WADC TECHNICAL REPORT 55-89
PART II

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

Part II. Hollingshead Fluid, No. 71852-B

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FOREWORD

This report was prepared by the Lubricants Section, Organic Materials Branch, Materials Laboratory, Directorate of Research, WADC. The work was accomplished under Project Number 7331 entitled "Hydraulic Fluids", Task Number 73313 entitled "Hydraulic Fluids" formerly RDO Number 613-15 entitled "Hydraulic Fluids, Lubricants, and Related Materials" with Oliver M. Ballentine as Project Engineer.

This report covers work conducted from June 1954 to November 1954. Data on other silicate-ester fluids submitted by the Hollingshead Corporation will be contained in forthcoming reports.

ABSTRACT

A physical and chemical laboratory evaluation has been conducted by the Materials Laboratory, Directorate of Research, Wright Air Development Center, on an experimental high temperature hydraulic fluid, 71852-B, which was developed by the Hollingshead Chemical Corporation. 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 proposed requirements as spontaneous ignition temperature, pour point, hydrolytic stability at 200°F, oxidation-corrosion, foaming, and viscosity-temperature requirements. The fluid however, failed to meet proposed requirements covering rubber compatibility. Insufficient fluid was available to perform the more significant 400°F hydrolytic stability test. This fluid had properties comparable to other fluids evaluated under the procedures contained in this report.

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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I. General Discussion on the Evaluation for the R. M. Hollingshead Corporation Experimental High Temperature Hydraulic Fluid, Number 71852-B.

A sample of an experimental high temperature hydraulic fluid was furnished by the R. M. Hollingshead Corporation for evaluation by this Center as a -65° to 400°F hydraulic fluid. The subject oil was assigned the Materials Laboratory code number MLO 54-541.

MLO 54-541, 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-H-8446. It is anticipated that some of the testing procedures and fluid requirements, established in this specification, will probably be changed as more experience is gained with the various classes of compounds under investigation by this Laboratory. Many of the fluid requirements will depend upon information obtained from future high temperature pump and cycling hydraulic mock-up tests.

Some of the more important tests and the results of these tests are given as follows:

Viscosity and Temperature Relationship

Viscosity measurements were conducted over a -65° to 400°F range. These data are presented in Table I. The viscometric requirements of the Proposed Specification MIL-H-8446 are 2500cs maximum at -65° and 2.5 minimum at 400°F. The 2500cs maximum at -65° was established because too high a viscosity would result in loss of pump efficiency in overcoming needless fluid friction. The 2.5cs value at 400°F has been established as an arbitrary value until sufficient hydraulic pump tests at 400°F have been performed to realistically determine the minimum viscosity a pump can tolerate and continue to maintain mechanical and volumetric efficiency under the parameters of temperature, pressure, and speed. MLO 54-541 exhibited a good viscosity-temperature slope having a measured viscosity of 3.4cs at 400°F and 2663.6cs at 65°F. The viscosity temperature data are tabulated in Table I, and given as thevviscosity temperature curve in Figure 1.

Spontaneous Ignition Temperature S.I.T.

The S. I. T. is defined as the temperature at which oil vapor and air ignite without the aid of an external energy source, such as a flame, spark, etc. Such conditions could 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-H-8446 is 700°F. The value obtained with MLO-54-541 was 815°F. The S.I.T. test was performed in the following manner. The sample of oil was added dropwise to a heated stainless steel disc. The steel disc was heated in 5°F increments until the temperature was high enough to spontaneously ignite the oil sample.

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 in this test were copper, steel, aluminum and magnesium alloys and pure anodic silver.

Specification MIL-H-8446 requires that the weight changes of the above metals, with the exception of copper, shall not exceed $\pm 0.2 mg/cm^2$ 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% from the original viscosity when measured at 210°F.

The data obtained on the MLO 54-541 fluid are given in Table I. In general MLO 54-541 exhibited good oxidation-corrosion stability under the conditions of this specification test. There was no apparent weight changes in any of the metal strips employed in this test. There was also no evidence of detrimental corrosion such as pitting, etching, etc. The 210°F viscosity change after the test was considered high (19.3%) even though this value fell within the specification limits of ±20%.

Thermal Stability

To provide some information on the thermal stability of MLO 54-541 fluid without the effect of metal catalysis, oil samples were sealed in glass tubes and placed in a constant temperature oven (400°F) for a period of 200 hours. It is estimated that a long service life fluid (e.g. 500 hour fluid) will not be exposed to its highest operating temperature (e.g. 400°F) for a period greater than 200 hours. At the termination of this test the MLO 54-541 fluid was examined for refractive index and viscosity changes. These data are given in Table I. Results from this test indicate that MLO 54-541 has excellent thermal stability. There were no significant changes in either physical property measurement, i.e. refractive index and viscosity, to suggest thermal instability.

