (MG. KOH/GM. OIL)										
ORIGINAL	0.2	0.3	0.1	0.1	0.8	0.1	0.1	0.1	0.9	0.0
FINAL	2.9	3.8	4.0	4.5	5.4	0.7	0.5	2.2	2.1	0.2
ASTM UNION COLOR										
ORIGINAL	7	6	6	6	6	4	2	2	2	2
FINAL	>8	>8	>8	>8	>8	>8	4	>8	>8	5
WT. % INSOLUBLE MATERIAL	2.8	0.4	0.3	0.8	TRACE	0.8	0.2	0.5	0.4	0.3
FINAL CATALYST CONDITION										
APPEARANCE	CORRODED	CORRODED	CORRODED	DULL	CORRODED	DULL	BRIGHT	DULL	CORRODED	DULL
COPPER	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	BRIGHT	DULL	DULL	DULL	DULL	BRIGHT	DULL	DULL	BRIGHT
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL	BRIGHT	DULL	DULL	DULL
MAGNESIUM	DULL	DULL	DULL	DULL	DULL	DULL	BRIGHT	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)										
COPPER	0.23	0.53	0.98	0.17	0.72	0.00	0.05	0.06	1.82	0.09
STEEL	0.00	+0.02	0.05	0.04	0.01	0.02	0.01	0.05	0.02	0.05
ALUMINUM	0.00	+0.02	0.02	0.02	0.00	0.00	0.01	0.05	+0.04	0.07
MAGNESIUM	0.00	+0.02	0.03	0.04	0.00	0.02	0.01	0.03	0.04	0.07

Table 39

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MINERAL OILS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
 TEST FLUIDS: MLO 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC).  
 MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC).  
 ADDITIVE: PAN = PHENYL-ALPHA-NAPHTHYLAMINE  
 PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE

TEST FLUID FLUID TREATMENT OXIDATION INHIBITOR, WT.-%	← - - - - - MLO 7026 - - - - - →		← - - - - - MLO 7027 - - - - - →	
	1.0 PAN	NONE 2.0 PRL 3605	SILICA GEL TREATED (1) 1.0 PAN	SILICA GEL TREATED (1) 1.0 PAN
OVERALL LIQUID LOSS, WT.-%	0	2	0	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+33	+58	+33	+19
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 4.6	0.2 4.7	0.1 4.1	0.0 3.3
WT.-% INSOLUBLE MATERIAL	2.3	2.1	0.1	0.3
FINAL CATALYST CONDITION APPEARANCE	DULL DULL DULL DULL	CORRODED DULL DULL DULL	DULL DULL BRIGHT BRIGHT	DULL DULL DULL DULL
WT. LOSS (MG/SQ. CM.)	0.11 +0.02 +0.01 +0.02	0.86 +0.04 +0.03 +0.05	0.16 0.00 0.00 0.01	0.11 0.00 +0.01 +0.04

(1) OIL PASSED THROUGH A COLUMN 1.6 INCHES IN DIAMETER AND 7 FEET HIGH CONTAINING ACTIVATED SILICA GEL AT APPROX. 150°F.  
 (OIL TO CLAY RATIO APPROX. 2 TO 1).

Table 40

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL FLUIDS AT 347°F.

TEST PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATES = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: NECTON 45 = A HIGHLY REFINED NAPHTHENIC MINERAL OIL (46 CENTISTOKES AT 100°F.)  
NECTON 50 = A HIGHLY REFINED NAPHTHENIC MINERAL OIL (65 CENTISTOKES AT 100°F.)

ADDITIVES: PAN = PHENYL-ALPHA-NAPHTHYLAMINE

PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE

TEST FLUID FLUID TREATMENT OXIDATION INHIBITOR, WT. %	NECTON 45		NECTON 50		SILICA GEL TREATED (1)	
	NONE 1.0 PAN	SILICA GEL TREATED (1) 1.0 PAN	NONE 2.0 PRL 3605	SILICA GEL TREATED (1) 1.0 PAN	NONE 2.0 PRL 3605	SILICA GEL TREATED (1) 2.0 PRL 3605
OVERALL LIQUID LOSS, WT. %	0	1	2	1	0	0
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+9	+24	+41	+24	+9	+9
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 2.2	0.3 2.8	0.0 2.3	0.3 2.8	0.5 1.8	0.5 1.8
WT. % INSOLUBLE MATERIAL	2.4	0.4	1.8	0.4	0.9	0.9
FINAL CATALYST CONDITION APPEARANCE	DULL DULL DULL COATED	DULL DULL BRIGHT DULL	CORRODED DULL DULL DULL	DULL DULL BRIGHT DULL	CORRODED DULL BRIGHT BRIGHT	CORRODED DULL BRIGHT BRIGHT
WT. LOSS (MG./SQ. CM.)	0.02 +0.02 +0.02 +0.11	0.06 0.02 0.00 0.02	2.07 +0.06 +0.03 +0.09	0.06 0.02 0.00 0.00	0.35 0.00 +0.01 +0.04	0.35 0.00 +0.01 +0.04

(1) OIL PASSED THROUGH A COLUMN 1.6 INCHES IN DIAMETER AND 7 FEET HIGH CONTAINING ACTIVATED SILICA GEL AT APPROX. 150°F.  
(OIL TO CLAY RATIO APPROX. 2 TO 1).



Table 41

## OXIDATION AND CORROSION CHARACTERISTICS OF A SATURATED MINERAL OIL AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: MLO 7096 = HYDROGENATED NECTON 50 (NAPHTHENIC MINERAL OIL).

ADDITIVE: PRL 3605 AND PRL 3606 = DITHIOCARBAMATE TYPE ADDITIVES OBTAINED FROM R. T. VANDERBILT COMPANY.  
PRL 3615 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM THE LUBRIZOL CORPORATION.

TEST FLUID		<----- MLO 7096 ----->				
OXIDATION INHIBITOR, WT. %		1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 PRL 3615	2.0 PRL 3605	1.0 ANTIOXIDANT 2246	2.0 PRL 3606
OVERALL LIQUID LOSS, WT. %		0	1	1	0	2
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.						
NEUT. NO. (MG. KOH/GM. OIL)						
ORIGINAL						
FINAL						
ASTM UNION COLOR						
ORIGINAL						
FINAL						
WT. % OIL INSOLUBLE MATERIAL						
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER						
STEEL						
ALUMINUM						
MAGNESIUM						
WT. LOSS (MG./SQ. CM.)						
COPPER						
STEEL						
ALUMINUM						
MAGNESIUM						
		DULL	CORRODED	DULL	DULL	DULL
		DULL	DULL	DULL	DULL	DULL
		BRIGHT	DULL	BRIGHT	DULL	DULL
		BRIGHT	DULL	DULL	DULL	DULL
		0.02	2.15	0.09	0.08	0.17
		0.03	+0.03	0.05	0.05	0.02
		0.00	+0.03	+0.02	0.02	0.02
		0.02	+0.05	0.01	0.03	0.00

Table 42

OXIDATION AND CORROSION CHARACTERISTICS OF PRIMOL D AT 347°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F.}$ ; TEST TIME = 72 HRS.; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: MLO 7144 = PRIMOL D, A WHITE OIL OBTAINED FROM THE ESSO RESEARCH AND ENGINEERING COMPANY.

ADDITIVE: PAN = PHENYL-ALPHA-NAPHTHYLAMINE  
PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE

TEST FLUID	<----- MLO 7144----->	
	1.0 PAN	2.0 PRL 3605
OXIDATION INHIBITOR, WT.%		
OVERALL LIQUID LOSS, WT.%	2	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+5	+2
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	0.0	0.2
FINAL	0.3	0.1
A.S.T.M. UNION COLOR		
ORIGINAL	2	0
FINAL	>8	3
WT.% INSOLUBLE MATERIAL	0.0	TRACE
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	DULL	DULL
STEEL	DULL	DULL
ALUMINUM	DULL	DULL
MAGNESIUM	BRIGHT	DULL
WT. LOSS (MG./SQ. CM.)		
COPPER	0.04	0.09
STEEL	0.00	0.02
ALUMINUM	0.05	0.02
MAGNESIUM	0.03	0.04

Table 43

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL FLUIDS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR;  
 TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH-SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: MLO 7075 = BUTYLATED NAPHTHALENE  
 PRL 70 = DIAMYL NAPHTHALENE  
 PRL 197 = TETRAMYL BENZENE

MLO 7028 = SOCONY VACUUM HEAT TRANSFER OIL 600

ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT CO.

TEST FLUID	MLO 7075 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	2.0 PRL 3605	PRL 70 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	PRL 197 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	MLO 7028 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	2.0 PRL 3605
OXIDATION INHIBITOR, WT. %						
OVERALL LIQUID LOSS, WT. %	2	2	0	1	1	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+284	+139	+72	+97	+203	+239
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.2 5.7	0.1 5.4	0.2 5.2	0.4 8.2	0.2 4.0	0.1 5.3
ASTM UNION COLOR ORIGINAL FINAL	>8 >8	>8 >8	5 >8	5 >8	7 >8	5 >8
WT. % INSOLUBLE MATERIAL	0.5	0.3	0.4	0.7	2.9	3.6
FINAL CATALYST CONDITION APPEARANCE	CORRODED DULL BRIGHT BRIGHT	CORRODED DULL BRIGHT DULL	CORRODED DULL BRIGHT BRIGHT	CORRODED DULL BRIGHT CORRODED	DULL DULL BRIGHT DULL	CORRODED DULL DULL DULL
WT. LOSS (MG./SQ. CM.)	0.26 0.02 0.01 0.01	1.64 +0.01 +0.02 +0.02	0.32 0.01 0.00 +0.02	1.07 0.02 0.00 0.36	0.16 0.01 0.01 0.05	2.32 +0.05 +0.01 +0.05

Table 44

OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL HYDROGENATED FLUIDS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
 TEST FLUIDS: MLO 7079 AND MLO 7087 = A HYDROGENATED CATALYTIC CYCLE STOCK EXTRACT  
 MLO 7091 = A HYDROGENATED COASTAL 500 EXTRACT  
 ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT CO.

TEST FLUID	< - - - - - MLO 7079 1.0 PHENYL-ALPHA NAPHTHYLAMINE	- - - - -> 2.0 PRL 3605	< - - MLO 7087- -> 1.0 PHENYL-ALPHA NAPHTHYLAMINE	< - - MLO 7091- -> 1.0 PHENYL-ALPHA NAPHTHYLAMINE
OXIDATION INHIBITOR, WT. %	0	0	1	0
OVERALL LIQUID LOSS, WT. %	+158	+373	+77	+198
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	0.0 1.2	0.3 3.0	0.2 2.1	0.1 4.7
NEUT. NO. (MG. KOH/GM. OIL ORIGINAL FINAL	> 8 > 8	> 8 > 8	6 > 8	4 > 8
ASTM UNION COLOR ORIGINAL FINAL	0.4	3.4	2.2	2.5
WT. % INSOLUBLE MATERIAL				
FINAL CATALYST CONDITION APPEARANCE	DULL DULL DULL DULL	CORRODED DULL BRIGHT DULL	DULL DULL DULL DULL	DULL DULL DULL DULL
WT. LOSS (MG./SQ. CM.)	0.02 0.02 0.02 0.02	0.96 0.05 0.05 0.04	0.00 0.02 0.02 0.02	0.12 0.01 0.03 0.02

*Contrails*

the 347°F. oxidation test. Viscosity change appears somewhat higher for these fractions than for conventionally refined naphthenic and paraffinic stocks.

6. Oxidation Characteristics of Special Mineral Oils from Phillips Petroleum Company. The Phillips Petroleum Company has submitted a series of seven mineral oils for evaluation of the effect of super-refining techniques. The samples are described in the following tabulation:

MLO Designation	Lubricant Description	Viscosity in Centistokes at	
		210°F.	100°F.
7125	Charge Stock for Sample A	8.5	68.6
7126	Highly Treated Oil Sample A	7.9	57.5
7127	Charge Stock for Sample B	23.1	318
7128	Highly Treated Oil Sample B	19.7	230
7129	Charge Stock for Samples C and D	43.2	861
7130	Highly Treated Oil Sample C	30.2	447
7131	Highly Treated Oil Sample D	32.7	507

Oxidation and corrosion test results at 347°F. are shown on Table 46. Phenyl-alpha-naphthylamine in 1.0 weight per cent concentration has been used in all of these fluids. The charging stocks show relatively good overall stability in this oxidation and corrosion test. In general, the highly treated oils show little improvement over the charging stocks. It should be noted that all of the highly treated oils exhibit high neutralization numbers. The high neutralization numbers may influence adversely the copper corrosion and property changes noted with the highly treated oils.

7. Oxidation and Corrosion Characteristics of Mineral Oil-Polyolefin Blend. Blends of polyolefins and paraffin mineral oils have been evaluated as shown on Tables 47 and 48. The data on Table 47 were obtained at an air rate of 10 liters per hour. The two Pennsylvania neutrals used in these blends form a solid sludge-like product when subjected to this oxidation test without additives. These data have previously been shown on Tables 26 and 27.

The results of the two blends without additives indicate that the polyolefin alters significantly the course of the oxidation. The overall results appear to be essentially the same as the oxidation characteristics of the polyolefin as shown on Table 29. As in the case of the polyolefin, the phenyl-alpha-naphthylamine is not effective as an oxidation inhibitor for the 50:50 blends of mineral oil with polyolefin.

Data for polyolefin and hydrogenated polyolefin blends with Barosa 43 and Kendall heavy neutral are shown on Table 48. As a reference point similar data for the blend components are shown on Tables 31 and 33. It can be seen from the data on Table 48 that 50:50 blends of Indopol L-50 with Barosa 43 and Kendall heavy neutral do not respond to the dithiocarbamate inhibitor. The resultant property changes and copper corrosion values are in both cases intermediate between the values obtained for the two components of the blend tested separately. A blend of hydrogenated Indopol L-50 with Barosa 43 has been evaluated with PAN in the 347°F. test. The results indicate good overall stability and no metal corrosion.

#### 8. Study of Additives in Mineral Oils and Esters at 347°F.

A group of 10 chemicals have been obtained from the Monsanto Chemical Company for evaluation as oxidation and corrosion inhibitors. It should be emphasized that these materials were not prepared specifically as oxidation inhibitors. Solubility in a paraffinic mineral oil and di-2-ethylhexyl sebacate is shown in Table 45.

Table 45

SOLUBILITY OF MONSANTO ADDITIVES IN MINERAL OILS AND ESTERS

ADDITIVE DESIGNATION	MELTING POINT, °F.	SOLUBILITY OF 1% ADD. IN BAROSA 43		SOLUBILITY OF 10% ADD. IN DI-2-ETHYLHEXYL SEBACATE	
		AT 200°F.(1)	AT 80°F.(2)	AT 200°F.(1)	AT 80°F.(2)
MLO 7153	<80	SOLUBLE	INSOLUBLE	PARTIALLY SOLUBLE	INSOLUBLE
MLO 7154	>480	INSOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
MLO 7155	>480	INSOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
MLO 7156	383	PARTIALLY SOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
MLO 7157	198	SOLUBLE	INSOLUBLE	SOLUBLE	INSOLUBLE
MLO 7158	385	PARTIALLY SOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
MLO 7159	316	PARTIALLY SOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
MLO 7160	>400	INSOLUBLE	INSOLUBLE	INSOLUBLE	INSOLUBLE
MLO 7161	<80	SOLUBLE	SOLUBLE	PARTIALLY SOLUBLE	PARTIALLY SOLUBLE
MLO 7162	<80	SOLUBLE	SOLUBLE	PARTIALLY SOLUBLE	PARTIALLY SOLUBLE

(1) SOLUBILITY WAS OBSERVED AFTER MIXING AT 200°F. FOR ONE HOUR.

(2) SOLUBILITY WAS OBSERVED AFTER COOLING TO 80°F. AND STORAGE FOR 24 HOURS AT THIS TEMPERATURE.

*Contrails*

Seven of the ten additives show partial or complete solubility in Barosa 43 after one hour of stirring at 200°F. These materials were evaluated at 1.0 weight per cent concentration in Barosa 43 at 347°F. The data are shown on Table 49. Comparable data for 1.0 weight per cent phenyl-alpha-naphthylamine in Barosa 43 are shown for comparison. All of the additives evaluated, with the exception of MLO 7153, are at least as effective as phenyl-alpha-naphthylamine in reducing neutralization number increase and/or dirtiness. Only MLO 7158 is effective in eliminating copper corrosion.

The six compounds showing some solubility in di-2-ethylhexyl sebacate have been tested for stable life at 347°F. The stable life curves are shown on Figure 3. The fluid properties after 72 hours or longer are shown on Table 50. Five of the compounds evaluated show a finite stable life in one weight per cent concentration in di-2-ethylhexyl sebacate. The additive (MLO 7155) which shows no stable life is insoluble in the ester. Additive MLO 7153 shows a stable life of the order of 90 to 100 hours. MLO 7154 shows a relatively high neutralization number increase but no sharp break indicative of the break point in the stable life after 72 hours. The remaining three fluids exhibit a stable life of the order of 20 to 40 hours. All of these additives show excessive copper corrosion in the ester.

The Monsanto compounds, in spite of relatively poor solubility characteristics, show the properties of antioxidants in both mineral oils and esters.

D. HIGH TEMPERATURE OXIDATION AND CORROSION STUDIES. Oxidation tests at 347°F. are designed to be primarily a measure of inhibitor effectiveness and stable life. High temperature oxidation and corrosion tests at 500°, 600°, and 700°F. are used to measure oxidation rate and oxygen tolerance. Inhibitors in this temperature range are, in general, ineffective and may contribute to property changes and corrosion because of the thermal and oxidative instability of the additive itself. A preliminary evaluation indicates that the mineral oils show excellent high temperature oxidation and corrosion characteristics relative to the various classes of fluids evaluated. Additional studies in the mineral oil class have been made to evaluate the effects of mineral oil and hydrocarbon type, additional super-refining techniques, and additives.

1. Oxidation and Corrosion at 500°F. A number of mineral oils, hydrocarbons, and additives have been evaluated in a 20-hour oxidation test at 500°F. using Spec. MIL-L-7808 oxidation and corrosion procedures and techniques. Paraffin, naphthene, and aromatic types of mineral oil, and polyolefin and aromatic hydrocarbons have been included in this study. Essentially all of the mineral oil and hydrocarbon materials discussed in the preceeding section have been included. The data for these tests are shown on Tables 51 through 62.

Data are presented for the polyolefins before and after hydrogenation. The effect of additives has also been studied. These data are shown on Tables 51 and 52. The rate of oxidation for the polyolefins

Table 46

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MINERAL OILS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: ALL TEST FLUIDS ARE MINERAL OILS OBTAINED FROM THE PHILLIPS PETROLEUM COMPANY. NOTE: ALL FLUIDS CONTAIN 1.0 WEIGHT PER CENT PHENYL-ALPHA-NAPHTHYLAMINE.

MLO 7125 = CHARGE STOCK FOR SAMPLE A.  
MLO 7126 = HIGHLY TREATED OIL, SAMPLE A.MLO 7127 = CHARGE STOCK FOR SAMPLE B.  
MLO 7128 = HIGHLY TREATED OIL, SAMPLE B.MLO 7129 = CHARGE STOCK FOR SAMPLES C AND D.  
MLO 7130 = HIGHLY TREATED OIL, SAMPLE C.  
MLO 7131 = HIGHLY TREATED OIL, SAMPLE D.

TEST FLUID	MLO 7125	MLO 7126	MLO 7127	MLO 7128	MLO 7129	MLO 7130	MLO 7131
OVERALL LIQUID LOSS, WT.-%	2	4	2	2	1	1	2
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+10	+11	+51	+27	+72	+12	+142
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 1.5	1.0 0.8	0.1 4.7	0.9 2.1	0.1 4.7	0.5 2.5	1.1 7.1
ASTM UNION COLOR ORIGINAL FINAL	4 >8	5 >8	5 >8	6 >8	8 >8	>8 >8	>8 >8
WT.-% INSOLUBLE MATERIAL	1.3	0.8	0.5	0.6	TRACE	0.8	TRACE
FINAL CATALYST CONDITION APPEARANCE	CORRODED	DULL	DULL	CORRODED	CORRODED	CORRODED	CORRODED
COPPER	DULL	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	BRIGHT	BRIGHT	BRIGHT	BRIGHT
ALUMINUM	DULL	DULL	DULL	BRIGHT	BRIGHT	BRIGHT	BRIGHT
MAGNESIUM	DULL	DULL	DULL	BRIGHT	BRIGHT	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)							
COPPER	0.22	0.11	0.08	0.29	0.45	0.65	0.83
STEEL	+0.03	+0.02	+0.02	+0.02	+0.05	0.05	+0.01
ALUMINUM	0.00	0.00	+0.04	+0.01	+0.01	+0.05	0.00
MAGNESIUM	+0.04	+0.02	+0.04	+0.01	+0.05	+0.05	+0.03



Table 47

## OXIDATION AND CORROSION CHARACTERISTICS OF MIXTURES AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $10 \pm 1$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUIDS: MLO 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC MINERAL OIL).

MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL).

MLO 7014 = INDOPOL L-50 (POLYBUTENE POLYMER OF 110 CENTISTOKES VISCOSITY AT  $100^\circ\text{F}$ .).

TEST FLUID (RATIO BY WT.) ANTI-OXIDANT, WT. %	<- - - 50:50 MLO NONE	7026:MLO 7014 - - - > 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	<- - - 50:50 MLO 7027:MLO 7014 - - - > NONE 1.0 PHENYL-ALPHA- NAPHTHYLAMINE
OVERALL LIQUID LOSS, WT. %	5	3	4
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F}$ .	+258	+173	+172
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 11.7	0.1 7.9	0.1 8.1
ASTM UNION COLOR ORIGINAL FINAL	3 >8	5 >8	5 >8
WT. % INSOLUBLE MATERIAL	0.3	0.4	0.4
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM MAGNESIUM	DULL DULL BRIGHT BRIGHT	DULL DULL DULL DULL	DULL DULL DULL BRIGHT
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM MAGNESIUM	0.01 +0.01 +0.01 +0.01	0.07 0.00 0.02 +0.02	0.09 0.00 +0.01 0.00

Table 48

OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MIXTURES AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;  
 CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
 TEST FLUIDS: MLO 7101 = HYDROGENATED MLO 7102  
 MLO 7102 = INDOPOL L-50 (POLYOLEFIN) OBTAINED FROM THE INDOLIL CHEMICAL COMPANY.  
 MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.  
 MLO 7071 = HEAVY NEUTRAL OBTAINED FROM THE KENDALL REFINING COMPANY  
 ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID (RATIO BY WT.)	50:50 MLO 7101: MLO 7093 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	50:50 MLO 7101: MLO 7093 2.0 PRL 3605	50:50 MLO 7102: MLO 7071 2.0 PRL 3605	50:50 MLO 7102: MLO 7071 2.0 PRL 3605
OXIDATION INHIBITOR, WT. %				
OVERALL LIQUID LOSS, WT. %	2	1	2	3
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+13	+5	+31	+52
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 0.7	0.1 0.1	0.1 2.9	0.2 3.7
ASTM UNION COLOR ORIGINAL FINAL	4 8	2 5	2 >8	4 >8
WT. % INSOLUBLE MATERIAL FINAL CATALYST CONDITION APPEARANCE	1.0	0.4	0.4	0.6
COPPER	DULL	DULL	CORRODED	CORRODED
STEEL	COATED	COATED	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL
MAGNESIUM	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)				
COPPER	+0.02	+0.05	1.21	0.62
STEEL	+0.09	+0.11	+0.08	+0.01
ALUMINUM	+0.06	+0.03	+0.04	+0.02
MAGNESIUM	+0.08	+0.02	+0.08	+0.06

Table 49

## EFFECT OF VARIOUS ADDITIVES ON THE OXIDATION AND CORROSION CHARACTERISTICS OF BAROSA 43 AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: BAROSA 43 (MLO 7121) = A PARAFFINIC MINERAL OIL OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.

ADDITIVES: PAN = PHENYL-ALPHA-NAPHTHYLAMINE.  
MLO 7153, 7156, 7157, 7158, 7159, 7161, AND MLO 7162 = A SERIES OF EXPERIMENTAL ADDITIVES OBTAINED FROM THE MONSANTO CHEMICAL COMPANY.

TEST FLUID ADDITIVE, WT. %	BAROSA 43									
	NONE	1.0 PAN	1.0 MLO 7153	1.0 MLO 7156	1.0 MLO 7157	1.0 MLO 7158	1.0 MLO 7159	1.0 MLO 7161	1.0 MLO 7162	
OVERALL LIQUID LOSS, WT. %	1	1	1	1	2	1	1	1	1	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+12	+9	+12	+16	+8	+13	+13	+7	+11	
NEUT. NO. (MG. KOH/GM. OIL)										
ORIGINAL	0.1	0.1	0.1	0.1	0.0	0.2	0.1	0.1	0.0	
FINAL	4.3	2.6	3.9	4.1	2.2	3.8	3.2	2.2	2.4	
WT. % INSOLUBLE MATERIAL	4.1	3.4	4.7	3.1	2.2	2.7	3.2	1.2	2.7	
FINAL CATALYST CONDITION										
APPEARANCE										
COPPER	CORRODED	DULL	CORRODED	CORRODED	CORRODED	DULL	CORRODED	CORRODED	CORRODED	
STEEL	DULL	DULL	DULL	DULL	DULL	DULL	COATED	DULL	COATED	
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL	COATED	DULL	DULL	
MAGNESIUM	DULL	DULL	DULL	COATED	DULL	DULL	COATED	DULL	DULL	
WT. LOSS (MG./SQ. CM.)										
COPPER	1.40	0.03	4.35	0.63	5.78	+0.03	0.38	5.80	2.40	
STEEL	0.08	+0.05	0.02	0.06	0.01	+0.06	+0.28	0.04	+0.12	
ALUMINUM	0.05	+0.05	0.04	0.02	0.02	+0.02	+0.14	0.00	0.01	
MAGNESIUM	0.07	+0.05	0.00	+0.14	+0.01	+0.11	+0.86	+0.03	0.02	

Figure 3

## OXIDATION STABILITY AT 347°F. OF DI-2-ETHYLHEXYL SEBACATE CONTAINING VARIOUS ADDITIVES

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME AS INDICATED; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID = DI-2-ETHYLHEXYL SEBACATE (MLO 7038).

ADDITIVES: MLO 7153, 7154, 7155, 7157, 7161, AND MLO 7162 = A SERIES OF EXPERIMENTAL ADDITIVES OBTAINED FROM THE MONSANTO CHEMICAL COMPANY.

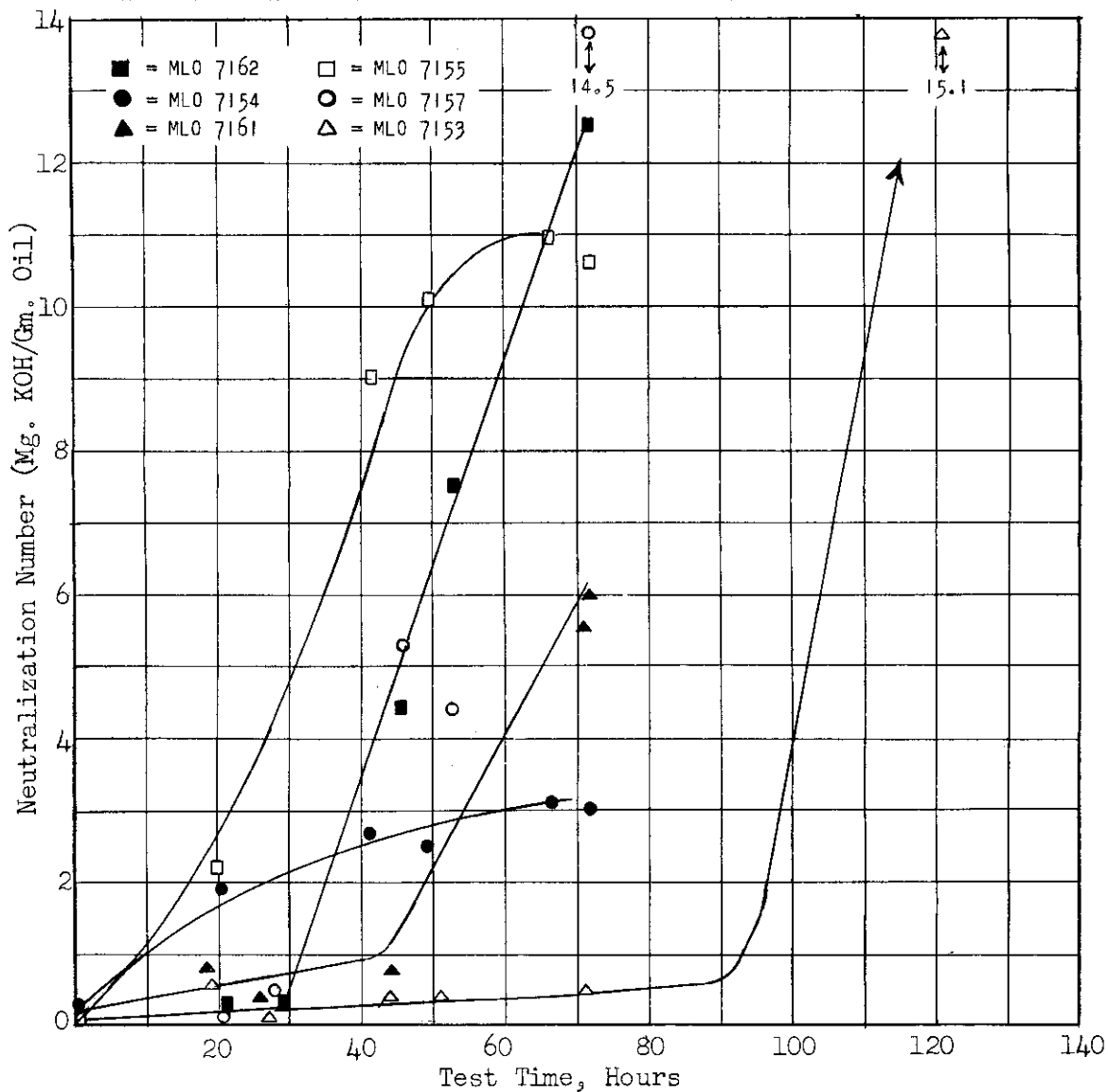


Table 50

## OXIDATION STABILITY AT 347°F. OF DI-2-ETHYLHEXYL SEBACATE CONTAINING VARIOUS ADDITIVES

TEST PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME AS INDICATED; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; AND CATALYSTS =

A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: DI-2-ETHYLHEXYL SEBACATE (MLO 7038).

ADDITIVES: MLO 7153, 7154, 7155, 7157, 7161, and MLO 7162 = A SERIES OF EXPERIMENTAL ADDITIVES OBTAINED FROM THE MONSANTO CHEMICAL COMPANY.

TEST FLUID OXIDATION INHIBITOR, WT. % TEST TIME, HOURS	DI-2-ETHYLHEXYL SEBACATE					1.0 MLO 7161				
	1.0 MLO 7155 72	1.0 MLO 7154 72	1.0 MLO 7153 116	1.0 MLO 7162 72	1.0 MLO 7157 72	1.0 MLO 7161 72	1.0 MLO 7157 72	1.0 MLO 7161 72	1.0 MLO 7157 72	1.0 MLO 7161 72
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+27	+4	+21	+20	+26	+9				
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 10.6	0.3 3.0	0.1 15.1	0.1 13.0	0.1 14.5	0.2 6.0				
ASTM UNION COLOR ORIGINAL FINAL	3 >8	3 >8	2 1/2 >8	2 1/2 >8	2 >8	3 1/2 >8				
WT. % OIL INSOLUBLE MATERIAL	0.7	0.7	0.6	0.8	0.8	0.3				
FINAL CATALYST CONDITION APPEARANCE	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED COATED COATED COATED	CORRODED COATED DULL DULL	CORRODED DULL COATED DULL				
WT. LOSS (MG./SQ. CM.)	3.55 +0.03 0.00 +0.01	1.84 +0.05 +0.06 0.12	9.65 +0.08 +0.01 +0.05	8.13 +0.62 +0.43 +0.16	28.35 +0.14 +0.07 +0.03	8.86 +0.05 +0.09 +0.07				

at 500°F. is about the same before and after hydrogenation. The use of phenyl-alpha-naphthylamine (PAN) or a dithiocarbamate antioxidant does not significantly lower the oxidation rate for these materials. There is some indication that oxygen tolerance is altered somewhat by hydrogenation and/or additives. Hydrogenation of the polyolefin tends to increase slightly the insolubles formed but decreases the change in viscosity noted. The use of PAN or dithiocarbamate increases slightly the formation of dirtiness in both base stocks.

In the case of the dithiocarbamate, the dirt formed is an orange colored finely divided material. This same type of deposit can be obtained from the thermal treatment of the dithiocarbamate additive in a thermally stable mineral oil or hydrocarbon.

In terms of overall behavior at 500°F., hydrogenation and additives do not improve the oxidative stability properties of the polyolefins. The use of the dithiocarbamate additive, PRL 3605, tends to promote copper corrosion in both the polyolefin and the hydrogenated stock.

A number of paraffinic mineral oils have been evaluated in the 500°F. oxidation test as shown in Tables 53, 54, 55, 56, and 57. A number of these paraffinic mineral oils have been studied before and after hydrogenation or silica gel adsorption, without additives. The results of these tests show that the hydrogenated and unhydrogenated oils have about the same oxidation stability at 500°F. That is, the rate of oxidation and the oxygen tolerance are not altered substantially by hydrogenation. In addition, both the hydrogenated and unhydrogenated mineral oils show good corrosion properties.

The effect of inhibitors on a paraffinic neutral are shown on Table 55. At best, the additives show a slight reduction in oxidation rate. In all cases this is accompanied by a reduction in oxygen tolerance based on dirtiness. Copper corrosion trends at 500°F. are less severe than at 347°F.

In addition to the tests at 500°F. with single inhibitors, a series of tests were conducted with a combination of hindered phenol and dialkyl acid phosphite as an oxidation inhibitor. This combination has been shown to have a synergistic effect as an oxidation inhibitor in the 347°F. tests with mineral oils and esters.

Oxidation tests at 500°F. have been conducted with Indopol L-50 (MLO 7014) and the two Pennsylvania extracted neutrals (MLO 7026 and MLO 7027) using a dialkyl acid phosphite-hindered phenol combination as an oxidation inhibitor. In one case Antioxidant 2246 was used as the hindered phenol, and in the other two cases Paranox 441 was used. Diisopropyl acid phosphite was the dialkyl acid phosphite used in all cases. These tests were conducted for 20 hours at an air rate of five liters per hour with the conventional metal catalysts. The results of these tests are compared on Table 58 with tests in which no oxidation inhibitor was used.

In general, no substantial improvement in oxidation stability is observed for blends containing the hindered phenol-dialkyl acid phosphite combination compared to the unblended fluids. The rate of oxidation, that is the amount of oxygen absorbed, is somewhat less in all cases, for the inhibited blends compared to the uninhibited fluids. However, the fluid properties after oxidation are not substantially different for the inhibited and the uninhibited fluids. In addition, there is a marked increase in the formation of insolubles for the blends containing the hindered phenol-dialkyl acid phosphite oxidation inhibitor compared to the uninhibited fluids. This same trend toward an increased formation of insolubles has also been noted for the cases where phenyl-alpha-naphthylamine was used as an oxidation inhibitor.

The effect of additives on Necton 50, a naphthenic mineral oil, is shown on Table 59. In no case are the overall properties of the fluid in this 500°F. oxidation test improved by the addition of additives. In several cases increased dirtiness and/or copper corrosion are noted.

Table 60 shows that hydrogenation of Necton 50 does not significantly alter the behavior in this oxidation test or the additive susceptibility of 500°F. Primol D, a highly refined white oil of the naphthenic class, exhibits good oxidation and corrosion characteristics at 500°F.

Two degrees of hydrogenation in an aromatic extract of cycle stock are compared with a butylated naphthylene in the 500°F. oxidation test on Table 61. MLO 7079 has been hydrogenated to the extent of 1.6 moles of hydrogen per mole of oil compared with 6.6 moles per mole for MLO 7087. The synthetic aromatic hydrocarbon MLO 7075 shows the lowest rate of oxidation and the smallest quantity of dirt formation. In the case of the hydrogenated cycle stock extracts, the higher the degree of hydrogenation the more severe the results of oxidation appear to be in this 500°F. test.

The seven mineral oils obtained from the Phillips Petroleum Company have been evaluated without additives at 500°F. as shown on Table 62. The highly refined oils MLO 7126, MLO 7128, MLO 7130, and MLO 7131 show about the same rate of oxidation as their respective base stocks. In all cases oxygen tolerance, as judged by dirtiness, is reduced for the highly treated oil over the base stock. No copper corrosion is noted at 500°F.

2. Oxidation and Corrosion at 600°F. Oxidation and corrosion tests at 600°F. have been conducted on the various mineral oil types listed above, both without additives and with 1.0 weight per cent phenyl-alpha-naphthylamine. These tests were conducted for a 20-hour test period with an air rate of five liters per hour using copper, steel, and aluminum catalysts. The results of these tests are found on Table 63.

In general, all of the fluids evaluated show reasonably good oxidation stability at 600°F. under these test conditions. The rate of oxidation, that is the amount of oxygen absorbed, is of about the same



order of magnitude for all mineral oils. Also, in terms of oxygen tolerance (the amount of fluid deterioration per unit of oxygen absorbed), there are only minor differences between the various fluids. The polybutene oil (MLO 7014) and the 50:50 mixtures of MLO 7014 with the Pennsylvania extracted neutrals tend to have a higher volatility loss than either the Pennsylvania neutrals alone or the naphthenic mineral oil (PRL 2052). The formation of oil insoluble material is not excessive for any of the neat fluids.

The addition of 1.0 weight per cent of phenyl-alpha-naphthylamine to each of the mineral oils does not improve the oxidation stability at 600°F. Phenyl-alpha-naphthylamine is an oxidation inhibitor which is effective at moderate temperatures. No significant reduction is observed in the rate of oxidation for blends containing the oxidation inhibitor compared to the non-additive fluids. In addition, a comparison of fluid properties after oxidation indicates that the oxygen tolerance is not improved by the presence of phenyl-alpha-naphthylamine. In some cases, a substantial increase in oil insoluble material is noted for fluids containing the oxidation inhibitor compared to the non-additive mineral oils.

E. EFFECT OF ACRYLOID POLYMERIC THICKENERS ON DIRTINESS AND SLUDGE DISPERSING PROPERTIES. It has been reported by the Wright Air Development Center that Spec. MIL-L-7808 fluids containing certain Acryloid thickeners show improved engine cleanliness over fluids without polymers in full scale engine testing. The improved engine cleanliness is attributed to the sludge dispersing characteristics of the polymer additive.

Other advantages of polymer thickened lubricants of the Spec. MIL-L-7808 type have been discussed in previous reports from this Laboratory on the development of PRL 3161 prior to the issuance of Spec. MIL-L-7808. These advantages are the improved viscosity-temperature characteristics resulting in higher viscosity at high temperature and a stable viscosity at -65°F. which is not subject to appreciable change with storage time. Since the development of PRL 3161 in 1950 and 1951, several additional Acryloid polymers of improved sludge dispersing types have become available. The Acryloid used in PRL 3161 is Acryloid HF-25. The additional Acryloids in this comparison are Acryloid HF-829 and Acryloid 966.

Oxidation and corrosion tests on typical Spec. MIL-L-7808 type fluids prepared with the various thickeners have been conducted at 347° and 500°F. to determine any evidence of reduced dirtiness found in engine tests. All of the Acryloids evaluated have been obtained from the Rohm and Haas Company. As a basis for comparison, Spec. MIL-L-7808 type fluids have been prepared from the same components except that no Acryloid was added.

The results of the 347°F. oxidation tests are shown on Table 65. These tests were conducted for the conventional 72-hour test period with



Table 51

## OXIDATION AND CORROSION CHARACTERISTICS OF INDOPOL L-50 AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.  
 TEST FLUID: MLO 7014 AND MLO 7102 = INDOPOL L-50 (POLYOLEFIN) OBTAINED FROM THE INDOL CHEMICAL COMPANY.  
 MLO 7101 = HYDROGENATED INDOPOL L-50.  
 ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID OXIDATION INHIBITOR, WT. %	<--- MLO 7014 --->		<--- MLO 7102 --->		<--- MLO 7101 --->	
	NONE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	NONE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	NONE	2.0 PRL 3605
LIQUID CHARGE, GRAMS	81	81	83	83	83	82
LIQUID LOSS, WT. %	13	8	12	19	13	13
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)						
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	26.2	26.2	26.2	26.2	26.2	26.2
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	7.2	7.6	9.1	8.8	7.6	7.6
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	1.18	1.26	1.47	1.42	1.24	1.24
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL						
FINAL	+63	+61	+93	+39	+4	+4
WT. % OIL INSOLUBLE MATERIAL (3)						
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)						
COPPER	0.01	+0.01	+0.03	0.00	0.00	0.01
STEEL	0.01	+0.01	0.02	+0.04	0.02	0.02
ALUMINUM	0.00	+0.02	+0.01	0.00	0.00	0.00

- (1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).  
 (2) AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.  
 (3) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.  
 (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 52

OXIDATION AND CORROSION CHARACTERISTICS OF A HYDROGENATED  
POLYOLEFIN AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID: MLO 7124 = HYDROGENATED POLYOLEFIN OBTAINED FROM THE INDOIL CHEMICAL COMPANY.

ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM THE R. T. VANDERBILT COMPANY.

TEST FLUID	<----- MLO 7124 ----->		
	-	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	2.0 PRL 3605
OXIDATION INHIBITOR, WT.%			
LIQUID CHARGED, GRAMS	81	80	80
LIQUID LOSS, WT.%	16	17	10
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (1) AT 130°F.	+6	+2	-14
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.1	0.1	0.1
FINAL	3.8	2.4	1.8
WT.% OIL INSOLUBLE MATERIAL (1)	0.5	1.3	1.5
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	DULL	DULL	CORRODED
STEEL	DULL	DULL	DULL
ALUMINUM	BRIGHT	DULL	DULL
WT. LOSS (MG./SQ. CM.)			
COPPER	0.05	0.02	0.51
STEEL	0.04	0.02	0.02
ALUMINUM	0.05	+0.01	0.02

(1) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF THE TEST. THE INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 53

**EFFECT OF HYDROGENATION ON THE OXIDATION AND CORROSION  
CHARACTERISTICS OF TWO MINERAL OILS AT 500°F.**

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.

MLO 7113 = HYDROGENATED MLO 7093.

MLO 7094 = AVIATION BRIGHT STOCK OBTAINED FROM THE STANDARD OIL COMPANY OF NEW JERSEY.

MLO 7118 = HYDROGENATED MLO 7094.

TEST FLUID	MLO 7093	MLO 7113	MLO 7094	MLO 7118
LIQUID CHARGED, GRAMS	84	83	83	82
LIQUID LOSS, WT. %	3	6	1	0
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	8.8	7.8	3.9	5.0
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	1.40	1.24	0.62	0.81
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+60	+59	+38	+17
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL	0.2	0.1	0.1	0.1
FINAL	2.9	4.2	1.3	4.3
WT. % OIL INSOLUBLE MATERIAL (3)	TRACE	TRACE	TRACE	0.1
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER	DULL	DULL	COATED	DULL
STEEL	BRIGHT	DULL	DULL	DULL
ALUMINUM	BRIGHT	DULL	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)				
COPPER	+0.06	0.03	+0.34	0.02
STEEL	0.08	0.00	+0.04	+0.02
ALUMINUM	0.00	+0.01	0.00	0.00

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 54

## OXIDATION AND CORROSION CHARACTERISTICS OF A PENNSYLVANIA SAE 30 MINERAL OIL AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7077 = A PENNSYLVANIA SAE 30 (PARAFFINIC MINERAL OIL)

MLO 7076 = HYDROGENATED MLO 7077

ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R.T. VANDERBILT CO.

TEST FLUID OXIDATION INHIBITOR, WT. %	<--- MLO 7077 ---> NONE	<--- MLO 7076 ---> NONE	<--- PRL 3605 ---> 2.0 PRL 3605
LIQUID CHARGED, GRAMS	86 1	86 3	85 1
LIQUID LOSS, WT. %	26.2 7.4	26.2 5.6	26.2 4.4
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)			
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	1.15	0.85	0.69
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)			
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+69	+54	+22
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.7 3.7	0.4 3.3	0.0 1.6
WT. % OIL INSOLUBLE MATERIAL (3)	0.1	TRACE	0.6
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM	DULL DULL BRIGHT	DULL DULL BRIGHT	CORRODED DULL DULL
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM	+0.05 0.06 0.03	0.74 0.03 +0.02	0.23 0.05 0.04

(1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 55

## OXIDATION AND CORROSION CHARACTERISTICS OF A PARAFFINIC MINERAL OIL AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID: MLO 7070 = A PARAFFINIC MINERAL OIL OBTAINED FROM THE KENDALL REFINING COMPANY.

ADDITIVE: PRL 3605 AND PRL 3606 = DITHIOCARBAMATE TYPE ADDITIVES OBTAINED FROM R. T. VANDERBILT CO.

PRL 3615 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM THE LUBRIZOL CORPORATION.

TEST FLUID OXIDATION INHIBITOR, WT. %	< - - - - - NONE	- - - - - 1.0 PHENYL-ALPHA- NAPHTHYLAMINE	- - - - - MLO 7070 1.0 PRL 3615	- - - - - 2.0 PRL 3605	- - - - - 2.0 PRL 3606
LIQUID CHARGED, GRAMS	85	86	84	86	85
LIQUID LOSS, WT. %	2	.1	2	4	1
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	8.0	5.0	4.6	3.8	4.7
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	1.25	0.78	0.73	0.59	0.74
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+78	+55	+73	+49	+56
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 4.5	0.3 1.5	0.9 3.3	0.1 2.2	0.1 2.5
WT. % OIL INSOLUBLE MATERIAL (3)	0.1	0.2	0.2	0.4	0.4
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM	DULL DULL BRIGHT	COATED DULL DULL	CORRODED DULL COATED	DULL DULL DULL	DULL DULL DULL
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM	0.04 0.04 0.07	0.17 0.02 0.02	2.19 +0.03 +0.16	+0.08 +0.01 +0.04	0.10 +0.01 0.01

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).(2) AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(3) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 56

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MINERAL OIL COMPOSITIONS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A ONE INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7014 = INDOPOL L-50 (POLYBUTENE POLYMER OF 110 CENTISTOKES AT 100°F.).

MLO 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC MINERAL OIL).

MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL).

MLO 7071 = A PENNSYLVANIA HEAVY NEUTRAL (PARAFFINIC MINERAL OIL).

TEST FLUID	MLO 7071	MLO 7026	MLO 7027	50:50 (BY WT.) MLO 7026:MLO 7014 1.0	50:50 (BY WT.) MLO 7027:MLO 7014 1.0
PHENYL- $\alpha$ -NAPHTHYLAMINE, WT. %	-	-	-	-	-
LIQUID CHARGED, GRAMS	86	84	83	83	82
LIQUID LOSS, WT. %	1	1	0	4	6
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GRAMS (1)	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GRAMS (1)	3.5	-	7.4	5.5	7.1
MOLS O <sub>2</sub> USED/426 GRAMS OF FLUID (2)	0.54	-	1.19	0.88	1.16
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+46	+50	+55	+67	+60
NEUT. NO. (MG. KOH/GM. OIL)	0.1	0.1	0.2	0.1	0.0
ORIGINAL	3.0	2.6	3.9	2.6	3.0
FINAL	NONE	0.1	0.1	TRACE	0.1
WT. % OIL INSOLUBLE MATERIAL (3)					
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT	DULL	BRIGHT	DULL
WT. LOSS (MG./SQ. CM.)					
COPPER	+0.05	0.00	0.02	0.03	+0.04
STEEL	0.03	+0.02	0.04	+0.01	+0.03
ALUMINUM	0.05	0.00	0.01	+0.01	0.00

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).(2) AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(3) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 57

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL FLUIDS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC MINERAL OIL).  
MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL).

TEST FLUID FLUID TREATMENT	<-----MLO 7026-----> NONE	SILICA GEL (4)	<-----MLO 7027-----> NONE	SILICA GEL (4)
LIQUID CHARGED, GRAMS	83	80	83	82
LIQUID LOSS, WT. %	0	4	0	7
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	9.1	5.6	7.4	6.9
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	1.46	0.93	1.19	1.12
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+50	+34	+55	+52
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL	0.1	0.1	0.2	0.2
FINAL	2.9	3.7	3.9	4.5
WT. % OIL INSOLUBLE MATERIAL (3)	TRACE	0.3	0.1	0.2
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER	DULL	BRIGHT	DULL	BRIGHT
STEEL	DULL	DULL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT	DULL	BRIGHT
WT. LOSS (MG./SQ. CM.)				
COPPER	0.00	0.00	0.02	0.01
STEEL	0.01	0.01	0.04	+0.04
ALUMINUM	0.05	+0.02	0.01	+0.02

(1) APPROX. AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HRS.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).(2) AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(3) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(4) OIL INSOLUBLE MATERIAL IS DETERMINED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(5) OIL PASSED THROUGH A COLUMN 1.6 INCHES IN DIAMETER AND 7 FEET HIGH CONTAINING ACTIVATED SILICA GEL AT APPROX. 150°F. (OIL TO SILICA GEL RATIO APPROX. 2 TO 1).

