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

PART IV. MLO 8200

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FOREWORD

This report was prepared by the Lubricants Section, Organic Materials Branch. It was initiated under Project No. 7331, "Hydraulic Fluids, Lubricants, and Related Materials," Task No. 73313, "Hydraulic Fluids." This work was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. O. M. Ballentine, 1st Lt H. M. Schiefer, and later Mr. R. J. Benzing acting as project engineers.

The period of work covered in this report is September 1954 to August 1956.

ABSTRACT

A physical and chemical evaluation has been conducted by the Materials Laboratory on an experimental high temperature hydraulic fluid, MLO 8200, which has a disiloxane base fluid. It was evaluated against the requirements of Specification MIL-H-8446 (USAF) and for higher temperature applications to 550°F. It passes all the requirements of MIL-H-8446 with the exception of rubber swell.

Some additional tests were performed on this fluid which indicated that, as for other silicate fluids tested, the hydrolytic stability at 400°F seems to be one of the major deficiencies of this type of fluid. It is apparent from mock-up and 400°F laboratory hydrolytic stability tests that the present hydrolytic stability test at 200°F needs revision. Some wear data were obtained with this fluid at 167° and 400°F which indicated that the fluid should have sufficient lubricity to lubricate high temperature pumps. Some minimum spontaneous ignition temperatures obtained with the fluid when sprayed from varying pressure levels into a glass vessel at atmospheric pressure showed that the ignition temperature of this fluid decreases to approximately 510°F at high pressures. This phenomenon is also present in other silicate base fluids tested under these conditions.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
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I. GENERAL DISCUSSION

The United States Air Force initiated a research and development program toward the development of a -65°F to 400°F hydraulic fluid in 1950. At that time a contract was negotiated with the California Research Corporation to investigate various classes of compounds suitable for such a high temperature hydraulic fluid. From the various classes of compounds studied, a final formulation was prepared using a disiloxane base fluid. This final formulation was coded MLO 8200 by the Materials Laboratory. The exact composition of the fluid is reported in Table I. The percentage of XF-371 in the final blend may vary with each batch and will depend upon the viscosity of the disiloxane base fluid and of the XF-371 itself.

MLO 8200 has been evaluated as a long term -65°F to 400°F fluid and a short term -65°F to 550°F fluid. Tests were performed in accordance with the procedure outlined in Specification MIL-H-8446 (USAF). Other tests were performed on this fluid to give additional information on its properties including those at 550°F.

Some of the more important tests conducted on MLO 8200 and their results are given as follows:

Viscosity-Temperature Relationship

Viscosity measurements were conducted over a -65°F to 500°F range. These data are presented in Tables II and IV along with the requirements of Specification MIL-H-8446 and plotted in Figure 2. MLO 8200 exhibits very good viscosity-temperature relationships in that it has a viscosity of 3.8 centistokes at 400°F, well above the 2.5 centistokes specification requirement, and a viscosity of 2557 centistokes at -65°F, which is very close to the 2500 requirement. The 500°F value of 1.76 to 2.00 centistokes represents a figure at which many hydraulic components show acceptable volumetric and mechanical efficiency.

Spontaneous Ignition Temperature (SIT)

Spontaneous ignition temperature is that temperature at which oil vapor and air ignite without the aid of an external high energy source such as a spark or flame. The specification test consists of dropwise addition of the test fluid on a heated surface of known temperature. The minimum temperature at which the fluid ignites is the spontaneous ignition temperature. A value of 700°F is required in Specification MIL-H-8446. The value obtained for MLO 8200 is 716°F which meets the requirements.

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The spontaneous ignition temperature data obtained in a 200 cc pyrex Erlenmeyer flask for MLO 8200 in air, using a fixed injector-to-hot surface distance and one atmosphere pressure are presented in Figure 1. These data indicate that a sharp drop in ignition temperature occurs when the fluid is ejected from pressure levels between 50 and 750 psig, and then levels out at an ignition temperature of approximately 500°F to 525°F at diesel injector pressures exceeding 750 psig. This reduction of spontaneous ignition temperature is an undesirable feature of this fluid, and is also reported for other silicate fluids tested under the above conditions.