Hydrolytic Stability

As an obvious corollary to the oxidation-corrosion stability test there exists the problem of the hydrolytic stability nature of hydraulic fluids. This is important since it is difficult to completely divorce fluids from water contamination either in storage, handling, or actual usage in aircraft.

The hydrolytic stability test being employed in Proposed Specification MIL-H-8446 is essentially the same type test found in the lower operating temperature hydraulic fluid specification, e. g. MIL-F-7100. In this test, the fluid sample is contaminated with 25% by weight of water and in the presence of a copper test specimen is agitated, 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 $\pm 0.5 mg/cm^2$. There shall be no pitting, etching, or visible corrosion on the surface of the copper. A slight discoloration will be permitted on this metal. 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% from the original value, when measured at 210°F. The amount of insoluble material, after the test, shall not be greater than 0.5 percent by weight of the original test fluid.

MLO 54-541 appeared to possess good hydrolytic stability characteristics. The copper weight change of $0.10 mg/cm^2$ meets, the specification requirement of $\pm 0.5 mg/cm^2$. Visual and microscopical examination of the copper strip gave no evidence of corrosion in the form of pitting or etching. The metal strip, however, was darkened considerably. Other properties such as acid number change and the percent of insolubles formed after the test were within specification limits. Those data are presented in Table I.

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 at vapor lock and consequent malfunction of the hydraulic system.

To eliminate the deleterious effect of excess foaming, Specification MIL-H-8446 requires that the total volume of oil and foam after a 5-minute blowing period at 200°F cannot exceed 600ml. The foam stability, i.e. the tendency of 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-H-8446. MLO 54-541 fluid exhibited satisfactory anti-foaming properties in this test. The total volume of oil and foam after a 5-minute blowing period was approximately 360 ml. The collapse time was determined to be 298 seconds.

Lubricity

In addition to its 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 75°C so that the lubricity could be compared to that of the present USAF hydraulic fluid meeting Military Specification MIL-0-5606. Rubbing speeds of 1200 rpm, jaw 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 the above parameters compare quite favorably with wear data from pump tests. The average wear scar diameters of the steels balls employed in this test when operated in the presence of the Hollingshead 71852-B fluid were 0.49, 0.67, and 0.87 millimeters at the jaw loads of 4, 10, and 40 kilograms, respectively. Wear evaluation under similar conditions of the present USAF hydraulic fluid meeting Military Specification MIL-0-5606 gave 0.24, 0.26 and 0.86mm wear scar diameters at the three increasing jaw loads. Specification MIL-0-5606 is a petroleum base oil containing slight concentrations of tricresyl phosphate, and a polymeric viscosity index improver, acryloid. If realistic conclusions can be made from this screening, low temperature test, MLO 54-541 is not as good a lubricant as Specification MIL-0-5606 at the lower loads but approaches the lubricity of this petroleum base fluid at higher loads.

Rubber Compatibility

Since many components of an aircraft hydraulic system, especially hydraulic pumps, contain rubber packing it is essential that the hydraulic fluid be sufficiently compatible with these elastomers to enable such materials to retain their original desired properties. Military Specification MIL-H-8446 requires that a swelling of standard non-plasticized synthetic rubber (L or R stock), by the test fluid shall be 25±5 percent when aged at 250°F for 70 hours. It is theorized that a 20 percent rubber swell will be required to insure an aged neoprene plasticized elastomer of maintaining a -65°F brittle point.

Rubber swell tests on the MLO 54-541 fluid indicate that this material is not sufficiently compatible with hydraulic system elastomers. The percent swell on 70 hour aged standard "R" and "L" stock elastomers was 10.5 and 9.2, respectively. These values are considerably lower than the desired 20 percent minimum.

II. Summary and Conclusions

A sample of the R. M. Hollingshead Corporation experimental fluid, 71852-B, has been evaluated by this Center as a high temperature hydraulic fluid (-65° to 400°F). This silicon containing fluid, designated by this Center as MLO 54-541, was evaluated against a proposed military specification referred to in this report as MIL-H-8446. It should be remembered that both the requirements and testing procedures outlined in this specification are subject to change as more experience and knowledge are gained in the area of high temperature hydraulic fluids and their system components.