Table 58

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL FLUIDS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;

CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7014 = INDOPOL L-50 (POLYBUTENE POLYMER OF 110 CENTISTOKES VISCOSITY AT 100°F.)

MLO 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC MINERAL OIL)

MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL)

TEST FLUID ADDITIVE, WT. %	MLO 7014			MLO 7026			MLO 7027		
ANTIOXIDANT 2246	-	-	1.0	-	-	-	-	-	-
PARANOX 441	-	-	-	-	-	-	-	-	-
DIIISOPROPYL ACID PHOSPHITE	-	-	0.5	-	-	-	-	-	-
LIQUID CHARGED, GMS.	81	13	81	83	0	85	83	0	83
LIQUID LOSS, WT. %	13	26.2	9	0	26.2	2	0	26.2	1
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	7.2	5.6	5.6	9.1	5.5	5.5	7.4	4.8	4.8
MOLS O <sub>2</sub> USED/426 GMS. FLUID (4)	1.18	0.93	0.93	1.46	0.86	0.86	1.19	0.77	0.77
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (2)									
AFTER REMOVAL OF ISOPENTANE INSOLUBLE MATERIAL (3)	+63		+69	+50		+52	+55		+38
AT 130°F.									
AFTER REMOVAL OF ISOPENTANE INSOLUBLE MATERIAL (3)	+71		+78	+49		+43	-		+41
AT 130°F.									
NEUT. NO. (MG. KOH/GM. OIL)									
ORIGINAL	0.0	2.2	0.4	0.1	0.1	0.1	0.2	0.3	0.3
FINAL	2.2	2.0	2.0	2.9	2.9	2.3	3.9	2.5	2.5
WT. % OIL INSOLUBLE MATERIAL (2)	TRACE		0.8	TRACE		0.4	0.1		0.6
WT. % ISOPENTANE INSOLUBLE MATERIAL (3)	0.1		1.0	0.3		0.5	0.6		0.7
FINAL CATALYST CONDITION									
APPEARANCE									
COPPER	DULL		DULL	DULL		COATED	DULL		DULL
STEEL	DULL		COATED	DULL		COATED	DULL		COATED
ALUMINUM	BRIGHT		COATED	BRIGHT		COATED	BRIGHT		COATED
WT. LOSS (MG./SQ. CM.)									
COPPER	0.01		0.02	0.00		+0.11	0.02		0.01
STEEL	0.01		+0.29	0.01		+0.39	0.04		+0.39
ALUMINUM	0.00		+0.28	0.05		+0.78	+0.01		+0.59

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HRS.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).(2) AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(3) OIL INSOLUBLE MATERIAL IS DETERMINED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THE ISOPENTANE INSOLUBLE MATERIAL IS THE SUM OF THE OIL INSOLUBLE MATERIAL PLUS THE ISOPENTANE INSOLUBLE MATERIAL. ISOPENTANE INSOLUBLE MATERIAL IS DETERMINED BY DILUTING A SAMPLE OF THE OXIDIZED FLUID (FROM WHICH THE OIL INSOLUBLE MATERIAL HAS BEEN REMOVED) IN ISOPENTANE AND WEIGHING THE INSOLUBLE MATERIAL WHICH SETTLES OUT.

(5) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF D1-2-ETHYLHEXYL SEBACATE IS 426.



Table 59

OXIDATION AND CORROSION CHARACTERISTICS OF NECTON 50 AT 500°F.

TEST FLUID: PRL 2052 = NECTON 50, A NAPHTHENIC MINERAL OIL  
 ADDITIVE: PRL 3615 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM LUBRIZOL CORPORATION.  
 PRL 3616 = A THIOPHOSPHATE TYPE ADDITIVE OBTAINED FROM AMERICAN CYANAMID CO.

TEST FLUID OXIDATION INHIBITOR, WT. %	< ----- &dashv; PRL 2052 ----- &dashv;		----- &dashv; PRL 3615 ----- &dashv;		----- &dashv; PRL 3616 ----- &dashv;		&dashv; PRL 3605 ----- >	
	NONE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0	1.0	1.0	1.0	2.0	
LIQUID CHARGED, GRAMS	87	86	88	87	87	87	87	
LIQUID LOSS, WT. %	2	1	0	0	0	0	0	
APPROX. AMOUNT OF O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2	26.2	26.2	
APPROX. AMOUNT OF O <sub>2</sub> USED, GMS. (1)	5.7	-	6.1	4.9	4.9	4.9	4.5	
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	0.87	-	0.34	0.75	0.75	0.75	0.69	
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+72	+84	+66	+69	+69	+69	+58	
NEUT. NO. (MG. KOH/GM. OIL)	0.1	0.1	1.0	1.8	1.8	1.8	0.0	
ORIGINAL	3.7	2.9	3.1	3.3	3.3	3.3	2.8	
FINAL	0.3	0.7	0.2	0.4	0.4	0.4	0.5	
WT. % OIL INSOLUBLE MATERIAL (3)								
FINAL CATALYST CONDITION								
APPEARANCE	COATED	DULL	CORRODED	CORRODED	CORRODED	CORRODED	DULL	
COPPER	DULL	DULL	DULL	DULL	DULL	DULL	DULL	
STEEL	BRIGHT	BRIGHT	DULL	DULL	DULL	DULL	BRIGHT	
ALUMINUM								
WT. LOSS (MG./SQ. CM.)								
COPPER	+0.21	+0.15	1.84	2.07	2.07	2.07	0.05	
STEEL	+0.02	+0.01	0.05	+0.05	+0.05	+0.05	0.03	
ALUMINUM	+0.03	+0.05	+0.02	+0.03	+0.03	+0.03	0.03	

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).
  - (2) AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
  - (3) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 60

## OXIDATION AND CORROSION CHARACTERISTICS OF HYDROGENATED NECTON 50 AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS: AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID: MLO 7066 AND MLO 7096 = HYDROGENATED NECTON 50 (NAPHTHENIC MINERAL OIL).

MLO 7144 = PRIMOL D, A HIGHLY REFINED NAPHTHENIC WHITE OIL

ADDITIVE: PRL 3605 = A DITHIOCARBAMATE TYPE ADDITIVE OBTAINED FROM R. T. VANDERBILT COMPANY.

TEST FLUID	MLO 7066	MLO 7096		MLO 7144
OXIDATION INHIBITOR, WT. %	NONE	2.0 PRL 3605		NONE
LIQUID CHARGED, GRAMS	86	86	86	85
LIQUID LOSS, WT. %	6	1	1	3
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	6.9	5.5	5.9	9.3
MOLS O <sub>2</sub> USED/426 GRAMS FLUID(2)	1.08	0.84	0.91	1.45
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+76	+58	+42	+67
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL	0.2	0.0	0.0	0.0
FINAL	3.3	2.4	2.4	2.8
WT. % OIL INSOLUBLE MATERIAL (3)	0.3	0.6	0.6	TRACE
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER	DULL	CORRODED	DULL	BRIGHT
STEEL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	BRIGHT
WT. LOSS (MG./SQ. CM.)				
COPPER	+0.01	0.49	0.10	0.02
STEEL	+0.02	+0.02	0.01	0.02
ALUMINUM	0.02	0.02	0.00	+0.02

- (1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FRE-  
QUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING  
THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLE-  
TION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA  
AND DRIED BEFORE WEIGHING.

Table 61

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MINERAL OILS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.  
 TEST FLUIDS: MLO 7079 = A HYDROGENATED CATALYTIC CYCLE STOCK EXTRACT.  
 MLO 7087 = A HYDROGENATED CATALYTIC CYCLE STOCK EXTRACT.  
 MLO 7075 = BUTYLATED NAPHTHALENES.

TEST FLUID	MLO 7079	MLO 7087	MLO 7075
LIQUID CHARGED, GRAMS	99	95	93
LIQUID LOSS, WT. %	0	2	1
APPROX. AMOUNT O <sub>2</sub> SUPPLIED GMS. (1)	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED GMS. (1)	6.4	6.3	3.2
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	0.87	0.97	0.45
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+222	+252	+165
NEUTRALIZATION NUMBER (MG. KOH/GM. OIL)			
ORIGINAL	0.3	0.2	0.1
FINAL	2.6	4.5	2.7
WT. % OIL INSOLUBLE MATERIAL (3)	0.6	2.0	0.2
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	DULL	BRIGHT	DULL
STEEL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	BRIGHT
WT. LOSS (MG./SQ. CM.)			
COPPER	0.02	0.05	0.09
STEEL	0.00	0.01	0.08
ALUMINUM	0.02	0.03	0.08

- (1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).  
 AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 62

## OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MINERAL OILS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: ALL TEST FLUIDS ARE MINERAL OILS OBTAINED FROM THE PHILLIPS PETROLEUM COMPANY.

MLO 7125 = CHARGE STOCK FOR SAMPLE A.

MLO 7127 = CHARGE STOCK FOR SAMPLE B.

MLO 7126 = HIGHLY TREATED OIL, SAMPLE A.

MLO 7128 = HIGHLY TREATED OIL, SAMPLE B.

NOTE: ALL FLUIDS CONTAIN 1.0 WT. PER CENT PHENYL-ALPHA-NAPHTHYLAMINE.

MLO 7129 = CHARGE STOCK FOR SAMPLES C AND D.

MLO 7130 = HIGHLY TREATED OIL, SAMPLE C.

MLO 7131 = HIGHLY TREATED OIL, SAMPLE D.

TEST FLUID	MLO 7125	MLO 7126	MLO 7127	MLO 7128	MLO 7129	MLO 7130	MLO 7131
LIQUID CHARGED, GRAMS	86	85	91	83	84	82	83
LIQUID LOSS, WT. %	0	0	2	0	0	0	1
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	5.1	4.6	3.7	4.4	3.6	3.1	4.4
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	0.80	0.73	0.56	0.71	0.57	0.50	0.70
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.							
NEUT. NO. (MG. KOH/GM. OIL)							
ORIGINAL	+60	+181	+50	+46	+94	+36	+90
FINAL	0.1	1.0	0.1	0.9	0.1	0.5	1.1
WT. % OIL INSOLUBLE MATERIAL (3)	2.2	1.6	1.0	1.4	1.4	1.5	2.4
FINAL CATALYST CONDITION	TRACE	0.6	0.1	0.4	0.2	0.6	0.3
APPEARANCE							
COPPER	COATED	DULL	COATED	COATED	COATED	COATED	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL	BRIGHT	DULL
WT. LOSS (MG./SQ. CM.)							
COPPER	+0.34	+0.07	+0.23	+0.11	+0.18	+0.07	0.00
STEEL	+0.02	0.07	+0.02	0.02	0.00	0.02	0.02
ALUMINUM	0.06	+0.01	0.01	+0.02	0.00	0.00	0.03

(1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR., S.T.P.) X TIME (HRS.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).  
AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF O<sub>2</sub> IS 32.  
ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Contrails

Table 63

# OXIDATION AND CORROSION CHARACTERISTICS OF SEVERAL MINERAL OIL COMPOSITIONS AT 600°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A ONE INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: PRL 2052 = NECTON 50 (A HIGHLY REFINED NAPHTHENIC MINERAL OIL, 65 CENTISTOKES AT 100°F.)

MLQ 7014 = INDOPOL L-50 (POLYBUTENE POLYMER OF 110 CENTISTOKES AT 100°F.)

MLQ 7026 = A PENNSYLVANIA EXTRACTED 300 NEUTRAL (PARAFFINIC MINERAL OIL)

MLQ 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL)

TEST FLUID	PRL 2052	MLQ 7014	MLQ 7026	MLQ 7027	50:50 (BY WT.) MLQ 7026:MLQ 7014	50:50 (BY WT.) MLQ 7027:MLQ 7014
PHENYL- $\alpha$ -NAPHTHYLAMINE, WT.-%	-	1.0	-	1.0	1.0	1.0
LIQUID CHARGED, GRAMS	88	82	86	84	83	83
LIQUID LOSS, WT.-%	10	38	16	20	25	36
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	12.8	13.1	-	12.7	12.1	11.0
MOLS O <sub>2</sub> USED/426 GRAMS OF FLUID (2)	1.94	2.10	-	2.02	1.95	1.76
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+193	+182	+57	+281	+104	+362
NEUT. NO. (MG. KOH/GM. OIL)	0.1	0.1	0.1	0.2	0.2	0.2
ORIGINAL	3.1	3.7	6.6	3.8	1.7	6.5
FINAL	0.9	0.2	0.6	0.3	0.1	0.8
WT.-% OIL INSOLUBLE MATERIAL (3)						
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	COATED	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	BRIGHT	BRIGHT	DULL
WT. LOSS (MG./SQ. IN.)						
COPPER	+0.12	+0.02	+0.09	+0.08	+0.05	+0.06
STEEL	0.07	0.08	0.07	0.22	0.03	+0.06
ALUMINUM	+0.01	+0.03	0.00	+0.02	+0.02	+0.02

- (1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).
- (2) AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (3) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

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an air rate of 10 liters per hour using copper, steel, aluminum, and magnesium catalysts. The results of these tests indicate that all of the Acryloid polymers tend to reduce the amount of sludge formed under these test conditions. In all cases the amount of sludge formed is less for blends containing the polymers than for the same blends without the polymer. In addition, no deleterious effect is noted that could be attributed to the presence of the Acryloids. That is, the neutralization number increase, volatility loss, and viscosity change are essentially the same for tests conducted with and without the polymers. In addition, no catalyst corrosion is obtained in any of these tests under these conditions.

The 500°F. oxidation and corrosion tests with these fluids are summarized on Table 66. These tests were conducted for a 20-hour test period with an air rate of five liters per hour using copper, steel, and aluminum catalysts. The results of these tests indicate that the polymers studied are still effective in reducing the formation of sludge. Acryloid HF-829 is the least effective of those evaluated. That is, tests conducted with Acryloid 966 in the blends containing 1.0 and 5.0 weight per cent tricresyl phosphate, and the test with Acryloid HF-25 in the blend containing 5.0 weight per cent tricresyl phosphate, show a substantial reduction in sludge formation compared to tests on the same blends which did not contain the polymers.

In the tests at 500°F., a substantial reduction in viscosity change is observed for the blends containing Acryloids compared to the blends without the polymer. This difference is undoubtedly due to incipient thermal instability of the polymers.

Previous studies conducted in this Laboratory have shown that Acryloid polymers are not entirely thermally stable at 500°F. In addition, a small amount of copper corrosion is obtained in these tests. This is typical of oxidation tests conducted on di-2-ethylhexyl sebacate blends containing tricresyl phosphate and is not attributed to the presence of the Acryloid polymers.

A section in Annual Report WADC TR 55-30 Pt 3 prepared by this Laboratory is devoted to a high temperature dirtiness study of di-2-ethylhexyl sebacate-base fluids containing tricresyl phosphate. The oxidation property changes in this study at 500°F. and 20 hours were about the same as those shown on Table 66 for comparable fluids except for the dirtiness values noted. Oil insolubles in this previous study were higher by a factor of three to five than in the current study. It should be emphasized that a new batch of tricresyl phosphate and an improved quality di-2-ethylhexyl sebacate have been used in the current studies. It is not readily apparent which material is responsible for the major portion of the improvements noted.

Additional tests have been conducted to determine whether these polymeric additives are effective in reducing sludge formation in mineral oils. As pointed out previously, mineral oils are one of the more promising types of materials available for high temperature hydraulic



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and lubricant systems. However, it has been shown in recent reports that sludge formation on oxidation in the intermediate temperature range (200° to 400°F.) can be relatively severe with these oils. The use of polymeric dispersants can be considered either as a replacement for super-refining to reduce dirtiness or as a supplement to super-refining to reduce further dirtiness in the intermediate temperature range.

Oxidation and corrosion tests on mineral oil compositions prepared with the two polymeric thickeners (Acryloid HF-829 and Acryloid 966) have been conducted at 347° and 500°F. Two different mineral oils, Barosa 43 and Esstic 45, were used as base stock materials for these studies. Barosa 43 is a well-refined paraffinic mineral oil and Esstic 45 is a well-refined naphthenic mineral oil. Both of these fluids were obtained from the Standard Oil Company of New Jersey.

The results of the 347°F. tests are shown on Tables 67 and 68. These tests were conducted for the conventional 72-hour test period with an air rate of five liters per hour using copper, steel, aluminum, and magnesium catalysts. The results of these tests show that both the Acryloid HF-829 and the Acryloid 966 are quite effective in reducing the amount of sludge formed for both mineral oil-base fluids under these test conditions. For example, the sludge formed for Barosa 43 (MLO 7093) is 3.4 weight per cent without the polymeric thickener. However, this value was reduced to 0.1 weight per cent with Acryloid 966 and to 0.3 weight per cent with Acryloid HF-829. Similarly, Esstic 45 has 2.4 weight per cent sludge under these test conditions without the polymeric thickeners, but shows only trace dirtiness with Acryloid 966 and 0.4 weight per cent sludge with Acryloid HF-829 as sludge dispersants.

With the exception of the sludge formation, no significant differences in fluid properties after oxidation are observed for the mineral oil blends with and without the polymeric thickeners. That is, the neutralization number increase, viscosity change, and volatility loss are essentially the same for the fluids with and without the additives. No substantial difference in the amount of catalyst corrosion is obtained in any of the tests under these conditions.

The 500°F. oxidation and corrosion tests for these fluids are tabulated on Tables 69 and 70. These tests were conducted for a 20-hour test period with an air rate of five liters per hour using copper, steel, and aluminum catalysts. In addition to the tests conducted with blends of the Acryloid polymers in Barosa 43 or Esstic 45 which contained only phenyl-alpha-naphthylamine as an oxidation inhibitor, two tests were conducted with and without Acryloid 966 in blends of Esstic 45 which contained 5.0 weight per cent of tricresyl phosphate.

The results of these tests again indicate that the polymeric thickeners are still effective in reducing the amount of sludge formed. This is particularly evident in the tests conducted with blends of Esstic 45. For the two tests containing only Esstic 45 and phenyl-alpha-naphthylamine, values of 2.3 and 1.1 per cent sludge were obtained.

However, for the same blend containing Acryloid 966 or Acryloid HF-829 the sludge values were reduced to 0.2 and 0.3 weight per cent, respectively.

Similarly, for the test conducted with Esstic 45 and tricresyl phosphate the sludge value was 1.1 weight per cent compared to 0.4 weight per cent for the comparable blend containing Acryloid 966.

For the 500°F. tests conducted with Barosa 43 neat or with the polymer additives, the sludge values are low in all cases. Again, no deleterious effects due to the presence of the polymeric thickeners are noted in the fluid properties after oxidation. A reduction in viscosity is obtained for the blends containing the Acryloid polymers. This is undoubtedly due to incipient thermal instability of the polymers. The volatility loss, the neutralization number, and the rate of oxidation (the amount of oxygen absorbed) are essentially the same for the tests with and without the Acryloid thickeners.

Two additional dispersants (Acryloids 794 and 917) have been evaluated in Barosa 43 at 347° and 500°F. as shown on Tables 71 and 72. The results indicate that Acryloids 794 and 917 are effective dispersants over the 347° to 500°F. temperature range. On the basis of these oxidation tests, there is no apparent difference in general effectiveness between the four Acryloids evaluated in the mineral oils.

The same test fluids shown on Table 71 have been used in panel coker studies. The panel coker results are shown on Table 64. These data indicate that the presence of the dispersant type Acryloid has only minor effects, if any, on the coking.

Table 64

## EFFECT OF ACRYLOID POLYMER ON PANEL COKING BEHAVIOR

TESTS CONDUCTED IN MODEL C COKER. TEST TIME = 8 HOURS; PANEL TEMPERATURE = 600°F.

TEST FLUID (CONC. IN WT.%)	WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID) ORIGINAL	FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.
BAROSA 43 (MLO 7093) A PARAFFINIC MINERAL OIL	162	0.2	1.4	195	253
BAROSA 43 + 1.0 PHENYL-ALPHA- NAPHTHYLAMINE					
+ 5.0 ACRYLOID 794	204	0.1	0.3	215	240
+ 5.0 ACRYLOID 966	116	0.2	0.5	225	240
+ 5.0 ACRYLOID 917	173	0.1	0.3	220	230

The products from these panel coker runs have been used to conduct additional 347°F. oxidation studies. The results of these oxidation tests are shown on Table 73. It can be seen by comparing Tables 71 and 73 that the panel coker products show increased property changes in viscosity and neutralization number over the unused fluids subjected to the same oxidation test. The dispersant activity of the Acryloids is not



measurably affected, however, by the panel coking pretreatment. The panel coker pretreatment does increase copper corrosion in all of the fluids tested. This increased metal corrosion does not appear to be caused by the Acryloid.

A series of six-hour thermal stability tests in the presence of copper, steel, and aluminum catalysts have been conducted with Acryloid blends of Esstic 45, Barosa 43, and hydrogenated Barosa 43. The tests were run in the glass thermal stability tubes under a nitrogen atmosphere. Results of the test are shown on Table 74. All of the Acryloid blends tested show more viscosity loss, neutralization number increase, and insoluble material following the test than do the mineral oil base stocks. In all blends containing Acryloid HF-829, substantial steel corrosion is noted along with relatively high sludge values. The blends of Acryloids 966, 917, and 794 show good corrosion characteristics and only a trace of dirtiness.

These data indicate that Acryloids 966, 917, and 794 exhibit good dispersing characteristics over the temperature range of 347° through 500°F. Thermal stability and corrosion characteristics at 700°F. of blends containing these dispersants exhibit good overall stability and corrosion properties.

F. EFFECT OF PHOSPHORUS TYPE LUBRICITY ADDITIVES ON THE OXIDATION AND CORROSION CHARACTERISTICS OF A MINERAL OIL. Data have been presented in previous yearly reports to show that phosphorus-containing lubricity additives are effective in improving lubricity at high temperatures as well as at low temperatures. Beneficial lubricity effects in esters and mineral oils at temperatures of 500° and 600°F. have been demonstrated in the Shell four-ball wear tester and the Vickers vane power steering pump. These data have been presented in WADC TR 55-30 Pt 3 and WADC TR 55-30 Pt 4. Data are also presented in this report to show that diisopropyl acid phosphite is effective as a lubricity additive in mineral oils at 700°F.

Adverse effects on the oxidation and thermal stability of esters at high temperatures have been shown to be due to the presence of phosphorus-containing lubricity additives. The presence of tricresyl phosphate or alkyl acid phosphites causes sharply increased sludging tendencies at 500°F. These studies have been extended to include phosphorus additives in mineral oils.

Oxidation and corrosion tests at 347°, 500°, and 600°F. have been conducted to determine the effect of tricresyl phosphate and diisopropyl acid phosphite on the oxidation and corrosion characteristics of Necton 60. Necton 60 is a well-refined naphthenic mineral oil obtained from the Standard Oil Company of New Jersey. In most cases 1.0 weight per cent Antioxidant 2246 was used as an oxidation inhibitor. Antioxidant 2246 is a hindered-phenol type oxidation inhibitor. The 347°F. tests were conducted using Spec. MIL-L-7808 test procedures and techniques. These tests were conducted for 72 hours at an air rate of five liters per hour using copper, steel, aluminum, and magnesium catalysts.

Table 65

EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808. TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347<sup>±</sup> 3°F.; AIR RATE = 10 ± 1 LITERS PER HOUR; TEST TIME = 72 HOURS; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID BASE STOCK TRICRESYL PHOSPHATE, WT. % ACRYLOID POLYMER, WT. %	DI-2-ETHYLHEXYL SEBACATE (MLO 7038) + 0.5 WT. % PHENOTHIAZINE					5.0 ACRYLOID 966	
	1.0	5.0	3.9 ACRYLOID HF - 25	5.0 ACRYLOID HF - 829	1.0 5.0 ACRYLOID HF - 829	5.0 ACRYLOID HF - 829	5.0 ACRYLOID 966
OVERALL LIQUID LOSS, WT. %	1	2	0	6	1	4	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F. AT 0°F.	+1 +2	+2 +4	+3 +5	+1 +4	0 —	-1 +1	-2 -4
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 1.1	0.1 1.3	0.2 1.6	0.3 1.2	0.1 1.5	0.1 1.2	0.1 1.6
ASTM UNION COLOR ORIGINAL FINAL	3 >8	2 >8	2 >8	3 >8	3 >8	3 >8	3 >8
WT. % INSOLUBLE MATERIAL	0.5	0.2	0.1	0.1	TRACE	TRACE	TRACE
FINAL CATALYST CONDITION APPEARANCE	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL DULL DULL	DULL DULL DULL DULL
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM MAGNESIUM	+0.08 +0.07 +0.06 +0.03	+0.03 0.02 0.01 0.02	+0.02 0.03 0.00 0.03	+0.05 +0.04 +0.03 +0.03	+0.01 0.00 0.03 0.02	0.03 +0.04 +0.02 +0.03	0.03 0.02 0.05 0.03

Table 66

## EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID	- - - - - 01-2-ETHYLHEXYL SEBACATE (MLO 7038) + 0.5 WT.-% PHENOTHIAZINE - - - - -									
	1.0	5.0	5.0	3.9 ACRYLOID HF - 25	5.0 ACRYLOID HF - 829	5.0 ACRYLOID HF - 829	5.0 ACRYLOID HF - 829	5.0 ACRYLOID 966	1.0	5.0 ACRYLOID 966
BASE STOCK										
TRICRESYL PHOSPHATE, WT.-%										
ACRYLOID POLYMER, WT.-%										
LIQUID CHARGED, GRAMS	90	91	91	91	90	90	90	90	90	91
LIQUID LOSS, WT.-%	1	4	3	3	4	8	3	7		
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2		26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	-	5.1	4.6	4.6	4.4	5.0	5.0	5.1		5.1
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	-	0.74	0.68	0.68	0.65	0.74	0.74	0.75		0.75
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3)										
AT 130°F.	+35	+67	+12	+12	-11	+8	-2	-9		
AT 0°F.	+67	+115	+80	+80	+17	+88	+40	+18		
NEUT. NO. (MG. KOH/GM. OIL)										
ORIGINAL	0.2	0.1	0.2	0.2	0.2	0.1	0.2	0.1		0.1
FINAL	15.5	18.0	12.3	12.3	13.6	23.2	13.9	10.1		10.1
WT.-% OIL INSOLUBLE MATERIAL (3)	1.2	0.8	0.4	0.4	0.8	0.9	0.3	0.3		0.3
FINAL CATALYST CONDITION										
APPEARANCE										
COPPER	CORRODED	DULL	DULL	DULL	CORRODED	CORRODED	CORRODED	CORRODED		CORRODED
STEEL	COATED	COATED	COATED	COATED	COATED	DULL	COATED	COATED		COATED
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL	BRIGHT	DULL		DULL
WT. LOSS (MG./SQ. CM.)										
COPPER	0.26	0.16	0.06	0.06	0.45	0.55	0.76	0.49		0.49
STEEL	+0.60	+0.19	+0.22	+0.22	+0.49	+0.03	+0.37	+0.30		+0.30
ALUMINUM	+0.02	+0.01	0.01	0.01	+0.03	+0.02	+0.02	+0.02		+0.02

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 67

EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL OF 34 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%)	<----- MLO 7093 ----->		
OXIDATION INHIBITOR	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE
ACRYLOID POLYMER	—	5.0 ACRYLOID 966	5.0 ACRYLOID HF-829
OVERALL LIQUID LOSS, WT.%	2	1	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+9	+12	+4
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.1	0.1	0.2
FINAL	2.6	4.7	2.8
ASTM UNION COLOR			
ORIGINAL	4	5	4
FINAL	>8	>8	>8
WT.% INSOLUBLE MATERIAL	3.4	0.1	0.3
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	DULL	DULL	DULL
STEEL	DULL	DULL	DULL
ALUMINUM	DULL	BRIGHT	BRIGHT
MAGNESIUM	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)			
COPPER	0.03	0.05	0.07
STEEL	+0.05	+0.03	+0.01
ALUMINUM	+0.05	+0.01	0.02
MAGNESIUM	+0.05	+0.04	0.01

Table 68

## EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID : MLO 7030 = ESS TIC 45, A NAPHTHENIC MINERAL OIL OF 45 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%)	MLO 7030	MLO 7030	MLO 7030	MLO 7030
OXIDATION INHIBITOR	-	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE
ACRYLOID POLYMER	-	5.0 ACRYLOID 966	5.0 ACRYLOID 966	5.0 ACRYLOID HF-829
OVERALL LIQUID LOSS, WT.-%	1	1	1	1
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+26	+19	+6	+8
NEUT. NO. (MG. KOH/GM. OIL)	0.2	0.2	0.2	0.2
ORIGINAL	4.9	3.0	6.1	4.6
FINAL				
ASTM UNION COLOR	3	4	4	4
ORIGINAL	>8	>8	>8	>8
FINAL				
WT.-% INSOLUBLE MATERIAL	1.9	2.4	TRACE	0.4
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER	CORRODED	DULL	CORRODED	DULL
STEEL	DULL	DULL	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT	BRIGHT	DULL
MAGNESIUM	BRIGHT	BRIGHT	DULL	DULL
WT. LOSS (MG./SQ. CM.)				
COPPER	0.36	0.11	0.26	0.12
STEEL	0.00	0.05	+0.01	+0.02
ALUMINUM	+0.03	+0.02	+0.02	+0.02
MAGNESIUM	0.00	+0.02	0.00	+0.01

Table 69

EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID: MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL OF 34 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%)	< - - - - - MLO 7093 - - - - - >		
OXIDATION INHIBITOR	NONE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE	1.0 PHENYL-ALPHA-NAPHTHYLAMINE
ACRYLOID POLYMER	NONE	5.0 ACRYLOID 966	5.0 ACRYLOID HF-829
LIQUID CHARGED, GRAMS	84	85	85
LIQUID LOSS, WT.%	3	1	0
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	-	3.6	3.7
MOLS O <sub>2</sub> USED/426 GMS. FLUID (2)	-	0.57	0.58
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+60	-3	-15
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.2	0.1	0.2
FINAL	2.9	2.3	2.3
WT.% OIL INSOLUBLE MATERIAL (3)	TRACE	0.1	0.1
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	DULL	DULL	DULL
STEEL	BRIGHT	DULL	DULL
ALUMINUM	BRIGHT	BRIGHT	DULL
WT. LOSS (MG./SQ. CM.)			
COPPER	+0.06	+0.05	+0.02
STEEL	0.08	0.02	0.03
ALUMINUM	0.00	0.02	0.02

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 70

## EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID: MLO 7030 = ESSIC 45, A NAPHTHENIC MINERAL OIL OF 45 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%) OXIDATION INHIBITOR	NONE			1.0 PHENYL-ALPHA-NAPHTHYLAMINE			1.0 PHENYL-ALPHA-NAPHTHYLAMINE			5.0 ACRYLOID 966			5.0 ACRYLOID HF-829			5.0 TRICRESYL PHOSPHATE			5.0 TRICRESYL PHOSPHATE			5.0 ACRYLOID 966		
	86	88	86	88	86	87	87	88	87	87	88	88	87	87	88	88	88	88	88	88	88	88	88	88
LIQUID CHARGED, GRAMS	2	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
LIQUID LOSS, WT.-%	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT OF O <sub>2</sub> SUPPLIED, GMS. (1)	4.8	-	4.5	-	4.5	3.7	3.7	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1
APPROX. AMOUNT OF O <sub>2</sub> USED, GMS. (1)	0.75	-	0.69	-	0.69	0.56	0.56	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62	0.62
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)																								
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3)	+115	+45	+102			+12																		
NEUT. NO. (MG. KOH/GM. OIL)	0.2	0.1	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
ORIGINAL	3.0	1.9	2.8	2.8	2.8	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3
FINAL	0.5	2.3	1.1	1.1	1.1	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
WT.-% OIL INSOLUBLE MATERIAL (3)																								
FINAL CATALYST CONDITION																								
APPEARANCE	COATED	COATED	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
COPPER	COATED	COATED	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	BRIGHT	BRIGHT	BRIGHT	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)	+0.10	+0.10	+0.07	+0.07	+0.07	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01	+0.01
COPPER	0.00	0.00	0.03	0.03	0.03	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
STEEL	0.03	0.00	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
ALUMINUM																								

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT(2) OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>(3) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF OI-2-

ETHYLHEXYL SEBACATE IS 426.

OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A

LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 71

EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 347°F.

TEST PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: BAROSA 43 (MLO 7093) = A PARAFFINIC MINERAL OIL OF 34 CENTISTOKES VISCOSITY AT  $100^\circ\text{F}$ .

TEST FLUID OXIDATION INHIBITOR, WT.% ACRYLOID POLYMER, WT.%	<----- BAROSA 43 -----> <----- 1.0 PHENYL-ALPHA-NAPHTHYLAMINE ----->			
	NONE	5.0 ACRYLOID 794	5.0 ACRYLOID 966	5.0 ACRYLOID 917
OVERALL LIQUID LOSS, WT.%	2	1	1	2
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F}$ .	+9	-3	+20	+5
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL	0.1	0.1	0.2	0.1
FINAL	2.6	3.0	3.3	4.2
ASTM UNION COLOR				
ORIGINAL	4	3 1/2	3 1/2	3 1/2
FINAL	>8	>8	>8	>8
WT.% OIL INSOLUBLE MATERIAL	3.4	0.3	TRACE	TRACE
FINAL CATALYST CONDITION APPEARANCE				
COPPER	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL
MAGNESIUM	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ.CM.)				
COPPER	0.03	0.07	0.11	0.14
STEEL	+0.05	+0.05	+0.02	+0.01
ALUMINUM	+0.05	+0.03	0.02	+0.03
MAGNESIUM	+0.05	+0.07	+0.01	+0.02



Table 72

## EFFECT OF ACRYLOID POLYMER ON FLUID DIRTINESS AT 500°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 + 5°F.; TEST TIME = 20 HRS.; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A ONE-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.  
 TEST FLUID: BAROSA 43 (MLO 7093) = A PARAFFINIC MINERAL OIL OF 3.4 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID OXIDATION INHIBITOR, WT. % ACRYLOID POLYMER, WT. %	NONE		NONE		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID	
	NONE		NONE		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID		5.0 ACRYLOID	
LIQUID CHARGED, GMS.	84	3	83	3	86	2	81	0	86	2	82	1	86	2	83	2
LIQUID LOSS, WT. %	26.2	8.8	26.2	3.9	26.2	7.3	26.2	3.4	26.2	4.5	26.2	2.8	26.2	4.5	26.2	3.8
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)																
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)																
MOLS O <sub>2</sub> USED/426 GMS FLUID (2)	1.40		0.63		1.14		0.56		0.69		0.46		0.69		0.61	
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+60		+51		+15		+14		-3		-7		+1		-6	
NEUT. NO. (MG. KOH/GM. OIL)																
ORIGINAL	0.2		0.1		0.2		0.2		0.1		0.1		0.1		0.1	
FINAL	2.9		1.8		2.4		2.1		2.0		1.9		2.2		2.1	
WT. % OIL INSOLUBLE MATERIAL (3)	TRACE		0.1		0.1		0.2		TRACE		0.1		0.1		0.1	
FINAL CATALYST CONDITION																
APPEARANCE																
COPPER	DULL		COATED		COATED		DULL		COATED		DULL		COATED		COATED	
STEEL	BRIGHT		DULL		DULL		DULL		DULL		DULL		DULL		DULL	
ALUMINUM	BRIGHT		DULL		BRIGHT		DULL		DULL		DULL		DULL		DULL	
WT. LOSS (MG./SQ. CM.)																
COPPER	+0.06		+0.10		+0.16		+0.05		+0.11		+0.02		+0.13		+0.12	
STEEL	0.08		0.05		0.00		0.01		0.00		+0.03		0.01		+0.05	
ALUMINUM	0.00		0.03		0.02		0.00		0.00		0.00		0.00		0.00	

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HRS.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GMS./LITER).

AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) MOLS O<sub>2</sub> FOR 426 GMS. OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 73

## OXIDATION AND CORROSION BEHAVIOR OF ACRYLOID-CONTAINING MINERAL OIL BLENDS AFTER PANEL COKER TESTS

TEST PROCEDURES AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: BAROSA 43 (MLO 7093) = A PARAFFINIC MINERAL OIL OF 3.4 CENTISTOKES VISCOSITY AT  $100^\circ\text{F}$ . IN ALL CASES, THE TEST FLUIDS HAVE BEEN SUBJECTED TO A PANEL COKER TEST PRIOR TO THIS OXIDATION TEST. PANEL COKER VALUES FOR THESE FLUIDS ARE ON TABLE 64.

TEST FLUID OXIDATION INHIBITOR, WT.% ACRYLOID POLYMER, WT.% PREVIOUS HISTORY OF FLUID	<div style="text-align: center;"> <div style="display: flex; justify-content: space-between;"> <span>&lt;-----BAROSA 43-----&gt;</span> <span>&lt;-----1.0 PHENYL-ALPHA-NAPHTHYLAMINE-----&gt;</span> </div> <div style="display: flex; justify-content: space-between;"> <span>1.0 PAN(1) NONE</span> <span>5.0 ACRYLOID 794</span> <span>5.0 ACRYLOID 966</span> <span>5.0 ACRYLOID 917</span> </div> <div style="text-align: center;">MODEL C PANEL COKER TEST</div> </div>			
OVERALL LIQUID LOSS, WT.%	2	2	2	2
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F}$ .	+9	+17	+144	+30
NEUT. NO. (MG. KOH/GM. OIL)				
ORIGINAL	1.0	0.2	0.5	0.3
FINAL	3.7	4.4	5.5	4.5
ASTM UNION COLOR				
ORIGINAL	>8	>8	>8	>8
FINAL	>8	>8	>8	>8
WT.% OIL INSOLUBLE MATERIAL	2.7	0.3	TRACE	TRACE
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER	CORRODED	DULL	CORRODED	CORRODED
STEEL	DULL	DULL	DULL	DULL
ALUMINUM	COATED	DULL	DULL	DULL
MAGNESIUM	COATED	DULL	DULL	DULL
WT. LOSS (MG./SQ.CM.)				
COPPER	0.78	0.13	0.33	0.88
STEEL	+0.08	+0.05	+0.04	0.00
ALUMINUM	+0.10	+0.05	+0.05	0.05
MAGNESIUM	+0.10	+0.05	+0.05	0.05

(1) FOR THIS TEST, 1.0 WEIGHT PER CENT PHENYL-ALPHA-NAPHTHYLAMINE WAS ADDED TO THE BAROSA 43 AFTER THE PANEL COKER TEST AND PRIOR TO THE OXIDATION TEST.

Table 74

## EFFECT OF ACRYLOID POLYMER ON THE THERMAL STABILITY OF SOME MINERAL OILS

ALL TESTS CONDUCTED IN GLASS TEST TUBES. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS THE PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID. APPROXIMATELY 0.25 LBS./SQ. IN. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED-IN CAPILLARY TUBE.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $700 \pm 10^\circ\text{F}$ .; TEST TIME = 6 HOURS; TEST FLUID CHARGED = 50 GRAMS; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7030 = ESSTIC 45, A WELL REFINED NAPHTHENIC MINERAL OIL  
MLO 7093 = BAROSA 43, A PARAFFINIC MINERAL OIL.  
MLO 7113 = HYDROGENATED BAROSA 43(1)

TEST FLUID	CENTISTOKE VISCOSITY AT $100^\circ\text{F}$ .		PER CENT LOSS IN WEIGHT(2)		CATALYST WEIGHT LOSS (MG./SQ. CM.)		NEUT. NO. (MG. KOH/GM. OIL)		OIL INSOLUBLE MATERIAL, WT. %
	ORIG.	FINAL	% CHANGE		COPPER	STEEL	ALUMINUM	ORIG.	FINAL
MLO 7030 + 1.0 PHENYL-ALPHA-NAPHTHYLAMINE +5.0 ACRYLOID HF-829 +5.0 ACRYLOID 966	75.6	26.5	-65	2	+0.03	0.71	0.02	0.2	0.6
	72.8	27.4	-62	2	+0.05	0.03	0.00	0.2	0.4
	33.0	23.2	-30	3	+0.20	+0.05	0.00	0.1	0.0
MLO 7093 + 1.0 PHENYL-ALPHA-NAPHTHYLAMINE +5.0 ACRYLOID HF-829 +5.0 ACRYLOID 966 (PRL AC 327) +5.0 ACRYLOID 966 (PRL AC 330) +5.0 ACRYLOID 794 +5.0 ACRYLOID 917	52.1	23.3	-55	4	+0.06	0.75	+0.02	0.2	0.8
	47.8	22.1	-54	2	+0.09	+0.01	0.01	0.1	0.4
	46.6	23.6	-49	1	+0.05	+0.04	0.00	0.2	0.4
	52.9	23.6	-55	2	+0.13	0.03	0.00	0.1	0.6
	53.9	21.9	-59	3	+0.16	+0.04	0.00	0.1	0.4
	32.1	21.8	-32	2	0.00	0.02	0.00	0.1	0.1
MLO 7113 (HYDROGENATED MLO 7093) +5.0 ACRYLOID HF-829 +5.0 ACRYLOID 966	46.6	21.1	-55	5	0.10	0.63	+0.02	0.1	0.5
	42.3	21.7	-49	3	0.00	0.04	0.00	0.1	0.5

(1) MATERIAL WAS HYDROGENATED USING A NICKEL CATALYST AT A MAXIMUM TEMPERATURE OF  $500^\circ\text{F}$ . AND A MAXIMUM PRESSURE OF 1600 P.S.I.G.

(2) LOSS IN WEIGHT IS GOOD TO  $\pm 1$  PER CENT OR  $\pm 0.5$  GRAMS.

The oxidation tests at both 500° and 600°F. were conducted for a 20-hour test period with an air rate of five liters per hour using copper, steel, and aluminum catalysts. The results of the tests at 347°, 500°, and 600°F. are tabulated on Tables 75, 76, and 77, respectively. The results of these tests indicate that the presence of 1.0 weight per cent tricresyl phosphate or 0.5 weight per cent diisopropyl acid phosphite does not affect seriously the oxidation characteristics of Necton 60 at either low or high temperatures.

In the 347°F. tests, the fluid properties and the amount of sludge formed are essentially the same for blends of Necton 60 containing tricresyl phosphate or diisopropyl acid phosphite as for the blend of Necton 60 and Antioxidant 2246 alone. The presence of Acryloid 966 in the blend of Necton 60 containing diisopropyl acid phosphite is effective in reducing the sludge formed for this fluid. That is, the blend containing Acryloid 966 has 0.5 weight per cent sludge compared to 3.6 weight per cent sludge for the blend without the polymer thickener. The effectiveness of Acryloid 966 in reducing sludge formation is discussed in detail in the preceding section of this report.

The tests conducted at 500° and 600°F. also show that no substantial differences in fluid properties are obtained for the blends containing tricresyl phosphate or diisopropyl acid phosphite compared to the same fluids without these additives. In all cases, the rate of oxidation (amount of oxygen absorbed), volatility loss, viscosity change, neutralization number increase, and the formation of oil insoluble material are of the same order of magnitude for all the fluids tested.

The addition of Acryloid 966 to the blend of Necton 60 containing Antioxidant 2246 and diisopropyl acid phosphite does not affect the oxidation stability of the fluid at 500°F.; however, a substantial amount of copper corrosion is obtained when Acryloid 966 is incorporated into the blend. This trend was observed for Acryloid 966 in the 500°F. test with Esstic 45 containing tricresyl phosphate (Table 70). This increased copper corrosion is attributed to thermal and/or oxidation products of the polymer thickeners.

These data show that the effect of the phosphorus type lubricity additives is different in order of magnitude in the ester versus the mineral oil. The bulk oil oxidation tests with the mineral oil (Necton 60) do not indicate deleterious effects for the phosphorus type additives. These blends will be carefully evaluated in other high temperature tests to determine the effect, if any, of the phosphorus type additive on oxidation, corrosion, coking, and thermal stability. Limited testing in more severe coking or deposition tests on metal surfaces indicates that the phosphorus type additives do have some deleterious effects in mineral oil blends at high temperatures.

G. CORROSION OF VARIOUS TYPES OF STEEL. It has been pointed out in previous reports that the SAE 1010 steel catalysts conventionally used in specification oxidation and corrosion tests are frequently corroded in high temperature oxidation or thermal stability tests by

ester-type synthetic fluids. Some exploratory studies have been conducted to determine whether or not this behavior is general in steel types more typical of those used in actual bearings and lubricant systems.

Four different steel types have been used to determine the effect of steel type on corrosion behavior. One-inch squares of SAE 1010 steel or stainless steel, and 0.5-inch diameter ball bearings fabricated from 52-100 bearing steel or M-10 tool steel, have been employed as catalysts. Test fluids comprise di-2-ethylhexyl sebacate and Hercoflex 600.

Oxidation tests at 500°F. have been conducted with two separate batches of di-2-ethylhexyl sebacate (PRL 3103 and MLO 7038) and with Hercoflex 600 (PRL 3542). These tests were conducted for a 60-hour test period using an air rate of five liters per hour. These test conditions are severe. The final fluid properties shown on Table 78 indicate considerable fluid deterioration.

In the tests conducted with di-2-ethylhexyl sebacate, each of the four steel types were used as catalysts. The test conducted with Hercoflex 600 contained one M-10 tool steel bearing and one 52-100 steel bearing as catalysts. The results of these oxidation tests are found on Table 78. In addition, comparable tests using the conventional copper, steel, and aluminum metal catalysts are included on Table 78.

The results of these tests indicate that significant differences in steel corrosion are obtained from different steel types. For example, in the tests conducted with di-2-ethylhexyl sebacate, the SAE 1010 steel and the 52-100 bearing steel are severely corroded, whereas the stainless steel and the M-10 tool steel are not corroded. Similarly, in the tests conducted with Hercoflex 600, the SAE 1010 steel and the 52-100 steel bearing are corroded and the M-10 tool steel bearing is not.

Thermal stability tests at 600°F. have also been conducted with di-2-ethylhexyl sebacate using the various types of steel as catalysts. These tests were conducted in the glass thermal stability apparatus for a six hour test period. These thermal stability tests are summarized in Table 79.

The results of these tests again indicate substantial differences in steel corrosion for the different types of steel catalysts. That is, both the SAE 1010 steel and the 52-100 steel bearing are corroded in the thermal stability tests with di-2-ethylhexyl sebacate, whereas the stainless steel and the M-10 tool steel bearing are essentially unaffected.

There have been indications that heavily loaded antifriction bearings running at moderately high temperatures with Spec. MIL-L-7808 fluid are showing shorter service life than anticipated. It is reported that many of the bearings after failure show severe surface pitting. There is some evidence that these more rapid bearing failures may be

related to the oil's viscosity, or to its hydrodynamic component of lubricity. It is significant to note that 52-100 bearings subjected to thermal and oxidative tests with Spec. MIL-L-7808 type fluids produce severe pitting of the type noted in the anti-friction bearing failures. Temperature conditions of the severity used in the thermal and oxidative tests have been shown to exist at the metal-to-metal contact surface in loaded bearings.

These facts suggest that stress corrosion of the bearing surface by the Spec. MIL-L-7808 lubricant may play a significant role in bearing failures. Laboratory thermal and oxidative tests show significant corrosion differences between 52-100 bearing steel and M-10 tool steel. Laboratory lubricity tests in the Shell four-ball wear and E.P. testers show about the same load carrying ability for 52-100 bearing steel and M-10 tool steel with ester-type lubricants. These data suggest that the relative importance of stress corrosion and the hydrodynamic properties (viscosity) of lubricants in antifriction bearing failures might be resolved by comparing the behavior of 52-100 steel bearings and M-10 tool steel bearings with Spec. MIL-L-7808 type lubricants. These same problems should also apply to loaded gears and other steel-on-steel bearings in quasi-hydrodynamic lubrication in jet engines or hydraulic systems at high temperatures.

H. THIN FILM OXIDATION TESTS AT 500° AND 600°F. A thin film type oxidation test has been used to evaluate the dirtiness behavior of several ester and mineral oil fluids. A similar test conducted at 347°F. has been used successfully to demonstrate dirtiness of the type encountered in the turbine bearing of an Allison turbo-prop engine. The dirtiness found in the bearing and in the thin film test was not adequately predicted by the conventional Spec. MIL-L-7808 oxidation test. The thin film test, as conducted at 347°F., is described in Appendix A of report PRL 6.1-Jan52 entitled "Development Work on Lubricants for Aircraft Turbine Engines" issued by this Laboratory.

This test comprises pumping the lubricant over a large area of metal surface simulating the flow of oil films over engine bearings when the air is also circulated over this metal surface. The air circulation can be varied independently and the amount of oxygen adsorbed measured quantitatively. Thermal effects can be evaluated by substituting nitrogen as the gas circulated.