These data indicate that special precaution should be taken when MLO 8200 is operated at high pressures and at temperatures above 500°F to prevent harmful fires. Further study is being carried out at the Bureau of Mines to determine the effect, if any, of metal containers, instead of pyrex glass, on the spontaneous ignition temperature-pressure characteristics of MLO 8200.

Oxidation and Corrosion

The oxidation and corrosion test was conducted in accordance with Federal Standard 791 method 5308 with modifications as indicated in Tables II and IV. The metals used in the 400°F test were copper, aluminum, steel, and silver.

Specification MIL-H-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 a maximum weight loss of ± 0.4 mg/cm^2. The oil shall not be oxidized to the extent of having changed more than ± 20% from the original viscosity when measured at 210°F.

The small change in physical and chemical properties of the oil oxidized at 400°F led to evaluation at higher temperatures, and additional tests were run at 550°F for ten hours. Except for viscosity loss (approximately 50% of original viscosity) the fluid has satisfactory properties. The utilization of this fluid in the 500°F to 550°F range should be in a hydraulic system which can tolerate reductions, during use, in viscosity of below 1.0 centistokes.

Thermal Stability

The thermal stability test data at 400°F in the absence of metals and at 550°F in the presence of metals as reported in Tables II and IV are both satisfactory. Again the only property that appears to suffer is the viscosity. This viscosity decrease is due to thermal degradation of the viscosity index improver. This reduction was verified in experiments run with the base oil, under identical conditions as the 400°F run, in which only slight viscosity decrease was experienced.

Hydrolytic Stability

The hydrolytic stability test requirements are as follows: The total change in weight of the copper strip shall not be greater than ± 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 0.5 mg 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 materials after the test shall not be greater than 0.5% by weight of the original test fluid.

MLO 8200 was well within the requirements of Specification MIL-H-8446 on hydrolytic stability. There was little change in fluid properties and little to no attack on the copper specimen.

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 hydrolysis studies in the ester type molecule. The products of silicate hydrolysis are usually not sufficiently reactive with KOH to give an accurate indication of the acid components formed.

Besides the hydrolytic stability test conducted at 200°F, some additional tests were performed with various concentrations of water at 400°F to determine the fluid's resistance to water under conditions more indicative of hydrolysis occurring in a closed hydraulic system at high temperatures. These tests were 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. It is possible with this test 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 curves can be termed the threshold of hydrolysis.

Two samples of MLO 8200 were tested in this test. The sample containing 5% water did not give any appreciable amount of solids after 24 hours whereas the sample with 6% water showed that hydrolysis occurred after 3 hours at 400°F giving a mass of solid material.

Mock-up tests conducted at 210°F with 2% water contamination, caused severe filter plugging after 80 hours of operation. This filter plugging resulted from solid formation due to hydrolysis of the fluid. It is apparent from the mock-up and high temperature test data (Parr Bomb) that hydrolytic instability at high temperatures seems to be one of the major deficiencies of this type of fluid, and that the present low temperature specification hydrolytic stability test needs revision. The evaluation of other silicate-ester fluids similar in physical and chemical properties to MLO 8200 indicates that hydrolysis is a general problem of the presently available fluids.

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, pump cavitation, 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 600 ml. The foam stability, i.e., the tendency of a fluid to retain its foam-liquid intersurface, is measured by the time required for the foam to dissipate under static conditions at 200°F. This time, known as collapse time, has been established as 10 minutes in Specification MIL-H-8446. MLO 8200 has a collapse time of 5 minutes but is marginal on the amount of foam that is formed initially (590 ml).

