WADC TECHNICAL REPORT 55-89
PART I

PHYSICAL AND CHEMICAL LABORATORY EVALUATION OF EXPERIMENTAL SILICATE-BASE HIGH-TEMPERATURE HYDRAULIC FLUIDS

Part I. Monsanto Fluid, OS-45

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FOREWORD

This report was prepared by the Lubricants Section, Organic Materials Branch and was initiated under Project No. 7331, "Hydraulic Fluids, Lubricants and Related Materials", Task No. 73313, "Hydraulic Fluids", formerly RDO No. 613-15, "Hydraulic Fluids" and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with O. M. Ballentine as project engineer.

This report covers work conducted from June 1954 to November 1954.

ABSTRACT

A physical and chemical evaluation has been conducted by the Wright Air Development Center, Directorate of Research, Materials Laboratory on an experimental high temperature hydraulic fluid, OS-45, developed by the Monsanto Chemical Company. This silicate base fluid was evaluated against proposed requirements for a -65° to 400°F hydraulic fluid for long life piloted aircraft. This fluid conformed to such proposed requirements as spontaneous ignition temperature, pour point, hydrolytic stability at 200°F, and foaming tendency. The oxidation and corrosion characteristics of the fluid at 400°F were good in all respects with the exception of a high acid number signifying some oxidation of the oil. The viscosity of OS-45, 1.2 cs at 400°F, failed to meet the desired specification value of 2.5 at 400°F. Additional hydrolytic stability tests at 400°F have shown that the fluid will break down in the presence of very small percentages of water. This instability at high temperatures seems to be one of the major deficiency of this type of fluid. It is apparent from the mock up and high temperature test data available, that the present hydrolytic stability test at 200°F needs revision. Fluid stability testing above 400°F indicated that OS-45 has marginal resistance to both oxidation and thermal stability, expecially when in the presence of metal catalysts. However this fluid is comparable to other silicate fluids evaluated under these requirements.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M.R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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General Discussion on the Evaluation of the Monsanto Chemical Company Experimental High Temperature Hydraulic Fluid MLO 54-540

A sample of an experimental hydraulic fluid, OS-45, was submitted by the Monsanto Chemical Company for evaluation by this Center as a high temperature hydraulic fluid. The subject oil was assigned a Materials Laboratory code number, MLO 54-540.

MLO 54-540 fluid, which is a silicate base fluid, was evaluated primarily against a proposed -65° to 400°F high temperature hydraulic fluid specification. At the present time this military specification has been assigned a temporary USAF number and will be referred to in this report as MIL-F-8446 (USAF). Since this is a proposed specification it is likely that some of the testing procedures and fluid requirements will be changed in accordance with results obtained from future high temperature pump and system mock-up tests.

Some of the more important tests conducted on OS-45 and the results of such tests are given as follows:

Viscosity - Temperature Relationship

Viscosity measurements were conducted over a -65° to 400°F range. These data are presented in Table 1. The viscometric requirements of the proposed Specification MIL-F-8446 are 2500cs maximum at -65°F and 2.5cs minimum at 400°F. A maximum of 2500cs at -65°F was established since too high a viscosity would result in loss of pump efficiency in overcoming needless fluid friction and would create too great a response time to actuate certain hydraulic system components. The 2.5cs value at 400°F is an arbitrary figure since there has been insufficient hydraulic pump tests at 400°F to realistically determine the minimum viscosity a pump can tolerate at this temperature and continue to maintain the required mechanical and volumetric efficiency under the parameters of temperature, pressure and speed. The viscosity of MLO 54-540 is 2229.5cs at -65°F and 1.20cs at 400°F. A viscosity-temperature curve is given in Figure I.

Spontaneous Ignition Temperature (S.I.T.)

This is the temperature at which oil vapor and air ignite without the aid of external sources such as a flame, spark, etc. Briefly this test is performed by dropping the test fluid on a heated surface and measuring the temperature required to spontaneously ignite the fluid. Such conditions would be present in an aircraft if a hydraulic fluid line was severed and allowed the fluid to be sprayed over a hot surface. The minimum S.I.T. value required by Specification MIL-F-8446 is 700°F. MLO 54-540 meets this requirement by exhibiting a S.I.T. value of 705°F.