Preliminary thin film tests have been conducted at 500° and 600°F. using 1000 square centimeters of aluminum jack chain as the metal surface. The jack chain is supported above the fluid level in the reservoir. A charge of test fluid comprising 100 milliliters is circulated at a rate of approximately 240 milliliters per minute over the metal surface. Either air or nitrogen at a rate of 10 liters per hour is circulated through the jack chain packing countercurrently to the descending fluid film.

Results from tests conducted at 500°F. are shown on Table 81. For the tests using air, the total dirtiness is approximately the same



Table 75

# EFFECT OF PHOSPHORUS TYPE LUBRICITY ADDITIVES ON THE OXIDATION AND CORROSION CHARACTERISTICS OF A NAPHTHENIC MINERAL OIL AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3°F.; TEST TIME = 72 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TEST FLUID: PRL 3467 = NECTON 60, A NAPHTHENIC MINERAL OIL OF 109 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%)	<-----PRL 3467----->			
	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246 1.0 TRICRESYL PHOSPHATE	1.0 ANTIOXIDANT 2246 1.0 TRICRESYL PHOSPHATE 5.0 ACRYLOID 966	1.0 ANTIOXIDANT 2246 0.5 DIISOPROPYL ACID PHOSPHITE
OXIDATION INHIBITOR	-	-	-	-
LUBRICITY ADDITIVE	-	-	-	-
ACRYLOID POLYMER	-	-	-	-
OVERALL LIQUID LOSS, WT.%	2	2	2	2
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+56	+59	+230	+35
NEUT. NO. (MG. KOH/GM. OIL)	0.2	0.3	0.2	0.2
ORIGINAL	4.2	4.0	4.5	2.5
FINAL				
ASTM UNION COLOR	3	3	3	3
ORIGINAL	>8	>8	>8	>8
FINAL				
WT. % INSOLUBLE MATERIAL	2.7	4.3	0.5	3.6
FINAL CATALYST CONDITION				
APPEARANCE				
COPPER	DULL	DULL	CORRODED	CORRODED
STEEL	DULL	DULL	DULL	COATED
ALUMINUM	BRIGHT	DULL	BRIGHT	COATED
MAGNESIUM	DULL	DULL	BRIGHT	COATED
WT. LOSS (MG./SQ. CM.)				
COPPER	0.09	0.15	0.27	0.61
STEEL	+0.05	+0.05	+0.02	+0.68
ALUMINUM	+0.05	+0.06	0.00	+0.23
MAGNESIUM	+0.05	+0.06	+0.03	+0.33

Table 76

# EFFECT OF PHOSPHORUS-TYPE LUBRICITY ADDITIVES ON THE OXIDATION AND CORROSION CHARACTERISTICS OF A NAPHTHENIC MINERAL OIL AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID = PRL 3467 = NECTON 60, A NAPHTHENIC MINERAL OIL OF 109 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%)		PRL 3467			
OXIDATION INHIBITOR		1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246
LUBRICITY ADDITIVE			1.0 TRICRESYL PHOSPHATE	0.5 DIISOPROPYL ACID PHOSPHITE	0.5 DIISOPROPYL ACID PHOSPHITE
ACRYLOID POLYMER					5.0 ACRYLOID 966
LIQUID CHARGED, GRAMS		85	87	87	87
LIQUID LOSS, WT. %		0	0	1	1
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)		26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)		4.7	4.7	4.2	4.3
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)		0.74	0.73	0.64	0.66
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3)					
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL		+75	+68	+55	+22
FINAL		0.2	0.3	0.4	0.4
WT. % OIL INSOLUBLE MATERIAL (3)		2.8	3.3	1.7	1.5
FINAL CATALYST CONDITION		0.1	0.1	0.3	0.3
APPEARANCE					
COPPER		COATED	COATED	DULL	CORRODED
STEEL		DULL	DULL	COATED	COATED
ALUMINUM		DULL	DULL	COATED	COATED
WT. LOSS (MG./SQ. CM.)					
COPPER		+0.12	+0.19	+0.03	1.17
STEEL		0.07	0.01	+0.33	+0.32
ALUMINUM		0.05	0.02	+0.25	+0.36

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.



Table 77

# EFFECT OF PHOSPHORUS TYPE LUBRICITY ADDITIVES ON THE OXIDATION AND CORROSION CHARACTERISTICS OF A NAPHTHENIC MINERAL OIL AT 600°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600 ± 5°F.; TEST TIME = 20 HOURS; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.;  
 CATALYST = A 1 INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.  
 TEST FLUID: PRL 3467 = NECTON 60, A NAPHTHENIC MINERAL OIL OF 109 CENTISTOKES VISCOSITY AT 100°F.

TEST FLUID (CONC. IN WT.%)		PRL 3467		PRL 3467		PRL 3467	
OXIDATION INHIBITOR		1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246
LUBRICITY ADDITIVE		1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246	1.0 ANTIOXIDANT 2246
LIQUID CHARGED, GRAMS		85	85	85	85	85	85
LIQUID LOSS, WT. %		14	11	11	11	11	11
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)		26.2	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)		10.2	8.9	8.9	8.9	8.9	8.9
MOLS O <sub>2</sub> USED/426 GMS. FLUID (2)		1.59	1.39	1.39	1.39	1.39	1.39
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3)							
NEUT. NO. (MG. KOH/GM. OIL)							
ORIGINAL							
FINAL							
WT. % OIL INSOLUBLE MATERIAL (3)							
FINAL CATALYST CONDITION							
APPEARANCE							
COPPER		COATED	COATED	COATED	COATED	COATED	COATED
STEEL		BRIGHT	DULL	BRIGHT	BRIGHT	BRIGHT	BRIGHT
ALUMINUM		COATED	DULL	BRIGHT	BRIGHT	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)							
COPPER		+0.22	+0.22	+0.33	+0.29	+0.32	+0.32
STEEL		0.05	0.05	0.07	+0.45	+0.76	+0.76
ALUMINUM		+0.13	0.00	0.02	+0.24	+0.74	+0.74

- (1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

Table 78

## OXIDATION AND CORROSION CHARACTERISTICS OF TWO ESTERS AT 500°F. USING A VARIETY OF METAL CATALYSTS

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 60 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYSTS ARE ONE-INCH SQUARES OF SHEET STOCK EXCEPT WHERE STEEL BALLS ARE INDICATED. (0.5 INCH DIAMETER BALL BEARING)

TEST FLUIDS: PRL 3103 = DI-2-ETHYLHEXYL SEBACATE

ML0 7038 = DI-2-ETHYLHEXYL SEBACATE

PRL 3542 = HERCOFLEX 600 (PENTAERYTHRITOL ESTER)

NOTE: EACH TEST FLUID CONTAINS 0.5 WEIGHT PER CENT PHENOTHIAZINE.

TEST FLUID	PRL 3103		ML0 7038	PRL 3542	
LIQUID CHARGED, GRAMS	91	90	90	97	97
LIQUID LOSS, WT. %	20	31	24	20	15
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	78.6	78.6	78.6	78.6	78.6
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	17.4	16.9	16.5	17.5	14.5
MOLS O <sub>2</sub> USED/426 GMS. FLUID (2)	2.60	2.50	2.44	2.42	1.99
% CHANGE IN CENTISTOKE VISCOSITY					
AT 130°F.	SOLID (3)	SOLID (3)	SOLID (3)	SOLID (3)	SOLID (3)
AT 0°F.	SOLID (3)	SOLID (3)	SOLID (3)	SOLID (3)	SOLID (3)
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.1	0.1	0.1	0.2	0.2
FINAL	36.2	23.6	50.0	12.5	16.3
WT. % OIL INSOLUBLE MATERIAL	- (3)	- (3)	- (3)	- (3)	- (3)
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	CORRODED	-	-	CORRODED	-
STEEL (1010)	CORRODED	CORRODED	CORRODED	CORRODED	-
ALUMINUM	BRIGHT	-	-	DULL	-
STAINLESS STEEL	-	DULL	DULL	-	-
STEEL BALL (52:100)	-	CORRODED	CORRODED	-	CORRODED
STEEL BALL (M-10 TOOL)	-	DULL	DULL	-	DULL
WT. LOSS (MG./SQ. CM.)					
COPPER	0.52	-	-	0.43	-
STEEL (1010)	3.26	9.84	3.66	8.80	-
ALUMINUM	0.09	-	-	0.00	-
STAINLESS STEEL	-	+0.11	+0.02	-	-
STEEL BALL (52:100)	-	8.44	7.77	-	79.00
STEEL BALL (M-10 TOOL)	-	0.00	+0.04	-	0.06

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OXIDIZED FLUID WAS AN INTIMATE MIXTURE OF OIL INSOLUBLE MATERIAL AND OXIDIZED LIQUID. OIL INSOLUBLES COULD NOT BE DETERMINED WITHOUT DILUTION.

Table 79

## THERMAL STABILITY OF DI-2-ETHYLHEXYL SEBACATE AT 600°F. USING VARIOUS TYPES OF STEEL CATALYSTS

TESTS ARE CONDUCTED IN GLASS TEST TUBES USING A 25 GRAM CHARGE OF TEST FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID -- APPROXIMATELY 0.25 LB./SQ. IN. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600°F.; TEST TIME = 6 HOURS; CATALYSTS ARE ONE-INCH SQUARES OF SHEET STOCK EXCEPT WHERE STEEL BALLS ARE INDICATED. (0.5 INCH DIAMETER BALL BEARING)

TEST FLUID	CENTISTOKE VISCOSITY AT 100°F.		% LOSS IN WEIGHT	CATALYST WT. LOSS (MG./SQ. CM.)					STEEL BALL (M-10 TOOL)	NEUT. NO. (MG. KOH/GM. OIL)		WT. % INSOLUBLE MATERIAL
	ORIG.	FINAL		COPPER	ALUMINUM	1010 STEEL	STAINLESS STEEL	STEEL BALL (52:100)		ORIG.	FINAL	
DI-2-ETHYLHEXYL SEBACATE (PRL 3483)	12.6	11.3	-10	-	-	-	-	-	-	0.1	25.3	-
DI-2-ETHYLHEXYL SEBACATE (PRL 3483)	12.6	10.7	-15	0.02	+2.08	7.00	-	-	-	0.1	11.4	1.1
DI-2-ETHYLHEXYL SEBACATE (PRL 3103)	12.8	11.1	-13	-	-	5.97	0.00	9.15	0.02	0.1	8.1	1.3
DI-2-ETHYLHEXYL SEBACATE (MLO 7038)	12.7	11.4	-11	-	-	5.16	0.01	9.12	0.00	0.1	8.6	1.1

for the diester and naphthenic mineral oil; it is somewhat less for the pentaerythritol ester (Hercoflex 600). Total insoluble material increases progressively for the diester, the diester-tricresyl phosphate blend (Spec. MIL-L-7808 type), and the diester-dialkyl acid phosphite blend. All of the ester type blends on Table 81 contain 0.5 weight per cent phenothiazine. There appears to be no appreciable change in the total dirtiness between the naphthenic mineral oil and the blend containing the dialkyl acid phosphite. In the tests using nitrogen, total dirtiness is greatly reduced. The same general relationships exist between the various fluids and blends evaluated.

Data obtained for the 600°F. tests are shown on Table 82. The total dirtiness, for tests using air, is considerably lower for the mineral oil and hydrocarbon fluids than for the diester and pentaerythritol ester (Hercoflex 600). In this case, the diester-dialkyl acid phosphite blend shows reduced dirtiness relative to the diester. The reason for this is not readily apparent. Values obtained with the Model C panel coker and the controlled atmosphere coker at 600°F. indicate increased dirtiness for the phosphite-containing blend. In the tests using nitrogen, the ester type fluids show increased dirtiness over the naphthenic mineral oil.

These preliminary data indicate that at 500°F., in terms of fluid dirtiness, the ester and mineral oil are about equal; at 600°F. the mineral oils show a definite advantage. This behavior is probably related to the relative difference in thermal stability between these fluid types at 600°F. As has been shown in various other laboratory type evaluations, this test again points out the advantage of eliminating, or severely limiting the amount of air in contact with fluids and lubricants at these high temperatures.

Some additional tests with the thin film technique have been carried out to determine the effect of such variables as gas rate, test temperature, and type of metal surface on the dirtiness characteristics of some fluids and lubricants.

The effect of air rate on chain coating has been investigated at both 500° and 600°F. test temperature in the thin film procedure. Table 80 is a tabulation of chain coatings observed using several different types of test fluids and air rates in the range of 1 to 30 liters per hour at each of the two test temperatures. These data indicate generally increasing chain coating with increasing available air at each of the two test temperatures.

Another comparison may be drawn from the data shown on Table 80. That is, by taking a constant air rate, for instance 10 liters per hour, the effect of test temperature on the chain coating tendencies of the various fluids can be shown. At 500°F., the dibasic acid ester and the two mineral oils show about the same coating; the pentaerythritol ester is somewhat lower; and the polyolefin and silicate hydraulic fluid (assumed from the 30 liter/hour value) show low values.

The differences in fluid dirtiness at 600°F. indicate a definite advantage for the mineral oils at this temperature. That is, the increase in chain coating for the 100°F. temperature rise is considerably less for the two mineral oils than for the remainder of the fluids. This behavior may be related to the thermal stability of the various types of fluids at 600°F.

Table 80

EFFECT OF AIR RATE ON CHAIN COATING IN THIN FILM OXIDATION TEST

Test Conditions Include: Test Temperature and Air Rate as Indicated;  
Test Time = 3 Hrs.; Pumping Rate = 240 ml./min.; Test Fluid Charged = 100 ml.; and Catalyst = 1000 Sq. Cm. of Aluminum Jack Chain.

Test Temperature, °F. Air Rate, Liters/Hour	Deposit on Chain, Mg.					
	500°F.			600°F.		
	1	10	30	5	10	30
Di-2-Ethylhexyl Sebacate*	57	134	195	752	884	947
Hercoflex 600 (PE Ester)*	24	63	83	225	372	521
Naphthenic Mineral Oil (Esstic 45)	5	100	183	35**	144	515
Paraffinic Mineral Oil (Pa. Neutral)	-	115	196	100	178	334
Polyolefin (Indopol L-50)	-	4	27	67	165	372
O.S. 45 Silicate Fluid	-	-	2	78	356	2038

\* Test fluid contains 0.5 wt.% phenothiazine.

\*\*Air rate for this test was 2.5 liters per hour.

More complete data for several fluids at different air rates are shown on Tables 83 and 84. Data for test fluids containing a dialkyl acid phosphite are included. Tests conducted at 500°F. are on Table 83. In general, the inclusion of the dialkyl acid phosphite increases the total dirtiness encountered under these conditions. Using nitrogen instead of air results in low values of dirtiness for the dibasic acid ester-dialkyl acid phosphite blend. The use of nitrogen with the naphthenic mineral oil-dialkyl acid phosphite blend does not materially reduce the dirtiness relative to corresponding tests using air.

Test results obtained at 600°F. are shown on Table 84. As pointed out above, all dirtiness values with the exception of the mineral oil, are increased at 600°F. and the dibasic acid ester-phenothiazine-dialkyl acid phosphite blend shows reduced dirtiness relative to the dibasic acid ester-phenothiazine blend. The reproducibility of the test, as shown by multiple tests under given conditions, is considered to be fair.

The effect of using different catalyst metals has been determined. Copper, steel, or aluminum jack chain has been used as the metal surface. The surface area was constant in all tests. The results of these tests, conducted at 600°F. with 10 liters of air per hour, are shown on Table 85. No clear cut differences in total insolubles formation relative to catalyst metal used can be pointed out with any of the test fluids used. The reproducibility for duplicate tests, in many cases, is relatively poor.

A few large catalyst area tests have been conducted in conjunction with the thin film studies. These tests incorporate the large catalyst area (1000 sq. cm.) provided by the jack chain but differ from the above tests in that they are static tests in which the jack chain is immersed in the test oil and the air is bubbled through the fluid from a point near the bottom of the tube. Steel jack chain has been used in these tests. Test results are on Table 86.

In the case of the dibasic acid ester and the pentaerythritol ester (Hercoflex 600), the total insolubles formation appears to be greater for the immersion type test. The oxygen absorption is approximately the same for the two types of tests. Steel corrosion (chain weight loss) is noted in the immersion type tests. Steel corrosion of this nature has been pointed out previously for these fluids under oxidation and thermal stability test conditions at 600°F.

The mineral oil, synthetic hydrocarbon (Indopol L-50), and the silicate hydraulic fluid appear to produce less insoluble material in the immersion type test than in the thin film type procedure.

Several conclusions may be drawn from these studies in the thin film type oxidation test. Both chain coating and general fluid dirtiness are increased with increasing air rate. Increased dirtiness with increasing temperature is influenced by fluid type. That is, in going from 500° to 600°F., for instance, the increase in dirtiness values for diesters is greater than that shown by mineral oils. This behavior is probably related to the relative thermal stabilities of the two types of fluids. The limited number of tests conducted using different catalyst metals (copper, steel, and aluminum) show no clear-cut relation between dirtiness and catalyst metal.

At 500°F. the ester and mineral oil fluids show about the same degree of dirtiness; at 600°F., the mineral oil shows a definite advantage. The addition of a dialkyl acid phosphite to the mineral oil causes no appreciable effect on the dirtiness of the blend at either 500° or 600°F. At 500°F., total dirtiness values increase progressively for the diester, the diester-tricresyl phosphate blend, and the diester-dialkyl acid phosphite blend. At 600°F., the diester-dialkyl acid phosphite blend appears to produce less insolubles than the diester alone. The air rate studies and the tests conducted with nitrogen instead of air point out the advantage to be gained by eliminating or restricting the amount of air in contact with fluids and lubricants at these operating temperatures.

Table 81

## THIN FILM OXIDATION TESTS AT 500°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 3 HOURS; GAS RATE (AIR OR  $\text{N}_2$ ) =  $10 \pm 1 \text{ L./HR.}$ ; TEST FLUID CHARGED = 100 ML.; PUMPING RATE = 240 ML./MIN.; AND CATALYST = 1000 SO. CM. OF ALUMINUM JACK CHAIN.

TEST FLUID (COMPOSITION IN WT.%)	DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE		SPEC. MIL-L-7808 TYPE FLUID (CONTAINS 5.0 TRI- CRESYL PHOSPHATE)		DI-2-ETHYLHEXYL SEB- ACATE + 0.5 PHENO- THIAZINE + 0.5 DI- ISOPROPYL ACID PHOSPHITE		HERCOFLEX 600 + 0.5 PHENOTHIAZINE		HERCOFLEX 600 + 0.5 PHENOTHIAZINE + 5.0 TRICRESYL PHOSPHATE		NAPHTHENIC MINERAL OIL (ESSTIC 45)		NAPHTHENIC MINERAL OIL + 0.5 DIISOPROPYL ACID PHOSPHITE	
	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$
GAS USED	90.0	90.0	91.0	91.0	91.0	90.0	97.0	98.0	100	98.0	87.5	86.5	85.5	87.0
LIQUID CHARGED, GMS.														
LIQUID LOSS, WT.%														
APPROX. AMOUNT $\text{O}_2$ SUPPLIED, GMS. (1)														
APPROX. AMOUNT $\text{O}_2$ USED, GMS. (1)	7.8	-	7.8	-	7.8	-	7.8	-	7.8	-	7.8	-	7.8	-
APPROX. AMOUNT $\text{O}_2$ USED, %	3.9	-	1.8	-	2.8	-	2.9	-	1.2	-	2.6	-	2.4	-
APPROX. AMOUNT $\text{O}_2$ USED, %	50	-	23	-	36	-	37	-	16	-	34	-	31	-
MOLES $\text{O}_2$ USED/426 GMS. OIL (2)	0.58	-	0.27	-	0.41	-	0.39	-	0.16	-	0.40	-	0.38	-
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF INSOLUBLES (3)														
NEUT. NO. (MG. KOH/GM. OIL)														
ORIGINAL														
FINAL														
OIL INSOLUBLE MATERIAL, MG. (3)														
CHAIN COATING, MG.														
TUBE COATINGS, MG. (4)														
TOTAL INSOLUBLE MATERIAL, MG.	217	10	359	18	572	98	126	178	103	10	228	11	240	188

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X  $\text{O}_2$  CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR  $\text{O}_2$ .

(2) THE MOLES OF  $\text{O}_2$  FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE  $\text{O}_2$  ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.



Table 82

## THIN FILM OXIDATION TESTS AT 600°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $600 \pm 10^\circ\text{F.}$ ; TEST TIME = 3 HOURS; GAS RATE (AIR OR  $\text{N}_2$ ) =  $10 \pm 1 \text{ L./HR.}$ ; TEST FLUID CHARGED = 100 ML.; PUMPING RATE = 240 ML./MIN.; AND CATALYST = 1000 SQ. CM. OF ALUMINUM JACK CHAIN.

TEST FLUID (COMPOSITIONS IN WT.%)	DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE		DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE + 0.5 DIISOPROPYL ACID PHOSPHITE		HERCOFLEX 600 + 0.5 PHENOTHIAZINE		NAPHTHENIC MINERAL OIL (ESSTIC 45)		PARAFFINIC MINERAL OIL (PA. NEUTRAL, ML0 7026)		INDOPOL L-50 (POLYBUTENE)	
	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$	AIR	$\text{N}_2$
GAS USED												
LIQUID CHARGED, GMS.	89.0	91.5	91.0	92.0	99.0	100	88.0	88.0	85.5	82.5	80.5	83.0
LIQUID LOSS, WT.%	25	7	29	9	20	3	11	3	6	15	29	29
APPROX. AMOUNT $\text{O}_2$ SUPPLIED, GMS. (1)												
APPROX. AMOUNT $\text{O}_2$ USED, GMS. (1)	7.8	-	7.8	-	7.8	-	7.8	-	7.8	7.8	7.8	7.8
	7.0	-	5.5	-	6.0	-	6.7	-	7.2	7.1	5.8	5.8
APPROX. AMOUNT $\text{O}_2$ USED, %												
MOLS $\text{O}_2$ USED/426 GMS. OIL (2)	90	-	71	-	76	-	84	-	92	90	74	74
	1.05	-	0.81	-	0.80	-	1.02	-	1.11	1.13	0.96	0.93
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF INSOLUBLES (3) AT 130°F.												
	+99	+7	+64	+16	+58	-11	+95	+4	+89	+89	+183	+249
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 18.2	0.1 6.9	0.2 29.8	0.2 19.3	0.1 8.8	0.1 1.1	0.2 1.6	0.2 0.2	0.1 0.3	0.1 1.5	0.1 0.7	0.1 1.2
OIL INSOLUBLE MATERIAL, MG. (3)	303	45	142	8	223	46	21	0	56	48	23	20
CHAIN COATING, MG.	480	31	216	95	335	51	144	5	214	178	165	122
TUBE COATING, MG. (4)	20	0	30	40	30	0	40	0	50	20	40	40
TOTAL INSOLUBLE MATERIAL, MG.	803	76	388	143	588	97	205	5	320	246	228	182

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X  $\text{O}_2$  CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN USED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR  $\text{O}_2$ .

(2) THE MOLS OF  $\text{O}_2$  FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE  $\text{O}_2$  ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.



Table 83

## EFFECT OF GAS RATE ON THIN FILM OXIDATION BEHAVIOR AT 500°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 500 ± 5°F.; TEST TIME = 3 HOURS; GAS RATE (AIR OR N<sub>2</sub>) AS INDICATED; TEST FLUID CHARGED = 100 ML.; PUMPING RATE = 240 ML. PER MINUTE; AND CATALYST = 1000 SQ. CM. OF ALUMINUM JACK CHAIN.

TEST FLUID (COMPOSITIONS IN WT.%)	DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE		DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE + 0.5 DIISOPROPYL PHOSPHITE		NAPHTHENIC MINERAL OIL (ESSTIC 45)		NAPHTHENIC MINERAL OIL + 0.5 DIISOPROPYL ACID PHOSPHITE		HERCOFLEX 600 + 0.5 PHENOTHIAZINE	
	AIR	NITROGEN	AIR	NITROGEN	AIR	NITROGEN	AIR	NITROGEN	AIR	NITROGEN
GAS RATE, LITERS/HOUR	1	10	1	10	1	10	1	10	1	10
LIQUID CHARGED, GMS.	90	90	91	91	87	87	88	87	97	97
LIQUID LOSS, WT.%	4	-	0	5	2	0	0	0	-	8
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	0.78	7.8	0.78	7.8	0.78	7.8	0.78	7.8	0.78	7.8
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	0.78(5)	3.9	0.78(5)	2.8	0.78(5)	2.6	0.78(5)	2.4	0.78(5)	2.9
APPROX. AMOUNT O <sub>2</sub> USED, %	100	50	100	36	100	33	100	31	100	37
MOLS O <sub>2</sub> USED/426 GMS. OIL (2)	0.12	0.58	0.11	0.41	0.12	0.40	0.12	0.38	0.11	0.39
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF INSOLUBLES (3) AT 130°F.	+6	+34	+6	+19	+3	+62	+7	+35	-5	+25
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 2.2	0.1 13.3	0.2 7.7	0.5 10.7	0.3 0.5	0.3 3.1	0.2 0.6	0.2 2.0	0.1 0.5	0.1 5.8
OIL INSOLUBLE MATERIAL, MG. (3)	16	48	53	85	26	52	18	40	18	33
CHAIN COATING, MG.	57	134	194	432	5	51	152	115	24	63
TUBE COATING, MG. (4)	5	35	20	55	0	125	45	50	0	30
TOTAL INSOLUBLES, MG.	78	217	267	572	31	228	215	240	42	126

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.

(5) THE EXHAUST GAS WAS NOT ANALYZED. ONE HUNDRED PER CENT OXYGEN USED HAS BEEN ASSUMED.

Table 84

## EFFECT OF AIR RATE ON THIN FILM OXIDATION AT 600°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $600 \pm 10^\circ\text{F}$ .; TEST TIME = 3 HOURS; AIR RATE AS INDICATED;  
 TEST FLUID CHARGED = 100 ML.; PUMPING RATE = 240 ML./MIN.; AND CATALYST = 1000 SO. CM.  
 OF ALUMINUM JACK CHAIN.

TEST FLUID (COMPOSITION IN WT.%)	D1-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE					D1-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE + 0.5 DIISOPROPYL ACID PHOSPHITE					HERCOFLEX 500 + 0.5 PHENO- THIAZINE				
	5		10			5		10			5		10		
AIR RATE, LITERS/HOUR	89	90	89	90		90	91	88	91	92	98	98	99	99	
LIQUID CHARGED, GMS	6	12	25	-		9	9	29	28	11	8	8	20	12	
LIQUID LOSS, WT.%															
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	3.9	3.9	7.8	7.8		3.9	7.8	7.8	7.8	7.8	3.9	3.9	7.8	7.8	
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	3.0	3.4	7.0	5.8		2.9	5.5	5.8	5.6	6.1	2.6	1.2	6.0	5.7	
APPROX. AMOUNT O <sub>2</sub> USED, %															
MOLS O <sub>2</sub> USED/426 GMS. OIL (2)	77	86	90	75		74	71	74	71	78	65	30	76	73	
	0.45	0.50	1.05	0.85		0.42	0.81	0.87	0.81	0.88	0.35	0.16	0.80	0.77	
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLES (3) AT 130°F.															
NEUT. NO. (MG. KOH/GM. OIL)															
ORIGINAL															
FINAL															
OIL INSOLUBLE MATERIAL, MG. (3)															
CHAIN COATING, MG.															
TUBE COATING, MG. (4)															
TOTAL INSOLUBLE MATERIAL, MG.	1172	1430	803	1423		338	388	427	418	637	662	355	588	767	

- (1) AMOUNT OF OXYGEN SUPPLIED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).  
 AMOUNT OXYGEN USED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.  
 (2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR  
 WEIGHT OF D1-2-ETHYLHEXYL SEBACATE IS 426.  
 (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES  
 ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.  
 (4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.

(TABLE 84 CONCLUDED ON NEXT PAGE)

Table 84 (concluded)

## EFFECT OF AIR RATE ON THIN FILM OXIDATION AT 600°F.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $600 \pm 10^\circ\text{F.}$ ; TEST TIME = 3 HOURS; AIR RATE AS INDICATED; TEST FLUID CHARGED = 100 ML.; PUMPING RATE = 240 ML./MIN.; AND CATALYST = 1000 SQ. CM. OF ALUMINUM JACK CHAIN.

TEST FLUID (COMPOSITIONS IN WT.%)		NAPHTHENIC MINERAL OIL (ESSTIC 45)		ESSTIC 45 + 0.5 DIISOPROPYL ACID PHOSPHITE		PARAFFINIC MINERAL OIL (PA. NEUTRAL, MLO 7026)		POLYBUTENE (INDOPOL L-50)		O.S. 45 SILICATE FLUID		
AIR RATE, LITERS/HOUR		2.5	<- -10- - ->	<- -10- - ->	88	89	88	<- -10- - ->	5	<- -10- - ->	5	<- -10- - ->
LIQUID CHARGED, GMS.		89	88	88	88	89	85	82	85	83	89	88
LIQUID LOSS, WT.%		4	11	14	-	-	2	15	7	29	10	22
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)		2.0	7.8	7.8	7.8	7.8	3.9	7.8	3.9	7.8	3.9	7.8
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)		0.4	6.7	6.9	-	-	2.5	7.1	2.1	5.8	1.4	5.5
APPROX. AMOUNT O <sub>2</sub> USED, %		19	84	88	-	-	67	90	53	74	35	71
MOLS O <sub>2</sub> USED/426 GMS. OIL (2)		0.66	1.02	1.04	-	-	0.41	1.13	0.32	0.96	0.20	0.82
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLES (3) AT 130°F.		+12	+95	+100	+84	+84	+24	+89	+5	+183	-3	+96
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL		0.2 0.5	0.2 1.6	0.2 2.5	0.3 2.4	0.3 2.4	0.1 1.4	0.1 1.5	0.0 0.4	0.1 0.7	0.0 7.4	0.0 2.0
OIL INSOLUBLE MATERIAL, MG. (3)		3	41	91	59	59	52	48	19	23	6	90
CHAIN COATING, MG.		35	144	156	278	278	100	178	67	165	78	356
TUBE COATING, MG. (4)		20	40	70	60	60	20	20	10	40	10	160
TOTAL INSOLUBLE MATERIAL, MG.		58	225	317	397	397	172	246	96	228	94	606

(1) AMOUNT OXYGEN SUPPLIED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).  
(2) AMOUNT OXYGEN USED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOL% OF  $O_2$  FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE  $O_2$  ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.

Table 85

## THIN FILM OXIDATION TESTS AT 600°F. USING SEVERAL CATALYST METALS

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $600 \pm 10^\circ\text{F.}$ ; TEST TIME = 3 HOURS; AIR RATE =  $10 \pm 1$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; PUMPING RATE = 240 ML. PER MINUTE; AND CATALYST = 1000 SQ. CM. OF JACK CHAIN OF METAL INDICATED.

TEST FLUID	DI-2-ETHYLHEXYL SEBACATE + 0.5 WT. % PHENOTHIAZINE				NAPHTHENIC MINERAL OIL (ESSTIC 45)				O.S. 45 SILICATE FLUID (MLO 7036)			
	COPPER	ALUMINUM	STEEL		COPPER	ALUMINUM	STEEL		COPPER	ALUMINUM	STEEL	
CATALYST METAL												
LIQUID CHARGED, GMS.	90	90	90		89	88	87		88	88	88	
LIQUID LOSS, WT. %	22	-	19		11	14	14		17	22	25	
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	7.8	7.8	7.8		7.8	7.8	7.8		7.8	7.8	7.8	
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	5.3	5.8	7.0		6.1	6.9	6.9		3.6	5.5	3.9	
APPROX. AMOUNT O <sub>2</sub> USED, %	68	75	90		79	88	88		46	72	50	
MOLS O <sub>2</sub> USED/426 GMS. OIL (2)	0.78	0.85	1.05		0.92	1.04	1.05		0.56	0.85	0.58	
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLES (3) AT 130°F.	+81	+112	+99		+89	+100	+130		+4	+36	+23	
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL	0.1	0.1	0.1		0.2	0.2	0.2		0.0	0.0	0.0	
FINAL	18.0	9.0	18.2		0.9	2.5	2.1		2.5	2.0	0.7	
OIL INSOLUBLE MATERIAL, MG. (3)	425	169	303		132	43	86		157	90	305	
CHAIN COATING, MG.	276	306	884		199	156	265		254	356	314	
TUBE COATING, MG. (4)	60	50	20		90	40	50		70	160	240	
TOTAL INSOLUBLE MATERIAL, MG.	761	525	803		421	317	401		481	606	859	

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.

Table 86

## COMPARISON OF PUMPING TYPE AND IMMERSION TYPE OXIDATION TESTS

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $600 \pm 10^\circ\text{F}$ ., TEST TIME = 3 HOURS; AIR RATE =  $10 \pm 1$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = 1000 SQ. CM. OF STEEL JACK CHAIN.

PUMPING TYPE TESTS. IN THESE TESTS, THE TEST FLUID IS PUMPED AT A RATE OF 240 ML. PER MINUTE AND IS DISCHARGED ABOVE THE JACK CHAIN CATALYST. THE FLUID IS DISTRIBUTED AS A THIN FILM OVER THE CHAIN AND AIR FLOWS COUNTERCURRENTLY THROUGH THE CHAIN.

IMMERSION TYPE TESTS. IN THESE TESTS, THE JACK CHAIN CATALYST IS IMMERSSED IN THE TEST OIL. AIR IS INTRODUCED NEAR THE BOTTOM OF THE TUBE.

TEST FLUID (COMPOSITIONS IN WT.%)	DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE		HERCOFLEX 600 + 0.5 PHENOTHIAZINE		NAPHTHENIC MINERAL OIL (ESSTIC 45)		POLYBUTENE (INDOPOL L-50)		O.S. 45 SILICATE FLUID	
	PUMPING	IMMERSION	PUMPING	IMMERSION	PUMPING	IMMERSION	PUMPING	IMMERSION	PUMPING	IMMERSION
LIQUID CHARGED, GMS	90	90	100	99	87	91	81	84	88	89
LIQUID LOSS, WT.%	19	12	21	14	24	4	10	18	25	36
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	-	6.0	6.7	6.5	7.4	5.6	5.1	6.0	4.6	4.9
APPROX. AMOUNT O <sub>2</sub> USED, % (2)	-	77	86	88	95	72	65	77	59	64
MOLS O <sub>2</sub> USED/426 GMS. OIL (2)	-	0.88	0.89	0.92	1.13	0.82	0.83	0.95	0.69	0.74
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF INSOLUBLES (3)										
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	+128	+52	+78	+104	+135	+98	+41	+2	+15	+196
OIL INSOLUBLE MATERIAL, MG. (3)	0.1	0.1	0.1	0.1	0.2	0.2	0.1	0.1	0.0	0.0
CHAIN COATING, MG.	12.0	9.8	6.2	8.6	1.4	1.7	0.7	0.7	1.5	2.1
TUBE COATING, MG. (4)	657	1556	199	341	91	45	11	18	142	305
TOTAL INSOLUBLES MATERIAL, MG.	262	-692	318	248	252	82	24	148	156	314
	70	60	30	70	40	20	30	30	90	20
	989	924	547	1357	383	147	282	196	388	132

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF O<sub>2</sub> FOR 426 GRAMS OF OIL HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

(4) THIS IS THE INSOLUBLE MATERIAL ADHERING TO THE GLASS TEST TUBE AFTER THE TUBE IS WASHED OUT WITH PETROLEUM NAPHTHA.

# Contrails

I. PANEL COKER STUDIES. A Model C and a modified (controlled atmosphere panel coker) Model C coker have been used as a method of evaluating relative coking or deposition trends for high temperature fluids. These units have been used to evaluate the effects of temperature, gas composition, and gas rate through the coker. In terms of fluid evaluation, the effects of base stock type, additives used, and fluid volatility have been compared. The tests discussed in this report are of an exploratory nature designed to illustrate better the relative importance of oxidation and thermal degradation in deposit forming trends of the various fluid classes.

1. Panel Coker Calibration. A one gallon sample of oil MLO 56-39 has been received from the Materials Laboratory, Wright Air Development Center. It was requested that this oil be used for further calibration of the Model C coker under standard operating conditions.

Values obtained for MLO 56-39 in the Model C panel coker at 600°F. and eight hours test time are shown in the following table. Values obtained for this oil by the Power Plant Laboratory, Wright Air Development Center are also listed. The values obtained indicate good agreement between the two laboratories.

PANEL COKER VALUES FOR MLO 56-39

	Date of Run	Deposit on Panel, mg.
Values Obtained by	29 Nov 55	162.6
Power Plant Laboratory	30 Nov 55	149.8
Values Obtained by	21 Feb 56	158.6
Pet. Ref. Laboratory	27 Feb 56	135.0

Earlier values obtained with the Model C panel coker for two fluids (GTO 38 and GTO 133) furnished by the Wright Air Development Center indicated relatively poor agreement between the two laboratories. A sample of GTO 38 was returned by this Laboratory to the Power Plant Laboratory for the determination of values on this particular sample of GTO 38.

2. Controlled Atmosphere Panel Coker. The panel coker test, as conducted under Spec. MIL-I-7808 conditions with a Model C coker, is an eight hour test at 600°F. during which time the fluid in the coker is generally completely replaced once because of volatility, oxidation, leakage, and thermal degradation. The unit is also open to the atmosphere with free air circulation. The resultant test is a complex function of oxidative and thermal effects, as well as many other variables difficult to control.



# Contrails

In an attempt to make a more realistic situation with better control of the variables, a Model C panel coker has been modified so that the atmosphere above the fluid can be carefully controlled and the rate of fluid loss reduced to the point where complete replacement of the test fluid in the unit occurs in 75 to 150 hours instead of about four hours under the regular Model C coker.

This modification consists of installing a running seal on the stirrer shaft where it enters the coker reservoir; using a gasket around the seal between the top of the reservoir and the panel holder; and installing a 1/4-inch tube at each end of the reservoir above the liquid level to allow for the entrance and exit of the controlled atmosphere.

Preliminary tests with this modified unit show that a gas flow of one liter per hour through the reservoir is sufficient to maintain a controlled atmosphere in the panel coker. A diagram of the controlled atmosphere panel coker is shown as Figure 4.

Test results with several representative fluids in the modified unit at an air rate of five liters per hour and in the Model C coker are shown on Table 87. These values are tabulated to point out the reduced fluid consumption and the higher reservoir oil temperature encountered for tests in the controlled atmosphere coker. The final neutralization number is, in general, lower following the tests conducted in the modified unit with an air rate of five liters per hour.

Data from the controlled atmosphere coker, using various nitrogen or air rates, are shown on Table 88. In general, the data from the controlled panel coker show that the severity of the test for the ester-type fluids is increased when fluid consumption is reduced. Under essentially a nitrogen atmosphere, Spec. MIL-L-7808 oil shows coke values of about the same order of magnitude as the Model C coker. Much of the thermal coking of Spec. MIL-L-7808 fluids is believed to be due to the phosphorus-containing lubricity additive used in these fluids. The di-2-ethylhexyl sebacate blend shows the same relative behavior but the coke values are lower. The naphthenic mineral oil, on the other hand, shows a reduction in coke as a direct function of the reduction in oxygen.

The silicate fluid shows about the same coking tendencies under nitrogen and air in the Model C coker. The values listed show some variation because of the crystalline form of the deposit which has a tendency to fall off the panel during the test and the cleaning procedure.

The use of an alkyl acid phosphite as a lubricity additive in esters and mineral oils affects adversely the coking tendencies of the resultant fluid. The data for these blends, as well as the silicate fluid, indicate that coking can be essentially a thermal phenomenon.

# Contrails

In a Model C coker, improved lubricity mineral oil and diester fluids (containing an alkyl acid phosphite) give approximately the same panel deposit. The controlled atmosphere panel coker gives increased values for the improved lubricity diester. The controlled atmosphere coker gives much lower coke values than the Model C for the alkyl acid phosphite in the mineral oil.

The fluid consumption rate is lower in the controlled atmosphere coker and the products of thermal deterioration and incipient oxidation are recirculated for a longer time than they are in the Model C coker. This lower consumption rate is believed to be more typical of actual jet engine lubrication experience.

3. Effect of Phosphoric or Phosphorous Acid Impurities on Panel Coker Values of Spec. MIL-L-7808 Type Fluids. Data have been presented in previous reports which indicate that phosphorus-containing antiwear additives incorporated in Spec. MIL-L-7808 fluids contribute more than any other component to dirtiness and loss in high temperature oxidation and thermal stability. Studies have indicated that phosphorus compounds containing acid groupings are more effective than aryl phosphorus compounds in increasing dirtiness and decreasing high temperature stability. It has been noted that increased acidity of the phosphorus compound does increase lubricity effectiveness.

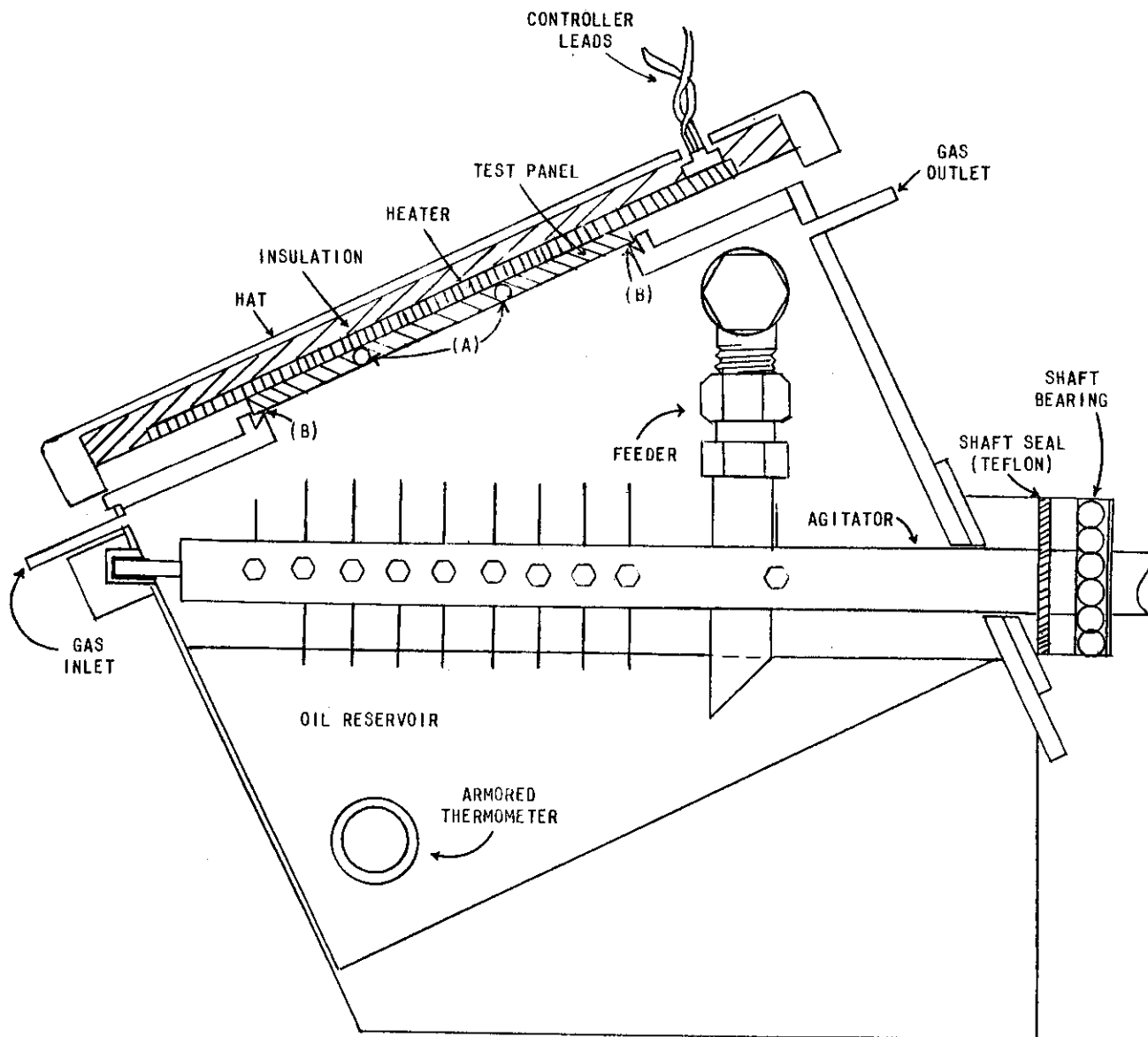
A study of the effect of small concentrations of phosphorous and phosphoric acid on the panel coking tendencies in Spec. MIL-L-7808 and MLO 7010 type compositions has been conducted in the Model C and controlled atmosphere cokers. These data are shown on Table 90. Phenothiazine or Paranox 441 (a hindered phenol) are used as the oxidation inhibitors in these blends. Paranox 441 alone does not provide sufficient stability to meet the Spec. MIL-L-7808 requirements for oxidation stability. Combinations of Paranox 441 and a dialkyl acid phosphite (MLO 7010 and GTO 120) have shown adequate oxidation stability for Spec. MIL-L-7808 requirements.

Data on Table 90 indicate that phosphoric and phosphorous acid in low concentrations cause marked increases in the coking tendencies of phenothiazine-inhibited di-2-ethylhexyl sebacate. The coking values in the Model C and the controlled atmosphere units are in good agreement in these tests. The amount of coking in both units increases steadily with acid concentration over the range of 0.01 to 0.2 weight per cent acid. A concentration of only 0.01 weight per cent phosphoric acid is sufficient to cause coking in excess of the limits of Spec. MIL-L-7808. A typical Spec. MIL-L-7808 oil contains about five weight per cent tricresyl phosphate. The amount of phosphoric acid type impurity necessary to cause excessive coking in an otherwise satisfactory fluid would be less than 0.5 weight per cent of the tricresyl phosphate used. Acidic materials of this sort can occur as an impurity in the tricresyl phosphate; they are produced by a hydrolysis type reaction in storage, and are produced as products of oxidative and thermal deterioration at elevated temperatures.



CONTROLLED ATMOSPHERE PANEL COKING TESTER

Scale: 3/4 inch = 1 inch



(A) = THERMOCOUPLE WELL  
(B) = KNIFE EDGE

Table 87

COMPARISON OF COKING VALUES OBTAINED IN THE CONTROLLED  
ATMOSPHERE PANEL COKER AND THE MODEL C COKER

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600°F. AND TEST TIME = 8 HOURS. AIR RATE IN THE  
CONTROLLED COKER = 5 LITERS/HOUR.

TEST FLUID (COMPOSITIONS IN WT.%)	COKER (1)	FLUID LOSS, ML.	MAX. OIL TEMP. IN RESERVOIR, °F.	WT. GAIN, MG.	NEUT. NO. (MG. KOH/GM. FLUID)	
					ORIG.	FINAL
DI-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE	C.A.	5	276	28	0.1	0.5
	MODEL C	180	218	17	0.1	1.4
HERCOFLEX 600 + 0.5 PHENOTHIAZINE	C.A.	10	263	11 <sup>(2)</sup>	0.1	0.1
	MODEL C	95	219	23	0.1	1.6
NAPHTHENIC MINERAL OIL	C.A.	25	317	109	0.0	1.2
	MODEL C	45	270	194	0.0	1.3
SILICATE FLUID	C.A.	10	294	179	0.1	3.8
	MODEL C	440	268	168	0.1	6.3

(1) C.A. INDICATES CONTROLLED ATMOSPHERE COKER.

(2) AIR RATE IN THIS CASE WAS 30 LITERS PER HOUR.

Table 88

## COMPARISON OF COKING VALUES OBTAINED IN THE CONTROLLED ATMOSPHERE PANEL COKER AND THE MODEL C COKER

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 600°F. AND TEST TIME = 8 HOURS. AIR OR NITROGEN RATES IN THE CONTROLLED COKER AS INDICATED. NITROGEN USED CONTAINS 0.5 PER CENT OXYGEN.

TEST FLUID (COMPOSITIONS IN WT.%)	<- - - - - NITROGEN RATE, LITERS/HOUR			CONTROLLED ATMOSPHERE COKER; WT. GAIN, MG. AIR RATE, LITERS/HOUR			- - - - -> AIR RATE, LITERS/HOUR			WT. GAIN, MG. MODEL C COKER
	1	5	5	1	5	5	1	5	30	
SPEC. MIL-L-7808 TYPE, BATCHES 1 2 3	-	42	100	-	-	-	-	-	-	20
	-	4.6	18	-	-	-	-	-	-	19
	-	18	40	-	-	-	-	-	-	28
D1-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE	1.9	3.6	28	39	-	-	-	-	85	6.6
D1-2-ETHYLHEXYL SEBACATE + 0.5 PHENOTHIAZINE + 0.5 DIISOPROPYL ACID PHOSPHITE	625	700	746	585	-	-	-	-	-	350
HERCOFLEX 600 + 0.5 PHENOTHIAZINE	-	-	-	-	-	-	-	-	11	23
NAPHTHENIC MINERAL OIL	1.8	5.9	120	40	-	-	-	-	186	194
NAPHTHENIC MINERAL OIL + 0.5 DIISOPROPYL ACID PHOSPHITE	-	100	145	-	-	-	-	-	-	327
SILICATE FLUID	-	495	179	-	-	-	-	-	-	168

The phosphoric and phosphorous acids in a Paranox 441-inhibited di-2-ethylhexyl sebacate do not cause increased coking in either the Model C or controlled atmosphere cokers. Coking behavior with this additive combination is similar to that reported for MLO 7010 and GTO 120 fluids in previous reports. In these latter fluids, a combination of diisopropyl acid phosphite and Paranox 441 is used as an oxidation inhibitor and lubricity additive. Coke deposits from phenothiazine-inhibited blends containing phosphorus additives tend to be spotty with large areas of clean metal surface on the panel.