Lubricity

In addition to the function of transmitting hydraulic pressure throughout the system, the hydraulic fluid must also serve as a lubricant to the various components in the system. As a preliminary screening test, the Shell 4-Ball Wear Tester was selected for evaluating lubricity and the Shell 4-Ball E. P. Tester was selected to study load-carrying ability. The wear tester was run at 75°C (167°F), 1200 rpm for two hours and at 400°F, 600 rpm for the same period of time.

The standard immediate seizure and weld point determinations were made on the E. P. tester. These data are reported in Table III. For comparative purposes, the following wear values for Specification MIL-O-5606 are included:

4-BALL DATA ON SPECIFICATION MIL-O-5606
(75°C, 1200 rpm, 2 hrs.)

4 kg	0.24 mm scar diameter
10 kg	0.26 mm scar diameter
40 kg	0.86 mm scar diameter

Although the wear resistance of the MLO 8200 fluid does not appear to be as good as that of Specification MIL-O-5606 fluid, the difference does not appear to be too significant.

The immediate seizure and weld points are relatively good for hydraulic fluids and are comparable in value to diester base stock engine oils.

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.

MLO 8200 exhibited very poor rubber compatibility in that it only gave 6% volume swell of standard "R" stock rubber (a particular neoprene formula) when tested at 250°F for 70 hours. The specification requirement for this test is 25 ± 5% volume swell, from which it is concluded that MLO 8200 gives only approximately one-third of the minimum swell required.

In addition to the specification rubber swell test at 250°F, some additional tests were performed with a plasticized neoprene type rubber (453-26c) in a closed system at 400°F. In this test, a plasticized neoprene type rubber was placed in a test tube filled with MLO 8200 fluid. The weight ratio of fluid to rubber was approximately 20 to 1. The test tube was then sealed, with a gas torch, and aged for 168 hours at 400°F. Such properties as tensile, volume swell, hardness, brittle point, and per cent elongation of the elastomer were measured after aging. The results of this testing are given in Table III. As indicated by the low swell obtained in the specification test with standard "R" stock, the fluid leached out the plasticizer in 453-26c elastomer giving a high brittle point (+350°F), high shore "A" hardness (94), and low per cent elongation (47) to the elastomer after aging. This test again indicates the incompatibility of MLO 8200 with the presently available high temperature neoprene elastomers and eliminates the use of this fluid with plasticized neoprene elastomers for the -65°F to 400°F application.

II. SUMMARY AND CONCLUSIONS

A sample of MLO 8200 has been evaluated by the Materials Laboratory for its physical and chemical properties. This silicate base fluid was evaluated against Specification MIL-H-8446 (USAF). Both the requirements and testing procedures are subject to change in this specification as more experience and knowledge are obtained in fluid and test development in the area of high temperature hydraulic fluids and their system components. In addition, the fluid was tested for properties at 500°F and 550°F as an evaluation of its suitability for potential use in missile applications at these temperatures.

MLO 8200 passes all laboratory specification tests under Specification MIL-H-8446 except the rubber compatibility test performed at 250°F. This fluid would excessively shrink finished neoprene packings.

MLO 8200 possesses poor high temperature hydrolytic stability as shown from results in a non-specification test at 400°F. However, this fluid is as good or better in high temperature hydrolytic stability as other silicate fluids which have been evaluated under the requirements for a -65°F to 400°F hydraulic fluid.

The spontaneous ignition temperature of MLO 8200 is 716°F at atmospheric pressure but decreases to approximately 510°F when the fluid is sprayed from a pressure greater than 750 psig into a glass vessel at atmospheric pressure. The largest decrease in the value of the spontaneous ignition temperature occurs at injection pressures between 200 and 750 psig.

MLO 8200 possesses good oxidation-corrosion resistance at 550°F for short periods of time, but the viscosity loss due to thermal conditions is high.

The results of the 4-ball tests indicate that MLO 8200 is not as good a lubricant as Specification MIL-O-5606 type fluids but that it should be satisfactory for hydraulic applications.

The anti-foaming tendencies of MLO 8200 are marginal in the amount of foam formed, but the fluid has a collapse time well within the requirements of Specification MIL-H-8446.