MLO 54-541 fluid has an excellent viscosity-temperature slope over the range of -65° to 400°F. The viscosity at -65°F (2664) is slightly greater than the 2500cs maximum permitted in Specification MIL-F-8446 but this is not considered too significant. MLO-54-541 is apparently non-corrosive to such metals as copper, aluminum, steel alloys, and pure silver under the parameter of 400°F oxidation. Although all of the metal strips, with the exception of silver acquired a stained appearance there was no indication of a corrosive attack on the metals, i.e. pitting, etching, etc. The fluid apparently underwent some polymerization as evidenced by a high viscosity increase (19.3%) but was not oxidized to the extent of forming insolubles. The thermal stability characteristics of this fluid are excellent. At the end of a 200-hour test at 400°F there was no significant change in the fluids refractive index or more important in the fluids' viscosity change. The 200°F hydrolytic stability test, given in Specification MIL-F-8446, showed that MLO 54-541 was resistant to the effects of water. However, it should be pointed out 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-H-8446 Specification test. This indicates that the present specification test may not simulate such conditions (e.g. hydrolysis produced by a 5.0% water contamination over a 30-day storage period). It may therefore be necessary to modify or even substitute a different test in Specification MIL-H-8446. Insufficient fluid was available to perform the more significant hydrolytic stability test at 400°F or to perform full scale mock up tests on MLO 54-541. The anti-foaming tendencies of MLO 54-541, and equally important, the ability to possess an unstable type foam also passed specification requirements. However, it should be pointed out that these foaming requirements may be made more restrictive if pump and hydraulic system tests now in progress indicate this would be necessary. MLO 54-541 failed to meet the rubber swell requirement of 20% on unplasticized "R" and "L" stock. This would indicate a greater possibility of fluid leakage, at the higher temperatures, than a high swelled "O" ring would permit. Since present day elastomers are plasticized to be usable over a -65° to 400°F temperature range, a low rubber swelling fluid would indicate the leaching out of the plasticizer with a resultant loss in low temperature properties. The results from the Shell-4-Ball Wear Test indicated that MLO 54-541 does not lubricate as well as the USAF Military Specification MIL-O-5606, petroleum base hydraulic oil, especially at the 4, and 10 kilogram jaw-loads. However, at the 40 kilogram load the lubricity of these two hydraulic fluids are essentially equivalent. It appears from these preliminary studies that MLO 54-541 fluid is a fair lubricant and should offer sufficient lubricating qualities to serve as a long service life hydraulic fluid.

In general MLO 54-541 possesses most of the fluid properties required in the proposed Military Specification MIL-H-8446 having the desired viscosity-temperature slope as well as the overall thermal, oxidative and hydrolytic stabilities. The greatest disadvantage of this fluid for the -65° to 400°F hydraulic fluid application is its lack of rubber compatibility with standard stock elastomers. The foaming tendencies of this fluid may also be questionable in high speed and high pressure pumps.

Table I

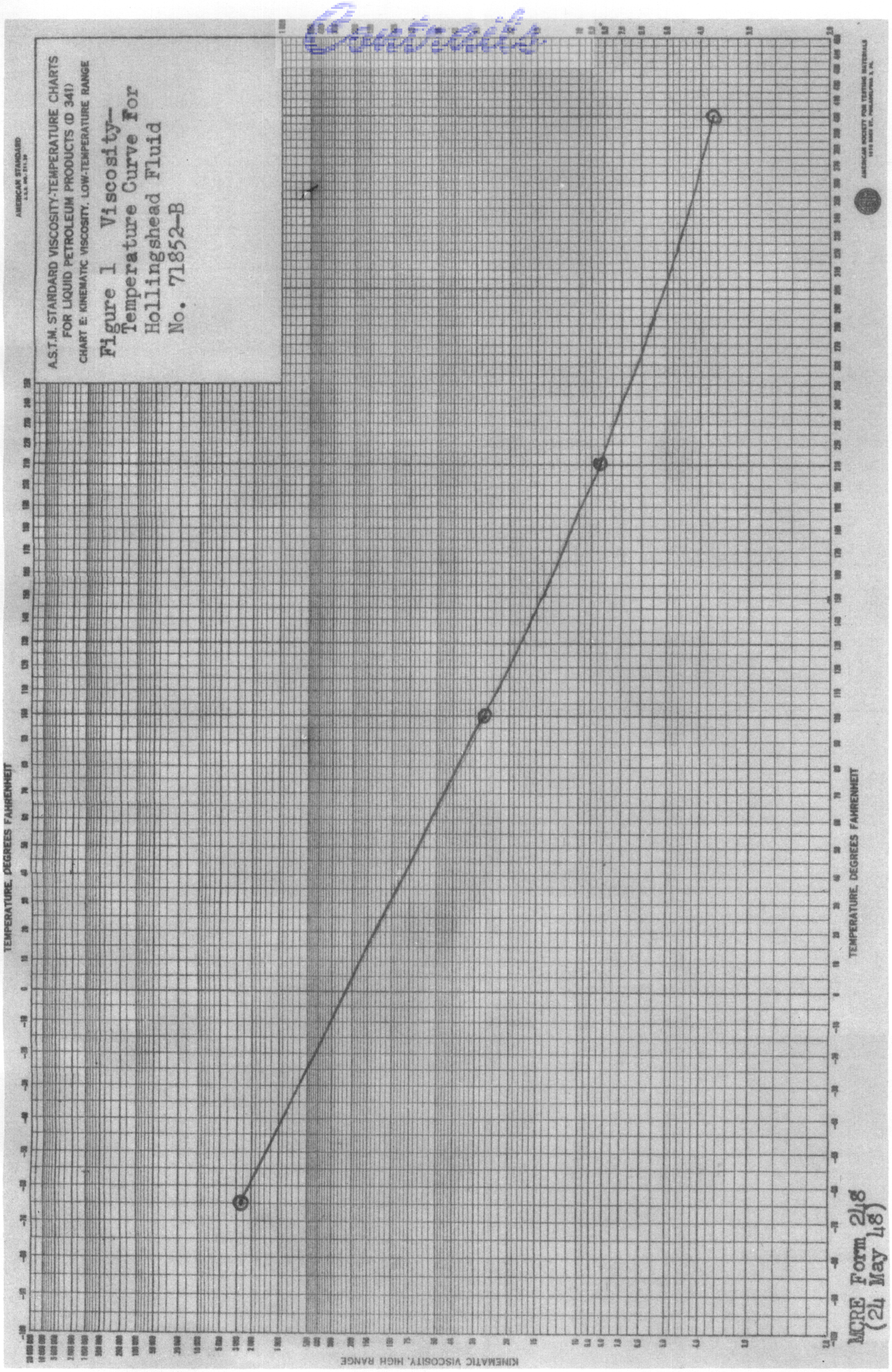
Physical and Chemical Properties of MLO 54-541 Compared to Proposed Requirements of Specification MIL-H-8446