Another striking difference between these blends inhibited with Paranox 441 and phenothiazine is the change in neutralization number during the panel coker test. In all cases, neutralization number change is about the same for a given inhibitor over the entire range of phosphoric and phosphorous acid concentration. The neutralization number increase for the blends containing Paranox 441 is much higher than for the blends containing phenothiazine. The same relative difference in neutralization number change has previously been noted in the Spec. MIL-L-7808 oxidation and corrosion tests comparing a typical phenothiazine-inhibited blend containing dialkyl acid phosphite with a similar blend containing Paranox 441 (hindered phenol). In these oxidation and corrosion tests, the increased neutralization number encountered with hindered phenol-inhibited blends is not indicative of more severe metal corrosion or larger changes in physical properties after oxidation.

These data indicate that traces of phosphoric or phosphorous acid in Spec. MIL-L-7808 compositions can materially affect panel coker values. The oxidation inhibitor type can alter markedly the coking tendencies of esters containing acidic phosphorus additives. Lubricity changes described in previous reports suggest the formation of acidic materials containing phosphorus during storage as well as in use at elevated temperatures. These laboratory data suggest the need for engine data to determine the benefits gained lubricity-wise from the phosphorus lubricity additives, and the improvement, if any, that can be obtained in terms of engine deposit from the use of hindered phenol type oxidation inhibitors instead of amine types.

4. A Comparison of Conradson Carbon and Panel Coker Values for Mineral Oils. A number of mineral oil fractions have been studied in the Model C panel coker and in the Conradson carbon residue (ASTM Method D189-52) tests for coke forming tendencies. The Conradson carbon test has been applied to lubricating oils, primarily in the automotive lubricants field, as a relative measure of coke or carbon forming tendencies.

Comparative data for these two coking tests are shown on Table 91. These data indicate that there is no apparent correlation between the panel coker values and the Conradson carbon values. In general, the Conradson carbon values increase with decreasing volatility of the mineral oil fractions. That is, Conradson carbon values show a general increase in going from neutrals - to bright stocks - to petroleum resins. Panel coker values show a general decrease with decreasing volatility over the same fluid range.

MLO 7083, MLO 7084, and MLO 7085 are the new and used Grade 1065 mineral oils from a J-57 jet engine test. Tests in the jet engine causing oxidation of the used samples are discussed in another section of this report. Conradson carbon values increase with use and oxidative deterioration for this Grade 1065 oil. The panel coker values remain essentially unchanged with use in the engine.

5. Coking Values of Blends. Several mineral oils and ester blends have been evaluated in the Model C and controlled atmosphere panel cokers. The results of these studies in the two panel cokers are shown on Table 92. Data are shown for di-sec-amyl sebacate. Data presented in previous reports indicate that di-sec-amyl sebacate is less thermally stable than di-2-ethylhexyl sebacate.

In general, thermal deterioration for diesters of secondary alcohols occurs about 100°F. lower than the same degree of thermal deterioration for diesters of primary alcohols. Little coking is encountered at 600°F. panel temperature for the di-sec-amyl sebacate alone. Blends of this ester with oxidation and antiwear additives cause a high coke deposition. Comparable blends with di-2-ethylhexyl sebacate cause very little coking.

Coking tendencies of 50:50 mixtures of Esstic 45 (naphthenic mineral oil) and esters of varying thermal stability have been determined. In all cases, the ester alone gives little or no coking. In combination with the mineral oil, coking values are directly proportional to the thermal stability of the ester component in both cokers. Only in the case of the di-sec-amyl sebacate blend, however, is the coke value as high or higher than that for the mineral oil alone.

Indopol L-50 (polybutene) is a synthetic hydrocarbon which absorbs oxygen readily to produce, at high temperatures, predominantly volatile oxidation products. Coking values for 50:50 blends of Indopol L-50 with mineral oils are in most cases higher than for the mineral oil component alone. The addition of small concentrations of various additives to Indopol L-50 also adversely affects coking tendencies.

These data adequately point up the sensitivity of coking tendencies to minor changes in composition. More evidence for relative coking trends in engines as a function of fluid composition would be helpful in assaying the relative merits of panel coker studies. Data obtained in high temperature oxidations and other high temperature stability tests do not in all cases show the same order of dirtiness as the panel coker.

The 13 large scale blends supplied to the Power Plant Laboratories have been evaluated in both cokers as shown on Table 93. These data indicate that all of the fluids except the synthetics (MLO 7058, MLO 7058A, and MLO 7061) fail to meet the Spec. MIL-L-7808C panel coker maximum values.

# Contrails

The values obtained in the Model C coker are typical of conventionally refined mineral oils evaluated previously. The polyolefin (MLO 7058) and the hydrogenated polyolefin (MLO 7058A) show excellent panel coker values. Aroclor 1248 (MLO 7061) shows somewhat higher coker values than those obtained for some previous batches of this fluid. The reason for this increased coker value is not apparent.

The three conventionally refined mineral oils (MLO 7055, MLO 7056, and MLO 7057) all show increases in coking value in both cokers when the 1.0 weight per cent PAN (phenyl-alpha-naphthylamine) oxidation inhibitor is added to give MLO 7055A, MLO 7056A, and MLO 7057A, respectively. Amine type oxidation inhibitors show the same general effect on Spec. MIL-L-7808 fluids.

The coker values obtained in the controlled atmosphere coker are generally lower than Model C values for the same fluid. Within the mineral oil and hydrocarbon class, the values from both cokers follow the same general pattern in aligning a series of fluids for coking severity.

6. High Temperature Coker Tests. A series of panel coker runs have been conducted with ester and mineral oil compositions at 700°F. panel temperature.. These data are presented on Tables 94 and 95. These data indicate the superiority of a pentaerythritol ester base stock over the dibasic acid ester in finished compositions.

The phenothiazine blends of both esters show only moderate coke values. The addition of tricresyl phosphate or a dialkyl acid phosphite to di-2-ethylhexyl sebacate causes an excessive increase in coking tendencies.

A comparison of mineral oil coking values at 600° and 700°F. can be made on Table 89. In general, there is a relatively small change in the coking value of the conventionally refined paraffinic stocks between 600° and 700°F. in the Model C coker. In some cases coking may be reduced at 700°F. over the 600°F. value. The values obtained in the controlled atmosphere coker are also lower than the values from the Model C coker at 700°F.

Table 89  
COMPARISON OF COKING VALUES AT 600° AND 700°F.  
All Tests Conducted in the Model C Coker; Test Time = 8 Hrs.

Test Fluid	Wt. gain, mg. at	
	600°F.	700°F.
MLO 7093 Barosa 43 (Paraffinic 180 Neutral)	158	200
MLO 7070 Kendall Medium Neutral	113	71.7
MLO 7078 Pennsylvania 180 Neutral	204	82.4
MLO 7071 Kendall Heavy Neutral	97.0	98.6
MLO 7077 Pa. Neutral-Bright Stock Blend	65.7	117
MLO 7094 Esso Aviation Bright Stock	116	76.0
MLO 7030 Esstic 45 (Naphthenic Mineral Oil)	154	239

7. Effect of Fluid Volatility on Panel Coking Value. A series of naphthenic mineral oils of increasing viscosity and boiling point range have been evaluated (Table 95). These materials are narrow boiling fractions. In the Model C coker, the volatile fractions show extremely high coking. The high viscosity, low volatility fractions show coking values typical of the mineral oils considered for high temperature lubricants.

The controlled atmosphere coker shows a somewhat different effect of volatility. The highly volatile PRL 2964 shows a very low coking value. MLO 7019 and PRL 2031 show high coking values, and the higher viscosity, low volatility stocks show much less coking tendency. The atmospheric pressure boiling ranges of the first three fluids are approximately 400° to 550°F. for PRL 2964, 475° to 675°F. for MLO 7019, and 575° to 750°F. for PRL 2031. The corresponding C.O.C. flash points are shown on Table 95.

The relative differences in coking as a function of volatility are attributable for the most part to vapor-phase versus liquid-phase oxidation. The fluids which contain large amounts of materials boiling below the 700°F. panel temperature, show high coking values because of the increased uptake of oxygen as vapor phase oxidation in the immediate vicinity of the panel. Where little vapor is formed, the much slower liquid phase oxidation accounts for normal coking values.

The difference between the coking of PRL 2964 in the two cokers is attributable to the difference in the accessibility to air. These same effects have been noted in the high temperature single pass lube rig with highly volatile materials. In a case where air is limited and the final boiling point is well below surface temperatures, vapor phase oxidation results in oil deterioration with little panel deposit. As the air supply is increased, panel dirtiness also increases.

The apparent relationship of coking and deposition on hot metal surfaces to fluid volatility indicates that satisfactory high temperature engine lubricants will probably be of the high viscosity-low volatility type. These data point up some of the shortcomings of lowering volatility and flash point requirements as a compromise to obtain better low temperature characteristics in a high temperature jet engine lubricant for use at 500° to 700°F. The effects of volatility on other stability tests at high temperatures have been indicated in previous reports. An attempt will be made to evaluate more carefully the effect of volatility in the various tests employed to measure overall stability.

J. SINGLE PASS HIGH TEMPERATURE LUBE RIG. A number of tests have been developed and/or used by this Laboratory to define high temperature stability of fluids. These tests include the following procedures:

1. Oxidation stable life,
2. Oxygen tolerance,
3. Oxidation rate,
4. Thin film oxidation test,



Table 90

## EFFECT OF PHOSPHORIC OR PHOSPHOROUS ACID IMPURITIES ON PANEL COKER VALUES AT 600°F.

TEST CONDITIONS INCLUDE: PANEL TEMPERATURE = 600°F. AND TEST TIME = 8 HRS. AIR RATE IN THE CONTROLLED ATMOSPHERE COKER = 5 LITERS PER HOUR.

TEST FLUID: DI-2-ETHYLHEXYL SEBACATE

<- - - ADDITIVE CONCENTRATION, WT. % - - ->				<- - - - MODEL C COKER - - - ->				<- - - - CONTROLLED ATMOSPHERE COKER - - - ->			
PHOSPHORIC ACID	PHOSPHOROUS ACID(1)	PHENO-THIAZINE	PARANOX 441	WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID)		MAXIMUM RESERVOIR TEMP., °F.	WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID)		MAXIMUM RESERVOIR TEMP., °F.
					ORIG.	FINAL			ORIG.	FINAL	
-	-	0.5	-	17	0.1	1.4	218	28	0.1	0.5	276
-	0.01	0.5	-	58.5	0.3	0.1	273	33.1	0.3	0.4	263
-	0.02	0.5	-	65.2	0.6	0.7	286	78.2	0.8	1.0	323
-	0.05	0.5	-	116	1.1	0.8	306	98.1	1.1	1.2	300
-	0.10	0.5	-	128	0.8	0.6	321	72.8	0.8	1.1	320
-	-	-	-	281	1.2	0.9	287	-	-	-	-
-	0.01	-	1.0	-	-	-	-	6.1	0.4	7.4	258
-	0.02	-	1.0	0.0	0.5	11.3	265	0.3	0.6	10.6	270
-	0.05	-	1.0	-	-	-	-	7.6	1.0	6.0	267
-	0.10	-	1.0	-	-	-	-	6.4	1.3	8.4	267
0.01	-	0.5	-	89.4	0.3	0.4	276	77.3	0.3	1.7	265
0.02	-	0.5	-	155	0.4	0.6	319	-	-	-	-
0.05	-	0.5	-	176	0.7	1.0	325	162	0.7	1.6	325
-	-	-	-	229	0.9	0.8	314	-	-	-	-
0.10	-	0.5	-	232	0.6	0.9	314	241	0.6	1.4	312
0.20	-	0.5	-	149	0.7	0.8	274	406	0.7	1.0	279
0.01	-	-	1.0	-	-	-	-	12.7	0.4	7.1	268
0.02	-	-	1.0	-	-	-	-	1.8	0.5	8.1	272
0.05	-	-	1.0	0.4	0.3	22.0	306	9.0	0.7	6.7	310
-	-	-	-	-	-	-	-	5.9	0.8	7.0	296
0.10	-	-	1.0	-	-	-	-	3.8	0.6	8.0	308
0.20	-	-	1.0	-	-	-	-	6.2	0.9	8.0	268

(1) PHOSPHOROUS ACID IS NOT COMPLETELY SOLUBLE AT CONCENTRATIONS ABOVE 0.01 WEIGHT PER CENT.

Table 91

## COMPARISON OF PANEL COKER VALUES AND CONRADSON CARBON RESIDUES FOR SEVERAL MINERAL OILS

PANEL COKER TEST CONDITIONS INCLUDE: PANEL TEMPERATURE = 600°F. AND TEST TIME = 8 HOURS.  
CONRADSON CARBON RESIDUE DETERMINED IN ACCORDANCE WITH A.S.T.M. METHOD D189-52.

TEST FLUID	CENTISTOKE VISCOSITY AT 100°F.	MODEL C PANEL COKER WT. GAIN, MG.	CONRADSON CARBON RESIDUE % CARBON
MLO 7078 PA. 180 NEUTRAL	38	204	0.04
MLO 7070 KENDALL MEDIUM NEUTRAL	36	113	0.01
MLO 7026 PA. 300 NEUTRAL	65	90.1; 94.1	0.04
MLO 7071 KENDALL HEAVY NEUTRAL	107	97.0	0.22
MLO 7069 KENDALL SAE 30, HD-1 MOTOR OIL	123	222	0.85
MLO 7072 KENDALL LIGHT RESIN	5900	5.8	2.86
MLO 7073 KENDALL HEAVY RESIN	16,000	6.3	5.18
MLO 7077 PA. NEUTRAL-BRIGHT STOCK BLEND	113	65.7	0.73
MLO 7076 MLO 7077 AFTER HYDROGENATION	97	10.8	0.06
MLO 7093 BAROSA 43 (PARAFFINIC 180 NEUTRAL)	34	158	0.04
MLO 7094 ESSO AVIATION BRIGHT STOCK	467	116	0.76
MLO 7030 ESSTIC 45 (HIGHLY REFINED NAPHTHENIC MINERAL OIL)	45	155; 194	0.03
MLO 7028 SOCONY-MOBIL HEAT TRANSFER OIL NO. 600	57	152	0.20
MLO 7083 GRADE 1065 AVIATION ENGINE OIL	104	79.2	0.15
MLO 7084 MLO 7083 AFTER 30 HOURS IN J-57 ENGINE TEST	124	88.5	0.51
MLO 7085 MLO 7083 AFTER 50 HOURS IN J-57 ENGINE TEST	132	62.4	0.77

Table 92

PANEL COKING VALUES FOR SEVERAL BLENDS

TEST CONDITIONS INCLUDE: PANEL TEMPERATURE = 600°F. AND TEST TIME = 8 HOURS. AIR RATE IN THE CONTROLLED ATMOSPHERE COKER = 5 LITERS PER HOUR.

TEST FLUID		MODEL C COKER WT. GAIN, MG.	CONTROLLED ATMOSPHERE COKER WT. GAIN MG.
PRL 3585	DI-SEC.-AMYL SEBACATE	5.5	18.7
	PRL 3585 + 1.0 PARANOX 441	125	91.6
	PRL 3585 + 1.0 PARANOX 441 + 0.5 DIISOPROPYL ACID PHOSPHITE	1040	1297 (1)
MLO 7038	DI-2-ETHYLHEXYL SEBACATE	9.8	-
	MLO 7038 + 1.0 PARANOX 441 + 0.5 DIISOPROPYL ACID PHOSPHITE	107	704
MLO 7030	ESSTIC 45 (HIGHLY REFINED NAPHTHENIC MINERAL OIL)	155	108
	50:50 (BY WT.) ESSTIC 45: HERCOFLEX 600	194	120
	50:50 (BY WT.) ESSTIC 45: DI-2-ETHYLHEXYL SEBACATE	100	24.4
	50:50 (BY WT.) ESSTIC 45: DI-SEC.-AMYL SEBACATE	129	29.7
MLO 7014	INDOPOL L-50 (LOW MOLECULAR WT. POLYOLEFIN)	313	123
MLO 7026	PA. 300 NEUTRAL	18.3	3.8
MLO 7070	KENDALL MEDIUM NEUTRAL	90.1	30.2
MLO 7071	KENDALL HEAVY NEUTRAL	113	-
	50:50 (BY WT.) MLO 7014:MLO 7026	97.0	-
	50:50 (BY WT.) MLO 7014:MLO 7070	145	84.2
	50:50 (BY WT.) MLO 7014:MLO 7071	227	101
MLO 7093	BAROSA 43 (PARAFFINIC 180 NEUTRAL)	189	65.0
	MLO 7093 + 2.0 PRL 3605 (DITHIOCARBAMATE TYPE ADDITIVE)	158	-
		410	213

(1) TEST TERMINATED AFTER 2.5 HOURS.

Table 93

PANEL COKING VALUES FOR EXPERIMENTAL LUBRICANTS FURNISHED TO THE POWER PLANT  
UNDER CONTRACT AF33(600)32853

TEST CONDITIONS INCLUDE: PANEL TEMPERATURE = 600°F. AND TEST TIME = 8 HOURS. AIR RATE IN THE CONTROLLED ATMOSPHERE COKER = 5 LITERS PER HOUR.

<-----TEST FLUID----->		<-----MODEL C COKER----->				<-----CONTROLLED ATMOSPHERE COKER----->					
DESIGNATION	DESCRIPTION	WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID) ORIG.	FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.	WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID) ORIG.	FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.
MLO 7055	HIGHLY REFINED NAPHTHENIC MINERAL OIL + ANTI-FOAM AGENT	168	0.1	1.1	270	250	64.8	0.1	0.9	0	300
MLO 7055A	MLO 7055 + 1.0 WT. % PAN(1)	186	0.1	1.5	145	240	150	0.1	1.4	10	290
MLO 7056	PENNSYLVANIA HEAVY; NEUTRAL + ANTI-FOAM AGENT	132	0.1	1.1	75	258	60.9	0.1	1.1	5	295
MLO 7056A	MLO 7056 + 1.0 WT. % PAN(1)	164	0.1	1.8	95	262	85.7	0.1	0.8	0	300
MLO 7057	PARAFFINIC 180 NEUTRAL + ANTI-FOAM AGENT	92.7	0.0	3.0	175	265	20.6	0.0	1.2	10	279
MLO 7057A	MLO 7057 + 1.0 WT. % PAN(1)	129	0.0	1.8	210	260	62.1	0.0	0.9	15	260
MLO 7058	LOW MOLECULAR WT. POLYOLEFIN + ANTI-FOAM AGENT	0.3	0.1	1.0	180	255	20.9	0.1	1.1	0	310
MLO 7058A	HYDROGENATED POLYOLEFIN + ANTI-FOAM AGENT + 1.0 WT. % PAN(1)	10.7	0.0	0.3	260	252	12.1	0.0	0.3	15	308
MLO 7059	50:50 (BY WT.) MLO 7056:MLO 7058	146	0.1	1.6	145	252	64.2	0.1	1.3	0	295
MLO 7059A	50:50 (BY WT.) MLO 7056A:MLO 7058A	136	0.1	0.4	170	242	90.2	0.1	0.5	0	305
MLO 7060	50:50 (BY WT.) MLO 7057:MLO 7058	116	0.0	1.7	235	250	45.8	0.0	1.0	5	303
MLO 7060A	50:50 (BY WT.) MLO 7057A:MLO 7058A	97.8	0.1	0.7	215	245	55.6	0.1	0.7	5	306
MLO 7061	AROCOLOR 1248 (CHLORINATED BIPHENYL)	32.7	0.0	0.0	75	285	25.0	0.0	0.0	0	315

(1) PHENYL-ALPHA-NAPHTHYLAMINE.

Table 94

PANEL COKING VALUES FOR SEVERAL FLUIDS AT 700°F.

TEST CONDITIONS INCLUDE: PANEL TEMPERATURE = 700°F. AND TEST TIME = 8 HOURS. AIR RATE IN THE CONTROLLED ATMOSPHERE COKER = 5 LITERS PER HOUR.

TEST FLUID		<--- MODEL C --->					<--- CONTROLLED ATMOSPHERE --->				
DESIGNATION	DESCRIPTION	WT. GAIN, MG.	NEUT. NO. GM. FLUID ORIG.	MG. KOH/ GM. FLUID FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.	WT. GAIN, MG.	NEUT. NO. GM. FLUID ORIG.	MG. KOH/ GM. FLUID FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.
MLO 7038	DI-2-ETHYLHEXYL SEBACATE	-	-	-	-	-	-	-	-	-	-
-	0.5 WT. % PHENOTHIAZINE IN MLO 7038	76	0.2	0.7	160	208	107	0.2	8.6	55	365
-	0.5 WT. % PHENOTHIAZINE + 5.0 WT. % TRICRESYL PHOSPHATE IN MLO 7038	1287	0.0	16.4	195	325	390	0.0	7.6	65	317
MLO 7010	1.0 WT. % PARANOX 441 + 0.5 WT. % DIISOPROPYL ACID PHOSPHITE + 3.5 WT. % ACRYLOID HF-25 IN MLO 7038	2225	0.5	28.2	310	328	1975	0.5	45.6	45	330
MLO 7064	HERCOFLEX 600 (PENTAERYTHRITOL ESTER)	2.0	0.1	10.4	330	305	1.1	0.1	5.6	45	326
-	0.5 WT. % PHENOTHIAZINE IN MLO 7064	31.7	0.1	4.7	290	295	15.7	0.1	5.6	35	337
-	0.5 WT. % PHENOTHIAZINE + 0.5 WT. % DIISOPROPYL ACID PHOSPHITE IN MLO 7064	89.8	-	8.0	255	300	16.6	-	17.0	45	328

Table 95

## PANEL COKING VALUES FOR SEVERAL MINERAL OILS AT 700°F.

TEST CONDITIONS INCLUDE: PANEL TEMPERATURE = 700°F. AND TEST TIME = 8 HOURS. AIR RATE IN THE CONTROLLED ATMOSPHERE COKER = 5 LITERS PER HOUR.

TEST FLUID		CENTISTOKE VISCOSITY AT 100°F.	COC FLASH POINT, °F.	<--- MODEL C COKER --->				<--- CONTROLLED ATMOSPHERE COKER --->			
DESIGNATION	DESCRIPTION			WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID) ORIG.   FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.	WT. GAIN, MG.	NEUT. NO. (MG. KOH/ GM. FLUID) ORIG.   FINAL	FLUID LOSS, ML.	MAXIMUM RESERVOIR TEMP., °F.
MLO 7093	BAROSA 43 (PARAFFINIC 180 NEUTRAL)	34	-	200 237	0.2   2.0 0.2   2.0	395 395	290 314	98.8 78.2	0.2   1.2 0.2   1.0	40 40	330 334
MLO 7070	KENDALL MEDIUM NEUTRAL	36	-	71.7	0.1   6.0	235	327	54.3	0.1   1.1	60	340
MLO 7078	PENNSYLVANIA 180 NEUTRAL	38	-	82.4 89.1	0.1   5.7 0.1   4.3	210 280	318 319	18.5 -	0.1   1.1 -   -	50 -	333 -
MLO 7071	KENDALL HEAVY NEUTRAL	107	-	98.6	0.1   2.3	190	283	5.2	0.1   1.4	35	303
MLO 7077	PA. NEUTRAL-BRIGHT STOCK BLEND	113	-	117	0.1   2.4	255	283	21.4	0.1   2.2	25	303
MLO 7094	ESSO AVIATION BRIGHT STOCK	467	-	76.0	0.1   2.5	65	301	8.0	0.1   1.1	35	284
MLO 7030	ESSTIC 45 (NAPHTHENIC MINERAL OIL)	45	-	239 575	0.1   5.3 0.1   5.2	225 235	362 355	129 166	0.1   1.6 0.1   1.5	35 45	353 352
PRL 3467	NECTON 60 (NAPHTHENIC MINERAL OIL)	109	-	256	0.1   3.7	335	305	19.1	0.1   2.1	45	322
PRL 2964	XCT WHITE OIL (NAPHTHENIC GAS OIL)	3	220	1047	0.1   2.2	740	345	10.6	0.1   1.3	0	335
MLO 7019	VOLTESO 36 (NAPHTHENIC GAS OIL)	9	310	1836	0.0   2.3	205	347	888	0.0   1.4	25	373
PRL 2031	NECTON 37 (NAPHTHENIC MINERAL OIL)	15	340	1957	0.1   1.7	170	362	926	0.1   1.2	0	382
PRL 2053	NECTON 42 (NAPHTHENIC MINERAL OIL)	35	375	184	0.0   3.7	190	370	193	0.0   1.3	0	365
PRL 3468	HUMBLE PALE D (NAPHTHENIC MINERAL OIL)	200	420	214	0.1   2.4	265	316	35.5	0.1   1.3	5	333

- Experiments*
5. Successive oxidation test,
  6. Panel coker,
  7. Controlled atmosphere panel coker,
  8. Single pass high temperature lube rig, and
  9. Thermal stability test in the presence of metals.

These tests are designed to evaluate various phases of high temperature fluid stability. Since the high temperature hydraulic and lubricant systems have not as yet been developed and evaluated with fluids, the degree of pertinence of the various test procedures can not adequately be assayed with respect to their application. It is apparent from current studies that various fluid types and finished compositions show significantly different ratings in the various tests.

The single pass high temperature lube rig is designed to rate fluids reproducibly in accordance with deposit formation on a hot metal surface at low rates of lubricant application in the presence of large quantities of air. Conditions of this sort might be obtained in bearings utilizing the single pass or expendable oil lubrication system which has been discussed in previous WADC technical reports from this Laboratory.

The pump and heat exchanger from the Petroleum Refining Laboratory high temperature fuel rig have been modified to simulate a one-pass lubricant system operating at 700°F. A flow diagram of the test system is shown on Figure 5. The heated member of the heat exchanger consists of a nine-inch section of half-inch steel or aluminum tubing. The heating source is saturated metaterephenyl vapor from the boiler. This technique provides even heating without danger of hot spots. Air and fluid are pumped in cocurrent flow through the heat exchanger.

Typical conditions investigated include: heat exchanger temperature = 707°F.; time = 5 hours; fluid-flow rate = 1 ml./min.; gas-flow rate = 10 l./hr.; gas used = air; and metal surface = aluminum. Some conditions have been modified in the course of testing. Gas rates of 30 liters of air per hour and 10 liters per hour of a 9:1 volume ratio of nitrogen to air have been used. Several tests have been made with steel instead of aluminum as the heat exchanger tube.

The coatings on the heat exchanger tube are generally uniform in this test procedure. Deposits on the heat exchanger and deterioration in the used oil are measured to determine relative lubricant behavior. The deposit on the tube is determined by a three-step method. The tube, as it is removed from the heat exchanger, is soaked in naphtha to remove residual oil and any loosely adhering deposit. The deposit is then recovered from the naphtha by centrifuging and is dried and weighed. The tube is then rubbed with solvents and tissue. The deposit removed is determined by weight difference. As a final step, any remaining deposit is burned from the surface of the tube with a flame and the amount removed determined by weight loss. The exit oil is collected in calibrated centrifuge tubes. After centrifuging, the water content of the sample is noted and residue is washed with naphtha, dried, and weighed. The neutralization number of the exit oil is measured.



Data for various classes of fluids and several types and grades of mineral oils are shown on Tables 96 and 97. These data have all been obtained at an air rate of 10 liters per hour. The mineral oils and synthetic hydrocarbons are among the best fluids evaluated in terms of overall dirtiness on the tube and in the exit oil. Aroclor 1248 also shows a low level of deposit with little evidence of fluid deterioration.

The esters evaluated show good dirtiness properties in the exit oil although neutralization number increase is high, indicative of thermal instability. The deposit on the tube as well as the neutralization number increase line up in accordance with the relative thermal stability of the esters. Polyester AP-52, which contains some secondary alcohol linkages, gives the highest deposit rating and the poorest thermal stability.

The neopentyl type esters, typified by Synthetics J-7 and Hercoflex 600, give the best thermal stability and the lowest deposit value on the tube. Di-2-ethylhexyl sebacate is intermediate among the ester types in both thermal stability and deposit formation.

The silicone and silicate fluids produce a high value of total deposits. These deposits, both in the oil and on the tube, are sand-like noncombustible materials. The silicone shows a relatively low tube deposit while the dirt in the exit oil is high. The silicates, on the other hand, show a higher per cent of the total deposit on the tube.

A series of mineral oils representing paraffinic, naphthenic, and aromatic types are shown on Table 97. The naphthenic and paraffinic series cover a range of viscosity grades. The higher the viscosity in these series, the lower the volatility and the higher the boiling range. Vapor phase oxidation has previously been discussed as a method of increasing oxygen tolerance at high temperatures. In the naphthenic mineral oil series, XCT white oil and Voltesso 36 boil below 700°F., while Necton 37 has components boiling both above and below 700°F. All of the paraffin oils boil above 700°F. The low boiling naphthenic mineral oils XCT white oil and Voltesso 36 give a very clean heat exchanger tube except for a small build-up of deposit around the inlet section of the tube. This is the area where fluid at room temperature enters the tube and is vaporized. There is essentially no evidence of deposit on the portion of the tube where vapor phase oxidation occurred.

Necton 37 shows a very heavy deposit. A similar phenomenon has been noted in the panel coker where surface temperatures within the boiling range of the fluid cause excessive deposit formation.

All of the paraffinic mineral oils evaluated give low deposit values in the rig test. In general, the values for the paraffinic stocks are lower than those for a naphthenic mineral oil of the same viscosity grade. The aromatic heat transfer oil produced more deposit than either of the other two mineral oil types evaluated. The polyolefins are about the same in deposit formation. This level of deposit on the tube is characteristic also of the paraffinic mineral oils of conventional refining.

*Contrails*

The effect of dispersant type additives in Kendall heavy neutral is shown on Table 98. Acryloids 829 and 966 and Du Pont Fuel Oil Additive No. 2 have been used as dispersants. In all cases the dispersant blend gives coking values of the same order of magnitude as the non-additive mineral oil base stock. Similar behavior was noted for the Acryloids in mineral oils in the panel coker studies.

Dialkyl acid phosphite has been used as a lubricity additive in mineral oils and also in combination with a hindered phenol as an oxidation inhibitor for the intermediate temperature range (200° - 400°F.). Data for these oils are shown on Table 99. These dialkyl acid phosphites have been shown to have a deleterious effect on dirtiness in high temperature oxidation tests and in the panel coker. The use of diisopropyl acid phosphite in the polyolefin and hydrogenated polyolefin causes a substantial rise in deposit on the tube. The use of Paranox 441 with diisopropyl acid phosphite in Kendall heavy neutral also causes a substantial rise in tube deposit. The use of the same additive combination in Necton 60 shows little change in tube deposit level.

Small quantities of n-octane or octene-2 have been added to the paraffinic and naphthenic oils containing the hindered phenol-diisopropyl acid phosphite combination. The use of an oxygen acceptor or vapor phase hydrocarbons to assimilate preferentially the oxygen to reduce dirtiness was not particularly effective in these cases.

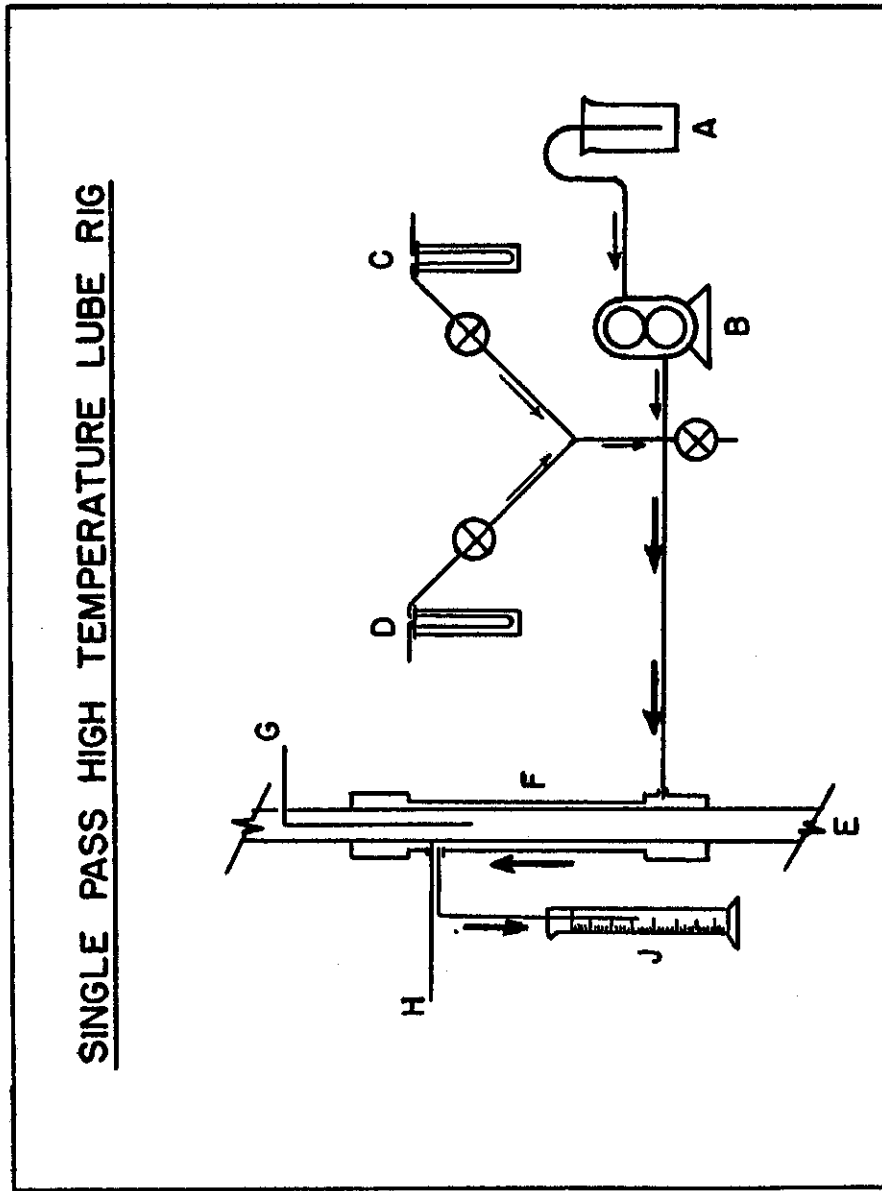
The effect of air rate on deposit formation has been studied for several mineral oils and esters. These data are tabulated on Table 100. Three air rates were employed. All of the data discussed previously is for an air rate of 10 liters per hour. In addition to this air rate, 1 and 30 liters of air per hour have been employed. In addition to the one liter of air per hour, nine liters of nitrogen per hour were used at the lowest air rate to keep up the overall gas rate.

A lowering of the total gas rate to one liter per hour would have a substantial effect on the amount of gas in the heat exchanger at any one time and therefore, on the residence time of the oil in the heat exchanger. The gas volume in the heat exchanger is 13 milliliters at ten liters per hour and 16 milliliters at 30 liters per hour.

The deposits at one liter of air per hour are substantially lower than for 10 liters of air per hour except in the case of the Hercoflex 600 type of ester. The increase in air rate to 30 liters per hour causes a large increase in deposition for the Hercoflex 600. The exit tube from the heat exchanger clogged from excessive dirt before the five hour run had completed with this ester.

For the mineral oils, the 30 liter per hour tends to magnify the differences noted at the lower air rate. The naphthenic mineral oil shows excessive deposits on the tube under these conditions while the paraffinics studied still show well.

Figure 5



→ = Gas Flow    → = Test Fluid Flow    → = Combined Gas and Test Fluid Flow

- A = Test Fluid Reservoir
- B = Zenith Pump
- C = Metered Gas Inlet
- D = Metered Gas Inlet

- E = 0.5 in. Diameter Aluminum or Steel Tube
- F = Test Fluid Heat Exchanger
- G = Thermocouple to Measure Heat Exchanger Temperature
- H = Thermocouple to Measure Outlet Fluid Temperature

Table 96

## SINGLE PASS HIGH TEMPERATURE LUBE RIG TESTS

TEST CONDITIONS INCLUDE: TEST TIME = 5 HOURS; HEAT EXCHANGER TEMPERATURE = 70°F.; AIR RATE = 10 + 1 LITERS PER HOUR; FLUID RATE = 1 ML./MIN.; AND RESIDENCE TIME FOR FLUID IN THE HEAT EXCHANGER = APPROX. 8 MINUTES. HEAT EXCHANGER SURFACE = ALUMINUM.

TEST FLUID (CONC. IN WT.%)	DEPOSIT ON TUBE, MG.	INSOLUBLES IN EXIT OIL, MG./100 GM.	WATER IN EXIT OIL, MG./100 GM.	NEUT. NO. INCREASE (MG. KOH/GM. OIL)
HERCOFLEX 600 (MLO 7064) + 0.5 PHENOTHIAZINE	16.3 17.4	50 64	- -	13.1 19.0
SYNTHETICS J-7 (MLO 7023)	23.7	330	800	18.0
SYNTHETICS J-7 (MLO 7023) + 0.5 DIISOPROPYL ACID PHOSPHITE + 0.5 PHENOTHIAZINE	10.1	14	380	18.0
POLYESTER AP-52 (PRL 3075)	1950	4900	167	72.0
TETRA-2-ETHYLHEXYL SILICATE (PRL 3455) (1)	576	900	288	14.1
O.S. 45-1 SILICATE FLUID (MLO 7037) (1)	212	29	42	13.5
ORONITE 8200 SILICATE FLUID (PRL 3506) (1)	732	0	0	1.6
ORONITE 8515 FLUID (MLO 7082) (1) (2)	1490	535	0	9.8
SILICONE 81406 (MLO 7017) (IMPROVED LUBRICITY)	25.4	1220	0	0.9
AROCLOR 1248 (PRL 3544)	9.8 (3)	0	0	0.5

(1) SAND-LIKE DEPOSIT ON TUBE AND IN EXIT OIL.

(2) HEAT EXCHANGER PLUGGED. TEST TERMINATED AFTER 3.5 HOURS.

(3) ONLY APPROXIMATELY 50 PER CENT OF THE TOTAL DEPOSIT WAS REMOVED FROM THE TUBE.

Table 97

SINGLE PASS HIGH TEMPERATURE LUBE RIG TESTS

TEST CONDITIONS INCLUDE: TEST TIME = 5 HOURS; HEAT EXCHANGER TEMPERATURE = 707°F.; AIR RATE = 10 ± 1 LITERS PER HOUR; FLUID RATE = 1 ML./MIN.; AND RESIDENCE TIME FOR FLUID IN THE HEAT EXCHANGER = APPROX. 8 MINUTES. HEAT EXCHANGER SURFACE = ALUMINUM.

TEST FLUID	DEPOSIT ON TUBE, MG.	INSOLUBLES IN EXIT OIL, MG./100 GM.	WATER IN EXIT OIL, MG./100 GM.	NEUT. NO. INCREASE (MG. KOH/GM. OIL)
PARAFFINIC MINERAL OILS				
KENDALL HEAVY RESIN (MLO 7073)	16.7	50	610	2.7
	24.7	51	380	1.5
KENDALL LIGHT RESIN (MLO 7072)	15.4	68	590	1.9
ESSO AVIATION BRIGHT STOCK (MLO 7094)	15.6	177	2180	2.0
KENDALL HEAVY NEUTRAL (MLO 7120)	13.8	82	1400	1.3
	12.5	66	1670	1.3
BAROSA 43 (MLO 7093)	31.2	42	745	0.7
NAPHTHENIC MINERAL OILS				
HUMBLE PALE D (PRL 3468)	102	470	990	1.5
NECTON 60 (PRL 3457)	61.6	-	-	-
	79.8	79	835	1.4
	61.7	176	840	1.3
NECTON 37 (PRL 2031)	412	23	1440	0.9
VOLTESSO 36 (MLO 7019)	36.3	79	2480	1.0
	19.2	40	2030	1.0
XCT WHITE OIL	14.9	92	2180	0.8
AROMATIC MINERAL OIL				
SOCOBY-MOBIL HEAT TRANSFER OIL (MLO 7028)	-(1)	-	-	-
	698	2130	455	0.5
	-(1)	-	-	-

(1) THERE WAS A LARGE AMOUNT OF SLUDGE AROUND THE TUBE. TUBE AND DEPOSIT DAMAGED DURING DISASSEMBLY.

Contrails

Table 98

EFFECT OF ACRYLOID POLYMER ON DIRTINESS IN THE HIGH TEMPERATURE LUBE RIG

TEST CONDITIONS INCLUDE: TEST TIME = 5 HOURS; HEAT EXCHANGER TEMPERATURE = 707°F.; AIR RATE = 10 ± 1 LITERS PER HOUR; FLUID RATE = 1 ML./MIN.; AND RESIDENCE TIME FOR FLUID IN THE HEAT EXCHANGER = APPROX. 8 MINUTES. HEAT EXCHANGER SURFACE = ALUMINUM.

TEST FLUID	DEPOSIT ON TUBE, MG.	INSOLUBLES IN EXIT OIL, MG./100 GM.	WATER IN EXIT OIL, MG./100 GM.	NEUT. NO. INCREASE (MG. KOH/GM. OIL)
KENDALL HEAVY NEUTRAL (MLO 7120) (PARAFFINIC MINERAL OIL)	13.8 12.5	82 66	1400 1670	1.3 1.3
+ 5.0 WT. % ACRYLOID HF-829	22.0	79	1630	2.5
+ 5.0 WT. % ACRYLOID 966	17.0	40	1290	0.7
+ 0.25 WT. % DU PONT FUEL ADDITIVE NO. 2	19.2	57	1430	1.5

Table 99

## EFFECT OF VARIOUS ADDITIVES ON DIRTINESS IN THE HIGH TEMPERATURE LUBE RIG

TEST CONDITIONS INCLUDE: TEST TIME = 5 HOURS; HEAT EXCHANGER TEMPERATURE = 707°F.; AIR RATE = 10 ± 1 LITERS PER HOUR; FLUID RATE = 1 ML./MIN.; AND RESIDENCE TIME FOR FLUID IN HEAT EXCHANGER = APPROX. 8 MINUTES. HEAT EXCHANGER SURFACE = ALUMINUM.

TEST FLUID (CONC. IN WT.%)	DEPOSIT ON TUBE, MG.	INSOLUBLES IN EXIT OIL, MG./100 GM.	WATER IN EXIT OIL, MG./100 GM.	NEUT. NO. INCREASE (MG. KOH/GM. OIL)
KENDALL HEAVY NEUTRAL (MLO 7120) (PARAFFINIC MINERAL OIL)	13.8 12.5	82 66	1400 1670	1.3 1.3
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441	128	928	1160	0.9
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441 + 10.0 OCTENE-2	145	175	1260	1.5
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441 + 10.0 n-OCTANE (1)	88.1	154	193	7.7
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441 + 10.0 n-OCTANE	82.0	160	1360	1.6
NECTON 60 (PRL 3467) (NAPHTHENIC MINERAL OIL)	61.6 79.8 61.7	- 79 176	- 835 840	- 1.4 1.3
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441	54.9	-	-	2.7
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441 + 10.0 OCTENE-2	80.3	146	1260	1.1
+ 0.5 DIISOPROPYL ACID PHOSPHITE + 1.0 PARANOX 441 + 10.0 n-OCTANE	103	116	1160	0.9
INDOPOL L-50 (MLO 7102) (POLYOLEFIN)	47.7 51.5 58.6	105 65 -	1400 500 -	1.0 0.8 -
POLYOLEFIN (MLO 7058) + 0.5 DIISOPROPYL ACID PHOSPHITE	46.1 188	125 61	680 1940	0.4 1.5
HYDROGENATED POLYOLEFIN + 1.0 PHENYL-ALPHA- NAPHTHYLAMINE (MLO 7058A) + 0.5 DIISOPROPYL ACID PHOSPHITE	33.8 291	143 145	2320 2630	0.8 0.7

(1) TEST TERMINATED AFTER 1.5 HOURS TEST TIME.



Table 100

EFFECT OF AIR RATE ON DIRTINESS IN THE HIGH TEMPERATURE LUBE RIG

TEST CONDITIONS INCLUDE: TEST TIME = 5 HOURS; HEAT EXCHANGER TEMPERATURE = 707°F.; AIR RATE AS INDICATED; FLUID RATE = 1 ML./MIN.; AND RESIDENCE TIME FOR FLUID IN HEAT EXCHANGER AS NOTED. HEAT EXCHANGER SURFACE = ALUMINUM.

TEST FLUID (CONC. IN WT.%)	AIR RATE, L./HR.	RESIDENCE TIME FOR FLUID IN HEAT EXCHANGER, MIN.	DEPOSIT ON TUBE, MG.	INSOLUBLES IN EXIT OIL, MG./100 GM.	WATER IN EXIT OIL, MG./100 GM.	NEUT. NO. INCREASE (MG. KOH/GM. OIL)
DI-2-ETHYLHEXYL SEBACATE (MLO 7038) + 0.5 PHENOTHIAZINE	1 (1) 10	8 8	253 1320	142 63	0 720	19.3 23.6
HERCÖFLEX 600 (MLO 7064) + 0.5 PHENOTHIAZINE	1 (1) 10 30 (2)	8 8 5	33.2 16.3 203	4 60 240	0 - 1400	10.2 13.1 23.9
INDOPOL L-50 (MLO 7102) (POLYOLEFIN)	1 (1) 10	8 8	12.8 47.7 51.5	0 105 65	0 1400 500	0 1.0 0.8
NECTON 60 (PRL 3467) (NAPHTHENIC MINERAL OIL)	1 (1) 10 30	8 8 5	7.9 79.8 61.7 1040	0 79 176 163	0 835 840 1770	0.2 1.4 1.3 1.5
KENDALL LIGHT RESIN (MLO 7072) (PARAFFINIC MINERAL OIL)	10 30	8 5	15.4 9.7	68 177	590 2280	1.9 3.0
ESSO AVIATION BRIGHT STOCK (MLO 7094) (PARAFFINIC MINERAL OIL)	10 30	8 5	15.6 17.3	177 376	2180 3580	2.0 6.1
KENDALL HEAVY NEUTRAL (MLO 7120) (PARAFFINIC MINERAL OIL)	10 30	8 5	12.5 100	66 1190	1670 3310	1.3 3.1

(1) GAS USED IN THIS TEST COMPRISED A 1:9 (BY VOL.) MIXTURE OF AIR AND NITROGEN. THAT IS, TOTAL GAS RATE WAS 10 LITERS PER HOUR.

(2) EXIT LINE PLUGGED. TEST TERMINATED AFTER 1.5 HOURS TEST TIME.

There is a larger differential among viscosity grades of the paraffinics studied. The higher viscosity-low volatility paraffinic stocks again appear to be the best of the fluids tested on deposit formation on hot metal surfaces.

K. IMPROVED THERMAL STABILITY TEST. Thermal stability and the corrosiveness of fluids under thermal stability conditions have been determined in several test procedures. An attempt has been made in this study to incorporate as many desirable features as possible into one test procedure. The conditions chosen for a single thermal stability and corrosion test are a stainless steel pressure cylinder of 46 milliliters capacity operated at  $700 \pm 10^\circ\text{F.}$  for six hours and including a one-half inch diameter bearing each of 52-100 bearing steel, M-10 tool steel, and Naval bronze. The test is conducted under a nitrogen atmosphere. The pressure in the cylinder is measured periodically for the six-hour test period. If a pressure of 300 p.s.i. gage is obtained, the test is terminated and the time recorded. The pressure after the test period is recorded at the test temperature ( $700^\circ\text{F.}$ ) and again at room temperature ( $70^\circ$  to  $80^\circ\text{F.}$ ). This latter value is indicative of the volatility of the thermal decomposition products. Assuming a decomposition to form one highly volatile fragment per molecule of test fluid, a pressure of 300 p.s.i. is indicative of 10 to 20 per cent fluid decomposition in the range of fluid molecular weights studied.

Using this test procedure, typical examples of various classes of fluids showing good overall high temperature behavior have been evaluated. These data are summarized on Table 101. These data again point up several pertinent facts concerning high temperature fluid behavior. All of the fluids evaluated form some volatile products on thermal decomposition in a period of six hours or less at  $700^\circ\text{F.}$  In all cases, with the possible exception of the chlorinated aromatic hydrocarbon, the decomposition products are volatile, inflammable materials.

The inflammability and explosive properties of these products will be of about the same order of magnitude as aviation gasoline. These properties of the products are quite different from the initial inflammability of the fluids.

In all cases except for the hydrogenated mineral oils and hydrocarbons, some metal corrosion and sludge formation is noted. It will be necessary to cope with these general properties of fluids at elevated temperatures by mechanical design considerations as well as fluid improvement in constructing hydraulic and lubricating systems for use in the elevated temperature range.