Oxidation and Corrosion

The oxidation and corrosion test was conducted in accordance with Federal Specification VV-L-791e Method 5308.3 with the following modifications; 400°F test temperature and 72 hour test duration. The metals employed were copper, steel and aluminum alloys and pure anode silver.

Specification MIL-F-8446 requires that the weight change of the above metals, with the exception of copper, shall not exceed ±0.2 mg/cm^2 of surface area after brushing, with copper having an allowance of ±0.4 mg/cm^2. The oil cannot be oxidized to the extent of having changed ±20 percent from the original viscosity when measured at 210°F. The data obtained on the MLO 54-540 fluid are given in Table I. In general MLO 54-540 possessed good oxidation-corrosion stability under the conditions of this specification test. There was no apparent weight change in the aluminum alloy and silver metals and the weight loss of the copper and steel alloys were within specification limits. Although each of the metals acquired a stained discoloration, this is not considered serious since there was no evidence of pitting or other phenomena associated with undesirable corrosion. The acid number change of the oil sample was high (6.6) greatly exceeding specification limits of 0.5. However, this high acid number was not associated with other changes in the fluid which would be expected of an highly oxidized sample; i.e. a large viscosity change and the formation of sludge or insolubles. It is possible that the high acid number formed in silicate type fluids is not inherently detrimental. Since MLO 54-540 fluid exhibited relatively good oxidation and corrosion stability at 400°F, a similar but more severe test was conducted at 550°F for a period of 24 hours. The results of this non-specification type test are given in Table I. The metals used in this test were stainless steel, aluminum alloy, chrome-molybdenum steel, copper-beryllium alloy and titanium. It is anticipated that these metals may be employed in systems operating at temperatures above 400°F. The air rate to the oil sample was the same as in the 400°F oxidation-corrosion test, i.e. 5 liter/hour.

At the termination of this test, all the metals with the exception of the aluminum alloy and the chrome-molybdenum steel strips met the specification corrosion requirements set forth for the 400°F test. The oil sample had undergone considerable degradation which was indicated by a high viscosity change, -30.0 percent when measured at 210°F, and a significant amount of sludge left in the test container.

Another 550°F test was conducted in which the ratio of air to oil sample was reduced to a point of simulating the concentration of air in a closed hydraulic system. This was accomplished by injecting 10 ml of air into the test container which contained 100 ml of oil sample. The test vessel was previously purged with clean nitrogen gas. At the end of 24 hours

MLO 54-540 underwent essentially the same degree of fluid degradation as was observed in the 550°F oxidation-corrosion test, i.e. a 39 percent decrease in viscosity (measured at 210°F). The aluminum alloy and chrome molybdenum metal strips also had a corroded appearance in this test.

The results of these tests indicate that MLO 54-540 would be marginal in applications at 550°F, especially for long periods and in the presence of oxygen. Since MLO 54-540 fluid initially possesses a low viscosity (0.9cs) at 550°F, even a slight viscosity decrease at this temperature may be significant in altering the lubricating power of the fluid for high speed and pressure hydraulic pumps.

In order to provide some information on the thermal stability of MLO 54-540 fluid, without the effect of metal catalysis, a series of MLO 54-540 samples were sealed in glass tubes and placed in a constant temperature oven (400°F) for a period of 360 hours. Samples were withdrawn periodically and examined for viscosity changes. These data are reported in Table II. The viscosity and fluid appearance of MLO 54-540 did not change appreciably under these testing conditions. At the end of 360 hours at 400°F the viscosity(210°F) had decreased approximately 10.0 percent. It was observed that the greatest viscosity change occurred within the first 24 hours of testing.

Hydrolytic Stability

As an obvious corollary to the oxidation-corrosion stability test there exists the problem of the hydrolytic stability characteristics of hydraulic fluids.