Tests	MLO 54-541	Specification MIL-H-8446 Requirements
1. Viscosity, (cs)		
-65°F	2663.6	2500 max
100°F	23.03	
210°F	7.61	
400°F	3.40	2.5* min.
2. Evaporation (%) (6.5 hrs. at 400°F)	16.6	
3. Flash Point, (°F)	380	
4. Fire Point, (°F)	450	
5. Spontanous Ignition Temperature (°F)	815	700 min.
6. Specific Gravity		
0°F	0.9779	
100°F	0.9346	
200°F	0.8950	
300°F	0.8570	
7. Pour Point, (°F)	Below-75	-75 Max.
8. Foaming		
Total Volume of Foam and Oil (militeres)	360	600 max
Collapse Time, seconds	298	600 max
9. Rubber Compatibility		
Rubber Swell (250°F, 70 hours)		
R Stock, %	10.5	20±5
L Stock %	9.2	20±5

* 1.0 cs value will be the absolute minimum value accepted.

Table I (Continued)

Tests	MLO 54-541	Specification MIL-H-8446 Requirement
10. Oxidation-Corrosion (400°F, 72 hours, Air Rate 5 l/hr.)		
Copper-Weight Change (mg/cm^2)	0.020	±0.4
-Appearance	Passes (Stained)	No pitting or etching
Aluminum-Weight Change (mg/cm^2)	0.012	±0.2
-Appearance	Passes (Stained)	No pitting or etching
Steel -Weight Change (mg/cm^2)	0.016	±0.2
-Appearance	Passes (Stained)	No pitting or etching
Silver -Weight Change (mg/cm^2)	0.02	±0.2
-Appearance	Passes	No pitting or etching
Percent Viscosity Change at 210°F	19.3	±20
Acid Number Increase, Mg KOH	0.16	0.5
Oil Appear	Passes (Black)	No Oil Separation
11. Thermal Stability (200 Hours, 400°F, Closed Tube)		
Refractive Index Change at 25°C	0.0000	
Viscosity Change at 210°F, cs	-0.20	
12. Hydrolytic Stability (200°F, 48 hours, 25% water)		
Copper - Weight Change (mg/cm^2)	0.10	0.5 max
	Passes (Stained)	No pitting or etching
Acid No., Mg/KOH/gm oil		
-Oil Layer Change	0.00	0.5 max
-Water Layer	0.10	0.5 max
Percent Insolubles	0.475	0.5 max
13. Lubricity - Shell-4-Ball Wear Test (75°C, 1200 rpm, 2 hours)		Specification MIL-0-5606 Hydraulic Fluid
Wear Scar Diameter, Millimeter at Jaw Loads of		
4 (Kg)	0.49	0.24
10 (Kg)	0.67	0.26
40 (Kg)	0.87	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 1 Viscosity—
Temperature Curve For
Hollingshead Fluid
No. 71852-B
TEMPERATURE, DEGREES FAHRENHEIT
KINEMATIC VISCOSITY, HIGH RANGE
MCRE Form 248
(24 May 48)
AMERICAN SOCIETY FOR TESTING MATERIALS