Mineral oils which exhibit good overall stability at high temperatures have been evaluated by this test procedure in somewhat more detail. The results of thermal stability tests conducted on the various paraffinic mineral oil compositions are found on Table 102.

The results of these tests indicate that there are only small differences in thermal stability for these mineral oils. The quantity of

gaseous material formed during the tests varies slightly. No neutralization number increase or insoluble material is obtained.

The viscosity change differs somewhat for the different mineral oils. The viscosity decrease and gas pressure are somewhat larger for the higher molecular weight mineral oils (higher viscosities). This effect has been observed in previous studies. It appears that the higher molecular weight fractions of the same mineral oil type after conventional refining are less stable thermally than the lower molecular weight fractions. With the exception of MLO 7094 (aviation bright stock), the amount of catalyst corrosion obtained is not severe. The M-10 tool steel and the bronze bearing are not affected appreciably and the 52-100 steel ball is corroded mildly by the majority of the test fluids. More severe steel corrosion is noted with MLO 7094 in that both the 52-100 steel and the M-10 steel are corroded.

The effect of hydrogenation on the thermal stability of several mineral oils has also been determined. These thermal stability tests were conducted under the same test conditions described above. In this study the effect of hydrogenation on a naphthenic mineral oil (Necton 50), a Pennsylvania SAE 30 (MLO 7077), a Coastal 500 extract, and a catalytic cycle stock extract have been investigated.

The fluid properties of the mineral oils before and after hydrogenation are summarized on Table 103. These materials were hydrogenated over a U.O.P. nickel catalyst at temperatures of 450° to 600°F. and pressures of 600 to 1800 p.s.i. for periods ranging from 4 to 14 hours. Hydrogenation under these conditions tends to saturate olefins and aromatic linkages and remove sulfur and nitrogen-containing impurities in the oil.

The degree of severity of hydrogenation is indicated by the amount of hydrogen absorbed per 500 grams of oil. It will be noted that the amount of hydrogen absorbed was considerably higher for the aromatic stocks (catalytic cycle stock extract and the Coastal 500 extract) than for the naphthenic (Necton 50) or paraffinic (Pennsylvania SAE 30) mineral oil.

The catalytic cycle stock extract was hydrogenated to two degrees of saturation. In one case a partial hydrogenation was carried to 2.52 moles of hydrogen per 500 grams of fluid. Catalyst poisoning stopped the hydrogenation at this point. The product from a partial hydrogenation was hydrogenated again over a new batch of U.O.P. nickel. The result of the two step hydrogenation was the absorption of 10.5 moles of hydrogen per 500 grams of fluid.

The results of the thermal stability tests with Necton 50 and the Pennsylvania SAE 30 mineral oil before and after hydrogenation and for the hydrogenated catalytic cycle stock extract and the Coastal 500 extract are tabulated on Table 104. The thermal stabilities of the catalytic cycle stock extract and the Coastal 500 extract were not determined. These stocks prior to hydrogenation were semifluid products at room temperature

containing relatively large quantities of non-hydrocarbon impurities.

The results of these tests indicate that there is no significant change in thermal stability for the hydrogenated Necton 50 or the hydrogenated Pennsylvania SAE 30 mineral oil compared to the unhydrogenated materials. In both cases the viscosity decrease, the neutralization number change, and the formation of volatile material are essentially the same before and after hydrogenation. A slight reduction in metal corrosion is noted for the hydrogenated materials although metal corrosion was not severe for the unhydrogenated materials.

The hydrogenated Coastal 500 extract appears to have about the same thermal stability as the Necton 50 and the Pennsylvania SAE 30 mineral oils. The hydrogenated samples of the catalytic cycle stock extract (MLO 7079 and MLO 7087) appear to be somewhat more thermally stable. Both MLO 7079 and MLO 7087 show small viscosity change values, and with MLO 7087 the pressure increase during the test and the residual pressure at room temperature are lower than for the other mineral oils evaluated. Partially or completely hydrogenated high molecular weight aromatic fractions from mineral oil show promise as stable high temperature hydraulic fluids where primary emphasis is placed on thermal and viscosity stability at high temperatures with only secondary emphasis on oxidation stability.

Several thermal stability tests with mineral oils containing dispersant type Acryloid polymers have been conducted at 700°F. in accordance with the improved procedure. These data are discussed in detail in the section on the effect of dispersant additives in mineral oils. The Acryloids, which contain an ester linkage, show relatively poor stability and cause steel corrosion when used as additives in mineral oils. These trends are not evident at temperatures up to 500°F. These data do point up the need for more studies at high temperatures of finished fluid formulations which show promise at temperatures of 500°F. and lower.

1. Thermal Stability of Several Low Molecular Weight Polyolefin Fractions. In report WADC TR 55-30 Pt 3, it is shown that the thermal stability of Indopol L-50 is somewhat poorer than that of a typical mineral oil. Indopol L-50 is a low molecular weight polyolefin commercially available from the Indoil Chemical Company.

Recently a new sample of a polyolefin and a hydrogenated fraction of the same material have been obtained. It has been indicated that these samples are a narrower boiling fraction of the polyolefin than that obtained previously. Thermal stability tests at 600° and 650°F. have been conducted on these new fractions to see whether or not any differences in thermal stability exist between the different samples.

Thermal stability tests at 600° and 650°F. in the glass apparatus and at 600°F. in the pressure cylinder were made. All tests were conducted for the conventional six-hour test period. The fluids evaluated include PRL 3440, the commercially available Indopol L-50; MLO 7119, a hydrogenated sample of PRL 3440; MLO 7123, the narrow boiling polyolefin

obtained recently from the Indoil Chemical Company; and MLO 7124, a hydrogenated sample of MLO 7123. MLO 7119 was hydrogenated at the Petroleum Refining Laboratory whereas MLO 7123 was hydrogenated by the Indoil Chemical Company. The results of the thermal stability tests for these materials are found on Table 105.

The results of these tests indicate that the hydrogenated and unhydrogenated samples of the narrow boiling polyolefin fractions (MLO 7124 and MLO 7123) are no more stable thermally than the sample of Indopol L-50 obtained previously (PRL 3440) and the hydrogenated sample of this material (MLO 7119). The volatility loss and the viscosity decrease are higher for MLO 7123 than for PRL 3440 at both 600° and 650°F. in the glass thermal stability apparatus. Similarly, the viscosity decrease is slightly higher for MLO 7123 than for PRL 3440 in the tests conducted in the pressure cylinder at 600°F. The hydrogenated samples MLO 7119 and MLO 7124 do not differ appreciably in thermal stability at 600°F. Reasonably good agreement was obtained for the viscosity decrease and volatility loss for these materials.

A comparison of the hydrogenated and unhydrogenated fluids indicates that in both cases (PRL 3440 and MLO 7123) hydrogenation improves the thermal stability slightly. For example, in the thermal stability tests at 600°F. in the pressure cylinder, the maximum system pressure is lower in both cases for the hydrogenated sample than for the unhydrogenated samples. In addition, the volatility loss is substantially reduced for MLO 7124 compared to MLO 7123 at both 600° and 650°F. in the glass thermal stability tests.

L. DENSITY DETERMINATIONS OVER THE TEMPERATURE RANGE OF 100° TO 500°F. In an effort to define better the physical properties of fluids and lubricants at high temperatures, the densities of some representative fluids have been determined over the range 100° to 500°F. A Lipkin pycnometer designed for use with volatile liquids has been used for the work. This is the pycnometer specified for use in ASTM Method D941-49 for the determination of the density of hydrocarbon liquids. The Lipkin pycnometer is described in detail in an article by M. R. Lipkin, J. A. Davison, W. T. Harvey, and S. S. Kurtz, Jr. in the Analytical Edition of Industrial and Engineering Chemistry, volume 16, page 55, 1944.

This type of pycnometer has been found to give good precision and reproducibility over the temperature range studied. It is believed that no difficulty would be encountered in extending the determinations to temperatures up to at least 700°F., providing the fluids involved possess adequate thermal stability and a vapor pressure of 300 mm. Hg or less at the desired temperature.

Constant temperature liquid baths have been used for the determinations at 100° and 210°F. A vapor bath of the type used by this Laboratory to determine high temperature viscosities has been employed for the determinations at 350° and 490°F. A description of the vapor bath is included in Annual Report WADC TR 55-30 Pt 3.

Table 101  
THERMAL STABILITY OF VARIOUS FLUIDS AT 700°F.

ALL TESTS CONDUCTED IN THE STAINLESS STEEL PRESSURE CYLINDER.  
TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $700 \pm 10^\circ\text{F}$ .; TEST TIME = 6 HOURS; TEST FLUID CHARGED = 20 ML.; AND CATALYST = A 0.5-INCH DIAMETER BALL BEARING EACH OF 52-100 STEEL, M-10 TOOL STEEL, AND NAVAL BRONZE. PRIOR TO THE START OF THE TEST THE SYSTEM IS PURGED WITH NITROGEN. THE SYSTEM IS THEN SEALED AND THE TEST STARTED.  
TOTAL VOLUME OF THE CYLINDER IS 46 ML. THE NITROGEN PRESSURE IN THE PRESSURE CYLINDER IS ABOUT 20 P.S.I. AT 700°F.

TEST FLUID	% CHANGE IN CENTISTOKE VISCOSITY AT 100°F.	CATALYST WT. LOSS, MG./SQ. CM. M-10 STEEL	52-100 STEEL	NAVAL BRONZE	NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL	FINAL	MAXIMUM SYSTEM PRESSURE, P.S.I.	FINAL PRESSURE AT ROOM TEMP., P.S.I.	INSOLUBLE MATERIAL WT. %
NEOPENTYL TYPE ESTER(1)	-2	0.04	12.00	0.40	0.2	42.7	290	108	0.3
NAPHTHENIC MINERAL OIL AFTER HYDROGENATION	-56	0.00	0.02	0.00	0.1	0.1	159	23	NONE
PARAFFINIC MINERAL OIL AFTER HYDROGENATION	-64	+0.08	+0.06	+0.02	0.1	0.1	101	20	NONE
AROMATIC MINERAL OIL AFTER HYDROGENATION	+5	0.00	0.00	0.00	0.3	0.3	64	10	NONE
CHLORINATED HYDROCARBON	-23	0.10	+0.30	1.60	0.1	0.1	35	4	0.7
SILICATE HYDRAULIC FLUID(1)	-68	0.00	0.00	0.10	0.2	0.7	267	90	TRACE
CHLORINATED SILICONE	-23	0.20	0.30	0.70	0.2	0.9	73	15	0.4

(1) TEST TERMINATED AT LESS THAN 3 HOURS TEST TIME.



Table 102

THERMAL STABILITY OF SEVERAL MINERAL OILS AT 700°F.

ALL TESTS CONDUCTED IN THE STAINLESS STEEL PRESSURE CYLINDER AT 700 ± 10°F.; TEST TIME = 6 HOURS; TEST FLUID CHARGED = 20 ML.; TOTAL VOLUME OF CYLINDER IS 46 ML.; CATALYST = A 0.5 INCH DIAMETER BALL BEARING EACH OF 52 - 100 STEEL, M-10 TOOL STEEL, AND NAVAL BRONZE. PRIOR TO THE START OF THE TEST THE SYSTEM IS PURGED WITH NITROGEN. THE SYSTEM IS THEN SEALED AND THE TEST STARTED. THE NITROGEN PRESSURE IN THE PRESSURE CYLINDER IS ABOUT 20 P.S.I. GAGE AT 700°F.

TEST FLUIDS: MLO 7026 = A PENNSYLVANIA 300 EXTRACTED NEUTRAL (PARAFFINIC MINERAL OIL)  
 MLO 7027 = A PENNSYLVANIA EXTRACTED 180 NEUTRAL (PARAFFINIC MINERAL OIL)  
 MLO 7070 = A PENNSYLVANIA MEDIUM NEUTRAL  
 MLO 7071 = A PENNSYLVANIA HEAVY NEUTRAL  
 MLO 7093 = BAROSA 43 (PARAFFINIC 180 NEUTRAL)  
 MLO 7094 = AVIATION BRIGHT STOCK

TEST FLUID	CENTISTOKE VISCOSITY AT 100°F.		% CHANGE	CATALYST WT. LOSS, MG./SQ. CM.			NEUT. NO. (MG. KOH/GM. OIL) ORIG.	MAX. SYSTEM PRESSURE, P.S.I.	FINAL PRESSURE AT ROOM TEMP., P.S.I.		INSOL. MATERIAL
	ORIG.	FINAL		M-10 STEEL	52-100 STEEL	BRONZE					
MLO 7026	65.2	25.9	-60	0.14	0.26	+0.04	0.1	186	36		TRACE
MLO 7027	27.6	15.5	-44	0.12	0.16	0.02	0.1	125	23		TRACE
MLO 7070	36.3	22.2	-39	+0.10	0.30	0.00	0.1	143	20		TRACE
MLO 7071	107	44.7	-58	0.04	0.18	0.02	0.1	140	22		TRACE
MLO 7093	33.7	20.7	-39	0.04	0.12	+0.04	0.2	126	16		TRACE
MLO 7094	467	65.9	-86	0.82	0.94	0.24	0.1	175	32		TRACE
	467	78.1	-83	0.34	0.74	+0.10	0.1	168	30		TRACE



Contrails

Table 103

PROPERTIES OF SEVERAL FLUIDS BEFORE AND AFTER HYDROGENATION

OILS WERE HYDROGENATED OVER U.O.P NICKEL CATALYST AT TEMPERATURES OF 450° TO 600°F. AND PRESSURES OF 600 TO 1800 P.S.I. FOR PERIODS RANGING FROM 4 TO 14 HOURS.

FLUID DES.	TEST FLUID	MOLS H <sub>2</sub> PER 500 GMS. OIL	CENTISTOKE VISCOSITY 100°F.	CENTISTOKE VISCOSITY 210°F.	A.S.T.M. SLOPE 100° TO 210°F.	VISCOSITY INDEX	POUR POINT °F.
PRL 2052	NECTON 50 (NAPHTHENIC MINERAL OIL)	-	65.2	7.19	0.787	68	-15
MLO 7066	PRL 2052 AFTER HYDROGENATION	0.45	64.3	7.15	0.786	69	-15
MLO 7077	PARAFFINIC MINERAL OIL (SAE 30 LUBE OIL)	-	113	12.2	0.682	106	+5
MLO 7076	MLO 7077 AFTER HYDROGENATION	1.13	97.2	11.2	0.683	108	+35
MLO 7083	CATALYTIC CYCLE STOCK EXTRACT	-	178	7.54	0.998	-206	+25
MLO 7079	MLO 7089 AFTER HYDROGENATION	2.52	89.9	5.78	0.981	-162	+15
MLO 7087	MLO 7089 AFTER HYDROGENATION	10.52	44.0	4.51	0.932	-116	-5
MLO 7092	COASTAL 500 LUBE OIL EXTRACT	-	578	15.6	0.910	-124	+30
MLO 7091	MLO 7092 AFTER HYDROGENATION	9.37	151	9.31	0.864	13	-5

Table 104

## THERMAL STABILITY OF SEVERAL MINERAL OILS AT 700°F.

ALL TESTS CONDUCTED IN THE STAINLESS STEEL PRESSURE CYLINDER AT  $700 \pm 10^\circ\text{F}$ .; TEST TIME = 6 HOURS: TEST FLUID CHARGED = 20 ML.; TOTAL VOLUME OF CYLINDER IS 46 ML.; CATALYST = A 0.5 INCH DIAMETER BALL BEARING EACH OF 52-100 STEEL, M-10 TOOL STEEL, AND NAVAL BRONZE. PRIOR TO THE START OF THE TEST THE SYSTEM IS PURGED WITH NITROGEN. THE SYSTEM IS THEN SEALED AND THE TEST STARTED. THE NITROGEN PRESSURE IN THE PRESSURE CYLINDER IS ABOUT 20 P.S.I. GAGE AT 700°F.

TEST FLUIDS: PRL 2052 = NECTON 50 (NAPHTHENIC MINERAL OIL)

MLO 7096 = HYDROGENATED NECTON 50

MLO 7077 = A PENNSYLVANIA SAE 30 MINERAL OIL

MLO 7076 = HYDROGENATED MLO 7077

MLO 7079 = A HYDROGENATED CATALYTIC CYCLE STOCK EXTRACT

MLO 7087 = A HYDROGENATED CATALYTIC CYCLE STOCK EXTRACT

MLO 7091 = A HYDROGENATED COASTAL 500 EXTRACT

TEST FLUID	CENTISTOKE VISCOSITY AT 100°F.		CATALYST WT. LOSS MG./SQ. CM.		NEUT. NO. (MG. KOH/GR. OIL)		MAX. SYSTEM PRESSURE, P.S.I.	FINAL PRESSURE AT ROOM TEMP, P.S.I.	INSOL. MATERIAL
	ORIG.	FINAL	M-10 STEEL	52-100 STEEL	ORIG.	FINAL			
PRL 2052	65.2	34.4	-	-	0.1	0.2	132	23	NONE
PRL 2052	65.2	25.6	0.10	0.14	0.1	0.2	142	26	TRACE
MLO 7096	63.2	28.1	0.00	0.00	0.2	0.1	159	23	NONE
MLO 7077	113	51.9	0.00	0.04	0.1	0.1	118	27	NONE
MLO 7076	97.2	35.5	+0.08	+0.06	0.1	0.1	101	20	NONE
MLO 7079	89.9	83.6	0.04	0.08	0.3	0.2	116	35	NONE
MLO 7087	44.0	45.6	0.00	0.00	0.2	0.2	64	10	NONE
MLO 7091	151.	65.4	0.00	0.02	0.1	0.1	138	18	NONE

Table 105

## THERMAL STABILITY OF SEVERAL POLYOLEFINS

TESTS CONDUCTED IN GLASS TEST TUBES CONSIST OF A TWENTY-FIVE GRAM CHARGE OF TEST FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 ML. OF TEST FLUID. THE SEAL ALLOWS THE PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 0.25 LB./SQ. IN.

TESTS CONDUCTED IN THE STAINLESS STEEL PRESSURE CYLINDER CONSIST OF A 20 ML. CHARGE OF TEST FLUID. THE TOTAL VOLUME OF THE CYLINDER IS 46 MLS. PRIOR TO THE START OF THE TEST, THE SYSTEM IS PURGED WITH NITROGEN. THE SYSTEM IS THEN SEALED AND THE TEST STARTED. IN THE STAINLESS STEEL PRESSURE CYLINDER, THE NITROGEN PRESSURE AT 600°F. IS APPROXIMATELY 20 P.S.I. GAGE.

TEST FLUID	TEST TEMP., °F.	TEST TIME, HRS.	TYPE OF APPARATUS (2)	CENTISTOKE VISCOSITY AT 100°F.		% LOSS IN WT. (1)	NEUT. NO. (MG. KOH/ GM. OIL)		MAX. SYSTEM PRESSURE, P.S.I.	FINAL PRESSURE AT ROOM TEMP., P.S.I.
				ORIGINAL	FINAL		ORIGINAL	FINAL		
PRL 3440	600	6	GLASS	111	97.6	-12	0.0	0.0	-	-
	600	6	P.C.	111	40.6	-63	0.0	0.0	138	8
	650	6	GLASS	111	34.3	-69	0.0	0.0	-	-
MLO 7119	600	6	GLASS	115	60.1	-48	0.0	0.0	-	-
	600	6	P.C.	115	52.6	-54	0.0	0.0	90	19
MLO 7123	600	6	GLASS	116	55.1	-53	0.0	0.0	-	-
(NARROW BOILING FRACTION)	600	6	P.C.	116	22.5	-81	0.0	0.0	115	25
	650	6	GLASS	116	28.4	-76	0.0	0.0	-	-
MLO 7124	600	6	GLASS	116	66.7	-43	0.0	0.0	-	-
	600	6	P.C.	116	62.2	-46	0.0	0.0	68	12
	650	6	GLASS	116	28.8	-75	0.0	0.0	-	-

(1) LOSS IN WEIGHT IS GOOD TO  $\pm 2\%$  OR 0.5 GRAMS.

(2) TEST CONDUCTED IN GLASS TEST TUBES DESIGNATED AS GLASS. TESTS CONDUCTED IN STAINLESS STEEL PRESSURE CYLINDER DESIGNATED AS P.C.

# Contrails

Any dissolved gases present in the fluid have been removed prior to the density determinations. This has been done by shaking the samples at a temperature of approximately 300°F. and a pressure of one millimeter mercury for 30 minutes. The sample is then cooled at the reduced pressure and the density determination is carried out immediately.

The pycnometer is filled by suction and placed in the 100°F. constant temperature bath. As the fluid reaches test temperature, the fluid level is adjusted by forcing any excess liquid out of the hooked end of the pycnometer. Frequent readings are made until a constant level is obtained. The sample is allowed to remain at test temperature for 30 minutes and a final volume reading is taken. The pycnometer is removed from the bath, wiped dry, cooled with a stream of dry air, and carefully weighted on an analytical balance. The pycnometer is then placed in the bath at the next higher temperature and the procedure repeated.

In calculating the density of the fluids, a correction is used to eliminate the effect of the buoyancy of the air. The results obtained, therefore, represent the density of the fluid in a vacuum. When determining the densities at relatively high temperatures, a further correction must be considered. This correction involves the expansion of the pycnometer bulb and capillaries at the test temperature resulting in a true volume larger than the apparent volume. A value of  $1 \times 10^{-5}$  cc./cc./°C. for the cubic coefficient of expansion of Pyrex glass was used in determining a correction factor for the volume at the various test temperatures.

Density determinations have been carried out at 100°, 210°, 350°, and 490°F. for fluids representative of each of the following classes of materials.

1. Hydrocarbons
2. Mineral Oils
3. Dibasic acid esters
4. Pentaerythritol esters
5. Silicate esters
6. Methyl phenyl silicones
7. Chlorine-containing silicones
8. Aroclor 1248
9. Phosphate esters

Values determined for the various fluids are shown on Table 106. All of the materials evaluated produce essentially a straight line function for the plot of density versus temperature. This is pointed out graphically on Figures 6, 7, and 8. On the basis of the data obtained, it appears that an extrapolation of data at low temperatures (e.g. 60° and 100°F.) would be adequate to define values at temperatures up to at least 500°F.

M. VAPOR PRESSURE MEASUREMENTS WITH SILANES. Five silane fluids have been received from the Materials Laboratory, WCRTR-1 of the Wright Air Development Center (WADC). These fluids are designated by the WADC as MLO 54-408(a), MLO 56-138, MLO 56-211, MLO 56-212, and MLO 56-280. It was requested that vapor pressure measurements over the range of 400°

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to 700°F. be conducted with these materials. These studies have been conducted in a vacuum equilibrium still which is described in Annual Report WADC TR 55-30 Pt 4.

Briefly, the apparatus consists of a stainless steel still provided with a Herculite glass window to allow observation of the fluid and stirrer under operating conditions. Provisions are made to allow the determination of vapor-liquid equilibrium data at various conditions of liquid hold-up for multi-component mixtures.

A heater-stirrer combination is designed to supply good heat transfer between the heater and the boiling liquid with a minimum of super heating or bumping.

A cold finger condenser extends into the still directly over a condensate cup. Condensation on the sides of the still is prevented by insulation and supplemental heating around the walls of the still.

Copper-constantan thermocouples are located in the still and in the air space between the resistance wire and the insulation around the outside of the still. Provision is made to measure pressures over a range of 0.1 to 760 mm. Hg.

The apparatus has been used successfully to determine vapor pressures over the range of 0.2 to 760 mm. Hg at temperatures up to 700°F. Tests are conducted by obtaining first the boiling point of the test fluid at an absolute pressure of 0.2 to 0.3 mm. Hg. The system is allowed to come to equilibrium. The pressure is then increased stepwise and an equilibrium established at each successive pressure increase. This procedure is repeated until a temperature of about 700°F. or a vapor pressure of 760 mm. is attained, or excessive thermal decomposition is attained.

Vapor pressure-temperature data for the silanes are tabulated on Table 107 and plotted graphically on Figures 9 and 10. These plots are based on the relationship  $\log P$  versus  $1/(t^{\circ}\text{C.} + 230)$  used by many investigators<sup>(1)</sup> to provide essentially a straight line relationship between vapor pressure and temperature.

The viscosity properties of the five fluids have been determined before and after the vapor pressure measurements. The values are shown on Table 108. Three of the fluids show a substantial viscosity decrease; one shows a substantial increase. Volatility loss or thermal deterioration may explain the viscosity increase. The viscosity decreases noted are believed to be attributable to thermal deterioration of the fluid in the equilibrium still.

The residence time in the still pot at various temperatures is shown on Table 109. It will be noted that liquid temperatures shown on Table 109 exceed the equilibrium temperatures indicated on Table 107. Temperatures shown on Table 107 are the highest temperatures at which a

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(1) Dreisbach and Spencer, Ind. Eng. Chem. 41, 176 (1949).

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vapor-liquid equilibrium at a given pressure could be obtained; those shown on Table 109 are the highest temperatures attained by the liquid in the still pot. That is, the inability to obtain a vapor-liquid equilibrium at the next higher pressure increment is indicative of a substantial thermal deterioration of the fluid. Those three fluids (MLO 54-408(c), MLO 56-280, and MLO 56-212) which were heated to the highest liquid temperature and for the longest total time are the three fluids incurring a substantial viscosity decrease during the vapor pressure-temperature determination.

It is apparent from these data that the silanes studied are not thermally stable in the temperature range of 650° to 760°F.

N. FOAMING CHARACTERISTICS OF HIGH TEMPERATURE LUBRICANTS.

Foaming has been a continuing problem in hydraulic and lubricant systems. Techniques for the evaluation of a fluid's foaming characteristics and the study of foaming with various classes of fluids and lubricants have been discussed in a series of technical reports from this Laboratory. These references are listed below:

- (1) Foaming Characteristics of Hydraulic Fluids and Special Lubricants, Report PRL 3.48-Dec49, dated December 7, 1949, under Contract Nord 7958(B).
- (2) Formal Report No. 7 on Hydraulic and Recoil Oils, dated April 8, 1943, to Section 11.2 of the National Defense Research Committee (pages 163-187).
- (3) Formal Report No. 8 on Hydraulic and Recoil Oils, dated September 24, 1943 to Section 11.2 of the National Defense Research Committee (pages 26-32).
- (4) Formal Report No. 9 on Hydraulic and Recoil Oils, dated April 11, 1944 to Section 11.2 of the National Defense Research Committee (pages 53-68).
- (5) Formal Report No. 10 on Hydraulic and Recoil Oils, dated December 12, 1944 to Section 11.2 of the National Defense Research Committee (pages 148-175).
- (6) Final Report WADC TR 55-30 Pt 1 on Fluids, Lubricants, Fuels, and Related Materials, dated November 22, 1952 under Contract AF33(038)18193 (pages 43-45).

Three types of tests have been used in these previous studies to evaluate foaming. The Viking pump test comprises a simple fluid system including a glass reservoir, a Viking gear pump, and a heat exchanger. The pressure can be reduced on the reservoir to duplicate high altitude conditions. Foaming is measured by the degree of bubble formation in the oil which is in turn measured by the reduction of light transmission through the reservoir due to the foaming. The critical measurement is the time required for the foam to disappear following a pressure reduction on the reservoir.

Table 106

## DENSITY OF VARIOUS FLUIDS OVER TEMPERATURE RANGE OF 100° TO 500°F.

DENSITY VALUES DETERMINED IN A PYCNOMETER OF THE TYPE SPECIFIED IN ASTM METHOD D941-49.  
VOLUME CORRECTED FOR GLASS EXPANSION AT TEST TEMPERATURE.

	TEST FLUID	DENSITY, GM./ML. ( $\pm 0.0003$ ) AT		
		100°F.	210°F.	490°F.
MLO 7028	D1-2-ETHYLHEXYL SEBACATE	0.9015	0.8575	0.7433
PRL 3542	HERCOFLEX 600 (PENTAERYTHRITOL ESTER)	0.9825	0.9352	0.8141
PRL 3455	TETRA-2-ETHYLHEXYL SILICATE	0.8682	0.8239	0.7068
MLO 7048	PHENYL-o-CHLOROPHENYL PHOSPHATE (1:2)	1.3372	1.2842	1.1493
PRL 3437	ESSTIC 45 (NAPHTHENIC MINERAL OIL)	0.8766	0.8385	0.7385
MLO 7026	PENNSYLVANIA 300 NEUTRAL MINERAL OIL	0.8574	0.8194	0.7211
MLO 7014	INDOPOL L-50 (POLYBUTENE)	0.8367	0.7990	0.6988
PRL 3554	AROCLOR 1248	1.4328	1.3728	1.2167
PRL 3497	METHYLPHENYL SILICONE (DOW)	0.9778	0.9268	0.8033
MLO 7016	IMPROVED LUBRICITY SILICONE 814D6 (G.E.)	1.0100	0.9567	0.8274



Figure 6

# DENSITY-TEMPERATURE RELATIONSHIP

DENSITY VALUES DETERMINED IN PYCNOMETER OF THE TYPE SPECIFIED IN ASTM METHOD D941-49. VOLUME CORRECTED FOR GLASS EXPANSION AT TEST TEMPERATURE.

TEST FLUIDS:  $\Delta$  = DI-2-ETHYLHEXYL SEBACATE (MLO 7038)

$\circ$  = ESSTIC 45, A HIGHLY REFINED NAPHTHENIC MINERAL OIL (PRL 3457)

$\square$  = TETRA-2-ETHYLHEXYL SILICATE (PRL 3455)

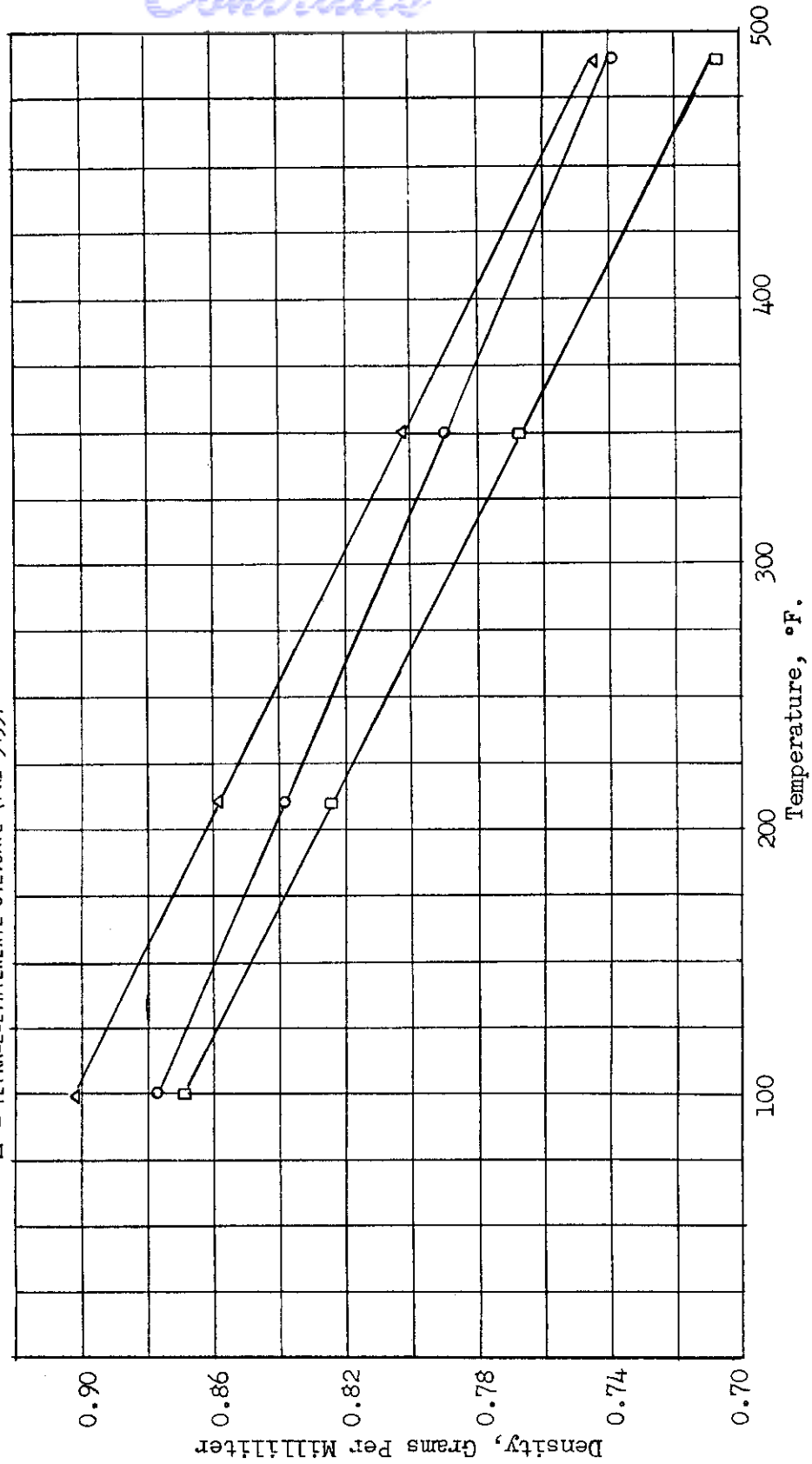


Figure 7

DENSITY-TEMPERATURE RELATIONSHIP

DENSITY VALUES DETERMINED IN A PYCNOMETER OF THE TYPE SPECIFIED IN ASTM METHOD D941-49. VOLUME CORRECTED FOR GLASS EXPANSION AT TEST TEMPERATURE.

TEST FLUIDS:  $\Delta$  = IMPROVED LUBRICITY SILICONE FLUID 81406 (MLO 7016)  
 $\circ$  = HERCOFLEX 600 (PRL 3542)

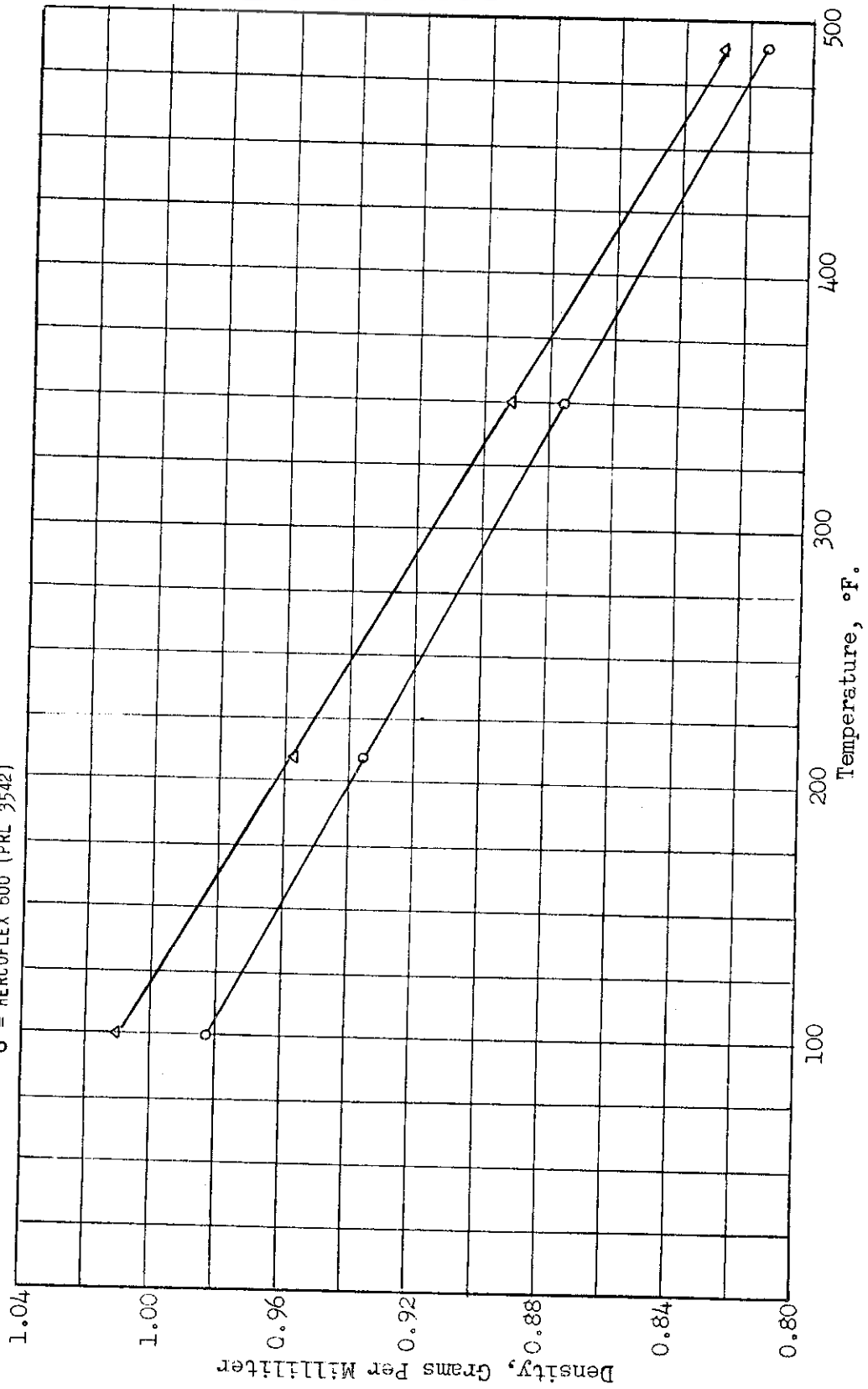


Figure 8  
DENSITY-TEMPERATURE RELATIONSHIP  
DENSITY VALUES DETERMINED IN PYCNOMETER OF THE TYPE SPECIFIED IN ASTM METHOD D941-49.  
VOLUME CORRECTED FOR GLASS EXPANSION AT TEST TEMPERATURE.

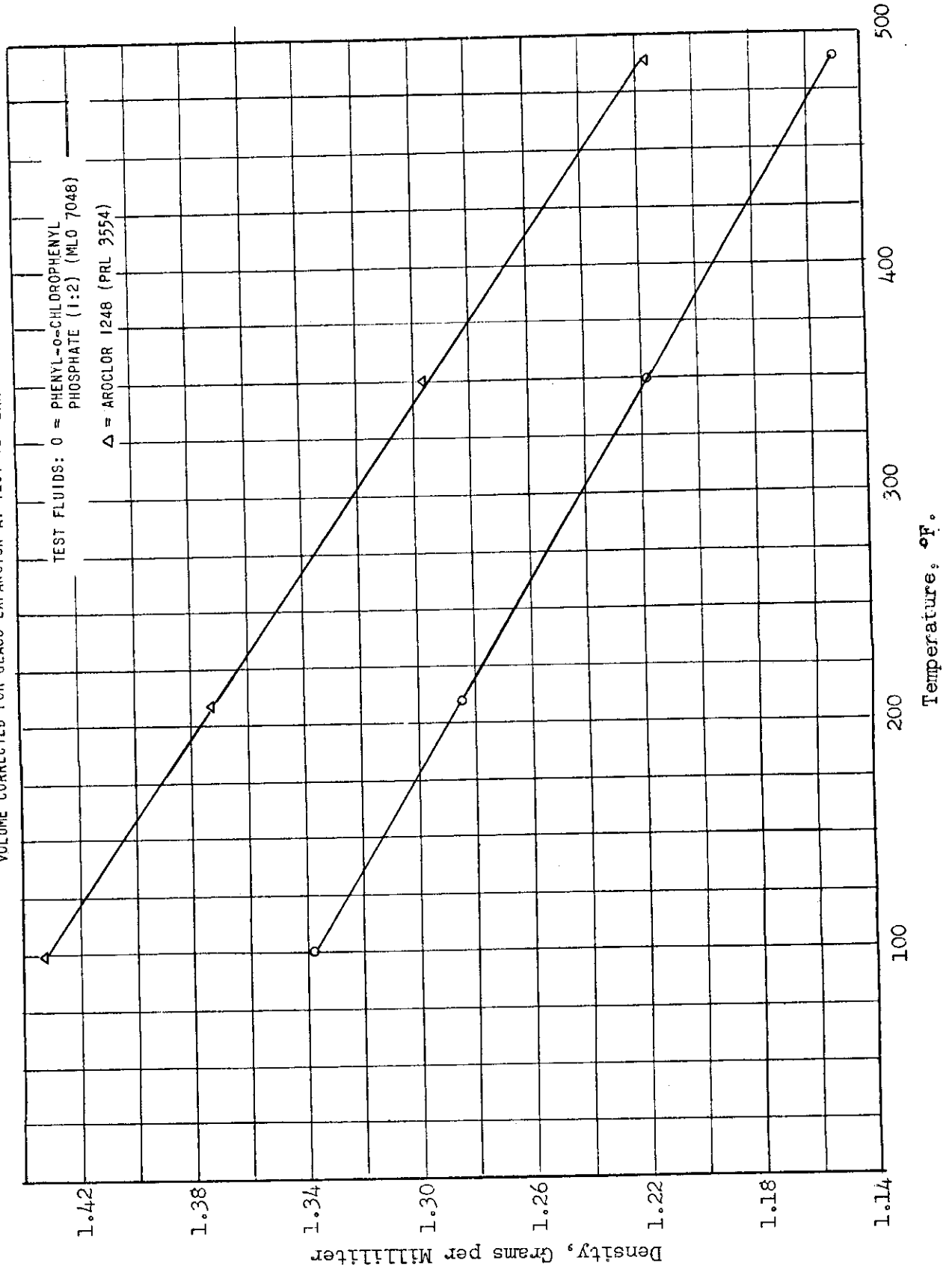
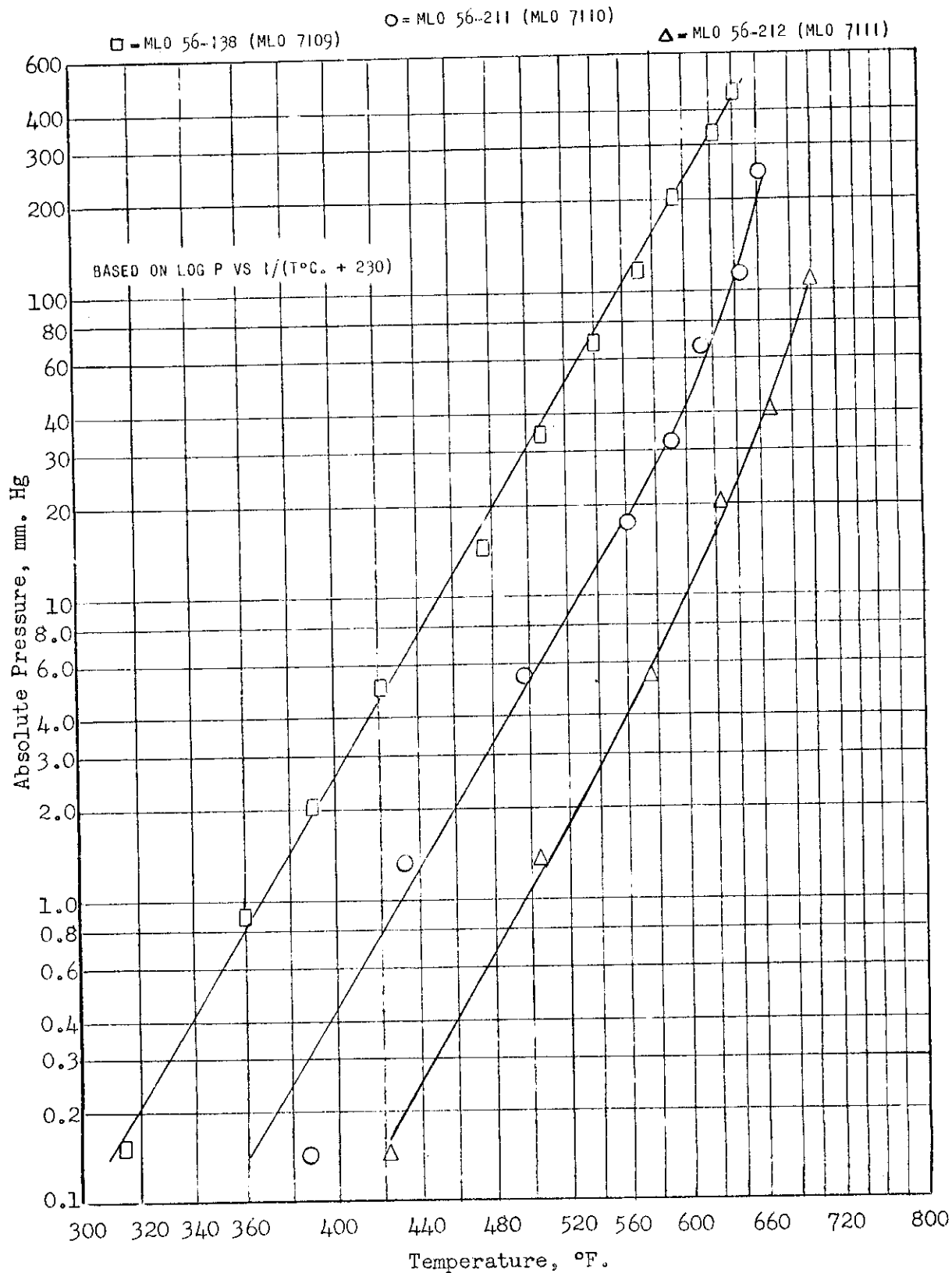


Table 107

VAPOR PRESSURE - TEMPERATURE RELATIONSHIP FOR SOME SILANES  
 VAPOR PRESSURE MEASUREMENTS MADE USING VACUUM EQUILIBRIUM STILL DESCRIBED IN REPORT (WADC TR 55-30 Pt 4).  
 VAPOR PRESSURE RUNS WERE TERMINATED WHEN THERMAL DETERIORATION BECAME APPARENT.

MLO 54-408(C) (MLO 7107)		MLO 56-280 (MLO 7108)		MLO 56-138 (MLO 7109)		MLO 56-211 (MLO 7110)		MLO 56-212 (MLO 7111)	
ABSOLUTE PRESSURE, MM. HG	REFLUX TEMP., °F.	ABSOLUTE PRESSURE, MM. HG	REFLUX TEMP., °F.	ABSOLUTE PRESSURE MM. HG	REFLUX TEMP., °F.	ABSOLUTE PRESSURE MM. HG	REFLUX TEMP., °F.	ABSOLUTE PRESSURE MM. HG	REFLUX TEMP., °F.
0.07	437	0.17	417	0.15	315	0.14	388	0.14	423
0.97	565	1.20	481	0.88	361	1.30	449	1.35	504
7.0	629	5.1	518	2.00	388	5.2	494	5.5	577
		14.4	551	5.0	424	17.0	561	20	626
		39.4	592	14.0	470	32	588	40	663
		80	631	32	509	65	611	104	699
				67	540	115	640		
				115	568	248	660		
				200	595				
				327	623				
				450	642				

# VAPOR PRESSURE-TEMPERATURE RELATIONSHIP FOR SEVERAL SILANES



VAPOR PRESSURE-TEMPERATURE RELATIONSHIP FOR SEVERAL SILANES

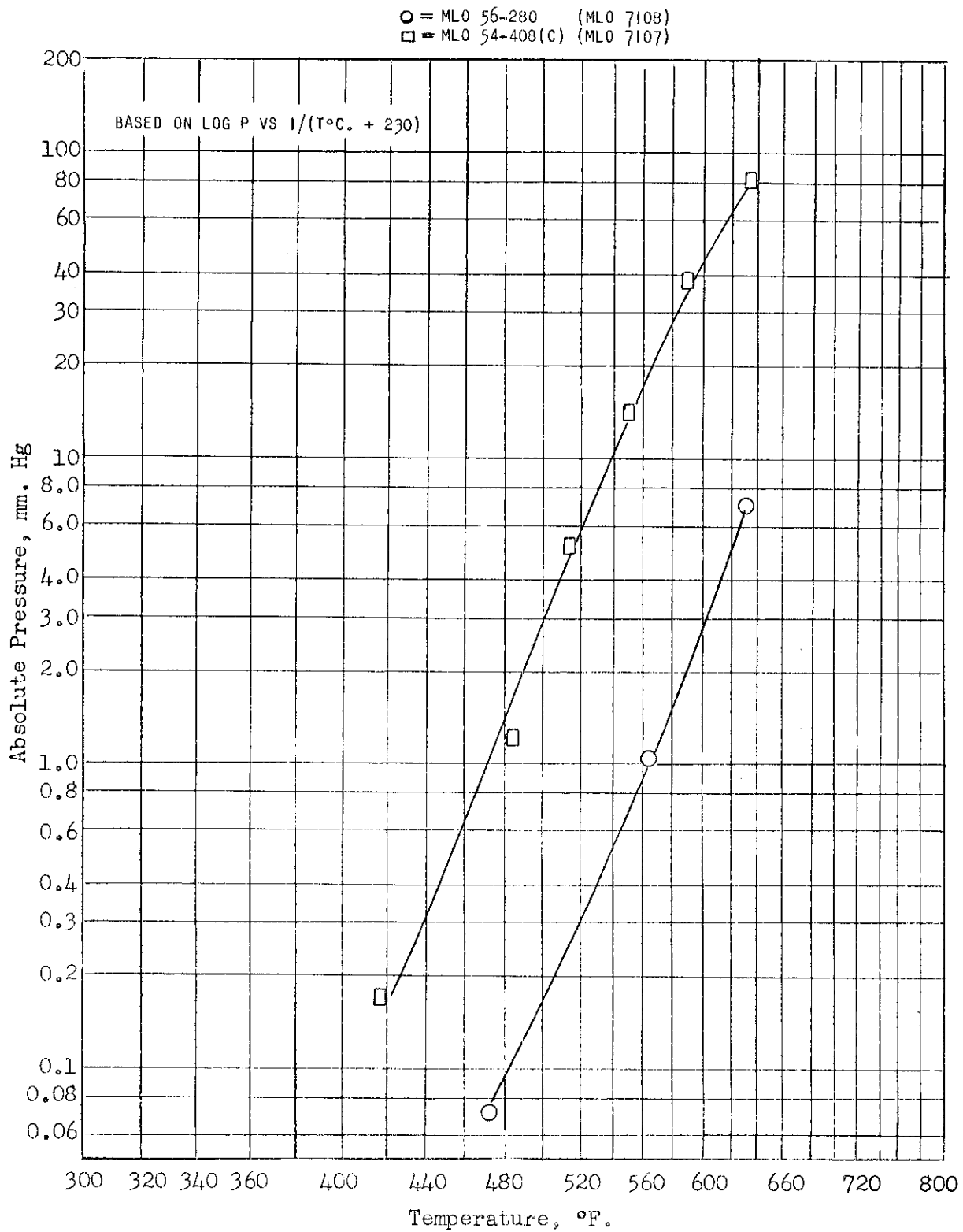


Table 108

VISCOSITY OF SEVERAL SILANE FLUIDS BEFORE AND AFTER  
VAPOR PRESSURE MEASUREMENTS

Fluid	- - - Centistoke Viscosity at 100°F. - - -		
	Original	Final	% Change
MLO 54-408(c)(MLO 7107	29.6	23.9	-19
MLO 56-280 (MLO 7108)	37.0	31.6	-15
MLO 56-138 (MLO 7109)	6.8	7.7	+13
MLO 56-211 (MLO 7110)	11.7	11.8	+ 1
MLO 56-212 (MLO 7111)	18.6	17.0	- 9



Table 109

THERMAL HISTORY OF SEVERAL SILANES DURING  
VAPOR PRESSURE DETERMINATIONS

Liquid Temp., (1) °F.	Heating Time, Hours (2)				
	MLO 56-138 (MLO 7109)	MLO 56-211 (MLO 7110)	MLO 54-408(c) (MLO 7107)	MLO 56-280 (MLO 7108)	MLO 56-212 (MLO 7111)
200	3.7	5.7	6.9	6.9	6.9
300	3.2	5.2	6.4	6.4	6.4
400	2.6	4.3	5.5	5.5	5.5
500	1.7	3.1	4.1	4.1	4.1
600	0.8	1.8	2.5	2.5	2.5
700	-	0.4	0.8	0.8	0.8
750	-	-	0.1	0.1	0.1

(1) Liquid temperature was measured by thermocouple in the still pot.