In a test for this phenomenon the fluid sample is contaminated with 25 percent by weight of water and in the presence of a copper test specimen is rotated at a temperature of 200°F for 48 hours.

The requirements of this test are as follows. The total change in weight of the copper strip shall not be greater than 0.5mg/cm^2. There shall be no pitting, etching, or visible corrosion on the surface of the copper. A slight discoloration will be permitted. After completion of the hydrolysis test the acid number increase of either the aqueous or oil layer shall not be greater than 0.5mg of potassium hydroxide per gram of oil. The viscosity of the oil layer shall not have changed more than ±20 percent from the original viscosity value when measured at 210°F. The amount of insoluble material, determined after the test, shall not be greater than 0.5 percent by weight of the original test fluid.

MLO 54-540 passed the hydrolytic stability test described in Specification MIL-F-8446. The copper weight loss was negligible and there was no evidence of pitting or etching. The viscosity change of the oil layer was only -1.25per cent (measured at 210°F), well within specification limits. There was no significant formation of sludge during this test. The acid numbers of both the water and oil layers were low, being 0.03 and 0.04 respectively. It is of interest to note that acid numbers do not have the same significance in silicate hydrolysis studies as one would expect in the hydrocarbon type molecule. The products of silicate hydrolysis are usually not sufficiently reactive with KOH to give an accurate indication of the acid components formed. An additional hydrolytic stability test was conducted on MLO 54-540 fluid to determine its resistance to water under conditions more indicative of hydrolysis occurring in a closed hydraulic system at 400°F. This test was conducted in a standard Parr, high pressure, rocking, hydrogenation apparatus. The oil samples, in which various concentrations of water were introduced, were made up to a total of 100 grams and placed, with a copper strip, in the bomb's reaction flask. Hydrolysis can be measured in this test by two methods. In the first method, the test is conducted for a specified period at which time fluid and metal changes are measured. In the second method it is possible to determine with a fair degree of accuracy, the time required for hydrolysis to occur. This is possible by observing pressure and temperature changes through appropriate recording devices connected directly to the sample area. A pressure drop usually denotes the utilization of water vapor in the process of hydrolysis, which in most cases results in an exothermic type reaction, i.e. increase in fluid temperature. Therefore a sharp break in both the pressure and temperature recording curves can be termed the threshold of hydrolysis.

Two MLO 54-540 fluid samples were tested in this hydrolysis test. The first sample contained 0.5 percent by weight water and was tested at 400°F for 200 hours without undergoing hydrolysis. The second sample, which contained 5 percent by weight water hydrolyzed in approximately three hours. Additional tests at the intermediate concentration of 1 and of 2% of water showed a threshold of hydrolysis of four hours for each concentration. Mock-up tests conducted at 210°F with 2% water contamination, caused severe filter plugging after 30.5 hours of operation. The filter plugging resulted from solid formation due to hydrolysis of the fluid.

The evaluation of other silicate ester fluids similar in physical and chemical properties to OS-45, indicate that hydrolysis is a general problem of the presently available fluids.

Tests were conducted on the MLO 54-540 fluid to determine the concentration of water that this material has a tendency to absorb at different temperatures. Not only is it important that the water concentration be below the concentration necessary to promote a hydrolysis type reaction but water also should not be present in sufficient quantity to be instrumental in causing high vapor pressure and corrosion to the hydraulic system. If a material can tie up water molecules without significantly effecting the properties of the resulting water containing fluids this would tend to reduce the concentration of free water. To determine the water absorbing tendency of MLO 54-540 fluid at different temperatures, samples of the oil were saturated with water. The free water was decanted and the oil samples containing water were then measured at different temperatures for their water content by the Carl Fischer Electrometric Titration Method of water determination. The results of this testing are given below.

Oil Temperature (°F)	Water Added to MLO 54-540	Water Content in MLO 54-540 (percent by weight)
80.6	0	0.005
80.6	Saturated	0.031
195	Saturated	0.067
400	Saturated	0.077

It is evident that very little water is absorbed by MLO 54-540 and for all practical purposes the fluid will have no effect on the concentration of water in a hydraulic system.