MLO 8200 exhibits good oxidative and viscosity properties at 550°F. Therefore, this fluid is presently being considered as a 550°F short term fluid. Other fluids of this general type, but with better rubber compatibility for special neoprene stocks, are receiving the most emphasis at 400°F.

TABLE I

Composition of MLO 8200 Fluid

Hexa (2-ethylbutoxy) disiloxane	93.38 weight %
XF-371 Silicone	4.6 weight %
p,p' dioctyldiphenylamine	2.0 weight %
Quinizarin	0.02 weight %

TABLE II

Physical and Chemical Properties of MLO 8200
Compared to the Requirements of Specification MIL-H-8446

Test	MLO 8200	Present Specification MIL-H-8446 Requirements
Viscosity at °F (cs)		
400	3.8	2.5 minimum
210	12.1	
100	33.8	
-65	2257	2500 maximum
Neutralization Number (mg KOH/gm oil)	0.01	0.2 maximum
Pour Point (°F)	Below -100	-75 maximum
Spontaneous Ignition Temperature (°F)	716	700 minimum
Foaming (Total volume of foam and oil (ml))	590	600 maximum
Collapse Time (seconds)	300	600 maximum
Rubber Compatibility at 250°F for 70 hours (% swell "R" stock)	6	25 ± 5
Hydrolytic Stability at 200°F, 48 hours (25 weight % of H_2O)		
Copper - weight change (mg/cm^2)	-0.02	0.5 maximum
appearance	Stain, no corrosion	No etching or pitting
Acid Number - oil layer	0.03	0.5 maximum
water layer	0.02	0.5 maximum
% Viscosity change at 210°F	-1.5	20
% Insolubles	0.02	0.5 maximum
Oxidation-Corrosion at 400°F, 72 hours (Air rate - 5 liters/hr)		
Weight change (mg/cm^2):		
Copper	-0.02	±0.4
Aluminum	-0.04	±0.2
Steel	-0.05	±0.2
Silver	0.0	±0.2

TABLE II (Continued)

Physical and Chemical Properties of MLO 8200
Compared to the Requirements of Specification MIL-H-8446

Test	MLO 8200	Present Specification MIL-H-8446 Requirements
Oxidation-Corrosion at 400°F, 72 hours (Air rate- 5 liters/hr)		
Appearance:		
Copper	Brown coating	No pitting or etching
Aluminum	No change	No pitting or etching
Steel	Bluish stain	No pitting or etching
Silver	No change	No pitting or etching
% Viscosity change at 210°F	-20	20
Acid Number Increase	0.5	0.5
Appearance of Oil	Dark brown but clear and free of solids	
Vapor Pressure at 400°F (mm Hg)	1.2	5 maximum
Low Temperature Stability	No separation or gelation when held at -65°F for 168 hours	No separation or gelation when held at -65°F for 72 hours

TABLE III

Properties of MLO 8200 of Importance to Hydraulic System Design

Test	MLO 8200
Flash Point (°F)	415
Fire Point (°F)	460
Specific Gravity (g/cc) at °F	
0	0.40
100	0.42
200	0.43
300	0.45
400	0.47
Specific Heat (BTU/lb/°F) at	
0	0.40
100	0.42
200	0.43
300	0.45
400	0.47
Compatibility with MIL-O-5606	100%
with water	Insoluble
Evaporation ($6^1/_2$ hrs at 400°F) (% loss)	22.3
Vapor Pressure (mm Hg) at 300°F	0.03
at 500°F	17
Compressibility (in^2/lb) at 77°F, 3000 psig.	5.3×10^{-6}
Lubricity	
Shell 4-Ball Wear Test (75°C, 1200 rpm, 2 hrs):	
Wear scar diameter, mm, at loads of	
4 Kg	.80
10 Kg	.98
40 Kg	1.47
Shell 4-Ball Wear Test (400°F, 600 rpm, 2 hrs)	
Wear scar diameter, mm, at loads of	
4 Kg	0.78
10 Kg	1.06
40 Kg	1.82
Shell 4-Ball Extreme Pressure Test (Steel on steel) Room temperature, 1 minute duration	
Seizure Point (Kg)	80
Weld Point (Kg)	120