(2) Heating time represents total hours liquid was at or above indicated temperatures.

# Contrails

The bomb froth and foam test consists of shaking 50 milliliters of test fluid under 1000 p.s.i. nitrogen pressure in a 500 milliliters capacity steel container fitted with a needle valve. After 30 minutes shaking, the bomb is inverted and the contents drained into a graduate at atmospheric pressure. The rate of disappearance of foam and the appearance of liquid are noted. This test is designed to simulate the effects created in a hydraulic system of sudden pressure release on a fluid from high pressure to atmospheric.

The aeration apparatus consists of a fitted glass filter (Ace Glass Company, 30 millimeters, E porosity) to which a 380 millimeters long, 38 millimeters i.d. glass reservoir is attached. The fluid to be tested is placed inside the tube on top of the filter plate, and dry air is passed through the filter from the bottom upward at a definite rate. The finely dispersed air causes the fluid to foam or increase in volume. The resultant increase in volume or head of foam is a function of the original height of fluid, the air rate, the filter porosity, the diameter of the foaming tube, and the tendency of the fluid to foam. By holding the other variables constant, the foaming tendencies of various fluids and anti-foam additives may be evaluated and compared at definite air rates. This is a dynamic test where an attempt is made to determine foaming characteristics in a state of dynamic equilibrium.

This test is designed to simulate best the effects of foaming caused by entrained air in an oil cycle as a result of spray lubrication, over scavenging in dry sump operation, and other air leakage or air entraining mechanisms in a dynamic cycle.

There is some indication that the same classes of additives and fluid components show about the same relative order of effectiveness in these three foaming tests. Foaming studies in the past have shown that pure compounds generally do not foam. In the case of diester-base hydraulic fluids (Spec. MIL-L-6387) and lubricants (Spec. MIL-L-7808), foaming, as indicated by these foaming tests, has not been a problem because of the simplicity of the composition of these fluids.

The mineral oils and synthetic hydrocarbons which have been studied as high temperature hydraulic fluids and lubricants exhibit poor foaming characteristics. In general, foaming in the more viscous mineral oils and hydrocarbons evaluated as high temperature fluids and lubricants can be more readily controlled than for hydraulic fluids and lubricants of the low viscosity type, e.g. Spec MIL-O-5606 and Grades 1010 and 1005 jet engine oils. Furthermore, foaming generally decreases with increasing oil temperature. Thus, the overall problem of foaming in high temperature applications of mineral oils appears to be similar to the problems studied in the preceding references.

A series of foaming tests have been conducted in the aeration tube to determine the effect of a high molecular weight methyl silicone on various classes of mineral oils and synthetic hydrocarbons. These fluids are of the same basic classes as those prepared by this Laboratory for use by the Power Plant Laboratory of WADC in mock-up and engine testing.

*Control*

The anti-foam additive used is a Dow Corning methyl silicone of 50,000 centistokes viscosity at 100°F. This silicone was dissolved in benzene to prepare a concentrate of 10 weight per cent silicone in benzene. The benzene concentrate was then added drop-wise to the mineral oil blend with vigorous agitation. The use of a benzene or other non-viscous silicone concentrate has been found to be essential in obtaining an effective dispersion of anti-foam additive in the final mineral oil blend. Previous studies have shown that high molecular weight silicones are more effective than lower molecular weight varieties in controlling foaming, particularly in mineral oils and hydrocarbons in the moderate viscosity range. The silicone used in this study is completely miscible with benzene. The 10 per cent concentration was chosen because of low viscosity and ease of dispersion in a well agitated oil.

Limits of solubility of this silicone in typical paraffinic and naphthenic neutrals are shown on Table 110. The solubility limits in both types of oil fall between 0.001 and 0.005 weight per cent of silicone in the blend. Solubility in this case is based on visual observation of the blend under strong illumination at room temperature (70° to 80°F.)

The limiting effective concentration of this silicone was also studied in the same two base oils. The results of these studies are shown on Table 111. It can be seen that both oils foam badly with no additive. Both oils respond to silicone anti-foam additives at concentrations as low as 0.00001 weight per cent. Maximum effectiveness in this foaming test is achieved with additive concentrations of 0.00005 per cent or greater. Concentrations of the order of 0.00005 to 0.001 per cent result in effective anti-foam action without adverse solubility problems at room temperature and above.

Foaming characteristics and foam stability for various mineral oils, synthetic hydrocarbons, and blends are shown on Tables 112 and 113. Barosa 43 and the Kendall heavy neutral are well refined paraffinic neutrals. Necton 42 is a highly refined naphthenic oil. The Indopol L-50 contains one double bond per molecule while the hydrogenated variety is completely saturated. All of the fluids tested without anti-foam additives show foaming tendencies and foam stability characteristics of mineral oils. In all cases, 0.001 weight per cent of the silicone anti-foam additive completely eliminates foaming at all air rates in the aeration tube procedure.

These data indicate that foaming can be eliminated in mineral oils and synthetic hydrocarbons of the type considered for use as high temperature fluids and lubricants. The persistence of the anti-foam characteristics with fluid use in high temperature applications has not been extensively studied. As more realistic limits for high temperature fluid use evolve, these conditions should be studied to determine the persistence of anti-foam properties.

Anti-foam properties at high temperatures are necessary to reduce air-oil contact area and contact time which influence oxidation rate and

Table 110  
SOLUBILITY STUDIES

Anti-Foam Additive: Dow Corning Type 200 Silicone of 50,000  
Centistokes at 100°F. (PRL 2095)

Procedure: The Anti-Foam Additive is added to the Mineral Oil as  
a 10 weight per cent solution of Silicone in Benzene.  
The oil is agitated vigorously during blending.

Base Oil	Additive Conc., Wt.%	Appearance
PRL 2053 Necton 42 (Naphthenic Mineral Oil)	0.001	Clear
	0.0025	Slightly Turbid
	0.005	Turbid
MLO 7101 Hydrogenated Indopol L-50 (Saturated Polyolefin)	0.001	Clear
	0.005	Turbid

Table 111

## EFFECT OF ANTI-FOAM ADDITIVE CONCENTRATION IN MINERAL OILS

ALL TESTS HAVE BEEN CONDUCTED IN THE AERATION TUBE AT ROOM TEMPERATURE (70° - 80°F.) USING DRIED AND FILTERED COMPRESSED AIR. THE HEIGHT OF FOAM AT EACH AIR RATE HAS BEEN MEASURED WHEN THE FOAM AND LIQUID LEVEL REACHED EQUILIBRIUM OR AFTER FIVE MINUTES, IF EQUILIBRIUM HAS NOT BEEN REACHED AFTER THIS TIME INTERVAL.

AERATION TUBE: A FINE POROSITY FRITTED GLASS FILTER (ACE GLASS, E POROSITY), 30 MM. IN DIAMETER WHICH HAS BEEN FITTED WITH A 380 MM. GRADUATED EXTENSION 38 MM. IN DIAMETER. TEST OIL IS PLACED TO A HEIGHT OF 50 MM. ON TOP OF THE FILTER PLATE.

ANTI-FOAM ADDITIVE: DOW CORNING TYPE 200 SILICONE OF 50,000 CENTISTOKES AT 100°F. (PRL 2095)

TEST FLUID	ADDITIVE CONC., WT.%	% INCREASE IN VOLUME (FLUID + FOAM) FOR INDICATED AIR RATE, LITERS/HR.			
		2	7	15	26
BAROSA 43 (PARAFFINIC 180 NEUTRAL)	NONE	140	580	>660	>660
	0.00001	46	110	100	80
	0.00005	16	20	20	16
	0.0001	14	16	16	14
	0.001	<10	<10	<10	<10
NECTON 42 (NAPHTHENIC MINERAL OIL)	NONE	68	320	300	244
	0.00002	20	26	24	20
	0.00005	<10	10	10	10
	0.001	<10	<10	<10	<10

## FOAMING CHARACTERISTICS OF SEVERAL MINERAL OILS

ALL TESTS HAVE BEEN CONDUCTED IN THE AERATION TUBE AT ROOM TEMPERATURE (70°-80°F.) USING DRIED AND FILTERED COMPRESSED AIR. THE HEIGHT OF FOAM AT EACH AIR RATE HAS BEEN MEASURED WHEN THE FOAM AND LIQUID LEVEL REACHED EQUILIBRIUM OR AFTER FIVE MINUTES, IF EQUILIBRIUM HAS NOT BEEN REACHED AFTER THIS TIME INTERVAL.

AERATION TUBE: A FINE POROSITY FRITTED GLASS FILTER (ACE GLASS, E POROSITY), 30 MM. IN DIAMETER WHICH HAS BEEN FITTED WITH A 380 MM. GRADUATED EXTENSION 38 MM. IN DIAMETER. TEST OIL IS PLACED TO A HEIGHT OF 50 MM. ON TOP OF FILTER PLATE.

ANTI-FOAM ADDITIVE: DOW CORNING TYPE 200 SILICONE OF 50,000 CENTISTOKES AT 100°F.  
(PRL 2095)

TEST FLUID	ADDITIVE CONC., WT.%	C'STOKE VISCOSITY AT 100°F.	% INCREASE IN VOLUME (FLUID + FOAM) FOR INDICATED AIR RATE, LITERS/HR.			
			2	7	15	26
MLO 7102 INDOPOL L-50 (POLYOLEFIN)	NONE 0.001	116	320 <10	>660 <10	460 <10	500 <10
MLO 7093 BAROSA 43 (PARAFFINIC 180 NEUTRAL)	NONE 0.001	33	150 <10	580 <10	>660 <10	>660 <10
MLO 7071 KENDALL HEAVY NEUTRAL	NONE 0.001	98	260 <10	300 <10	300 <10	300 <10
- 50:50 (BY WT.) INDOPOL L-50 BAROSA 43	NONE 0.001	56	280 <10	520 <10	430 <10	440 <10
- 50:50 (BY WT.) INDOPOL L-50 KENDALL HEAVY NEUTRAL	NONE 0.001	106	260 <10	296 <10	180 <10	200 <10
MLO 7101 HYDROGENATED INDOPOL L-50	NONE 0.001	112	340 <10	640 <10	640 <10	660 <10
PRL 2053 NECTON 42 (NAPHTHENIC MINERAL OIL)	NONE 0.001	35	68 <10	320 <10	300 <10	244 <10

# FOAM STABILITY OF SOME MINERAL OILS

ALL TESTS HAVE BEEN CONDUCTED AT ROOM TEMPERATURE (70° - 80°F.). FILTERED AND DRIED COMPRESSED AIR IS PASSED THROUGH THE FILTER PLATE AND OIL SAMPLE AT A RATE OF 7 LITERS PER HOUR. WHEN EQUILIBRIUM IS REACHED BY THE FOAM AND LIQUID COLUMN, OR AFTER 5 MINUTES IF EQUILIBRIUM HAS NOT BEEN REACHED AFTER THIS TIME INTERVAL, THE HEIGHT OF THE FOAM AND LIQUID COLUMN IS NOTED AND THE AIR SUPPLY IS SHUT OFF AND THE LINES TO THE TUBE VENTED. THE HEIGHT OF THE FOAM AND LIQUID ARE THEN READ AT 15 SECOND INTERVALS UNTIL ALL OF THE FOAM HAS DISAPPEARED.

AERATION TUBE: A FINE POROSITY, FRITTED GLASS FILTER (ACE GLASS, E POROSITY) 30 MM. IN DIAMETER IS FITTED WITH A 380 MM. GRADUATED EXTENSION 38 MM. IN DIAMETER. TEST OIL IS PLACED TO A HEIGHT OF 50 MM. ON TOP OF THE FILTER PLATE.

TEST FLUID	VOLUME OF FOAM PER VOLUME OF LIQUID	TIME IN MIN. FOR DISAPPEARANCE OF INDICATED % OF INITIAL FOAM		AV. RATE OF DISAPPEARANCE OF INDICATED % OF FOAM, ML/MIN. 0-50% 0-90%	
		50%	90%	0-50%	0-90%
MLO 7102 INDOPOL L-50 (POLYOLEFIN)	>9.8	8.5	14.0	22	24
MLO 7093 BAROSA 43 (PARAFFINIC 180 NEUTRAL)	7.0	2.3	4.3	76	73
MLO 7071 KENDALL HEAVY NEUTRAL	4.0	3.0	7.2	33	25
- 50:50 (BY WT.) INDOPOL L-50: BAROSA 43	6.8	3.6	6.0	47	51
- 50:50 (BY WT.) INDOPOL L-50: KENDALL HEAVY NEUTRAL	3.9	3.2	7.0	31	25
MLO 7101 HYDROGENATED INDOPOL L-50	7.4	9.0	16.5	21	20
PRL 2053 NECTON 42 (NAPHTHENIC MINERAL OIL)	4.2	1.0	2.0	105	95

*Contrails*

ultimately fluid life. This factor of foaming becomes critical only at temperatures above which useful fluid life is expected to exceed the stable life or induction period of the fluid.

O. LUBRICITY TESTS AT 700°F. Modification of a Shell four-ball wear tester to allow testing at 500° to 700°F. has been described in Annual Report WADC TR 55-30 Pt 4. Preliminary testing at 700°F. and more extensive tests at 500° and 600°F. are also discussed in that report. Preliminary tests at 700°F. with silicate esters, neopentyl type esters, and Aroclor 1248 have been unsuccessful because of the high decomposition rate and/or volatility. That is, the initial 30 milliliters charge is completely lost before the hour run is completed. To allow for testing volatile and unstable compositions, a hypodermic tube from a graduated burette is sealed into the steel cylindrical tube forming the gas seal. The flow of make-up fluid can be controlled with a glass stopcock at the bottom of the burette. Make-up oil rates have been determined on the basis of fluid loss measured in preliminary tests. The close ball pot-to-spindle clearance precludes the use of a chicken feeder type of fluid level control.

1. Shell Four-Ball Wear Tests. A series of tests at 700°F. has been conducted with mineral oils. These tests were made using M-10 tool steel balls. The results of the high temperature tests are compared with values obtained with the same fluids at 167°F. using 52-100 steel balls on Table 114. Wear data at 700°F. for the non-additive mineral oils appears to be comparable to test results at 167°F. for the one and ten kilogram loadings. At 40 kilograms load, the 700°F. values are substantially higher than the 167°F. values. In both the Kendall heavy neutral and the Necton 60, the 0.5 weight per cent diisopropyl acid phosphite is very effective in reducing wear values at the higher loadings. That is, diisopropyl acid phosphite in mineral oils has been shown conclusively to maintain its effectiveness as a lubricity additive over the entire test range of 167° to 700°F. in the Shell four-ball wear tester. The two Kendall resins show good overall lubricity at 700°F. without additives.

High temperature lubricity values (700°F.) for silicate ester fluids, Aroclor 1248, and chlorinated silicone are compared with values at 600°F. on Table 115. The data with the silicate fluids were obtained at a rate of 45 milliliters per hour of replenishing fluid to the ball pot. A rate of 55 milliliters per hour was used for Aroclor 1248. Even at this high replenishing rate, it was difficult to maintain sufficient fluid level in the pot for running. The improved lubricity silicone was run without fluid replenishing.

The wear data for Aroclor 1248 and the silicate fluids are of about the same order of magnitude at 600° and 700°F. The improved lubricity silicone shows a slight trend toward reduced wear at high loadings at 700°F. compared with 600°F. The use of diisopropyl acid phosphite as an additive in the improved lubricity silicone does not improve the lubricity characteristics. The diisopropyl acid phosphite is quite effective as a lubricity additive for mineral oils at 700°F.



*Continued*

A 50:50 mixture of improved lubricity silicone and mineral oil plus diisopropyl acid phosphite additive has been evaluated at 700°F. These data indicate that the poorer lubricity properties of the silicone again dominate. The 40 kilograms load value is quite high in this case. Values of this magnitude are indicative of seizure with recovery. With the silicates and silicones particularly, the 40 kilograms tests appear to be close to the point of seizure. Therefore, large variations in wear scar are indicative of seizure versus no seizure and not poor reproducibility in wear values.

The high temperature behavior of silicones and silicates in general bears out the trend established in low temperature investigations. That is, these base materials show a poor additive susceptibility. The relatively poor lubricity of silicones and silicates in combination with established lubricity additives and bulk quantities of good lubricants indicates that the silicon-containing materials may participate in the chemical reaction on the bearing surface to form a surface film which resists further chemical attack from the established lubricants under boundary conditions. For example, ferrous alloys of silicon of low melting point and high corrosion resistance are known. The effect of additives and bulk constituents in silicates and silicones will be studied further to determine additional effects of these materials in boundary lubrication.

2. Evaluation of Oronite High Temperature Hydraulic Fluid in the PRL High Temperature Pump Test Unit. A three gallon sample of Oronite High Temperature Hydraulic Fluid 8515 has been received from the Wright Air Development Center for evaluation in the PRL high temperature pump test unit. Pump tests at both 400° and 500°F. have been conducted with this fluid. The results of these tests are summarized on Table 116. Similar data for Silicate Fluid O.S. 45, Silicate Fluid MLO 8200, and for di-2-ethylhexyl sebacate are also included for purposes of comparison. The results of these previous tests, as well as a description of the high temperature pump test unit and the operating procedure, are discussed in detail in Annual Reports WADC TR 55-30 Pt 3 and WADC TR 55-30 Pt 4.

The test with the 8515 fluid at 400°F. was conducted for the conventional four-hour test period at a pressure of 700 p.s.i. Shortly after the fluid reached test temperature, a small amount of colloidal metal was observed in the reservoir and persisted during the test period. During the test, the flow rate decreased from 3.8 to 3.3 gallons per minute. Following completion of the test, the pump was dismantled and the vanes were weighed. The average weight loss was 1.9 milligrams per vane. This is considered to be a moderate amount of wear. In addition, visual observation of the pump parts indicated that a considerable amount of wear had occurred on the cam ring and the valve plate.

The test at 500°F. was also conducted for the conventional test period; however, under these test conditions the system pressure could not be maintained at 700 p.s.i. Colloidal metal was observed again in the reservoir during the course of the test. The flow rate decreased steadily from an initial value of 2.1 g.p.m. at 500°F. to less than 1.0 g.p.m. As a result, the system pressure could not be maintained at 700 p.s.i. and

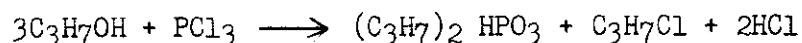
*Continued*

the pressure decreased steadily from 700 p.s.i. to 450 p.s.i. during the four-hour test period. After completion of the test, the vanes were weighed and the pump parts were inspected. The average weight loss per vane, 2.1 milligrams, was only slightly higher; however, both the cam ring and the valve plate were severely worn, and the metal surfaces where the major portion of wear occurred were extremely rough.

A comparison of these results with the two silicate fluids evaluated previously, indicates that there is no appreciable improvement in operation for the Oronite High Temperature Hydraulic Fluid 8515 over Silicate Fluid O.S. 45 or Silicate Fluid MLO 8200. That is, the formation of colloidal metal and a relatively large decrease in flow rate were noted for both O.S. 45 and fluid 8515 at 400°F.; and none of the silicate fluids operated satisfactorily at 500°F. Comparable data for di-ethylhexyl sebacate show good operation at both 400° and 500°F. In both cases some decrease in flow rate was obtained, but satisfactory operation for the four-hour test period at 700 p.s.i. was maintained. In addition, no substantial amount of valve plate or cam ring wear was observed after completion of the tests.

P. PREPARATION OF DIISOPROPYL ACID PHOSPHITE. Diisopropyl acid phosphite has been found to have good overall properties for improving the lubricity of jet engine lubricants. This material gives improved storage stability in ester-base compositions compared with alkyl acid phosphites prepared from primary alcohols. The isopropyl group is small enough to provide an acid phosphite effective as a lubricity additive in small concentrations. Thus far, no commercial source of diisopropyl acid phosphite has been found. The quantities of this additive required for MLO 7010 and GTO 120 fluids formulated for "mock-up" testing have been prepared in the laboratory.

Preparation of diisopropyl acid phosphite is in accordance with the following equation:



The preparation is conducted in a conventional three-neck flask fitted with an air driven stirrer and inlet and outlet tubes for the circulation of dry nitrogen through the flasks. Dry isopropyl alcohol is added to the flasks, the agitator started, a stream of dry nitrogen started, and the flask chilled to 5°C. or lower. Any conventional cooling method such as an ice-salt bath or a dry ice-acetone bath may be used to maintain the temperature of the flask below 10°C. Phosphorus trichloride is added to the flask dropwise through a dropping funnel. The rate of addition is controlled so that the flask temperature does not exceed 10°C. The preparation is conducted with 10 per cent excess of alcohol.

After the addition of the phosphorus trichloride is complete, the reaction mixture is allowed to warm up to room temperature. The mixture is transferred to a vacuum distillation flask. A heating mantle is attached and as much volatile material as possible is removed at about

Table 114

## LUBRICITY CHARACTERISTICS OF SOME MINERAL OILS IN THE SHELL FOUR-BALL WEAR TESTER

TEST CONDITIONS INCLUDE: TEST TIME = 1 HOUR; TEST SPEED = 620 R.P.M.; TEST TEMPERATURE AS INDICATED; BEARINGS = M-10 TOOL STEEL BEARINGS (0.5 INCH DIAMETER) SKF INDUSTRIES GRADE NO. 1, PRL BATCH NO. 2 OR 52-100 STEEL BEARINGS (0.5 INCH DIAMETER) SKF INDUSTRIES GRADE NO. 1, PRL BATCH NO. 12.

THE TESTS AT 700°F. WERE CONDUCTED UNDER A CONTROLLED ATMOSPHERE. THIS WAS ACCOMPLISHED BY INTRODUCING DRY AIR AT A RATE OF 0.7 LITERS PER HOUR INTO THE GAS SPACE OVER THE BALL POT.

<-----TEST FLUID----->		<-----AVERAGE WEAR SCAR DIAM., MM.----->					
DESIGNATION	DESCRIPTION	M-10 TOOL STEEL BEARINGS 700°F.		52-100 STEEL BEARINGS 167°F.		40 KG.	
		1 KG.	40 KG.	1 KG.	40 KG.	1 KG.	40 KG.
MLO 7093	BAROSA 43 (PARAFFINIC 180 NEUTRAL)	0.20	1.17	0.20	0.46	0.20	0.72
PRL 3467	NECTON 60 (NAPHTHENIC MINERAL OIL)	0.24	1.54	0.22	0.55	0.22	0.63
-	1.0 WT.-% PARANOX 441 + 0.5 WT.-% DIISOPROPYL ACID PHOSPHITE IN PRL 3467	0.27	0.57	0.18	0.30	0.18	0.46
MLO 7071	KENDALL HEAVY NEUTRAL	0.22	1.30	0.20	0.43	0.20	0.59
-	1.0 WT.-% PARANOX 441 + 0.5 WT.-% DIISOPROPYL ACID PHOSPHITE IN MLO 7071	0.22	0.39	0.19	0.32	0.19	0.45
MLO 7072	KENDALL LIGHT RESIN	0.19	0.97	0.17	0.28	0.17	0.54
MLO 7073	KENDALL HEAVY RESIN	0.19	0.95	-	-	-	-

Table 115

## LUBRICITY CHARACTERISTICS OF SEVERAL FLUIDS IN THE SHELL FOUR-BALL TESTER

TEST CONDITIONS INCLUDE: TEST TIME = ONE HOUR; TEST SPEED = 620 R.P.M.; TEST TEMPERATURE AS INDICATED; BEARINGS = M-10 TOOL STEEL BEARINGS (0.5-INCH DIAMETER) SKF INDUSTRIES GRADE NO. 1, PRL BATCH NO. 2.  
ALL TESTS WERE CONDUCTED UNDER A CONTROLLED ATMOSPHERE. THIS WAS ACCOMPLISHED BY INTRODUCING DRY AIR AT A RATE OF 0.7 LITERS PER HOUR INTO THE GAS SPACE OVER THE BALL POT.

TEST FLUID	TEST TEMP., °F.	AVERAGE WEAR SCAR DIAM., MM. M-10 TOOL STEEL BEARINGS		
		1 KG.	10 KG.	40 KG.
ORONITE 8200 FLUID (PRL 3506)	600	0.42	0.58	1.71
	700(1)	0.31	0.64	2.51
0.5 45-1 HYDRAULIC FLUID (MLO.7037)	600	0.98	1.17	1.43
	700(1)	0.38	0.52	1.11
AROCLO 1248 (MLO 7061)	600	0.55	0.83	1.20
	700(2)	0.51	0.81	-
SILICONE 81406 (MLO 7017)(IMPROVED LUBRICITY)	600	0.33	0.91	0.97
	700	0.33	0.76	0.74
SILICONE 81406 + 0.5 DIISOPROPYL ACID PHOSPHITE	700	0.41	0.84	0.87
NECTON 60 (PRL 3467)(NAPHTHENIC MINERAL OIL)	700	0.24	0.58	1.54
NECTON 60 + 0.5 DIISOPROPYL ACID PHOSPHITE	700	0.27	0.30	0.57
50:50 (BY WT.) SILICONE 81406:NECTON 60 + 0.5 DIISOPROPYL ACID PHOSPHITE	700	0.35	0.89	2.60

(1) TEST FLUID REPLENISHED AT THE RATE OF 45 ML. PER HOUR.

(2) TEST FLUID REPLENISHED AT THE RATE OF 55 ML. PER HOUR.

Table 116  
EVALUATION OF ORONITE HIGH TEMPERATURE HYDRAULIC FLUID 8515 IN THE PRL  
HIGH TEMPERATURE PUMP TEST UNIT  
TEST PRESSURE = 700 P.S.I. UNLESS OTHERWISE INDICATED IN  
FOOTNOTES.

TEST FLUID	TEST TEMP., °F. (PUMP INLET TEMP.)	TEST TIME AT TEST TEMP., HRS.	FLOW RATE TEST TEMP., G.P.M.	FLUID VISC. AT TEST TEMP., CS.	AV. WT. LOSS PER VANE, MG.
ORONITE HIGH TEMPERATURE HYDRAULIC FLUID 8515	400	4	3.8 - 3.3	3.0	1.9 (1)
	500	4	2.1 - 1.0(2)	2.1	2.2 (2)
SILICATE FLUID O.S. 45	100	4	5.8	12.0	0.2
	300	4	4.9	1.9	0.7
	400	4	4.3 - 2.8	1.2	1.5 (1)
	500	0.5(3)	1.8 - 1.2	0.9	1.3 (3)
SILICATE FLUID MLO 8200	500	-(4)	1.6 - 1.0 (4)	2.6	4.0 (4)
DI-2-ETHYLHEXYL SEBACATE + 0.5 WT. % PHENOTHIAZINE	100	4	5.8	12.7	0.4
	300	4	5.4	1.8	0.4
	400	4	4.9 - 4.6	1.0	+ 0.3
	500	4	3.4 - 2.7	0.8	2.3

- (1) EXCESSIVE CAM RING AND VALVE PLATE WEAR NOTED AFTER COMPLETION OF TEST. A SMALL AMOUNT OF COLLOIDAL METAL WAS OBSERVED IN RESERVOIR DURING OPERATION.
- (2) DURING OPERATION AT 500°F. COLLOIDAL METAL WAS OBSERVED IN THE RESERVOIR. THE FLOW RATE DROPPED STEADILY FROM 2.1 TO LESS THAN 1.0 G.P.M. AS A RESULT, THE SYSTEM PRESSURE COULD NOT BE MAINTAINED AT 700 P.S.I. AND DROPPED STEADILY TO 450 P.S.I. DURING THE COURSE OF THE RUN.
- (3) OPERATION AT 500°F. AND 700 P.S.I. WAS TERMINATED AFTER 0.5 HOURS BECAUSE STEADY OPERATION COULD NOT BE MAINTAINED. COLLOIDAL METAL APPEARED IN RESERVOIR DURING TEST. EXCESSIVE VALVE PLATE WEAR WAS NOTED AFTER COMPLETION OF TEST.
- (4) THE TEST WAS TERMINATED WHEN THE TEMPERATURE REACHED 490°F. BECAUSE COLLOIDAL METAL WAS OBSERVED IN THE RESERVOIR AND THE PRESSURE DROPPED RAPIDLY FROM 700 P.S.I. TO 600 P.S.I. IN A 10 MINUTE PERIOD. IN ADDITION THE FLOW RATE DROPPED BELOW 1.0 G.P.M. AND SEVERE CAM RING AND VALVE PLATE WEAR WAS OBSERVED AFTER COMPLETION OF THE TEST.

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15 mm. Hg pressure (water aspirator) and a maximum pot temperature of 50°C. The volatile material includes hydrochloric acid, isopropyl chloride, and isopropyl alcohol.

The flask is then connected to a high vacuum system and the desired product distilled at about 1 mm. Hg absolute pressure. A dry ice cold trap and an Ascarite absorber tube are included in the vacuum line to protect the vacuum pump from traces of volatile and corrosive reaction products.

The product boiling between 55° and 65°C. at 1 mm. Hg absolute pressure is predominantly diisopropyl acid phosphite with a refractive index at 20°C. of  $1.4080 \pm 0.0010$ . A yield of 60 to 70 per cent of theoretical is typical for a preparation conducted in this manner.

A technique for the estimation of acid phosphite purity from neutralization numbers has been described in Annual Report WADC TR 55-30 Pt 2. The acid group in a dialkyl acid phosphite cannot be readily titrated in a normal neutralization number procedure. This lack of acidity has been explained by keto-enol isomerism or hydrogen bonding. In any event, an equilibrium appears to exist between an acid form and a non-acid form in which the latter dominates. Destroying the equilibrium by removing the acid form with excess base does give complete titration of an acid phosphite. These conditions are sufficiently mild so that no saponification is apparent. The procedure includes the addition of an excess of sodium butylate to the acid phosphite. The mixture is allowed to stand for 25 hours and then back titrated with a solution of hydrochloric acid in butanol. Using these techniques, the diisopropyl acid phosphite fractions described give neutralization numbers indicative of 95+ per cent purity.

Sufficient diisopropyl acid phosphite has been prepared to meet current requests for high lubricity jet engine lubricant samples. Preliminary lubricity tests indicate that the new batch is an effective lubricity additive.

## Q. PREPARATION OF LARGE SCALE MINERAL OIL AND HYDROCARBON

BLENDS. Extensive high temperature testing in the Laboratory has been reported for mineral oils and hydrocarbons as well as several other classes of fluids in WADC Technical Reports 55-30 Part 3 and 55-30 Part 4. The mineral oils and hydrocarbons in particular show good high temperature properties. Current studies indicate that stability properties at intermediate temperatures (200° to 400°F.) of conventionally refined mineral oils and unsaturated hydrocarbons can be improved by further refining, e.g., hydrogenation or silica gel percolation. In general, these additional refining steps have relatively little effect on the properties measured at 500° to 700°F.

Conventionally refined mineral oils and polyolefin hydrocarbons are available in large quantity in contrast to the research quantities of the improved stability stocks prepared by additional refining. To extend the scope of the laboratory evaluation of these mineral oil and hydrocarbon



# Contrails

stocks, two gallon samples of a number of fluids were prepared and sent to the Materials Laboratory, WCRTRI, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio. Quantities of phenyl-alpha-naphthylamine and Paranox 441 oxidation inhibitors were supplied along with the following fluids:

- (1) Esstic 45, naphthenic mineral oil (PRL 3457)
- (2) Pennsylvania 180 neutral mineral oil (MLO 7027)
- (3) Pennsylvania 300 neutral mineral oil (MLO 7026)
- (4) Indopol L-50, polybutene (MLO 7014)
- (5) 50:50 Blend of MLO 7014 and MLO 7027 (MLO 7054)
- (6) 50:50 Blend of MLO 7014 and MLO 7026 (MLO 7053)
- (7) Aroclor 1248, chlorinated aromatic hydrocarbon (PRL 3544)
- (8) Hercoflex 600, pentaerythritol ester (MLO 7064)

As indicated, blends 7 and 8 are not mineral oils or hydrocarbons.

On the basis of these preliminary blends, a series of 12 blends of 100 gallons each have been prepared as typical mineral oil and hydrocarbon compositions. In addition, 225 gallons of Aroclor 1248 were procured.

These materials have been supplied to the Power Plant Laboratory for large scale mock-up testing at high temperatures. The compositions of the blends are shown on Table 117. There are six basic compositions representing paraffinic and naphthenic mineral oil, saturated and unsaturated polyolefin hydrocarbons, and blends of the paraffinic mineral oils and polyolefin. For each basic composition, there is one blend which contains an oxidation inhibitor and one which does not. All of the blends contain a silicone anti-foam additive which successfully eliminates foaming in the Specification VV-L-791e test procedure.

Inspection properties for these blends are also shown on Table 117. These compositions were chosen from conventionally refined stocks with high temperature behavior as the primary criterion for selection. Low temperature properties have not been emphasized in this study. Preliminary tests with mineral oil and hydrocarbon compositions of this type have shown them to be superior to Spec. MIL-L-7808 fluids in stability and general behavior in laboratory tests at 500° and 700°F.

In the range of 200° to 400°F., laboratory evaluation has shown that Spec. MIL-L-7808 fluids are superior in stability to these mineral oil and hydrocarbon compositions. It should be emphasized that current studies indicate that super-refining techniques, such as hydrogenation, will produce mineral oils that are comparable to Spec. MIL-L-7808 fluids in stability in the region of 200° to 400°F. Synthetic hydrocarbon blend MLO 7058A is typical of improved stability mineral oils. MLO 7058A is a hydrogenated polyolefin hydrocarbon with an oxidation inhibitor.

The relationship between Spec. MIL-L-7808 fluids, blends MLO 7055 through MLO 7060, and super-refined mineral oils (hydrogenated)

# Contrails

has been well established on the basis of laboratory tests. Mock-up engine testing with blends MLO 7055 through MLO 7060 will establish their relationship with Spec. MIL-L-7808 fluids in larger scale tests. These results should be of practical importance in establishing the pertinence of various laboratory test techniques. The desirability of super-refining mineral oils should also be predicted by careful correlation of the mock-up and engine test results.

In addition to super-refining, the beneficial effects of dispersant type Acryloids on mineral oils and hydrocarbons have been demonstrated. These effects are noted as a reduction in dirtiness under oxidative conditions in the temperature range of 347° to 500°F. The laboratory data indicate the desirability of including several examples of super-refined mineral oils and hydrocarbons and mineral oils containing an effective Acryloid dispersant in the "mock-up" and jet engine evaluation program undertaken with the 12 large scale mineral oil and hydrocarbon blends shown on Table 117.

R. USED AVIATION ENGINE OILS. This Laboratory has received three series of used jet engine oils from a J-57 engine. The samples were supplied by the Power Plant Laboratory, WCLPF-2, of Wright-Patterson Air Force Base, Ohio. Two sets of samples are Grade 1065 engine oil (mineral Oil) taken from engine tests of 50 and 72 hours duration. For the 50-hour test, the engine was operated at 250°F. oil inlet temperature. The 72-hour test was conducted at 300°F. oil inlet temperature. The third set of samples is from a 100-hour engine test at 300°F. oil inlet temperature using phenothiazine-inhibited di-2-ethylhexyl sebacate as the test fluid.

The time in the engine and some properties of the used 1065 mineral oils are shown on Table 118. The same general property changes are shown for both the 50 and 72-hour tests. The property changes noted with increasing time in the engine, i.e. increases in neutralization number, viscosity, and Conradson carbon residue, are indicative of oxidation. Used samples from the 50-hour test show somewhat more severe oxidative deterioration. These samples have been evaluated more extensively than those from the 72-hour test.

The panel coker values do not change appreciably with use. On the basis of successive oxidation tests at 500°F. which have been reported and discussed in recent WADC technical reports under Contract AF33(616)2851, moderate oxidative deterioration of mineral oils would have less effect on panel coking tendencies than would similar deterioration of a Spec. MIL-L-7808 jet engine lubricant.

The oxidation and corrosion test results for the new and used samples of Grade 1065 mineral oil are shown for 72 hours at 347°F. on Table 119 and for 20 hours at 500°F. on Table 120. All tests were made using Spec. MIL-L-7808 oxidation and corrosion test methods and techniques.

It should be noted that the property changes after 50 hours in the engine are somewhat less severe than for 72 hours at 347°F. or 20 hours



Table 117

PROPERTIES OF EXPERIMENTAL LUBRICANTS FURNISHED TO THE POWER PLANT LABORATORY UNDER CONTRACT AF33(600)32853

ITEM NO.	QUANTITY	DESIGNATION	DESCRIPTION	CENTISTOKE VISCOSITY AT		ASTM SLOPE (210°-100°F.)	VISCOSITY INDEX	NEUT. NO. (MG. KOH/ GM. OIL)	POUR PT., °F.	FLASH PT., °F.
1	100 GAL.	MLO 7055	HIGHLY REFINED NAPHTHENIC MINERAL OIL + ANTI-FOAM AGENT	1.33	5.65	44.2	61.3	0.1	-25	390
2	100 GAL.	MLO 7055A	MLO 7055 + 1.0 WT.% PAN(1)	1.32	5.58	43.5	59.6	0.1	-25	390
3	100 GAL.	MLO 7056	PENNSYLVANIA HEAVY NEUTRAL + ANTI-FOAM AGENT	2.09	10.6	98.3	98.8	0.1	+15	500
4	100 GAL.	MLO 7056A	MLO 7056 + 1.0 WT.% PAN(1)	2.06	10.4	96.3	97.9	0.1	+15	500
5	100 GAL.	MLO 7057	PARAFFINIC 180 NEUTRAL + ANTI-FOAM AGENT	1.45	5.52	32.8	116	0.0	+15	405
6	100 GAL.	MLO 7057A	MLO 7057 + 1.0 WT.% PAN(1)	1.44	5.47	32.5	115	0.0	+15	405
7	100 GAL.	MLO 7058	LOW MOLECULAR WT. POLYOLEFIN + ANTI-FOAM AGENT	2.08	11.1	116	87.0	0.1	-25	335
8	100 GAL.	MLO 7058A	HYDROGENATED POLYOLEFIN + ANTI-FOAM AGENT + 1.0 WT.% PAN(1)	2.03	10.8	112	86.1	0.0	-30	345
9	100 GAL.	MLO 7059	50:50 (BY WT.) MLO 7056:MLO 7058	2.08	10.8	106	93.0	0.1	0	375
10	100 GAL.	MLO 7059A	50:50 (BY WT.) MLO 7056A:MLO 7058A	2.05	10.6	103	93.1	0.1	0	405
11	100 GAL.	MLO 7060	50:50 (BY WT.) MLO 7057:MLO 7058	1.72	7.54	56.4	105	0.0	+5	365
12	100 GAL.	MLO 7060A	50:50 (BY WT.) MLO 7057A:MLO 7058A	1.70	7.42	55.6	103	0.2	+5	380
13	225 GAL.	MLO 7061	AROCLOR 1248 (CHLORINATED BIPHENYL)	0.71	3.26	46.7	-	0.1	+19	380

(1) PHENYL-ALPHA-NAPHTHYLAMINE.

(2) CENTISTOKE VISCOSITY AT 400°F. CALCULATED USING "TABLES FOR DETERMINATION OF ASTM SLOPE AND PREDICTION OF VISCOSITIES" COMPILED BY LAWRENCE T. EBY, ESSO RESEARCH AND ENGINEERING COMPANY.

# Contrails

at 500°F. for the unused sample. Sludge forming tendencies of the engine and the bench tests could not be compared since the sludge in the used engine oil had already been removed by filtration and deposition on metal surfaces during the engine test.

The 30- and 50-hour samples show more severe oxidative deterioration than the unused oil under both sets of oxidative test conditions. In terms of viscosity change, copper corrosion, and sludge formation, further oxidation of the used samples is relatively more severe at 347°F. than at 500°F. This trend again tends to point up the fact that mineral oils show relatively good behavior at 500°F. and higher.

The properties and time in the engine for the used diester fluid are shown on Table 121. It should be emphasized that the ester fluid is 0.5 weight per cent phenothiazine in di-2-ethylhexyl sebacate and not a commercial Spec. MIL-L-7808 jet engine lubricant. The used fluids show a substantial neutralization number increase and a small change in viscosity. Again sludge formation can not be quantitatively measured for the used samples because of the sludge removal in the engine. The relatively high neutralization number and low viscosity increase may be indicative of combined thermal and oxidative deterioration. It has been indicated that the ester fluid has been used for 100 hours at 300°F. oil inlet temperatures.

Oxidation and corrosion tests with the new and used diester lubricant are shown on Tables 122 and 123. These data are for 347° and 500°F., respectively. In the comparison of oxidation stability in the engine and in the 347° and 500°F. oxidation tests, different criteria are used for the mineral oil and ester fluids.

In the case of the 1065 mineral oil, the stable life at 347°F. is well under 72 hours. The stable life for the diester fluid is about 250 to 300 hours at 347°F. under Spec. MIL-L-7808 test conditions. At 500°F. neither fluid type has any appreciable stable life.

The problem for the ester-base fluid at 347°F. is to determine the extent of depletion of the stable life of the used fluids at 347°F. The data for MLO 7135 indicate that, after only 25 hours in the J-57 engine, the ester fluid has a stable life of slightly less than 72 hours. This represents a 75 per cent depletion of the initial stable life of the fluid in 25 hours in the engine at only 300°F. oil inlet temperature. The 50 and 100 hour samples show little if any evidence of stable life at the 347°F. test temperature. It is believed that the improvement noted in the 75 hour sample over the 50 and 100-hour samples is due to the addition of make-up oil close to the sampling time.

A comparison of the 500°F. oxidation and corrosion test results of the used 1065 mineral oils and those of the diester fluids is of interest. There is more evidence of actual oxidative deterioration in the used mineral oil samples than in the used ester samples. The used mineral oils, however, show superior oxygen tolerance to the used ester fluids. This relationship between the two fluid types is also evident in successive

oxidation tests at 500°F. This relationship is not predicted from simple 500°F. oxidation tests of increased severity, i.e. test times longer than 20 hours. The data would suggest that in actual engine use bulk oil temperatures do not adequately portray the severity or type of oxidative deterioration in the engine.

1. Laboratory Tests Simulating Engine Severity. The behavior of used engine oils suggests that spray lubrication on large metal surfaces, short oil residence times at high temperatures, or some other phenomenon may cause the unexpected type and degree of deterioration of Spec. MIL-L-7808 type oils at relatively low measured bulk oil temperatures.

A number of laboratory tests simulating test conditions which may exist in isolated parts of the engine are available for study. A program has been undertaken to determine the relative roles of such tests on subsequent 347°F. oxidation and corrosion tests. This approach may point up the more pertinent procedures by their similarity to the engine tests in producing property changes. The tests of this type considered in this investigation are: a bulk oil oxidation at 400°F. where stable life depletion rate approximates that noted in the engine; a thin film oxidation and corrosion test where spray lubrication on a metal surface is simulated; the panel coker test; a high temperature thermal stability test; a short time high temperature oxidation test; and a successive oxidation test in which the contaminant is produced from a high temperature test and used in a 347°F. test. Only a portion of this program has been completed. Most of this preliminary work has been done with the phenothiazine-inhibited di-2-ethylhexyl sebacate used in the J-57 engine test.

An extrapolation of the log of stable life versus temperature for a phenothiazine-inhibited di-2-ethylhexyl sebacate fluid indicates that the temperature required for a stable life of about 25 hours is 400°F. The stable life at 347°F. is in the range of 200 to 300 hours. Several 24-hour tests at 400°F. have been conducted with MLO 7038 (di-2-ethylhexyl sebacate) plus 0.5 weight per cent phenothiazine and MLO 7083 (Grade 1065 engine oil) using Spec. MIL-L-7808 oxidation and corrosion procedures and techniques.

The results of these tests are shown on Table 125. In all cases the stable life of the diester fluid appears to be less than 24 hours at 400°F. Neutralization number increase, sludge formation, and copper corrosion are indicative of incipient oxidative deterioration.

The mineral oil (MLO 7083) shows evidence of incipient oxidative deterioration under these same conditions. It is interesting to note that dirt formation is appreciable greater for the ester than the mineral oil under these conditions. These tests show the effect of considering the bulk oil temperature in the engine as being sufficiently high to produce incipient oxidation in a diester fluid in 24 hours.

Since the bulk oil temperature is controlled at a lower temperature for these tests, i.e. 250° or 300°F. and only portions of the oil are subjected to short time exposure to higher temperature, other techniques

Table 118

PROPERTIES OF GRADE 1065 AVIATION ENGINE OILS FROM J-57 ENGINE TEST

HISTORY OF SAMPLE FLUID DESIGNATION HOURS IN J-57 ENGINE	UNUSED FLUID MLO 7083 NONE	USED FLUID FROM 50 HOUR TEST IN J-57 ENGINE MLO 7084		USED FLUID FROM 72.5 HOUR TEST - IN J-57 ENGINE MLO 7132 MLO 7133 MLO 7134		
		30	50	25	50	72.5
CENTISTOKES VISCOSITY AT						
100°F.	104	124	132	84.6	95.6	102
130°F.	47.7	54.6	57.8	39.9	44.1	46.5
210°F.	11.4	12.3	12.8	9.93	10.6	11.0
ASTM SLOPE (210° TO 100°F.)	0.692	0.700	0.696	0.700	0.701	0.702
PER CENT VISCOSITY INCREASE AT 100°F. (1)	-	-	6	-	10	16
NEUTRALIZATION NUMBER (MG. KOH/GM. OIL)	0.1	1.6	3.0	1.1	1.4	1.2
PANEL COXER DEPOSIT, MG.	79	89	62	-	-	-
CONRADSON CARBON RESIDUE, %	0.15	0.51	0.77	0.30	0.38	0.30

(1) VALUES CALCULATED IN EACH CASE FROM THE VALUE DETERMINED FOR THE FIRST SAMPLE TAKEN FROM THE ENGINE.

Table 119

## OXIDATION AND CORROSION CHARACTERISTICS OF GRADE 1065 AVIATION ENGINE OILS FROM J-57 ENGINE TESTS

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F.}$ ; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1-INCH SQUARE EACH OF METALS INDICATED.