Foaming

It is essential that the hydraulic fluid be in complete liquid form to insure adequate transmittance of pressure throughout a hydraulic system. The tendency for a liquid to foam while under pumping conditions creates a definite hazard of vapor lock and consequent malfunction of the hydraulic system.

To eliminate the deleterious effect of excess foaming, Specification MIL-F-8446 requires that the total volume of oil and foam after a 5 minute blowing period at 200°F cannot exceed 600 ml. The foam stability, that is the tendency for a fluid to retain its foam-liquid interface, is measured by the time required for the foam to dissipate under static conditions at 200°F. This time, which is referred to as the collapse time, has been established at a maximum of 10 minutes in Specification MIL-F-8446. MLO 54-540 fluid exhibited good anti-foaming properties in this test. The total volume of oil and foam after a 5 minute blowing period was approximately 230 ml. The collapse time was determined to be 61 seconds.

Lubricity

In addition to this primary function of transmitting hydraulic pressure throughout the hydraulic system of the aircraft, the hydraulic fluid has the secondary duty of lubricating the hydraulic pump and other hydraulic system components. There is no universally accepted method for the measurement of lubricity. For preliminary lubricity studies the Shell-4-Ball Wear Tester was selected as the best choice for evaluating the wear properties of a hydraulic fluid. This testing was conducted at two temperatures. First, at 75°C and under standard testing conditions so that the lubricity could be compared to that of the present USAF hydraulic fluid, Specification MIL-O-5606. In the second test, the temperature was increased to the highest operating temperature desired of the fluid, i.e. 400°F. Rubbing speeds of 1200 rpm, loads of 4, 10, and 40 Kg and a test time of 2 hours were chosen since previous experience has indicated that the results obtained under these conditions compare quite favorably with wear data from pump tests. The average scar diameters of the steel balls employed in this test after lubrication with MLO 54-540 were 0.38, 0.58, and 1.08 millimeters at loads of 4, 10, and 40 kilograms respectively. Wear evaluation under similar conditions with the present USAF hydraulic fluid meeting Military Specification MIL-O-5606 gave 0.24, 0.26 and 0.86mm wear scar diameters under the three increasing loads. Specification MIL-O-5606 is a petroleum base oil containing lubricity additives.

If realistic conclusions can be made from this screening, low temperatures test, MLO 54-540 is not as good a lubricant as Specification MIL-O-5606 type fluids. However the difference in lubricity of the two fluids at the different jaw loads may not be too significant considering the light loads that are experienced in hydraulic systems.

Rubber Compatibility

Since many components of an aircraft hydraulic system, especially hydraulic pumps, contain rubber packings and gaskets, it is essential that a hydraulic fluid be sufficiently compatible with these elastomers to enable such materials to retain their original desired properties. Military Specification MIL-F-8446 requires that a swelling of standard synthetic rubber (L or R stock), by the test fluid, shall be 25±5 percent when aged at 250°F for 70 hours. Briefly the test is conducted by measuring the difference in the water displacement of the rubber specimen before and after lubricant immersion for one week. This difference divided by the water displacement before immersion in the oil sample gives the increase in volume of the rubber specimen. The rubber swell with MLO 54-540 was found to be 14 percent which was 6 percent below the minimum specification requirement. Additional non-specification rubber tests were conducted in an attempt to simulate the effect that fluids have on elastomers in a closed system at 400°F. In this test a plasticized neoprene type rubber was placed in a test tube filled with MLO 54-540 fluid. The weight ratio of fluid to rubber was approximately 8 to 1. The test tube was then sealed, with a gas torch, and aged for 168 hours at 400°F. Such properties as volumes swell, hardness and the brittle point of the elastomer were measured after aging. The results of this testing are given in Table I. MLO 54-540 is relatively compatible with the plasticized elastomer used in this test as shown by a -52°F brittle point of the elastomer after the aging of the sample. However -65°F brittle point would be desired in this test. It is theorized that a 25 percent rubber swell will be required in the 250°F specification test to insure an aged neoprene plasticized elastomer of maintaining a -65°F brittle point.