TABLE III (Continued)

Properties of MLO 8200 of Importance to Hydraulic System Design

Test	MLO 8200
Thermal Stability at 400°F (Closed tube and no metals)	
% Viscosity change at 210°F after 200 hours	-40
Final Neutralization Number	0.21
Fluid Appearance	Clear
Hydrolysis Test at 400°F in Parr Hydrogenation Bomb	
5% Water	24 hours with negligible amount of solids formed
6% Water	Hydrolysis occurred after 3 hours
Rubber Compatibility (400°F, 168 hours, 453-26c Elastomers, Closed System)	
Tensile (lb/in^2)	1300
% Rubber Swell	12.7
Hardness, Shore "A"	94
Brittle Point (°F)	+30
% Elongation	47

TABLE IV

Properties of MLO 8200 at 550°F

Test	MLO 8200	
Viscosity at 500°F (cs)	2.00 to	1.76
Thermal Stability at 550°F in presence of metals:		
Length of test (hours)	10	24
Weight change (mg/cm²):		
Titanium	0.0	-0.06
Copper-Beryllium	0.0	-0.10
Aluminum	0.0	-0.04
Stainless Steel	0.0	-0.06
Chrome-Molybdenum Steel	-.03	0.05
Appearance:		
Titanium	Shiny	Shiny
Copper-Beryllium	Shiny	Shiny
Aluminum	Shiny	Shiny
Stainless Steel	Shiny	Shiny
Chrome-Molybdenum Steel	Shiny and blue	Shiny and blue
Viscosity at 210°F (cs) after test	7.23	4.18
Viscosity change at 210°F (%)	-38.4	-64.3
Acid Number Increase	0.26	0.0
Appearance of Oil	Free of solids	Free of solids
Weight Loss of Oil (%)	1.3	1.9
Oxidation-Corrosion at 550°F for 10 hrs (Air rate - 5 liters/hr)		
Length of test (hours)	10	24
Weight change of metals (mg/cm²):		
Titanium	0.0	-0.01
Copper-Beryllium	0.0	-0.07
Aluminum	0.0	0.0
Stainless Steel	0.0	0.0
Chrome-Molybdenum Steel	0.0	-0.04
Appearance:		
Titanium	Shiny	Shiny
Copper-Beryllium	Dull	Brownish stain
Aluminum	Shiny	Shiny
Stainless Steel	Shiny	Shiny
Chrome-Molybdenum Steel	Blue stain	Blue stain

TABLE IV

Properties of MLO 8200 at 550°F

Test	MLO 8200	
Oxidation-Corrosion at 550°F for 10 hours (Air rate - 5 liters/hr)		
Viscosity at 210°F (cs) after test	6.14	5.48
Acid Number Increase	0.28	0.34
Viscosity change at 210°F (%)	-47.6	-54.1
Appearance of Oil	Free of solids	Free of solids
Weight Loss of Oil (%)	4.4	4.1
Rubber Compatibility (550°F, 3 hours, 453-9A Elastomer, Closed System)	Original	Final
Tensile, (lb/in²)	2000	830
% Elongation	170	197
Hardness, Shore "A"	79	63

Bibliography

1. WADC TR 54-191, Fourth Annual Report on "The Development of a High Temperature Aircraft Hydraulic Fluid" under Contract AF 33(038)-9831 with California Research Corporation, December 1954

2. WADC TR 54-458, "Development of a Rubber for Service in Contact with Experimental Hydraulic Fluids at 400°F", December 1954