HISTORY OF SAMPLE FLUID DESIGNATION HOURS IN J-57 ENGINE	UNUSED FLUID MLO 7083 NONE	USED FLUID FROM 50 HOUR TEST IN J-57 ENGINE MLO 7084 30		USED FLUID FROM 72.5 HOUR TEST IN J-57 ENGINE MLO 7132 25		USED FLUID FROM 72.5 HOUR TEST IN J-57 ENGINE MLO 7133 50		MLO 7134 72.5
		1	2	3	1	2	3	
OVERALL LIQUID LOSS, WT. %	1							2
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F.}$	+49	+256	+326	+59	+90	+79		
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	5 >8	>8 >8	>8 >8	>8 >8	>8 >8	>8 >8		
WT. % OIL INSOLUBLE MATERIAL	1.3	0.5	1.4	1.4	1.2	1.0		
CONRADSON CARBON RESIDUE, WT. % (1) ORIGINAL FINAL	0.15 0.84	0.51 -	0.77 -	0.30 -	0.38 -	0.30 -		
FINAL CATALYST CONDITION APPEARANCE	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL	CORRODED DULL DULL DULL		
WT. LOSS (MG./SQ. CM.)	0.90 +0.01 0.01 0.00 -	1.94 +0.01 0.00 0.00 -	0.74 0.01 0.01 0.02 -	0.53 +0.01 +0.04 +0.07 0.00	0.23 0.00 +0.02 +0.02 0.00	0.31 0.00 +0.02 +0.03 0.00		

(1) CONRADSON CARBON RESIDUE DETERMINED ON OIL INSOLUBLES-FREE FLUID IN ACCORDANCE WITH ASTM PROCEDURE D189-41.

Table 120

## OXIDATION AND CORROSION CHARACTERISTICS OF GRADE 1065 AVIATION ENGINE OILS FROM J-57 ENGINE TESTS

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
 TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

HISTORY OF SAMPLE FLUID DESIGNATION NUMBER OF HOURS IN J-57 ENGINE	UNUSED FLUID MLO 7083 NONE	USED FLUID FROM 50 HOUR TEST IN J-57 ENGINE		USED FLUID FROM 72.5 HOUR TEST IN J-57 ENGINE TEST	
		MLO 7084 30	MLO 7085 50	MLO 7132 25	MLO 7133 50
LIQUID CHARGED, GRAMS	85 2	84 1	85 1	86 2	87 2
LIQUID LOSS, WT. %	26.2 4.5	26.2 3.9	26.2 7.6	26.2 5.7	26.2 5.1
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GM. (1)					
APPROX. AMOUNT O <sub>2</sub> USED, GM. (1)					
MOLS O <sub>2</sub> USED/426 GRAMS FLUID (2)	0.69	0.62	1.19	0.88	0.78
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+34	+60	+71	+78	+90
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.1	1.6	3.0	1.1	1.4
FINAL	2.5	4.2	4.7	6.7	7.5
WT. % OIL INSOLUBLE MATERIAL (3)	TRACE	0.1	0.3	0.1	0.3
FINAL CATALYST CONDITION					
APPEARANCE	DULL DULL BRIGHT	DULL DULL BRIGHT	CORRODED DULL BRIGHT	CORRODED COATED COATED	DULL COATED COATED
WT. LOSS (MG./ SQ. CM.)					
COPPER	+0.04	0.01	0.20	0.49	0.11
STEEL	0.02	0.05	0.05	+0.10	+0.12
ALUMINUM	0.00	0.00	0.02	+0.19	+0.23

(1) AMOUNT OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HR.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER).

AMOUNT OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.

(2) THE MOLS OF OXYGEN FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF

DI-2-ETHYLHEXYL SEBACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

*Continued*

Table 121

## PROPERTIES OF PLEXOL 201 FROM J-57 ENGINE TESTS

HISTORY OF SAMPLE		UNUSED FLUID					USED FLUID FROM 100 HOUR TEST IN J-57 ENGINE				
FLUID DESIGNATION		ML0 7038		ML0 7135		ML0 7136		ML0 7137		ML0 7138	
HOURS IN J-57 ENGINE TEST		NONE		25		50		75		100	
CENTISTOKES VISCOSITY AT											
100°F.		12.7		13.3		13.6		13.4		13.9	
130°F.		8.00		8.32		8.49		8.38		8.67	
210°F.		3.35		3.45		3.48		3.44		3.52	
ASTM SLOPE (210° TO 100°F.)		0.698		0.697		0.701		0.702		0.702	
PER CENT VISCOSITY INCREASE AT 100°F. (1)		-		-		2		1		4	
NEUTRALIZATION NUMBER (MG. KOH/GM. OIL)		0.0		1.2		3.9		2.6		5.0	

(1) VALUES CALCULATED FROM THE FIRST SAMPLE TAKEN FROM THE ENGINE.



Table 122

OXIDATION AND CORROSION CHARACTERISTICS OF PHENOTHIAZINE-INHIBITED  
PLEXOL 201 FROM J-57 ENGINE TEST

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^{\circ}\text{F.}$ ; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, MAGNESIUM, AND SILVER-PLATED STEEL.

NOTE: TEST FLUID USED FOR ENGINE TEST COMPRISES PLEXOL 201 PLUS 0.5 WEIGHT PER CENT PHENOTHIAZINE. UNUSED FLUID SHOWN ALSO CONTAINS 0.5 WEIGHT PER CENT PHENOTHIAZINE.

HISTORY OF FLUID FLUID DESIGNATION HOURS IN J-57 ENGINE	UNUSED FLUID MLO 7038 NONE	USED FLUID FROM 100 HOUR TEST IN J-57 ENGINE			
		MLO 7135 25	MLO 7136 50	MLO 7137 75	MLO 7138 100
OVERALL LIQUID LOSS, WT.%	3	3	4	4	5
% CHANGE IN CENTISTOKE VISCOSITY AT $130^{\circ}\text{F.}$	+1	+1	+32	+10	+30
NEUT. NO. (MG. KOH/GM. OIL)					
ORIGINAL	0.2	1.2	3.9	2.6	5.0
FINAL	1.1	3.2	19.8	10.3	19.3
ASTM UNION COLOR					
ORIGINAL	2	>8	>8	>8	>8
FINAL	>8	>8	>8	>8	>8
WT.% INSOLUBLE MATERIAL	0.4	0.2	0.4	0.5	0.4
FINAL CATALYST CONDITION					
APPEARANCE					
COPPER	COATED	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL
MAGNESIUM	COATED	DULL	DULL	DULL	DULL
SILVER	-	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)					
COPPER	+0.12	+0.09	0.08	0.05	0.09
STEEL	+0.05	+0.09	+0.04	+0.05	+0.03
ALUMINUM	+0.03	+0.08	+0.03	+0.04	+0.02
MAGNESIUM	+0.12	+0.05	+0.08	+0.05	+0.05
SILVER	-	+0.02	+0.02	0.02	0.00

Table 123

## OXIDATION AND CORROSION CHARACTERISTICS OF PHENOTHAZINE-INHIBITED PLEXOL 201 FROM J-57 ENGINE TEST

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F}$ .; TEST TIME = 20 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

NOTE: TEST FLUID USED FOR ENGINE TEST COMPRISES PLEXOL 201 PLUS 0.5 WEIGHT PER CENT PHENOTHAZINE. UNUSED FLUID SHOWN ALSO CONTAINS 0.5 WEIGHT PER CENT PHENOTHAZINE.

HISTORY OF FLUID FLUID DESIGNATION HOURS IN J-57 ENGINE	UNUSED FLUID MLO 7038 NONE	USED FLUIDS FROM 100 HOUR TEST IN J-57 ENGINE			
		MLO 7135 25	MLO 7136 50	MLO 7137 75	MLO 7138 100
LIQUID CHARGED GRAMS LIQUID LOSS, WT.-%	91 8	89 9	90 8	91 6	90 6
APPROX. AMOUNT O <sub>2</sub> SUPPLIED, GMS. (1)	26.2	26.2	26.2	26.2	26.2
APPROX. AMOUNT O <sub>2</sub> USED, GMS. (1)	5.3	5.6	7.4	-	7.2
MOLS O <sub>2</sub> USED/426 GMS. FLUID (2)	0.78	0.84	1.11	-	1.07
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL OF OIL INSOLUBLE MATERIAL (3) AT 130°F.	+87	+78	+132	+50	+78
NEUT. NO. (MG. KOH/GM. OIL) ORIGINAL FINAL	0.1 8.8	0.2 24.8	3.9 14.2	2.6 17.2	5.0 21.6
WT.-% OIL INSOLUBLE MATERIAL (3)	0.3	1.0	0.5	0.6	1.2
FINAL CATALYST CONDITION APPEARANCE COPPER STEEL ALUMINUM	DULL COATED DULL	CORRODED DULL DULL	CORRODED DULL DULL	CORRODED DULL DULL	DULL DULL DULL
WT. LOSS (MG./SQ. CM.) COPPER STEEL ALUMINUM	0.02 +0.97 +0.03	0.29 0.01 +0.09	0.53 +0.05 +0.05	0.30 0.00 0.03	0.19 0.03 +0.02

(1) AMOUNT OF OXYGEN SUPPLIED CALCULATED AS FOLLOWS: AIR RATE (L./HR. AT S.T.P.) X TIME (HRS.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). THE AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.(2) MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF OI-2-ETHYLHEXYL SEDACATE IS 426.

(3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUIDS IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLE MATERIAL IS WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.

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have also been used to study effective acceleration of oxidative deterioration at 347°F.

Preliminary treatments evaluated for their effect on subsequent 347°F. oxidation and corrosion behavior include: thermal stability tests at 500° and 600°F. for six hours under a nitrogen atmosphere; panel coking tests for eight hours at 600°F. panel temperature; oxidation at 500°F. for six hours at five liters of air per hour; and oxidation at 400°F. for 24 hours at five liters of air per hour.

The effects of these separate preliminary tests on MLO 7038 fluid are shown on Tables 124, 125, 126, and 127. The thermal stability and panel coker test results are typical of di-2-ethylhexyl sebacate fluids and commercial Spec. MIL-L-7808 fluids evaluated previously. Thermal stability results at 500°F. show only a moderate increase in neutralization number. At 600°F., di-2-ethylhexyl sebacate shows evidence of excessive thermal deterioration and steel corrosion. The panel coker values at 600°F. panel temperature are well within the Spec. MIL-L-7808 limits indicative of satisfactory coking behavior in this test. The neutralization number increase in the panel coker test is again moderate.

Table 124

## PANEL COKER VALUES FOR DI-2-ETHYLHEXYL SEBACATE

Test Conditions Include: Test Temperature = 600°F. and Test Time = 8 Hours.  
Air Rate in the Controlled Coker = 5 Liters per Hour.

Test Fluid	Coker(1)	Wt. Gain, mg.	Fluid Loss, ml.	Max. Oil Temp. in Reservoir, °F.	Neut. No. (Mg. KOH/gm. Oil)	
					Orig.	Final
Di-2-Ethylhexyl Sebacate (MLO 7038) +	C.A.	13.4	135	255	0.1	0.3
0.5 Wt.% Phenothiazine	Model C	23.8	30	314	0.1	0.6

(1) C.A. = Controlled atmosphere.

The six-hour oxidation test at 500°F. shows evidence of incipient oxidation as indicated by a relatively large neutralization number increase. The 500°F. oxidation test forms essentially no dirt or sludge. The 24-hour oxidation test at 400°F. shows a moderate increase in neutralization number coupled with a small amount of sludge.

All of these thermally and oxidatively treated ester products were then subjected to a 72-hour oxidation and corrosion test at 347°F. These oxidation and corrosion test results are shown on Table 128. These results can be compared directly with the oxidation and corrosion test results conducted with the used jet engine oil samples. To aid in determining the effect of the pretreatment on inhibitor depletion, tests with additive and non-additive di-2-ethylhexyl sebacate samples are shown on

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Table 128 for comparison. The non-additive ester fluid shows large viscosity and neutralization number increases, copper and magnesium corrosion, but little dirt formation.

The phenothiazine-inhibited ester shows good overall oxidation and corrosion stability with no pretreatment. The products from the 500°F. thermal stability test and the Model C and controlled atmosphere panel cokers show up as well as or better than the unused inhibited ester in this 347°F. test. The oxidized fluid from the 400°F. treatment shows incipient copper corrosion with high values for neutralization number and viscosity increases. Sludge formation is no greater for this pretreated sample than for the unused inhibited fluid.

The fluid oxidized at 500°F. shows a somewhat different deterioration pattern. This sample shows only moderate increases in viscosity and neutralization number with high sludge formation and copper corrosion. The sample pretreated by a 600°F. thermal stability gives high corrosion and an extremely high viscosity change. These factors are attributed to the extensive thermal decomposition in the thermal test.

These preliminary data on laboratory pretreatment to simulate the type of deterioration encountered in engine tests show certain trends. Oxidation and corrosion characteristics of the used engine oils do not follow the pattern of a non-additive ester. The panel coker and 500°F. thermal stability tests used have little adverse influence on subsequent oxidation and corrosion at 347°F. of the inhibited ester. The pretreated oil from the 500°F. oxidation shows the highest value for dirtiness in subsequent oxidation. This trend is in keeping with the values from successive oxidation tests conducted with esters at 500°F. These data have been presented in detail in Annual Report WADC TR 55-30 Pt 4 from this Laboratory.

This trend noted in successive oxidation tests at 500°F. and in the oxidation at 347°F. of the samples pretreated at 500°F. may suggest the cause of the relatively high rate of sludge formation in the J-57 engine in the first 25 hours of running at high oil inlet temperatures. The high temperature oxidative pretreatment will be used for further tests employing small concentrations of contaminant from the high temperature oxidation product in unused ester fluid for evaluation at 347°F. Low temperature (347°F.) PRL thin film oxidation and corrosion tests will be conducted to determine the pertinence of this test in simulating engine severity.

S. HIGH TEMPERATURE EVALUATION OF SPEC. MIL-L-6387 HYDRAULIC FLUID. Spec. MIL-L-6387 covers an ester-base hydraulic fluid designed for high temperature use under severe lubricity conditions in the Sundstrand constant speed alternator drive. Since the development of Spec. MIL-L-7808 jet engine oil, both types of fluids have been used successfully in alternator drives. It has been demonstrated in report WADC TR 55-30 Pt 1 from this Laboratory that a single fluid composition can be formulated to meet both Spec. MIL-L-7808 and MIL-L-6387. These ester fluids have good lubricity characteristics at elevated temperatures

Table 125

## OXIDATION AND CORROSION CHARACTERISTICS OF TWO FLUIDS AT 400°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $400 \pm 3^\circ\text{F.}$ ; TEST TIME = 24 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUIDS: MLO 7038 = DI-2-ETHYLHEXYL SEBACATE + 0.5 WT.% PHENOTHIAZINE.  
MLO 7083 = AVIATION ENGINE OIL GRADE 1065.

TEST FLUID	< - - - MLO 7038 - - - >			< - - - - MLO 7083 - - - - >		
OVERALL LIQUID LOSS, WT. %	2	2	4	1	3	3
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+3	+2	+4	+21	+20	+28
NEUT. NO. (MG. KOH/GM. OIL)						
ORIGINAL	0.1	0.1	0.1	0.0	0.0	0.0
FINAL	2.8	3.0	4.9	2.9	2.6	2.6
ASTM UNION COLOR						
ORIGINAL	2	2	2	5	5	5
FINAL	>8	>8	>8	>8	>8	>8
WT. % INSOLUBLE MATERIAL	0.4	0.4	0.4	TRACE	TRACE	TRACE
FINAL CATALYST CONDITION						
APPEARANCE						
COPPER	DULL	COATED	DULL	CORRODED	CORRODED	CORRODED
STEEL	DULL	DULL	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)						
COPPER	+0.05	+1.63	+0.09	0.79	0.94	0.88
STEEL	+0.08	+0.09	0.05	0.04	0.02	+0.04
ALUMINUM	0.01	+0.02	+0.05	0.04	0.02	0.03

Table 126

## THERMAL STABILITY OF DI-2-ETHYLHEXYL SEBACATE

TESTS WERE CONDUCTED IN GLASS TEST TUBES FOR A 6-HOUR TEST PERIOD. TEST FLUID CHARGE AS INDICATED. THE TUBES WERE SEALED WITH A U-TUBE CONTAINING APPROX. 3 MLS. OF TEST FLUID. THE SEAL ALLOWS THE PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROX. 0.25 LBS./SQ. INCH. CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM. TEST IS CONDUCTED UNDER A NITROGEN ATMOSPHERE.

TEST FLUID	TEST TEMP., °F.	LIQUID CHARGE, GMS.	CENTISTOKE VISCOSITY AT 100°F. ORIG.   FINAL   % CHANGE	% LOSS IN WT. (1)	OIL INSOLUBLE MATERIAL, WT. %	NEUT. NO. (MG. KOH/GM. OIL) ORIG.   FINAL	CATALYST WT. LOSS (MG./SQ. CM.) COPPER   STEEL   ALUMINUM
DI-2-ETHYLHEXYL SEBACATE (MLO 7038) + 0.5 WT. % PHENOTHIAZINE	500	130	12.70   -   -	1	TRACE	0.1   1.0	0.03   +0.02   +0.01
	500	136	12.70   12.80   +1	0	0.0	0.1   1.0	+0.01   +0.15   0.02
	600	87	12.70   13.06   +3	2	0.4	0.1   18.5	0.05   7.67   +0.02
	600	90	12.70   13.01   +2	2	0.4	0.1   21.8	0.05   8.34   +0.23

(1) LOSS IN WEIGHT OF THE FLUID IS GOOD TO  $\pm 0.5\%$  OR 0.5 GRAMS.

Table 127

OXIDATION AND CORROSION CHARACTERISTICS OF DI-2-ETHYLHEXYL  
SEBACATE AT 500°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $500 \pm 5^\circ\text{F.}$ ; TEST TIME = 6 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 150 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, AND ALUMINUM.

TEST FLUID	DI-2-ETHYLHEXYL SEBACATE (MLO 7038) + 0.5 WT.% PHENOTHIAZINE	
LIQUID CHARGED, GM.	134	135
LIQUID LOSS, WT.%	1	2
APPROXIMATE AMOUNT OF O <sub>2</sub> SUPPLIED, GMS.(1)	7.8	7.8
APPROXIMATE AMOUNT OF O <sub>2</sub> USED, GMS.(1)	1.7	1.7
MOLS O <sub>2</sub> USED/426 GRAMS OF FLUID (2)	0.17	0.17
% CHANGE IN CENTISTOKE VISCOSITY AFTER REMOVAL(3) OF OIL INSOLUBLES AT 130°F.	+7	+7
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	0.1	0.1
FINAL	4.3	4.7
WT.% OIL INSOLUBLE MATERIAL(3)	TRACE	TRACE
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	DULL	DULL
STEEL	COATED	COATED
ALUMINUM	BRIGHT	BRIGHT
WT. LOSS (MG./SQ. CM.)		
COPPER	0.15	0.13
STEEL	+0.86	+0.78
ALUMINUM	+0.01	0.02

- (1) AMOUNT OXYGEN SUPPLIES CALCULATED AS FOLLOWS: AIR RATE (L/HR. AT S.T.P.) X TIME (HRS.) X O<sub>2</sub> CONTENT (FRACTION) X 1.43 (GM./LITER). AMOUNT OF OXYGEN CONSUMED DETERMINED BY FREQUENT SAMPLING OF EXHAUST GASES AND ANALYSIS FOR O<sub>2</sub>.
- (2) MOLS OF O<sub>2</sub> FOR 426 GRAMS OF FLUID HAS BEEN TABULATED AS A COMMON BASIS FOR COMPARING THE O<sub>2</sub> ABSORBED. THE MOLECULAR WEIGHT OF DI-2-ETHYLHEXYL SEBACATE IS 426.
- (3) OIL INSOLUBLE MATERIAL IS REMOVED BY CENTRIFUGING OXIDIZED FLUID IMMEDIATELY AFTER COMPLETION OF TEST. THE OIL INSOLUBLES ARE WASHED WITH A LOW BOILING PETROLEUM NAPHTHA AND DRIED BEFORE WEIGHING.



Table 128

**EFFECT OF PRELIMINARY TREATMENT ON OXIDATION AND CORROSION CHARACTERISTICS  
OF DI-2-ETHYLHEXYL SEBACATE AT 347°F.**

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEST TEMPERATURE = 347 ± 3 °F.; TEST TIME AS INDICATED; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

NOTE: EXCEPT FOR THE UNUSED FLUIDS NOTED, THE TEST FLUIDS ARE THE PRODUCTS FROM THE VARIOUS TESTS INDICATED.

TEST FLUID OXIDATION INHIBITOR, WT. % PREVIOUS HISTORY OF FLUID	← - - - - - NONE - - - - - → UNUSED		← - - - - - DI-2-ETHYLHEXYL SEBACATE (MLO 7038) - - - - - → UNUSED		- - - - - 0.5 PHENOTHIAZINE - - - - - AT 400°F. (SEE TABLE 125)		- - - - - 0.5 PHENOTHIAZINE - - - - - AT 500°F. (SEE TABLE 127)		- - - - - THERMAL STABILITY TEST AT 500°F.   AT 600°F. (SEE TABLE 126)		- - - - - PANEL COKER TEST C COKER   CA COKER (SEE TABLE 124)	
TEST TIME, HOURS	36	72	72	72	72	72	72	72	72	72	72	72
OVERALL LIQUID LOSS, WT. %	2	-	3	-	-	-	-	6	-	-	-	-
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+28	+230	+1	+34	+11	-	-	-(11)	+2	+2	+2	+2
NEUT. NO. (MG. KOH/GM. OIL)												
ORIGINAL	0.1	0.1	0.1	3.5	4.3	1.0	1.0	18.7	0.3	0.3	0.6	0.6
FINAL	9.5	20.5	1.1	20.1	6.7	1.2	1.2	26.0	1.4	1.4	1.3	1.3
ASTM UNION COLOR												
ORIGINAL	2	2	2	>8	>8	3	>8	5	>8	>8	>8	>8
FINAL	5	>8	>8	>8	>8	>8	>8	>8	>8	>8	>8	>8
WT. % INSOLUBLE MATERIAL	TRACE	0.2	0.4	0.4	3.1	0.1	0.1	SOLID(1)	0.2	0.2	0.4	0.4
FINAL CATALYST CONDITION												
COPPER	DULL	CORRODED	COATED	CORRODED	CORRODED	DULL	DULL	CORRODED	DULL	DULL	DULL	DULL
STEEL	DULL	DULL	DULL	DULL	DULL	DULL	DULL	CORRODED	DULL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL	DULL	DULL	DULL	DULL	CORRODED	DULL	DULL	DULL	DULL
MAGNESIUM	CORRODED	CORRODED	COATED	DULL	BRIGHT	DULL	DULL	CORRODED	DULL	DULL	DULL	DULL
WT. LOSS (MG./SQ. CM.)												
COPPER	0.04	0.65	+0.12	0.43	10.6	0.05	0.05	8.53	0.03	0.03	0.02	0.02
STEEL	0.02	0.04	+0.05	0.03	0.04	0.00	0.00	0.22	+0.01	+0.01	+0.08	+0.08
ALUMINUM	0.04	0.02	+0.03	0.00	0.05	+0.01	+0.01	0.07	0.01	0.01	+0.05	+0.05
MAGNESIUM	0.54	36.4	+0.12	0.05	0.03	0.00	0.00	48.30	+0.05	+0.05	+0.05	+0.05

(1) MATERIAL FOLLOWING THE TEST WAS AN INTIMATE MIXTURE OF TEST FLUID AND SOLIDS. OIL INSOLUBLE MATERIAL COULD NOT BE DETERMINED WITHOUT DILUTION.

(as high as 500° to 600°F.), a wide liquid range, and relatively good thermal stability for temperatures up to 550°F.

A commercial sample of Spec. MIL-L-6387 fluid has been used as the test fluid for a high temperature hydraulic pump test by the Hamilton Standard Division of United Aircraft Corporation. A sample has been obtained for evaluation after 102 hours running time at 275°F. oil inlet temperature in the hydraulic pump unit. The properties of the new and used oil are shown on Table 129. A moderate increase in neutralization number and a decrease in viscosity are noted for the used fluid.

The viscosity decrease is a shear effect caused primarily by fluid turbulence and attrition in the loading valve used to maintain the operating pressure of 3000 p.s.i. The viscosity decrease of 16 per cent after 102 hours test time is considered to be satisfactory shear stability for fluids of this type. The viscosity decrease for a typical Spec. MIL-O-5606 hydraulic fluid is more than twice that of the Spec. MIL-L-6387 fluid for the same degree of shear severity. That is, the Spec. MIL-O-5606 fluid would incur 35 to 40 per cent viscosity decrease due to the shear effects in the equivalent 102 hour test at 3000 p.s.i. In the viscosity decrease due to shear, the viscosity decreases rapidly in the first cycles through the shearing device and then shows little additional viscosity change with increasing fluid use after about the first 1000 or 2000 test cycles.

The increase in neutralization number appears to be primarily due to thermal rather than oxidative changes. Oxidation and corrosion tests have been conducted with the original and used samples of Spec. MIL-L-6387 fluid. Stable life and 72-hour tests have been made as shown on Tables 130 and 131 and Figure 11. These data indicate that the unused Spec. MIL-L-6387 fluid has a stable life of approximately 140 hours at 347°F. The used sample from the 102-hour hydraulic system test shows a stable life of approximately 100 hours. These data would indicate that the neutralization number increase in the used Spec. MIL-L-6387 fluid is caused primarily by thermal effects and not oxidation.

Thermal tests at 347° and 500°F. with Spec. MIL-L-6387 and Spec. MIL-L-7808 type lubricants are shown on Table 132. These data show that neutralization numbers of the same order of magnitude as those obtained after 102 hours operation at 275°F. oil reservoir temperature can be obtained in thermal stability tests at 347°F. for 72 hours. These tests on the original fluid and the used fluid indicate that the increase in neutralization number is greater for the first 72-hour test period than for subsequent 72-hour test periods. These data suggest a satisfactory life of the order of 300 to 400 hours for Spec. MIL-L-6387 in an air-free hydraulic unit operating at 275°F. oil reservoir temperature.

The esters available for use in the preparation of Spec. MIL-L-6387 hydraulic fluid are limited because of the 7500 centistoke viscosity limit at -65°F. coupled with the required high temperature viscosities. Spec. MIL-L-7808 has a 13,000 centistoke viscosity limit at -65°F. This allows considerably more freedom of choice in ester base stocks. The

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Spec. MIL-L-7808 viscosity requirements of 13,000 centistokes maximum at -65°F. are equivalent to a viscosity of 7500 centistokes at -58°F. In other words, the loss in low temperature viscosity between Spec. MIL-L-6387 and MIL-L-7808 is only 7°F. while the benefits in ester choice and high temperature stability may be appreciable.

Comparable thermal stability data for a di-2-ethylhexyl sebacate ester (Spec. MIL-L-7808 type fluid) and for Spec. MIL-L-6387 fluids are shown on Table 132 at 347° and 500°F. The di-2-ethylhexyl sebacate fluid (MLO 7038) shows substantially better stability to change in neutralization number at both 347° and 500°F. The MLO 7038 composition shows no magnesium corrosion under the 347°F. test conditions in contrast to the high magnesium corrosion values for the Spec. MIL-L-6387 fluid in the test.

All of the laboratory data obtained, thus far, indicate that a satisfactory ester-base hydraulic fluid can be formulated for use in the temperature range of -65° to +400°F. and perhaps higher. It appears that a small increase in -65°F. viscosity in the Spec. MIL-L-6387 fluid will allow substantial improvements in high temperature fluid stability. The retention of a separate ester type hydraulic fluid specification as opposed to Spec. MIL-L-7808 jet engine oil will allow more careful quality control which may successfully eliminate such problems a storage stability from the hydraulic fluid.

T. CONCLUSIONS. Mineral oils and hydrocarbons as a class show very good high temperature stability in comparative laboratory evaluations. Conventionally refined mineral oils show better over-all oxidative behavior at 500°F. and above than at 347°F. Dirtiness and lack of inhibitor susceptibility are primarily responsible for the relatively poor oxidation stability at 347°F. A combination of additives and additional refining does improve considerably the properties obtainable in 347°F. oxidation tests where stable life and induction period are the governing criteria of oxidation.

Hydrogenation over a nickel catalyst is more effective than silica gel percolation in improving inhibitor susceptibility. Both of these treatments tend to remove non-hydrocarbon impurities (i.e. sulfur, nitrogen, and oxygen-containing impurities) and unsaturated hydrocarbons from mineral oils.

Conventionally refined paraffinic and naphthenic mineral oils and olefinic and aromatic hydrocarbons have all been substantially improved in stability by hydrogenation over U.O.P. nickel catalyst at 500°F. and 1000 p.s.i. hydrogen pressure. Hydrogenation proceeds most readily with the polyolefin hydrocarbon and least readily with the cycle stock extract. The paraffinic and naphthenic mineral oils are intermediate in terms of hydrogenation difficulty. The catalytic cycle stock extract contained sufficient non-hydrocarbon impurities to produce catalyst poisoning after only partial hydrogenation. More complete hydrogenation was accomplished by a two stage hydrogenation in which the bulk of the catalyst poisons were removed in the first step.

Table 129

PROPERTIES OF NEW AND USED SAMPLES OF SPEC. MIL-L-6387 HYDRAULIC FLUID

Test Fluids Received From Hamilton Standard Division of United Aircraft Corporation, Windsor Locks, Connecticut.

Test Fluids: MLO 7139 = Unused Sample of Spec. MIL-L-6387A Hydraulic Fluid.

MLO 7141 = Used Sample of Spec. MIL-L-6387A Hydraulic Fluid Taken From a High Temperature Hydraulic Pump Test After 102 Hours Running Time at 275°F. Oil Inlet Temperature.

Test Fluid	MLO 7139	MLO 7141
Centistoke Viscosity at 100°F. 130°F. 210°F.	15.3 10.0 4.41	12.8 8.42 3.71
Per Cent Viscosity Decrease at 100°F.	-	16
ASTM Slope (100° to 210°F.)	0.596	0.634
Neutralization Number (Mg. KOH/Gm. Oil)	0.1	0.8

OXIDATION AND CORROSION CHARACTERISTICS OF NEW AND USED SPEC.  
MIL-L-6387 HYDRAULIC FLUIDS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = 72 HOURS; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML.; AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.  
TEST FLUIDS: MLO 7139 = UNUSED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD  
MLO 7141 = USED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD.  
(102 HOURS TEST TIME IN A HIGH TEMPERATURE PUMP TEST AT  $275^\circ\text{F}$ . OIL INLET TEMPERATURE).  
MLO 7038 = DI-2-ETHYLHEXYL SEBACATE + 0.5 WT.% PHENOTHIAZINE.

TEST FLUID	MLO 7038	MLO 7139	MLO 7141
OVERALL LIQUID LOSS, WT.%	3	3	2
% CHANGE IN CENTISTOKE VISCOSITY AT $130^\circ\text{F}$ .	+1	+5	+10
NEUT. NO. (MG. KOH/GM. OIL)			
ORIGINAL	0.2	0.1	0.8
FINAL	1.1	1.3	5.4
ASTM UNION COLOR			
ORIGINAL	2	3	5
FINAL	>8	>8	>8
WT.% INSOLUBLE MATERIAL	0.4	0.4	1.2
FINAL CATALYST CONDITION			
APPEARANCE			
COPPER	COATED	DULL	DULL
STEEL	DULL	DULL	DULL
ALUMINUM	DULL	DULL	DULL
MAGNESIUM	COATED	DULL	CORRODED
WT. LOSS (MG./SQ. CM.)			
COPPER	+0.12	0.08	0.02
STEEL	+0.05	+0.01	+0.01
ALUMINUM	+0.03	+0.02	+0.03
MAGNESIUM	+0.12	+0.01	3.00

Table 131

OXIDATION STABILITY OF NEW AND USED  
SPEC. MIL-L-6387 HYDRAULIC FLUIDS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.  
TEST CONDITIONS INCLUDE: TEST TEMPERATURE =  $347 \pm 3^\circ\text{F}$ .; TEST TIME = AS INDICATED; AIR RATE =  $5 \pm 0.5$  LITERS PER HOUR; TEST FLUID CHARGED = 100 ML., AND CATALYST = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM AND MAGNESIUM.  
TEST FLUIDS: MLO 7139 = UNUSED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD.  
MLO 7141 = USED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD.  
(102 HOURS TEST TIME IN A HIGH TEMPERATURE PUMP TEST AT 275°F. OIL INLET TEMPERATURE).

TEST FLUID	MLO 7139	MLO 7141
TEST TIME, HOURS	185	168
OVERALL LIQUID LOSS, WT.%	-	-
% CHANGE IN CENTISTOKE VISCOSITY AT 130°F.	+25	+509
NEUT. NO. (MG. KOH/GM. OIL)		
ORIGINAL	0.1	0.8
FINAL	21.2	16.8
ASTM UNION COLOR		
ORIGINAL	3	5
FINAL	>8	>8
WT.% INSOLUBLE MATERIAL	1.9	5.9
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	CORRODED	CORRODED
STEEL	DULL	DULL
ALUMINUM	DULL	BRIGHT
MAGNESIUM	COATED	CORRODED
WT. LOSS (MG./SQ. CM.)		
COPPER	0.29	0.86
STEEL	+0.05	0.04
ALUMINUM	+0.03	0.00
MAGNESIUM	+0.14	38.60

Figure 11

OXIDATION STABILITY OF NEW AND USED SPEC. MIL-L-6387 HYDRAULIC FLUIDS AT 347°F.

TEST PROCEDURE AND TECHNIQUES IN ACCORDANCE WITH SPEC. MIL-L-7808.

TEST CONDITIONS INCLUDE: TEMPERATURE = 347 ± 3°F.; TEST TIME AS INDICATED; AIR RATE = 5 ± 0.5 LITERS PER HOUR; TEST FLUID CHARGED = 100 MLS.; AND CATALYSTS = A 1-INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM AND MAGNESIUM.

▲ MLO 7139 = UNUSED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD.

○ MLO 7141 = USED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD (102 HOURS TEST TIME IN HIGH TEMPERATURE PUMP TEST AT 275°F. OIL INLET TEMPERATURE).

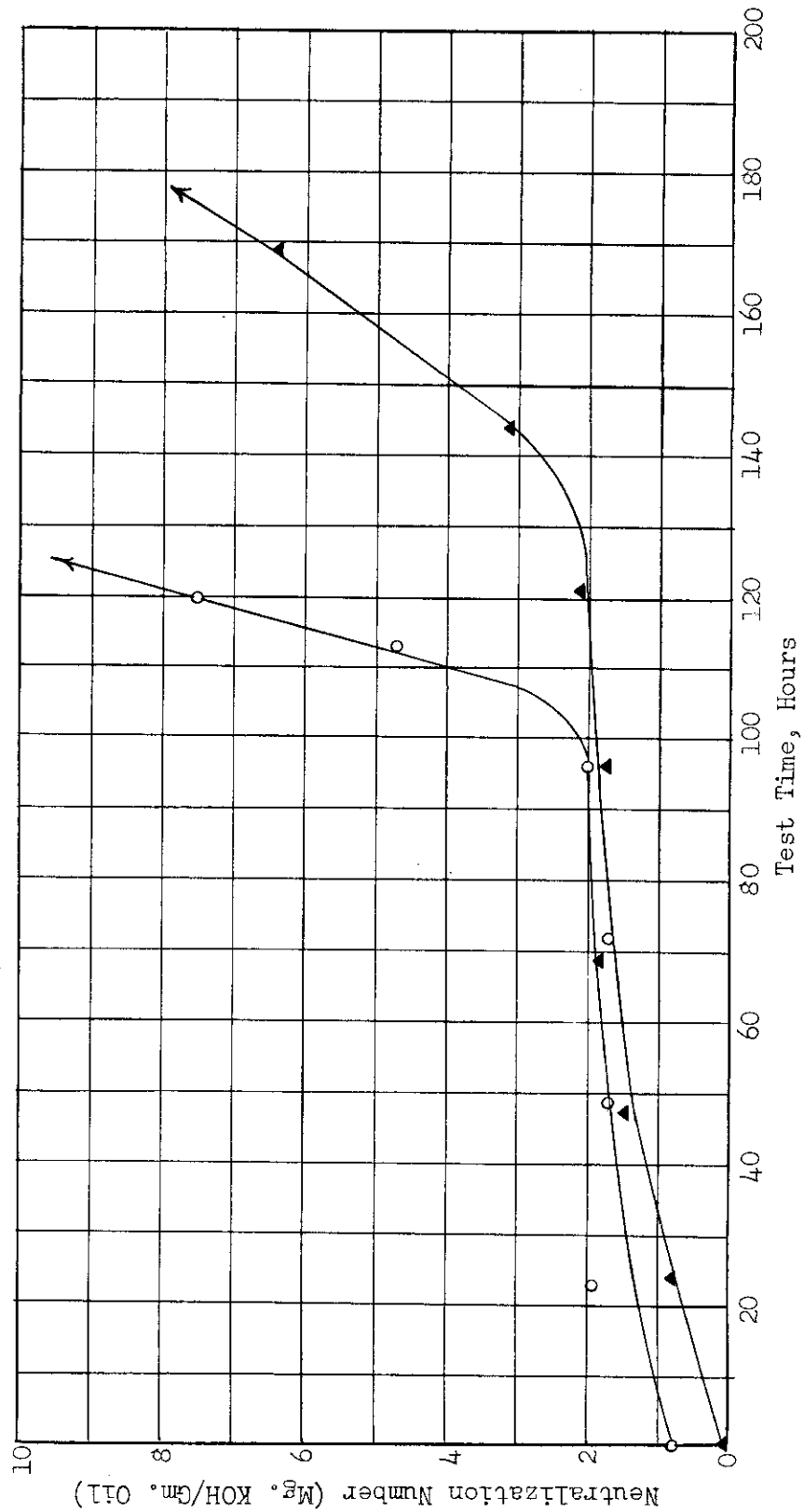




Table 132

## THERMAL STABILITY OF NEW AND USED SPEC. MIL-L-6387 HYDRAULIC FLUIDS

TESTS CONDUCTED AT 347°F. CONTAIN A 100 ML CHARGE OF FLUID. THE TEST TUBES ARE SEALED WITH A U-TUBE CONTAINING APPROXIMATELY 3 MLS. OF TEST FLUID. THE SEAL ALLOWS THE PASSAGE OF GAS OUT OR AIR IN WITH PRESSURE CHANGES GREATER THAN 4 INCHES OF FLUID - APPROXIMATELY 0.25 LBS./SQ. IN. PRIOR TO THE START OF THE TEST, THE VOLUME OF THE SEALED TUBE IS REPLACED APPROXIMATELY 4 TIMES WITH NITROGEN. THE NITROGEN IS INTRODUCED ABOVE THE SURFACE OF THE TEST FLUID BY MEANS OF A SEALED IN CAPILLARY TUBE. CATALYSTS = 1 INCH SQUARE EACH OF COPPER, STEEL, ALUMINUM, AND MAGNESIUM.

TESTS CONDUCTED AT 500°F. CONTAIN A 25 GMS. CHARGE OF TEST FLUID. PROCEDURE USED IS IDENTICAL WITH THAT USED FOR 347°F. TESTS EXCEPT THAT NO CATALYSTS ARE USED IN 500°F. TESTS.

TEST FLUIDS: MLO 7139 = UNUSED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD.

MLO 7141 = USED SPEC. MIL-L-6387A HYDRAULIC FLUID RECEIVED FROM HAMILTON STANDARD (102 HOURS TEST TIME IN HIGH TEMPERATURE PUMP

TEST AT 275°F. OIL INLET TEMPERATURE.

MLO 7038 AND PRL 3103 = DI-2-ETHYLHEXYL SEBACATE

PRL 2996A = DI-2-METHYLBUTYL ADIPATE

PRL 2996A = 01-3-METRYLBUTYL ADIPATE												
FLUID	TEST TEMP., °F.	TEST TIME, HRS.	CENTISTOKE VISC. AT 100°F.		% LOSS IN WEIGHT	CATALYST WT. LOSS MG./SQ. CM.			NEUT. NO. MG. KOH/GM. OIL		WT. % INSOLUBLE MATERIAL	
			ORIGINAL	FINAL		CHANGE	COPPER	STEEL	ALUMINUM	MAGNESIUM		ORIG.
MLO 7139	347	72	15.3	15.3	0	0.03	0.09	0.02	0.62	0.1	0.7	TRACE
MLO 7139	347	144	15.3	15.1	-1	0.06	0.09	+0.09	2.22	0.1	0.8	0.1
MLO 7139	500	20	15.3	12.0	-22	-	-	-	-	0.1	7.8	0.1
MLO 7141	347	72	12.8	12.9	+1	0.07	0.03	+0.01	2.92	0.8	1.0	TRACE
MLO 7141	347	144	12.8	12.9	+1	0.05	0.33	0.00	3.12	0.8	1.1	TRACE
MLO 7038	347	72	12.8	12.9	+1	+0.01	+0.06	+0.07	+0.08	0.1	0.3	0.0
MLO 7038	347	144	12.8	12.8	0	0.00	+0.01	+0.04	+0.07	0.1	0.2	0.0
PRL 3103	500	20	12.8	12.8	0	-	-	-	-	0.1	2.8	-
PRL 2996A	500	20	4.87	5.06	+4	-	-	-	-	0.2	7.9	-

Hydrogenation has little effect on the viscosity and viscosity-temperature characteristics of well refined naphthenic and paraffinic mineral oils and polyolefin hydrocarbons. The pour point tends to increase with hydrogenation for paraffin hydrocarbons and paraffinic mineral oils. This latter effect is primarily a wax problem. Hydrogenation of cycle stock extract and lube oil extract results in substantial lowering of viscosity level, improvement in viscosity-temperature characteristics, and lowering of the pour point. The pour point in this case is a viscous pour.

A neopentyl glycol type ester (Hercules J-20) is essentially the same as a pentaerythritol ester (Hercoflex 600) in high temperature oxidation characteristics and thermal stability.

The thermal and oxidative stability of bis (1-methyl cyclohexyl-methyl) sebacate have been studied and compared with typical esters. Under Spec. MIL-L-7808 conditions, oxidation stability of phenothiazine-inhibited bis (1-methyl cyclohexyl methyl) sebacate and a typical diester are of the same order of magnitude. In 500° and 600°F. oxidation and 700°F. thermal tests in the presence of metals, the bis (1-methyl cyclohexyl methyl) sebacate is comparable to several other neopentyl type esters of the pentaerythritol or neopentyl glycol type. In the 700°F. thermal tests, the bis (1-methyl cyclohexyl methyl) sebacate forms a solid rubbery decomposition product compared with a product that is part volatile and part liquid which is typical of the pentaerythritol and neopentyl glycol type esters.

The same order of magnitude of thermal stability in esters has been shown for a series of neopentyl type esters in which the hydroxy groups vary from one to four in the alcohol portion of the molecule and both mono and di-basic acids have been used.

Bis tridecyl sebacate is a more viscous grade of diester than di-2-ethylhexyl sebacate. The thermal and oxidative stability is essentially the same for bis tridecyl sebacate and di-2-ethylhexyl sebacate.

The isosebacic acid esters examined have essentially the same thermal stability characteristics as the chain type dibasic acid esters typified by di-2-ethylhexyl sebacate. These esters show less oxidation stability at 347° and 500°F. than the esters currently used to prepare Spec. MIL-L-7808 fluids.

The chlorinated phosphate esters evaluated are similar in properties to compounds of this class evaluated previously. Thermal stability and high temperature oxidation are adversely affected by the presence of metals. The metals (copper, steel, and aluminum) are all severely corroded by these phosphate esters in 500°F. oxidation tests.

High molecular weight fractions of mineral oils (Kendall resins) derived from Pennsylvania crude oil show promising oxidation and corrosion characteristics at 347° and 500°F. These materials show good thermal stability at 600°F. but relatively poor stability at 700°F. For a given series of mineral oil types, it has been noted that thermal stability

tends to decrease gradually with increasing molecular weight or viscosity grade.

Air rate is not a critical variable in the study of oxidation characteristics within the stable life of the fluid under Spec. MIL-L-7808 type test conditions. Air rate does become a critical factor when the stable life of the fluid is exceeded and oxidation occurs. An air rate of five liters per hour has been chosen as a standard air rate for the evaluation of oxidation and corrosion characteristics at 347°F.

The unsaturated polyolefins evaluated show essentially no stable life at 347°F. and little or no inhibitor response. The unsaturated polyolefins do show good oxygen tolerance at 347°F. as indicated by the very low sludging tendencies in a 72-hour test. Completely hydrogenated polyolefins show excellent inhibitor response to both phenyl-alpha-naphthylamine and dithiocarbamate type additives at 347°F. Stable life values of the hydrogenated polyolefin blends are comparable to those of high quality Spec. MIL-L-7808 fluids.

Oxidation properties of the conventionally refined mineral oils in the 72-hour test at 347°F. are generally poor. Several oxidation inhibitor types produce improved oxidation behavior under these conditions. In general, however, sufficient inhibitor response is not obtained with these conventionally refined mineral oil fractions to obtain a stable life of 72 hours at 347°F. or to pass the property change limitations of Spec. MIL-L-7808 under these test conditions. Inhibitor effectiveness at 347°F. for the dithiocarbamate inhibitors is a strong function of mineral oil type and degree of refining. The highest order of effectiveness for this type of inhibitor appears to be completely saturated paraffinic mineral oils or synthetic paraffin hydrocarbons. Other additives evaluated, such as phenyl-alpha-naphthylamine (PAN) and hindered phenol types, show less specificity.

Hydrogenation and silica gel adsorption have been compared as super-refining treatments for paraffinic and naphthenic mineral oils. Hydrogenation produces mineral oils of excellent inhibitor susceptibility and low sludging or dirtiness trends at 347°F. Silica gel percolation tends to show the same order of effectiveness in reducing sludging and dirtiness. Silica gel treatment is not as effective as hydrogenation in improving additive susceptibility.

A series of four highly treated stocks and the original charging stocks obtained from the Phillips Petroleum Company have oxidation characteristics at 347°F. typical of conventionally refined mineral oils. The highly treated stocks show no significant improvements over the charging stocks.

A number of experimental compounds from the Monsanto Chemical Company have been evaluated as oxidation inhibitors in mineral oils and esters. Several of these materials show activity as oxidation inhibitors.

# Contrails

Oxidation studies on conventionally refined mineral oil fractions at 500° and 600°F. show good oxygen tolerance and a moderate oxidation rate. In general, polyolefins, such as Indopol L-50, show about the same oxidation rate but better oxygen tolerance than conventionally refined mineral oil fractions. Oxygen tolerance in this comparison is based primarily on dirtiness.

Hydrogenation, silica gel adsorption, and acid extraction do not affect significantly the oxygen tolerance or oxidation rate of the paraffinic, naphthenic, and aromatic mineral oils evaluated. The hydrogenated polyolefin shows the same oxidation rate but lower oxygen tolerance than the unsaturated polyolefin.

The use of oxidation inhibitors in mineral oils and polyolefins at 500° and 600°F. does not reduce substantially the oxidation rate, but in general does increase slightly the dirtiness for a given amount of oxygen assimilated. The final properties after oxidation at 500° and 600°F. of a 50:50 mixture of Indopol L-50 polyolefin with a conventional mineral oil are dictated primarily by the polyolefin component.

Five Acryloid polymers have been shown to have a significant effect in the reduction of dirtiness or sludge forming tendencies under oxidation conditions in both diester and mineral oil base stocks. The sludge dispersing effects have been noted in all 347° and 500°F. tests. In the 500°F. oxidation tests, the unusually good viscosity stability is due to the competing reactions of viscosity increase of the base stock by oxidation and the viscosity decrease of the polymer by incipient thermal instability.

The dispersant Acryloids do not affect materially the panel coking tendencies of the base stocks. Panel coking tests as a fluid pretreatment do not reduce the effectiveness of the Acryloid dispersant in subsequent bulk oil oxidation tests. Thermal stability tests at 700°F. show that most of the Acryloids evaluated do not contribute to steel and copper corrosion under these conditions but do undergo substantial viscosity decrease.

Tricresyl phosphate and diisopropyl acid phosphite show little or no effect on the bulk oil oxidation tests at 347°, 500° and 600°F. in a naphthenic mineral oil. These phosphorus additives show a substantial increase in dirt formation in comparable oxidation tests at 500° and 600°F. in diester base stocks. The use of Acryloid 966 is also effective as a dispersant to reduce dirt formation in 347° and 500°F. oxidation tests with naphthenic mineral oil blends containing tricresyl phosphate or diisopropyl acid phosphite.

Esters, at 600°F. and above in thermal tests, and at 500°F. and above in severe oxidation tests, corrode SAE 1010 steel conventionally used in Spec. MIL-L-7808 oxidation tests. This appears to be a characteristic of esters of dibasic acid and mono-functional alcohols as well as esters of di- or poly-functional alcohols and monobasic acids.

# Contrails

In view of the severe corrosion noted in thermal tests, this difficulty appears to stem from the mono- or di-functional organic acid. Additional corrosion tests under thermal and oxidative conditions indicate that 52-100 bearing steel is corroded under the same conditions and to about the same degree as SAE 1010 steel.

Stainless steel and M-10 tool steel are not significantly corroded or attacked under these same thermal and oxidative conditions.