Summary and Conclusions

A sample of the Monsanto Chemical Corporation high temperature hydraulic fluid, MLO 54-540 (OS-45), has been evaluated by this Center for chemical properties. This silicate base fluid was evaluated against a proposed military specification referred to in this report as MIL-F-8446. Both the requirements and testing procedures are subject to change in this specification as more experience and knowledge is obtained in fluid and test development in the area of high temperature hydraulic fluids and their system components.

The MLO 54-540 fluid has excellent viscosity characteristics at the lower temperatures but exhibits questionable viscosity at temperatures of 400°F and higher. Pump tests are being conducted at 400°F on MLO 54-540 to help answer some questions on the relationship of viscosity and lubricity.

MLO 54-540 fluid exhibits fair oxidation and corrosion properties at 400°F in the presence of such metals as silver, steel, copper and aluminum. There was no gum or insolubles formed in this test nor was there any indication of corrosion on the metals.

However, at a higher temperature (550°F) corrosion was noted on both the aluminum and chrome-molybdenum steel alloys. The oil samples underwent oxidation resulting in a significant viscosity change and the formation of sludge. Eliminating the entrance of fresh air to the oil sample apparently had little effect on diminishing either of these two adverse properties. However, no sludge was noted in this test. It appears that MLO 54-540 possesses excellent thermal stability at 400°F for a considerable period. The fluid when exposed to 400°F for 360 hours underwent only a 10% decrease in viscosity, when measured at 210°F.

The hydrolytic stability characteristics of MLO 54-540 when evaluated in accordance with the 200°F test set forth in Specification MIL-F-8446 showed that the fluid was resistant to the effect of water. However at higher temperatures, 400°F, and in the presence of 5 percent water this high temperature hydraulic fluid underwent hydrolysis to form an insoluble mass within 3 hours.

It should be mentioned that the silicates represent a class of compounds that are inherently by structure susceptive to hydrolysis. From past experience with the silicate fluids it was discovered that even slight concentrations of water over a period of time brought about hydrolytic instability of a fluid that formerly met the requirements of MIL-F-8446 Specification test. This indicates that the present specification test may not simulate such conditions (e.g. hydrolysis produced by a 5% water contamination over a 30 day storage period) It may therefore be necessary to modify or even substitute a different test in Specification MIL-F-8446.

MLO 54-540 fluid is relatively compatible with a plasticized elastomeric packing that has been proposed for the -65° to 400°F hydraulic system. This elastomer had a -52°F brittle point after aging 168 hours at 400°F with MLO 54-540 fluid. This is only several degrees higher than the -65°F brittle point desired in this test.

The anti-foaming tendencies of MLO 54-540 fluid, and equally important, the ability to refrain from forming a stable foam indicates that little trouble should be anticipated from this fluid under cycling conditions encountered in a hydraulic system.

The results from the Shell-4-Ball Wear Test indicate that MLO 54-540 does not lubricate as well as the USAF Military Specification MIL-O-5606 petroleum base hydraulic oil. However MLO 54-540 is a fair lubricant and should offer sufficient lubricity to serve as a long service life hydraulic fluid.

Table I

Physical and Chemical Properties of MLO 54-540 Compared to Proposed Requirements of Specification MIL-F-8446

Tests	MLO 54-540	Present Specification MIL-F-8446 Requirements
1. Viscosity, cs.		
400°F	1.20	2.5 min*
210°F	3.98	
100°F	12.16	
-65°F	2229.5	2500 max
2. Flash Point °F	365	
3. Fire Point °F	425	
4. Acid No. (Mg KOH)	0.08	0.2 max
5. Specific Gravity		
0°F	0.8147	
60°F	0.8922	
100°F	0.8760	
200°F	0.8380	
300°F	0.7970	
6. Pour Point, °F	Below - 75	-75 max
7. Spontaneous Ignition Temperature, °F	705	700 min
8. Evaporation, % (6 1/2 hours at 400°F)	21.23	
9. Foaming		
Total Volume of Foam and Oil, ml.	230	600 max
Collapse Time, Seconds	61	600 max