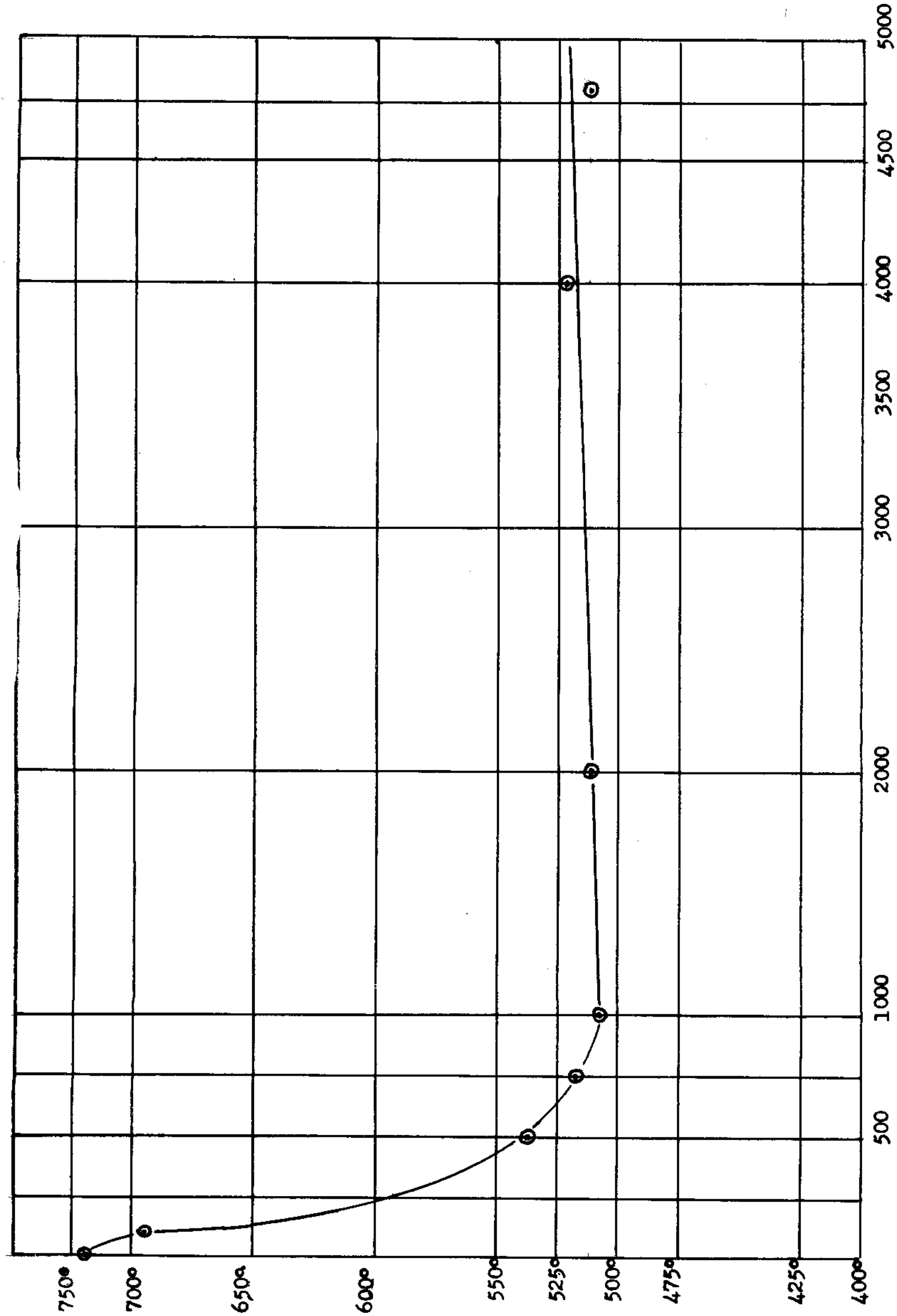

Figure 1
Diesel Injector Pressure, psig
Minimum spontaneous ignition temperature of MLO 8200 in air at one atmosphere pressure as a function of diesel injector pressure, P.S.I.G.