Corrosion of the SAE 1010 and 52-100 bearing steel by the ester type fluids shows up as severe surface pitting. This type of corrosion may be a factor in the relatively rapid failure rate of heavily loaded anti-friction bearings and gears running at elevated temperatures with Spec. MIL-L-7808 ester lubricants.

A PRL thin film oxidation test has previously been used to demonstrate correlation of fluid behavior in a laboratory test and in jet engine bearings. This PRL thin film test produces the same type of deposits observed in the panel coker. About the same level of deposits are formed at 500°F. in this test with ester-base fluids of the Spec. MIL-L-7808 type and conventionally refined mineral oil fractions. A comparison of depositions at 600°F. in this thin film test shows less deposits for well refined mineral oils compared with Spec. MIL-L-7808 type ester fluids. This behavior appears to be related to the thermal stability of the respective fluids. The advantage to be gained by eliminating or restricting the amount of air in contact with fluids and lubricants at these operating temperatures is again pointed out by these tests.

Cooperative calibration studies on the Model C panel coker indicate that the severity of the unit used by this Laboratory is essentially the same as those units used by the Power Plant Laboratory of WADC.

A controlled atmosphere panel coker has successfully been built and operated. In terms of oil consumption and recycle of oxidation and thermal decomposition products, this unit more nearly simulates jet engine behavior than the Model C coker. This controlled atmosphere coker is capable of demonstrating the thermal and oxidative components of deposit formation. In general, for a given set of conditions in the controlled atmosphere panel coker, ester-base fluids of the Spec. MIL-L-7808 type show somewhat more severe deposit formation than in the Model C coker, while mineral oil-base fluids show less severe coking.

Trace quantities of phosphoric and phosphorous acid in Spec. MIL-L-7808 composition can materially affect the panel coker values. The type of oxidation inhibitor can alter markedly the coking tendencies of esters containing acidic phosphorus additives. That is, dibasic acid ester-phenothiazine blends show increased coking with increasing phosphoric or phosphorous acid concentration; dibasic acid ester-Paranox 441 blends do not show increased coking in the presence of phosphoric or phosphorous acid.

Data obtained for a number of mineral oil fractions indicate no apparent correlation between panel coker values and Conradson carbon



values (ASTM Method D189-52).

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Blends of a mineral oil with esters of varying thermal stability give panel coker values which are directly proportional to the thermal stability of the ester component. Blends of additives and mineral oils with a polyolefin synthetic hydrocarbon give panel coker values in excess of those obtained by testing the components neat.

Panel coker results for the 12 large blends of mineral oils and hydrocarbons indicate that the large blends are typical of the mineral oils and hydrocarbons previously evaluated in the cokers. The conventionally refined mineral oils and mineral oil-hydrocarbon blends have Model C coker values somewhat higher than the Spec. MIL-L-7808 limits. Both the polyolefin and the hydrogenated polyolefin show very low panel coker values in both cokers.

Spec. MIL-L-7808 fluids show an excessive increase in coking when the panel temperature is raised to 700°F. Conventionally refined mineral oils show little change in coker values between 600° and 700°F. panel temperatures. The volatility of the mineral oil test fluid has been shown to have a marked effect on panel coking values. In general, the lower the volatility level (the higher the viscosity grade or molecular weight) the lower the panel coking value.

The PRL high temperature fuel rig has been effectively modified to a one-pass lubricant rig. This unit has been used for evaluating lubricants under oxidative conditions on a 700°F. metal surface. Tube deposit as well as fluid deterioration can be determined with the unit. Mineral oils, synthetic hydrocarbons, and pentaerythritol esters show good overall properties with respect to tube deposits and fluid deterioration in preliminary tests.

Spec. MIL-L-7808 fluid shows a high tube deposit but little evidence of sludge in the exit oil. The silicate-base fluid and improved lubricity silicones produce substantial quantities of sand-like non-combustible deposits on the tube and in the exit oil. Volatility has been shown to be an important factor in the behavior of fluids in this rig. Hydrocarbons and mineral oils boiling substantially below 700°F. produce little deposit on the tube. It is indicated that vapor-phase oxidation dominates under these conditions producing less coke and more water and gaseous products of oxidation.

Dispersant type polymeric additives have little or no effect on the level of deposit formation of mineral oils in this rig test. The alkyl acid phosphite lubricity additives do, however, have a substantial adverse effect on deposit formation of mineral oils.

Air rate is a critical variable controlling test severity in this one-pass rig. Data obtained at 30 liters of air per hour tend to magnify the smaller differences observed at one and ten liters of air per hour. In all cases, increasing air rate produces increased deposition on the heat exchanger as well as increased evidence of deterioration in the used oil.

Thermal stability tests in the PRL pressure cylinder have been modified to contain metal catalysts. Tests at 700°F. under a nitrogen atmosphere show evidence of some metal corrosion (either bronze or steel) for all of the test fluid types except the hydrogenated mineral oils and hydrocarbons. Tests at 700°F. in the pressure cylinder adequately demonstrate thermal deterioration of all the materials tested to produce volatile products. These volatile products are, in almost all cases, of a highly inflammable character.

The thermal stability of a series of paraffinic mineral oils appears to be a function of the molecular weight of the mineral oil fractions. That is, the higher molecular weight fractions of a conventionally refined mineral oil type appear to be less stable thermally than the lower molecular weight fractions. Additional refining (hydrogenation) causes no significant change in the thermal stability of a naphthenic or a paraffinic mineral oil. Partially or completely hydrogenated high molecular weight aromatic fractions from mineral oil show promise as stable high temperature hydraulic fluids where primary emphasis is placed on thermal and viscosity stability at high temperatures with only secondary emphasis on oxidation stability.

Thermal stability studies on hydrogenated and unsaturated polyolefins indicate no appreciable difference in thermal stability. Narrow boiling distilled fractions of polyolefins show no improvement in thermal stability over non-distilled products. The use of different olefins as starting materials for the preparation of polyolefins appears to effect larger changes in thermal stability than does hydrogenation or distillation of the polymer products.

Density determinations with a Lipkin pycnometer of the type specified in ASTM Method D941-49 appear adequate for fluids of adequate thermal stability and vapor pressures of 300 mm. Hg or less at the test temperature. Density determinations over the temperature range of 100° to 500°F. have been determined without difficulty. The Lipkin pycnometer can be conveniently used in a high temperature vapor bath of the type previously described for high temperature viscosity measurement.

The density-temperature function appears to be essentially a straight line for all of the fluid types evaluated over the range of 100° to 500°F. These data indicate that an adequate extrapolated value for high temperature density may be determined from density values at lower temperatures (e.g. 68° and 100°F. or 100° and 210°F.).

Vapor pressure measurements have been made successfully on five silane fluid samples in the PRL vacuum equilibrium still. These vapor pressure studies indicate that these silanes are not thermally stable in the 650° to 750°F. range.

Foaming can be eliminated in mineral oils and synthetic hydrocarbons of the type considered for use as high temperature fluids and lubricants through the use of a high molecular weight methyl silicone anti-foam agent. The persistence of the anti-foam characteristics with



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fluid use at high temperature needs investigation. When fluid life is expected to exceed the induction period of the fluid, anti-foam properties are necessary to reduce air-oil contact area and contact time which influence oxidation rate.

Lubricity tests in the Shell four-ball wear tester at 700°F. indicate that diisopropyl acid phosphite is still an effective lubricity additive for mineral oils. Non-additive mineral oils show wear values at one and ten kilograms load at 700°F. with M-10 balls comparable to those obtained at the same loadings with 52-100 steel balls at 167°F. The 40 kilogram wear values for non-additive mineral oils at 700°F. are relatively high.

Special techniques have been developed for adding make-up fluid to the four-ball pot during the test. This technique is useful for evaluating fluids which show a high volatility loss or excessive thermal breakdown to volatile fragments. Lubricity values for the improved lubricity silicone, Aroclor 1248, and the silicate ester-base fluids at 700°F. are about the same or slightly better than the 600°F. values.

Oronite High Temperature Hydraulic Fluid 8515 shows essentially the same behavior in the PRL high temperature pump test unit at 400° and 500°F. as that obtained previously with silicate fluids MLO 8200 and O.S. 45. That is, the formation of colloidal metal, a relatively large decrease in flow rate, and excessive valve plate and cam ring wear are noted for all three of these fluids. Under the same test conditions, di-2-ethylhexyl sebacate shows satisfactory operation in the pump.

A suitable method for the preparation of diisopropyl acid phosphite has been used to prepare sufficient material for incorporation in the test program on lubricity.

A total of 12 one-hundred gallon blends of conventionally refined mineral oils and synthetic hydrocarbons have been prepared for mock-up and engine testing. The test program for these large scale blends should provide additional correlation between small scale laboratory tests and end use applications for high temperature jet engine lubricants. Some of the principles involved in this series of blends are (1) the effectiveness of anti-foam additives, (2) the effectiveness of oxidation inhibitors in conventionally refined mineral oils and a paraffinic hydrocarbon, (3) the effect of hydrogenation, (4) the use of an olefinic hydrocarbon neat and in mineral oil blends as an oxygen acceptor, and (5) the effect of mineral oil type on high temperature behavior.

Used fluids from J-57 engine tests with Grade 1065 mineral oil and phenothiazine-inhibited di-2-ethylhexyl sebacate have been evaluated. Property changes in the used engine oils indicate more oxidative deterioration in the engine at 250° and 300°F. bulk oil temperatures than is predicted by bulk oil oxidation tests at 347°F. of the Spec. MIL-L-7808 type. There is more evidence of actual oxidative deterioration in the used mineral oil samples than in the used ester samples. The used mineral oils show superior high temperature oxygen tolerance to the used ester

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fluids. This behavior between the two fluids can also be shown in the 500°F. successive oxidation tests.

Panel coker tests at 600°F. and thermal stability tests at 500°F. are not effective in reducing oxidation stability of the ester fluid to the extent noted in the used engine oils. A 24-hour oxidation test at 400°F. and six-hour oxidation tests at 500°F., do reduce stability to subsequent 347°F. oxidation and corrosion tests in the same general fashion as noted with the used engine oils. Trends noted in 500°F. successive oxidation and corrosion tests, and in 347°F. oxidation of samples pretreated by a 500°F. oxidation test suggest a cause for the relatively high rate of sludging in the early stages of a typical J-57 engine test with Spec. MIL-L-7808 fluid at high oil inlet temperatures.

Evaluation of a commercial Spec. MIL-L-6387 fluid in a high temperature hydraulic unit shows the same order of magnitude of useful life as that which can be predicted from laboratory thermal stability tests at 347°F. The use of di-2-ethylhexyl sebacate as a base stock for a hydraulic fluid of the Spec. MIL-L-7808 engine oil type offers considerable advantage in useful life over the current Spec. MIL-L-6387 fluid on the basis of Laboratory bench tests.

U. FUTURE WORK. Essentially all of the tests utilized by this Laboratory for the evaluation of fluids and lubricants in the high temperature range have been developed by this Laboratory as a portion of the previous test program. Further efforts will be directed toward the reduction of these test procedures to a significant reproducible test suitable for adoption as a standard test technique. Tests for which such standard test techniques can be evolved on a quantitative basis from the preliminary studies conducted, thus far, are:

1. Viscosity
2. Density
3. Thermal Stability
4. Vapor Pressure
5. Rate of Oxidation
6. Oxygen Tolerance
7. Lubricity
8. Metal Corrosion

The choice of final conditions of test severity for rate of oxidation, metal corrosion, oxygen tolerance, and lubricity will require further test data from actual high temperature hydraulic fluid and jet engine lubricant applications. A continuing series of high temperature stability tests involving combined functions of oxidation, thermal deterioration, and metal corrosion will be studied in this program. These tests include the following procedures:

1. Thin film oxidation test
2. Successive oxidation test
3. Controlled atmosphere panel coker
4. Single pass high temperature lube rig

# Contrails

These test techniques are designed to simulate certain portions of a lube system and/or methods of lubricant use not common in current practice, such as a single or limited pass lube system. The pertinence of these tests can not definitely be established until practical high temperature lube systems exist at least in prototype equipment. Results of these types of tests are of considerable exploratory value in emphasizing certain advantages and disadvantages among the various high temperature lubricant types. Such test techniques will be developed further.

Base stock studies, thus far, have shown that hydrocarbons, mineral oils, esters, silicones, silicates, and chlorinated aromatic hydrocarbons give promise as high temperature fluids and lubricants. Emphasis will be placed on the further study of improved stability of hydrocarbons and esters as a function of molecular structure, and improved mineral oil stability as a function of additional refining techniques, such as extraction, hydrogenation, and adsorption.

Certain desirable structural features of esters and synthetic hydrocarbons are evident from the results of this current study. Attempts will be made to obtain esters and synthetic hydrocarbons with desirable structures for good thermal stability and high oxygen tolerance.

Conventionally refined mineral oils show good high temperature properties but lesser stability to oxidation than Spec. MIL-L-7808 fluids at 350°F. Additional refining techniques, including hydrogenation, silica gel adsorption, and new additive combinations, yield mineral oils of improved inhibitor susceptibility for moderate temperatures (350°F.) and good high temperature stability. These additional refining processes will be applied to a variety of mineral oil types in an attempt to determine the extent of base stock improvement that can be obtained.

Emphasis will also be placed on the hydrogenation of certain aromatic mineral oil fractions which can be obtained by solvent extraction. These aromatic extracts exhibit excellent viscosity stability at 700°F. and may be desirable as high temperature hydraulic and radiation stable fluids and lubricants.

Most of the studies on high temperature fluids, thus far, have been concentrated on base stock materials or base stock plus additive for one specific test, i.e. lubricity, oxidation rate, corrosion, or oxygen tolerance. Emphasis in the future program will be directed to the complete evaluation of finished compositions including one or more base stocks with additives.

Specific studies have shown that base stock blends of improved oxygen tolerance can be prepared from a good oxygen acceptor and a good solvent for oxidation products.

Additive studies have shown that lubricity can be improved at 700°F., oxidation rate can be reduced for a time at high temperature, oxidation and corrosion can be effectively prevented at 350°F., foaming can be eliminated or reduced, and sludging tendencies can be reduced

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(sludge dispersant additive) at 350° and 500°F. Complete evaluations of formulations encompassing these desirable effects will be conducted to determine the limitations as well as the advantages of various additives and base stock combinations.

As a result of these high temperature studies, departure from current hydraulic and lubricant system design and use has been suggested to take full advantage of fluid and lubricant properties. Some of these suggestions include:

1. A two oil system comprising one oil for start up and a second oil for high temperature operation.
2. Lubricant dilution for start up plus regular lubricant for running.
3. Oil cooling plus oil lubrication plus oil expended overboard or to the burners.
4. Oil vaporization for cooling plus oil lubrication plus oil expended overboard or to the burners.
5. Water or halocarbon cooling plus oil lubrication plus oil expended overboard.
6. Halocarbon blanketing plus oil cooling plus oil lubrication plus oil expended overboard.

High temperature evaluations involving these various combinations of components will be made using test devices and techniques discussed previously.

In order to understand better the problems involved in high temperature hydraulic and jet engine lube systems, better integration of system design and prototype testing with fluid and lubricant development is desired. This Laboratory plans to cooperate with the Wright Air Development Center, jet engine manufacturers, and aircraft component industries in evaluating pertinent used fluid samples of established history from high temperature hydraulic and jet engine lube systems. Cooperation of this sort is the only way available to the research laboratory to follow fluid development into practical applications and consequently determine the effectiveness of laboratory evaluation techniques in the development of new fluids. Thus far, this kind of cooperation has provided valuable data on the degree of lubricity required and the deterioration encountered in existing hydraulic and jet engine systems. Further cooperative studies can profitably be made.

In addition to the laboratory evaluation of used commercial fluids from full scale equipment, the fluids which have been exhaustively evaluated in the laboratory should also be evaluated in mock-ups and service equipment on an expanded scale. With adequate cooperation on the part of the Air Force and industry this portion of the program will be continued and expanded.

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II. MISCELLANEOUS

A. TECHNICAL PAPERS AND REPORTS. Several reports and technical papers have been prepared during the period covered by this report. Some of these have been presented at technical meetings of various organizations.

1. WADC Technical Reports. Two WADC Technical Reports have been issued. The first of these, designated as WADC Technical Report 56-224, is entitled "High Temperature Lubricant Studies" and is dated May 1956. The material included in WADC TR 56-224 was presented as a paper at the Annual Meeting of the American Society of Lubrication Engineers at Pittsburgh, Pennsylvania in April 1956. The following two paragraphs comprise the abstract taken from WADC TR 56-224.

"Basic information on the behavior of hydraulic fluids and lubricants is needed at temperatures above 400°F. if successful operation of engines and hydraulic systems is to be achieved at high temperatures. It is necessary first to develop reliable test procedures and techniques covering the measurement of viscosity, lubricity, corrosion, and oxidation and thermal stability at temperatures of 500° to 700°F. Several fluids were evaluated in this manner. These fluids are representative of the different chemical classes, such as silicones, silicates, hydrocarbons and mineral oils, diesters, and halogenated aromatic hydrocarbons.

"It was found that the property differences between the various types of fluids at 500° to 700°F. were much narrower than exist in the 200° to 350°F. range. Data are presented showing the inadequacy of extrapolating 200° to 350°F. data as a means of predicting performance at 500° to 700°F. The behavior of fluids and lubricants in the 200° to 350°F. range has been quite well defined in recent years for the materials used in this study. However, there is a paucity of information at 500° to 700°F. This paper attempts to remedy somewhat this situation."

The second of these reports, designated as WADC Technical Report 56-254, is entitled "Mineral Oils as High Temperature Fluids and Lubricants" and is dated June 1956. This report summarizes the portion of the work relative to mineral oils conducted by this Laboratory from October 1951 through December 1955. The following paragraphs comprise the abstract taken from WADC TR 56-254.

"In a survey of fluids and lubricants for use at 400° to 700°F., certain types of mineral oils were found to be among the most promising materials studied. While synthetic lubricants have better performance characteristics than most mineral oils and hydrocarbons in the moderate temperature range (200° to 400°F.), they do not possess this margin of superiority in the high temperature range (400° to 700°F.). Certain synthetics have better viscosity-volatility and viscosity-temperature characteristics than hydrocarbons.



# Contrails

"Some mineral oils and diesters of the Spec. MIL-L-7808 type have good lubricity properties at high temperatures. Using a hydraulic pump and a laboratory wear tester, it has been found that there are lubricity additives that are still effective at 500° to 700°F. in mineral oils and diesters.

"Mineral oils and certain types of hydrocarbons have better thermal stability and oxygen tolerance than many synthetics at 600° to 700°F. The concept of oxidation stability, or inertness toward oxygen, is not normally applicable at these temperatures. Oxidation rate and oxygen tolerance are better criteria for judging oxidative deterioration at 500° to 700°F.

"It appears that some additives and additional refining techniques (such as hydrogenation) will be beneficial in improving the oxidation behavior of mineral oils and certain other hydrocarbons at moderate and high temperatures.

"The thermal and oxidative components of high temperature coking on metal surfaces are demonstrated in a controlled atmosphere panel coker. It may be necessary to control the atmosphere in engine parts requiring lubrication at 500° to 700°F. because of the possibility of spontaneous ignition of the lubricant or of fragments from its decomposition.

"In a lubrication system where the oil is used for a limited time and then discarded (perhaps to the burners) it is necessary to know the effect of contaminating the fresh oil with oxidized or deteriorated oil. Data have been obtained indicating that this effect need not be serious with esters and mineral oils at 500°F."

2. Paper Concerning ASTM Slope. A paper entitled "The Use of ASTM Slope for Predicting Viscosities" by E. E. Klaus and M. R. Fenske has been published in the ASTM Bulletin (ASTM Bulletin 215, 87, July 1956). This paper was prepared for presentation at the Symposium on Viscosity-Index Systems sponsored by the American Society for Testing Materials in February at Dallas, Texas. The report is designated by this Laboratory as P.R.L.-5-56. This paper is believed to be of general interest in the field of military hydraulic fluids and lubricants, since the viscosity-temperature system proposed in this paper is suitable for use over the temperature interval of -65° to 700°F. Most conventional viscosity-temperature systems are tied to measured viscosities at 210° and 100°F. The following paragraphs comprise the abstract from report P.R.L.-5-56.

"A great number of methods for expressing the viscosity-temperature characteristics have been proposed, but there are always shortcomings in application or measurement. The ASTM slope and viscosity index are compared here with percentage change of viscosity with temperature, and the advantages and shortcomings of ASTM slope are discussed. Methods are presented for measuring kinematic viscosities over the range of 700° to -65°F.

"Data are given showing the limitations of the ASTM viscosity-temperature chart over this temperature range. The fluids studied were

mineral oils, esters of organic and inorganic acids, and polymer-containing oils. The errors obtained by using ASTM slope and the ASTM viscosity-temperature chart to obtain viscosities by interpolation and extrapolation are discussed, and methods for eliminating these difficulties are given. The use of measured viscosities at the temperature extremes encountered in a specific application is recommended."

### 3. Paper Concerned with Chemical Structure and Lubrication.

A paper entitled "Chemical Structure and Lubrication" by E. E. Klaus and M. R. Fenske has been prepared for presentation at the Symposium on Additives in Lubricants sponsored by the Petroleum Division, American Chemical Society in Atlantic City, New Jersey in September 1956. This paper is designated as report P.R.L.-9-56. The following comprises the abstract from report P.R.L.-9-56.

"The three types of lubrication, boundary, hydrodynamic, and quasi-hydrodynamic, are illustrated using three hydraulic pumps and the Shell four-ball wear machine. Usually correlations between the wear on pump parts can be made with the wear scars at 1 or 10 kilograms from the wear machine. This machine has been modified to permit wear tests at temperatures up to 700°F.

"Tricresyl phosphate is an effective anti-wear agent in some ester type lubricants and in mineral oils. However, the nature of the base stock influences considerably its effectiveness, probably because other components are competing for the bearing surfaces. This is illustrated using various esters and other components such as anti-rust and detergent additives.

"Examples are given of chemically corrosive additives in contrast to chemical polishing agents as well as the effects on lubricity of fluids differing widely in chemical structure.

"Wear data are presented for the vane pump and four-ball machine at temperatures of 500° to 600°F."

4. Paper for Air Force-Navy-Industry Conference. A paper entitled "Studies of Hydraulic Fluids and Lubricants at Elevated Temperatures" was prepared for presentation at the Air Force-Navy-Industry conference on aircraft lubricants at San Antonio, Texas on November 5-7, 1956. Preprints of this paper were made and copies sent to the Wright Air Development Center, WCRTR-1 in October 1956.

B. PREPARATION OF EXPERIMENTAL BLENDS. A number of large blends have been prepared and distributed during the period covered by this report. The preparation of these blends was requested by the Wright Air Development Center. The formulations prepared include:

1. Two mineral oil hydraulic fluid formulations (25 gallons each) suitable for use at 600° to 700°F. in a hydraulic system mock-up,



2. A 10-gallon sample of GTO 120, a high lubricity fluid meeting the Spec. MIL-L-7808 requirements,
3. A total of twelve 100-gallon mineral oil and synthetic hydrocarbon blends for evaluation by the Power Plant Laboratory, Wright Air Development Center, and
4. A 225-gallon sample of a chlorinated aromatic hydrocarbon type fluid.

The blends listed in item three above have been discussed previously in this report. The chlorinated aromatic hydrocarbon (item four) was procured from the commercial supplier and shipped to the Wright Air Development Center without blending by this Laboratory.

Several smaller blends have also been prepared and shipped to various organizations.

1. Experimental Hydraulic Fluids. Thirty (30) gallons of each of two experimental hydraulic fluids have been prepared. These fluids are designated as MLO 7116 and MLO 7117. Twenty-five (25) gallons of each of these fluids have been shipped to Mr. Don Reinke of the Sundstrand Aviation Corporation, Rockford, Illinois. The remaining five (5) gallons of each fluid has been sent to the Materials Laboratory, WCRTR-1 of the Wright Air Development Center.

It has been indicated that these fluids are to be tested by the Sunstrand Corporation under Contract AF33(616)3126 at temperatures up to 650° to 700°F. Some of the pertinent physical properties of these fluids are shown in Table 133.

Table 133

PHYSICAL PROPERTIES OF TWO EXPERIMENTAL HYDRAULIC FLUIDS

Properties	MLO 7116	MLO 7117
Centistokes Viscosity at		
700°F.	-	0.5
500°F.	2.1	1.1
350°F.	4.7	2.4
210°F.	19.3	9.7
100°F.	179	110
32°F.	2700	2500
ASTM Slope (210° to 100°F.)	0.607	0.776
Flash Point, C.O.C., °F.	390	430
Fire Point, C.O.C., °F.	430	490
Pour Point, °F.	-25	-10
Neut. No.(mg. KOH/g. Oil)	0.1	0.1
Specific Gravity at 60°/60°F.	0.89	0.90

MLO 7116 and MLO 7117 are primarily narrow boiling fractions of naphthenic mineral oils. The two oils differ in composition in that MLO 7116 contains a small quantity of a polybutene thickener in addition to the oxidation inhibitor, while MLO 7117 contains only an oxidation inhibitor. Mineral oils of this general type show good thermal stability under a nitrogen atmosphere at temperatures up to about 700°F. In the region of 700° to 750°F., these mineral oils produce a small amount of volatile products and gas. The residue, however, is a liquid and is free from sludge in the six-hour thermal stability tests shown on Table 134. The large viscosity loss appears to be due to the presence of a relatively small amount of volatile material formed by pyrolysis.

It will be noted from the data on Table 134 that fluids of the MLO 7116 type are not as good as MLO 7117 in thermal stability at temperatures of 600°F. and above. This difference in thermal stability is due to thermal effects on the polymeric additive. There are no indications that undesirable solids or deposits are formed with either composition in a thermal stability test up to 750°F.

The above discussion of fluids MLO 7116 and MLO 7117 relates to thermal stability in the absence of oxygen. Data showing the effects of a limited amount of oxygen at 500°F. on the properties of these fluids are shown on Table 135. Oxygen absorption of 0.2 mole per mole of fluid is equivalent to a minimum volume of approximately 30 cubic feet of air at 0°F. and 760 mm. Hg pressure in a system containing a five gallon charge. These values assume that all of the air reacts with the fluid. This requires intimate air-oil mixing for extended periods of time. The data on Table 135 indicate that small quantities of oxygen in the system do not result in serious corrosion, viscosity increase, or sludge formation for either of the mineral oil compositions.

All of the fluids and lubricants evaluated in high temperature stability tests tend to produce some small fragments on thermal decomposition. This phenomenon is not limited to esters and mineral oils. It is, therefore, advisable to consider inflammability and explosive vapor-air mixtures from the standpoint of these decomposition fragments.

2. GTO 120 Gear Lubricant. Ten (10) gallons of GTO 120, a high load carrying oil, have been prepared and sent to the Power Plant Laboratory, WCLPF of the Wright Air Development Center. GTO 120 is a high lubricity fluid which meets the Spec. MIL-L-7808 requirements and which gives Ryder gear values in excess of 3000 pounds per inch. It has been indicated that this batch of GTO 120 is to be used for further rig tests at the Power Plant Laboratory.

3. Fluid Samples Distributed. A one-quart sample of GTO 38 has been returned to the Power Plant Laboratory, Wright Air Development Center. GTO 38 is a fluid used for interlaboratory calibration of the Model C panel coker. Calibration studies conducted by this Laboratory are discussed in the section of this report pertaining to panel coker studies. This sample was returned to the Power Plant Laboratory in order that coker calibration might be checked with a specific sample of the fluid.

Table 134

THERMAL STABILITY CHARACTERISTICS OF SOME EXPERIMENTAL HIGH TEMPERATURE HYDRAULIC FLUIDS

Thermal stability tests have been conducted in all glass tubes under a nitrogen atmosphere. The tubes are sealed with a U-tube containing the test fluid. The seal allows the passage of gas out or in with a pressure drop across the U-tube of more than 4 inches of the fluid.

TEST FLUID DESIGNATION	MLO 7116 MINERAL OIL -- BASE HYDRAULIC FLUID --					MLO 7117 NARROW BOILING MINERAL -- -- OIL FRACTION -- --				
TEST TEMPERATURE, °F.	500	600	650	700		600	650	700	750	
TEST TIME, HRS.	20	6	6	6		6	6	6	6	
WT. OF FLUID CHARGED, GMS.	25	100	25	100		45	25	100	25	
% LOSS IN WEIGHT	0	2	8	5		2	0	2	14	
NEUTRALIZATION NUMBER (MG. KOH/GM. FLUID)										
ORIGINAL	0.0	0.0	0.0	0.0		0.1	0.1	0.1	0.1	
FINAL	0.0	0.1	0.4	0.7		0.1	0.1	0.1	0.1	
% CHANGE IN CENTISTOKE VISCOSITY AT 100°F.	-7	-43	-69	-85		+1	-12	-40	-84	
APPEARANCE OF FLUID AFTER TEST	CLEAR	CLEAR	CLEAR	CLEAR		CLEAR	CLEAR	CLEAR	CLEAR	

Table 135

OXIDATION AND CORROSION CHARACTERISTICS OF SOME HIGH TEMPERATURE  
HYDRAULIC FLUIDS AT 500°F.

Procedures and techniques in accordance with Spec. MIL-L-6387

TEST CONDITIONS: TEST TEMPERATURE = 500 $\pm$  5°F.; TEST TIME = 24 HOURS; AIR  
RATE = 0.6 LITER/HOUR; CATALYST = ONE INCH SQUARES OF  
COPPER, STEEL AND ALUMINUM.

TEST FLUID	MLO 7116 MINERAL OIL-BASE HYDRAULIC FLUID	MLO 7117 NARROW BOILING MINERAL OIL FRACTION
LIQUID CHARGE, MOLES	0.20 <sup>(1)</sup>	0.20 <sup>(1)</sup>
MOLES OF O <sub>2</sub> SUPPLIED	0.12	0.12
MOLES OF O <sub>2</sub> USED	0.04	0.05
MOLES O <sub>2</sub> USED/MOLE FLUID	0.20	0.25
WEIGHT PER CENT LIQUID LOSS	0.0	1.1
PER CENT CHANGE IN VISCOSITY AT 130°F.	-43	+10
AT 0°F.	-	-
NEUT. NUMBER (MG. KOH/GM. FLUID)		
ORIGINAL	0.0	0.1
FINAL	1.1	1.2
WEIGHT PER CENT INSOLUBLE MATERIAL	TRACE	0.1
FINAL CATALYST CONDITION		
APPEARANCE		
COPPER	COATED	COATED
STEEL	DULL	DULL
ALUMINUM	BRIGHT	DULL
WEIGHT LOSS (MG./CM. <sup>2</sup> )		
COPPER	+0.05	+0.12
STEEL	0.05	0.03
ALUMINUM	0.01	0.00

(1) MOLECULAR WEIGHT OF THIS FLUID ESTIMATED TO BE 425.

# Contrails

One-pint samples of each of two experimental hydraulic fluids (MLO 7116 and MLO 7117) have been sent to Chance Vought Aircraft, Incorporated, Dallas 22, Texas. A discussion of the compositions and properties of these fluids is included in part B-1 above.

C. SHELL FOUR-BALL WEAR TESTS. Lubricity tests in the Shell four-ball wear tester have been conducted with three test fluids furnished by the Materials Laboratory, Wright Air Development Center (WADC). These fluids are designated by WADC as GTO 38, GTO 133, and MLO 9840. GTO 38 and GTO 133 have been evaluated under each of the test conditions shown in the table below. MLO 9840 has been tested under the first two conditions. The bearings used in all of the tests are SKF Grade No. 1 E.P. bearings (52-100 steel) of 0.5-inch diameter. Tests were conducted at loads of 1, 4, 10 and 40 kilograms.

	Spindle Speed, r.p.m.	Test Time, Hours	Test Temp., °F.      °C.	
(1)	620	1	167	75
(2)	1200	2	167	75
(3)	1200	2	400	204

Lubricity tests in the Shell four-ball wear tester for GTO 38, GTO 133, and MLO 9840 are shown on Table 136. Three test conditions were used in this study. The first set of test conditions includes: one hour test time, 75°C. (167°F.) test temperature, and 620 r.p.m. test speed. This is the standard test used by this Laboratory. These conditions were chosen on the basis of a study of variables conducted previously at this Laboratory. The 620 r.p.m. test speed was found to give the best reproducibility in this Laboratory's wear tester. Poor reproducibility at higher test speeds was caused, at least in part, by vibration.

The one-hour test time was chosen on the basis that, in all cases evaluated, the large percentage of the wear occurring in a two hour test had already occurred after one hour of running. The 75°C. (167°F.) test temperature was about the lowest test temperature which could be maintained for the test period over the entire load range without cooling. Since these test conditions are used in all standard PRL evaluations, they have also been included in this study.

In accordance with the WADC request, two hour tests at 1200 r.p.m. and 75°C. (167°F.) and 400°F. were also conducted.

The agreement between values obtained for one hour at 620 r.p.m. and two hours at 1200 r.p.m. can be seen from the data on Table 136. These data show that GTO 38 and GTO 133 exhibit excellent anti-wear properties. The silicone lubricant MLO 9840 shows very high wear at loads of 4, 10, and 40 kilograms. The behavior of GTO 38 and GTO 133 with increasing temperature is typical of dibasic acid esters containing tricresyl phosphate as the anti-wear additive.

Table 136

STUDIES WITH THE SHELL FOUR-BALL WEAR TESTER

Test Conditions of Time, Temperature, and Speed are Indicated.

Ball Bearings Used are SKF Grade 1 E.P. Bearings of 0.5 Inch

Diameter (PRL Batch # 12).

Test Fluids GTO 38, GTO 133, and MLO 9840 furnished by Materials Laboratory, WADC.

Test Fluid	- - - Average Wear Scar Diameter, mm. at - - - - -			
	1 Kg.	4 Kg.	10 Kg.	40 Kg.
1 Hour - 75°C. (167°F.) - 620 r.p.m.				
GTO 38	0.15	0.19	0.23	0.36
GTO 133	0.16	0.22	0.22	0.38
MLO 9840	0.32	0.75	1.43	2.92
2 Hours - 167°F. - 1200 r.p.m.				
GTO 38	0.18	0.21	0.25	0.36
GTO 133	0.18	0.19	0.22	0.49
MLO 9840	0.37	0.91	1.78	3.44
2 Hours - 400°F. - 1200 r.p.m.				
GTO 38	0.17	0.20	0.24	0.72
GTO 133	0.22	0.22	0.30	0.66

*Contrails*

D. EVALUATION OF SILICONE FLUIDS FOR NON-NEWTONIAN BEHAVIOR.

Four silicone fluids have been received from the Materials Laboratory at Wright Air Development Center. These fluids are designated as MLO 56-161, MLO 56-320, MLO 56-491, and MLO 56-492. It has been requested that these fluids be evaluated for non-Newtonian behavior at high rates of shear.

Two units are available at the Petroleum Refining Laboratory for non-Newtonian studies at high shear rates. These units are the PRL high shear viscometer and the PRL low pressure viscometer. Both of these units are capillary-type viscometers. The method used to supply the driving head differs for the two units.

In the PRL high shear viscometer, the fluid is circulated through the capillaries by means of a Hycon Model 67A-050 piston pump. The pressure drop through the capillaries is dependent on the flow rate of the pump.

In the PRL low pressure viscometer, the driving head is an inert gas pressure (nitrogen). In addition, the range of shear rates attainable differs for the two units. The PRL high shear viscometer can be used to obtain shear rates from 50,000 to 1,000,000 reciprocal seconds. The maximum shear rate attainable in the PRL low pressure viscometer is about 80,000 reciprocal seconds. Consequently, for non-Newtonian studies at high rates of shear the PRL high shear viscometer is normally used. However, in previous studies at this Laboratory, it has been found that silicone fluids do not have the lubricity requirements necessary to lubricate the piston pump over the pressure range required with this unit. For this reason, the non-Newtonian studies on the silicone fluids received from the Wright Air Development Center have been investigated using the PRL low pressure viscometer.

It has been demonstrated that for a given fluid at the same shear rate, the viscosity measured by the low pressure viscometer and by the high shear viscometer are identical. Further, methods for the extrapolation of non-Newtonian behavior as a function of shear rate have been used successfully and experimentally checked with the two units. This technique is illustrated on Figure 12 for PRL 2914 and a typical Spec. MIL-O-5606 hydraulic fluid.

The silicone fluids designated as MLO 56-161 and MLO 56-320 have been evaluated for non-Newtonian behavior at 0° and 100°F. in the PRL low pressure viscometer. The fluids designated as MLO 56-491 and MLO 56-492 were evaluated only at 100°F. The results of these determinations are found on Table 137.

The limits of error in these measurements are  $\pm 2$  per cent. On this basis, the results for the silicones indicate that they are essentially Newtonian in this range of shear rate or shearing stress. For purposes of comparison, the value for Spec. MIL-O-5606 hydraulic fluid for the same shearing stress is included on Table 137. It should be pointed out that recent studies indicate that non-Newtonian behavior is primarily a function of shearing stress rather than shear rate.



This comparison shows that for a shearing stress sufficient to produce 17 per cent viscosity decrease in a Spec. MIL-O-5606 fluid, essentially no viscosity decrease can be measured for the silicones.

E. FLUIDS FOR USE AS STANDARDS. A sample of Acryloid polymer prepared for use in the preparation of shear stability reference fluid Spec. MIL-F-5602 has been evaluated. Samples of several fluids have been distributed by this Laboratory for use as calibrating fluids on an inter-laboratory basis. Two fluids have been distributed as calibration fluids for the Shell four-ball wear tester and one fluid as a low temperature viscosity standard for viscosity determinations at -40° and -65°F.

1. Shear Stability Reference Polymer. A sample of HFS-5 Acryloid for use in the preparation of shear stability reference fluid Spec. MIL-F-5602 has been received by this Laboratory for shear stability tests. This material was prepared by the Rohm and Haas Company. Permanent shear tests in the PRL Hycon pump test unit have been conducted with Spec. MIL-F-5602 type fluids prepared from HFS-4 and HFS-5 polymeric thickener. Results of these tests using a sharp edge orifice loading device are shown on Table 138. The data were measured at 800 and 1500 p.s.i. pressure drop across the orifice in tests of 5000 cycles duration. Comparable values for a Spec. MIL-O-5606 fluid (PRL 2286) are also shown as a basis of comparison. Values for viscosity decrease versus pumping time for the two reference fluids are plotted on Figure 13.

The results show that a typical commercial Spec. MIL-O-5606 fluid and a shear stability reference fluid based on HFS-4 polymer give about the same permanent viscosity loss under identical test conditions. The limits of reproducibility in this type of test are considered to be  $\pm 1.5$  percentage units in viscosity decrease. The values obtained for the fluid prepared with HFS-5 polymer are slightly higher than similar values for the fluid formulated with HFS-4. Over the pressure range covered, the HFS-5 type reference fluid shows less shear stability than a typical Spec. MIL-O-5606 fluid by a factor equal to approximately twice the limits of error in this test procedure.

On the basis of these tests it has been recommended that Acryloid HFS-5 be qualified for use in the preparation of Spec. MIL-F-5602 shear stability reference fluid.

2. Standard Fluids for Wear Tester. A one-pint sample of each of two fluids (PRL 3207 and PRL 3462) has been sent to Mr. F. C. Ferris, Chemistry Department, Ford Motor Company, Dearborn, Michigan. These fluids represent a good non-additive lubricant and a lubricant containing an anti-wear additive. PRL 3207 and PRL 3462 have been distributed to several laboratories over the past few years and interlaboratory reproducibility has been good.

3. Low Temperature Viscosity Standard. This Laboratory has furnished a four-ounce sample of PRL 2815 low temperature viscosity standard to each of the following organizations. PRL 2815 is calibrated as a standard for viscosity determinations at -40° and -65°F. The primary

# Contrails

purpose of this standard is the establishment of a common temperature scale for viscosity determinations at these low temperatures.

Douglas Aircraft Co., Inc.  
Santa Monica, California

Royal Lubricants Company (2 samples)  
Hanover, New Jersey

Eastman Chemical Products, Incorporated  
Kingsport, Tennessee

Dr. M. Fainman  
Standard Oil Company of Indiana  
Whiting Research Laboratories  
P. O. Box 431  
Whiting, Indiana

Allison Division  
General Motors Corporation  
Plant No. 5  
Indianapolis, Indiana

Commander  
Wright Air Development Center  
ATTN: WCRTR-1  
Wright-Patterson Air Force Base, Ohio

Baker Caster Oil Company  
35 Avenue A  
Bayonne, New Jersey

Figure 12

VISCOSITY-SHEAR CHARACTERISTICS FOR TWO POLYMER FLUIDS AT 100°F.

TEST FLUIDS: PRL 2914, A POLYMER SOLUTION CONTAINING 4.7 WT.-% ACRYLOID HF-160 IN A MINERAL OIL.

● = VALUES OBTAINED IN PRL HIGH SHEAR VISCOMETER.

○ = VALUES OBTAINED IN PRL LOW PRESSURE VISCOMETER.

PRL 2286, A COMMERCIAL SPEC. MIL-O-5606 HYDRAULIC FLUID.

□ = VALUES OBTAINED IN PRL HIGH SHEAR VISCOMETER.

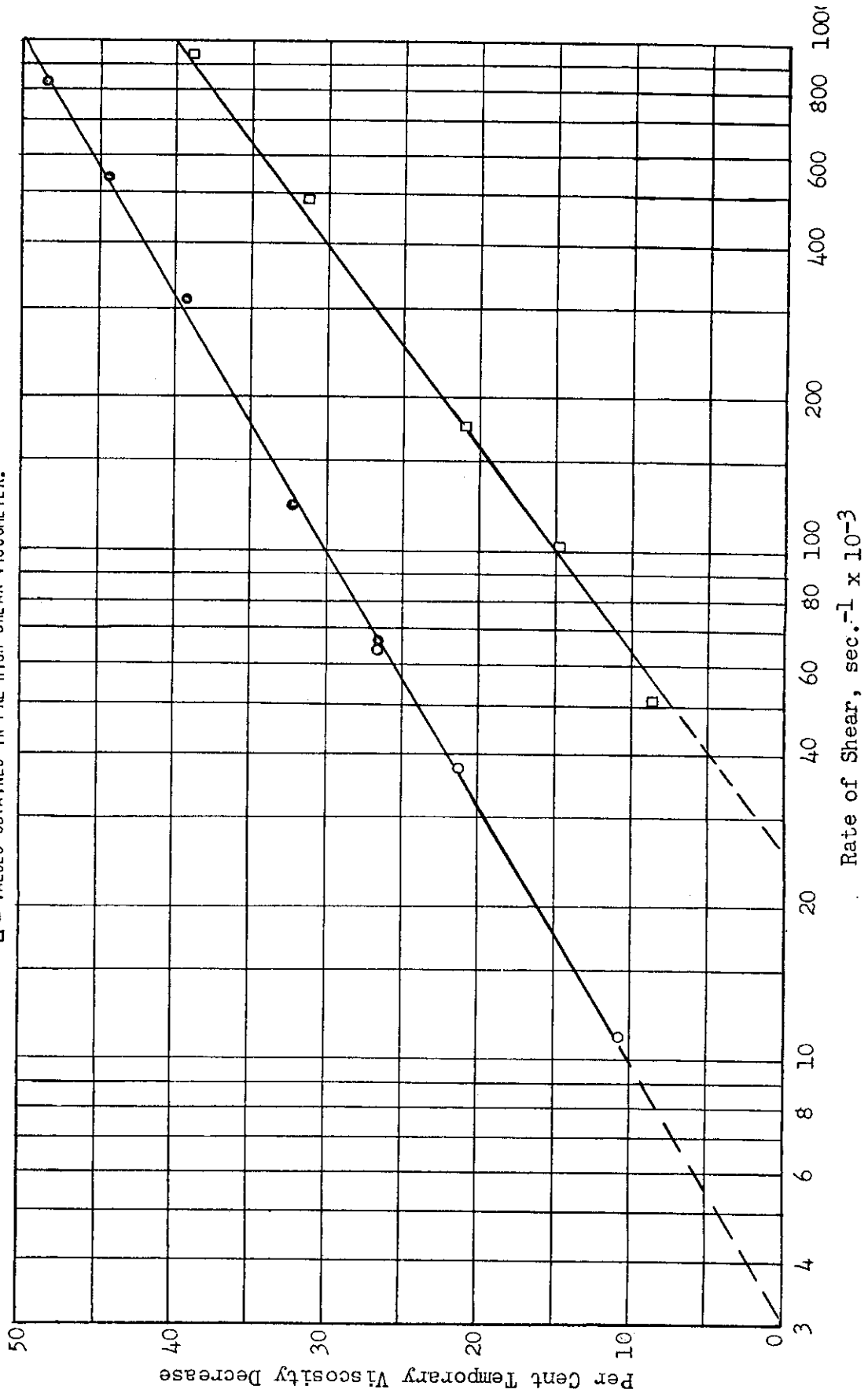


Table 137

## VISCOSITY-SHEAR CHARACTERISTICS OF SEVERAL SILICONE FLUIDS

SILICONE FLUIDS EVALUATED IN THE PRL LOW PRESSURE VISCOMETER.

SPEC. MIL-0-5606 HYDRAULIC FLUID EVALUATED IN THE PRL HIGH SHEAR VISCOMETER.

TEST FLUID	TEMP., °F.	LOW SHEAR VISCOSITY CENTISTOKES	VISCOSITY CENTIPOISES	APPARENT VISC., CENTIPOISES	% VISC. DECREASE	PRESSURE DROP ACROSS CAPILLARY, P.S.I.	SHEARING STRESS, DYNES/CM. <sup>2</sup> (1)	RATE OF SHEAR, SEC. <sup>-1</sup> (2)
SILICONE FLUID DESIGNATION MLO 56-161	0	199	208	207	0.5	295	13,900	6,700
	100	30.4	30.9	31.0	+0.3	295	14,300	46,000
MLO 56-320	0	102	102	102	0.0	295	13,600	13,600
	100	25.8	25.0	24.3	2.8	295	14,300	58,600
MLO 56-491	100	44.3	47.2	46.7	1.2	295	14,000	30,000
MLO 56-492	100	40.2	43.4	42.2	2.8	295	14,200	33,600
COMMERCIAL SPEC. MIL-0-5606 HYDRAULIC FLUID	100	14.2	11.8	9.80	17	--	14,000	120,000

(1) CALCULATED FROM NEWTON'S LAW:  $F/A = \mu \frac{dv}{dx}$ WHERE  $F/A$  = SHEARING STRESS IN DYNES PER SQ. CM. $\mu$  = VISCOSITY IN POISES. $\frac{dv}{dx}$  = RATE OF SHEAR, SEC.<sup>-1</sup>.

(2) THE RATE OF SHEAR DESIGNATED IS THE MAXIMUM RATE OF SHEAR AT THE CAPILLARY WALL. THIS VALUE IS APPROXIMATELY TWO TIMES THE AVERAGE RATE OF SHEAR EXISTING THROUGHOUT THE CAPILLARY CROSS SECTION.

Table 138

## PERMANENT VISCOSITY DECREASE DUE TO SHEAR

All Tests Conducted at 100°F. (Pump Inlet Temperature) in the PRL Hycon Pump Test Unit. In all Cases the Flow Rate was 2600  $\pm$  50 ml./min.

Test Fluids: MLO 7098 = 7.2 wt.% Acryloid HFS-5 in a 3.5 Centistoke Base Stock at 100°F.  
 MLO 7099 = 7.1 wt.% Acryloid HFS-4 in a 3.5 Centistoke Base Stock at 100°F.  
 PRL 2286 = A Typical Commercial Spec. MIL-O-5606 Hydraulic Fluid Obtained from The Standard Oil Company of New Jersey.

Test Fluid	MLO 7098	MLO 7099	PRL 2286
Centistoke Viscosity at 100°F.	14.2	14.1	14.1
% Permanent Decrease Due to Shear <sup>(2)</sup> At 800 p.s.i.	30.5	25.5	25.0
At 1000 p.s.i.	36.0 <sup>(1)</sup>	31.5 <sup>(1)</sup>	29.5 <sup>(1)</sup>
At 1500 p.s.i.	40.5	38.5	35.5

- (1) Estimated from a plot of per cent permanent viscosity decrease versus pressure drop across the orifice.  
 (2) Decrease in viscosity after 5000 cycles through the orifice.

Figure 13

PERMANENT VISCOSITY DECREASE DUE TO SHEAR FOR TWO SHEAR STABILITY REFERENCE FLUIDS  
IN THE PRL HYCON PUMP TEST UNIT AT 100°F.

ALL TESTS CONDUCTED IN HYCON PUMP TEST STAND AT 100°F. (PUMP INLET TEMPERATURE) USING AN ORIFICE AS THE PRESSURE LOADING DEVICE.

- $\Delta$  O ML0 7098 = 7.2 WT.% ACRYLOID HF S-5 IN A 3.5 CENTISTOKE (100°F.) BASE STOCK  
 $\blacktriangle$  ML0,7099 = 7.1 WT.% ACRYLOID HF S-4 IN A 3.5 CENTISTOKE (100°F.) BASE STOCK