* 1.0 cs value will be the absolute minimum value accepted

Table I, Continued

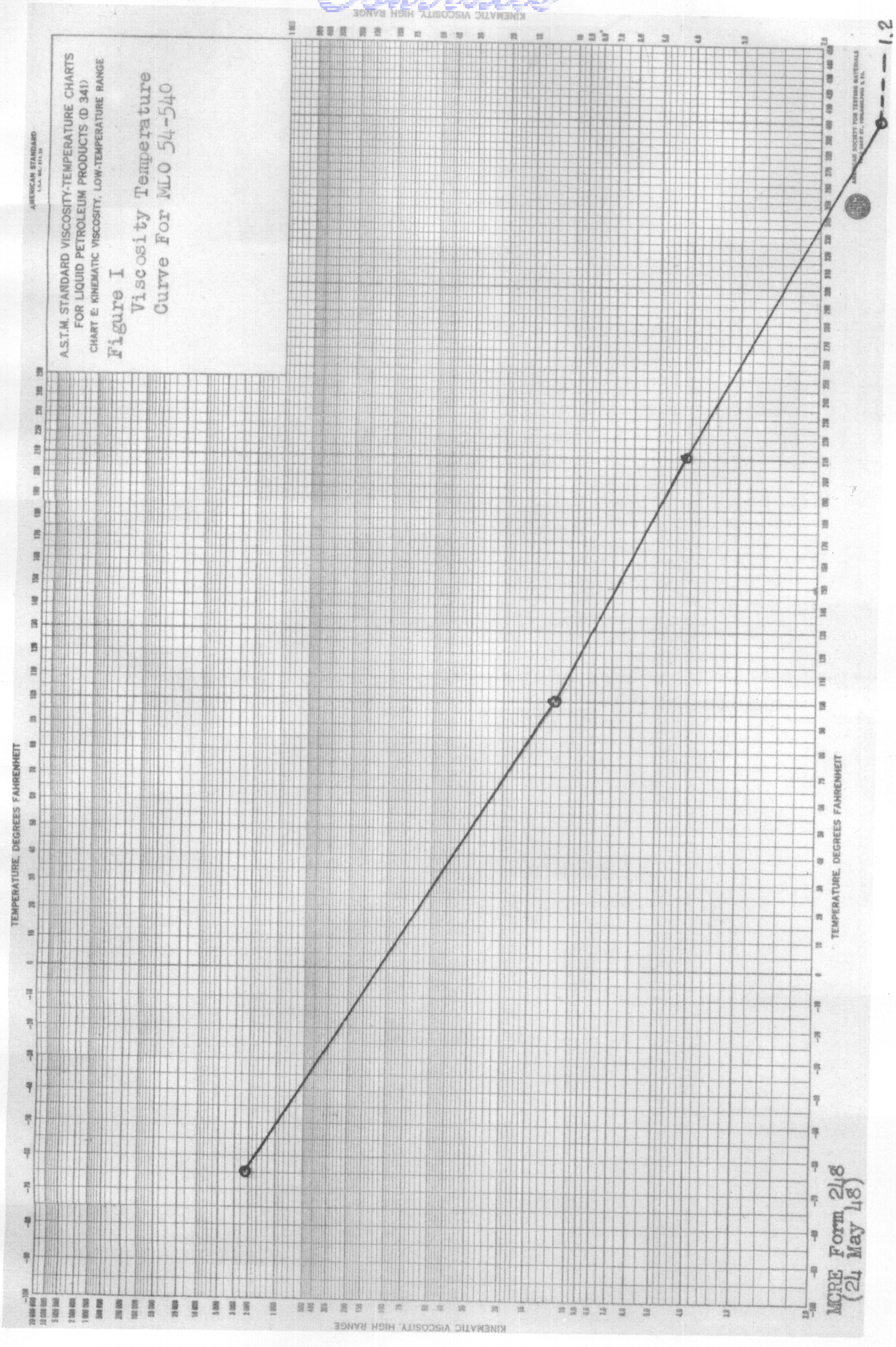
Tests	MLO 54-540		Present Specification MIL-F-8446 Requirements
10. Rubber Compatibility			
a. At 250°F - 70 hours Rubber Swell (Standard R Stock)	14%		25±5%
b. At 400°F - 168 hours			
Rubber Swell (453-26c elastomer)	22%		
Hardness	62		
Brittle Point, °F	-52		
11. Oxidation - Corrosion			
a. (At 400°F, 72 hours, Air Rate - 5 l/hr.)			
Copper - Weight Change, mg/cm²	-0.16		± 0.4
- Appearance	Stained		No pitting or etching
Aluminum - Weight Change	0.0		± 0.2
- Appearance	Stained		No pitting or etching
Steel - - Weight Change	-0.10		± 0.2
- Appearance	Stained		No pitting or etching
Silver - Weight Change	0.0		± 0.2
- Appearance	Stained		No pitting or etching
Percent Viscosity Change at 210°F	10		± 20
Acid Number Increase	6.6		0.5
Oil Lost During Test, %	13.0		
Oil Appearance	Black		
b. At 550° - 24 hours	Air 5 l/hour	.04% Oxygen in System	
Copper Beryllium- Weight Change, mg/cm²	+0.08	+ .016	
- Appearance	Stained	Stained	
Chrome Molybdenum-Weight Change	-0.44	-0.344	
-Appearance	Passes	Slight Stain	
Aluminum - -Weight Change	-0.008	+0.008	
-Appearance	Etched	Etched	
Steel -Weight Change	+0.016	+0.064	
-Appearance	Passes	Passes	
Titanium -Weight Change	+.008	+0.04	
-Appearance	Passes	Passes	