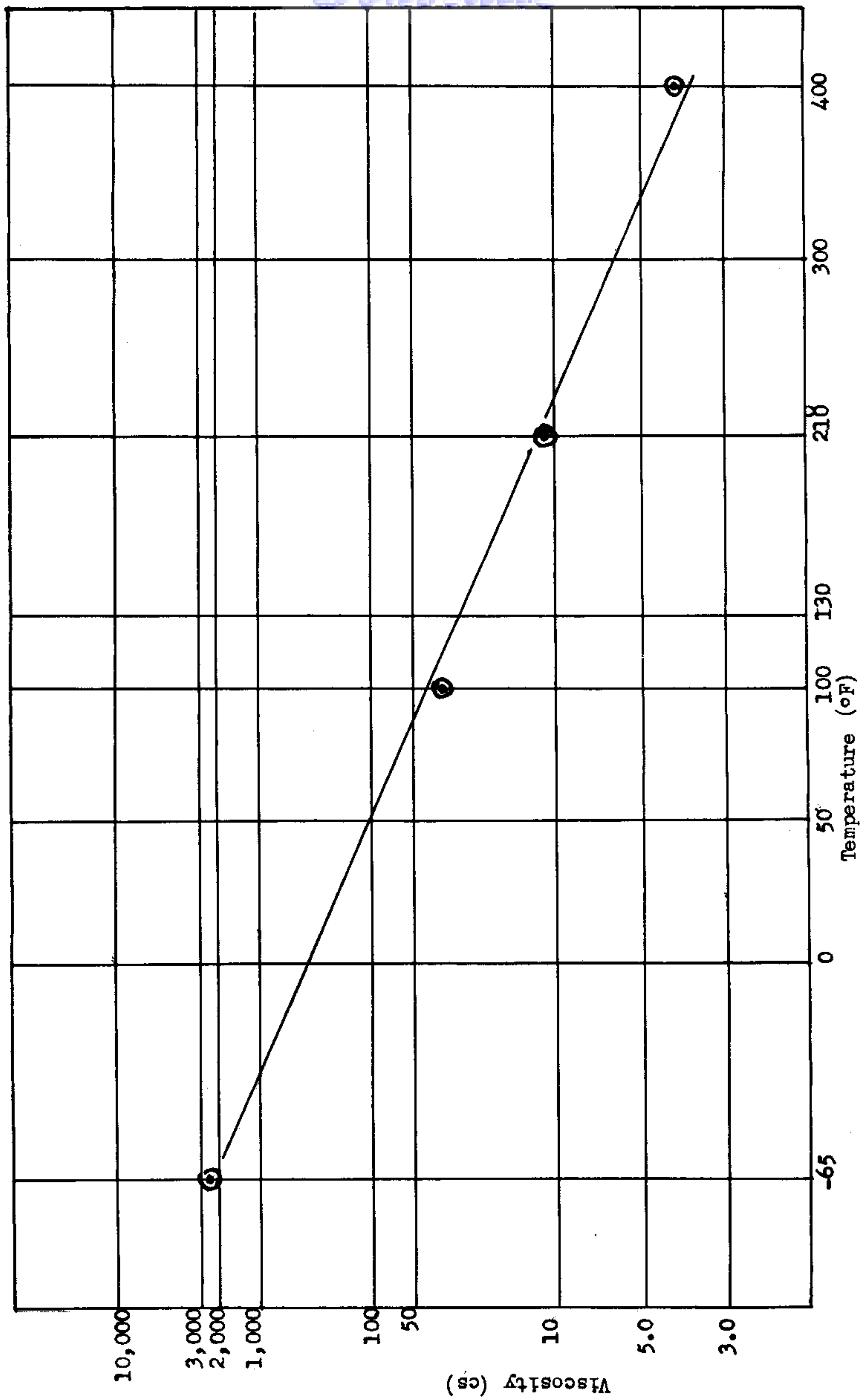

Figure 2
Viscosity-Temperature Properties of MLO 8200