Table I, Continued

Tests	FLO 54-540		Present Specification MIL-F-8446 Requirements
Viscosity Change, % at 210°F	-30	-39	
Acid Number Change	+ 0.42	-0.04	
Oil Appearance	Black (Sludge in Oil)	clear (no sludge)	
12. Hydrolytic Stability			
a. At 200°F, 48 hours, 25% by wt. of Water			
Copper - Weight Change, mg/cm^2	- 0.02		0.5 max
- Appearance	Stained		No etching or pitting
Acid No - Oil Layer Change	+ 0.04		0.5 max
- Water Layer	0.03		0.5 max
Percent Viscosity Change at 210°F	-1.25		± 20
Percent Insolubles	0.092		0.5 max
b. At 400°F			
200 hours - 0.5% by wt. of water			
Copper - Weight Change, mg/cm^2	0.03		
- Appearance	Stained		
Percent Insolubles	0.13		
3 hours - 5.0% by wt. of water			
Copper - Weight change, mg/cm^2	1.9		
- Appearance	Stained		
Oil Appearance	Completely hydrolyzed		
13. Thermal Stability (Closed tube and no metals)			
Number of Hours at 400°F	Viscosity (210°F)		
0	3.90		
24	3.71		
48	3.62		
96	3.65		
264	3.53		
336	3.52		
360	3.51		

Table I, Continued

Tests	MLO 54-540	Specification MIL-0-5606 Hydraulic Fluid
14. Lubricity, Shell-4-Ball Wear Test (75°C, 1200 rpm, 2 Hours)		
Wear Scar Diameter, Millimeters at Jaw Loads of		
4 Kg.	0.38	0.24
10 Kg.	0.58	0.26
40 Kg.	1.08	0.86


AMERICAN STANDARD
A.S.T.M. STANDARD VISCOSITY-TEMPERATURE CHARTS
FOR LIQUID PETROLEUM PRODUCTS (D 341)
CHART E: KINEMATIC VISCOSITY, LOW-TEMPERATURE RANGE
Figure I
Viscosity Temperature
Curve For MLO 54-540
KINEMATIC VISCOSITY, HIGH RANGE
TEMPERATURE, DEGREES FAHRENHEIT
1.2
MCRE Form 248
(24 May 48)