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EFFECT OF EXTREME CONDITIONS ON THE BEHAVIOR OF LUBRICANTS AND FLUIDS

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FOREWORD

This report was prepared by Midwest Research Institute, 425 Volker Boulevard, Kansas City, Missouri 64110, under USAF Contract No. AF 33(615)-3484, Phase I. This contract is in effect from 1 January 1966 to 31 December 1968. The contract is a portion of the mission under BPSN Nos. 66(687340-734008-62405514) and 66(687343-734302-62405514).

The work was monitored by the Air Force Materials Laboratory under the direction of project engineer Mr. F. C. Brooks.

Technical Report AFML-TR-67-8, Parts I and II, covered work for the 1966 and 1967 calendar years. This report concerns the work conducted during the 1968 calendar year. The manuscript was released by the author in December 1968 for publication.

The author gratefully acknowledges the assistance of Mr. Vern Hopkins for the technical direction of the project, Mr. Andrew D. St. John for the analytical work, Mr. A. J. Bossert for the design and development of the high vacuum bearing wear rig, Mr. F. J. Barker for the pressure-viscosity studies, and Mr. Deane H. House who conducted much of the experimental work.

This technical report has been reviewed and is approved.

R. L. ADAMCZAK, Chief
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ABSTRACT

Four shear/thermal stability pump loop experiments were conducted between 400° and 600°F with XF-1-0301 fluorosilicone. This fluid is reasonably stable up to 450°F, will form solids without appreciable property changes at 500°F, and will undergo physical property changes when used at 600°F. XF-1-0301 satisfactorily lubricated an aircraft-type piston pump at 400°F but permitted excessive wear at 500°F.

The bulk modulus of XF-1-0301 was determined to 450°F and 10,000 psig. In general, the isothermal secant bulk modulus for this fluid is higher than a polymeric perfluorinated fluid and a chlorinated phenyl methyl silicone, about the same as ML0-8200 disiloxane and a phenyl methyl silicone, and lower than the bulk moduli of petroleum base fluids, an ester of TMP, a silane, and a 5P4E polyphenyl ether.

Comparative four-ball tests were run at 400°F with XF-1-0301, a polymeric perfluorinated fluid, a deep dewaxed mineral oil, and chlorinated and unchlorinated phenyl methyl silicones. In addition, new and used fluids, other than the silicones, were also studied. The tests indicated that at 400°F the XF-1-0301 fluid will have lubricating characteristics similar to the polymeric perfluorinated fluid and the deep dewaxed mineral oil and superior to the silicones. There was no great difference in the results of the tests run in new fluids and those run in fluids which had been used in pump loop experiments.

Fabrication of a high vacuum simulated bearing wear rig was completed. This rig will be used in existing vacuum systems to evaluate, primarily, solid lubricants to 1500°F.

Pressure-viscosity data for the diester bis(2-ethyl hexyl)sebacate were determined to 90,000 psig at 100°F and to 140,000 psig at 210°F. Agreement of these data with those of previous investigators is good. In addition, the viscosity and density of two fluids, a 5P4E polyphenyl ether and a chlorinated phenyl methyl silicone, were determined to 300°F. The maximum pressure of the work with the polyphenyl ether was 70,000 psig and the maximum pressure of the work with the silicone was 90,000 psig. Both fluids became too viscous for higher pressure work.

(This abstract is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of Fluid and Lubricant Materials Branch, MANL, Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson AFB, Ohio 45433.)

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I.

INTRODUCTION

Fluids and lubricants which are potentially superior to those now commonly used in high performance aircraft are being studied over a wide range of environmental conditions to determine the effects of these conditions on their properties and characteristics. Knowledge gained as a result of these studies can be used to aid in specifying applications or in the further development of fluids and lubricants to meet the requirements of both existing and future aircraft and aerospace vehicles. Overall activities on this three-year program include experimental investigations and/or apparatus development in the following areas.

1. Shear stability of experimental fluids at elevated temperatures;

2. Lubrication behavior of hydraulic fluids;

3. Bearing lubrication with solid film lubricants;

4. Pressure-viscosity phenomena;

5. Bulk modulus characteristics;

6. Rolling and sliding friction studies of fluids;

7. Extreme pressure behavior of lubricants;

8. Lubricity; and

9. Design and development of a 550°F/5,000-psig hydraulic circuit and a high vacuum simulated bearing wear rig.

Project activities during 1968, the third year of a three-year effort, included work in the following areas:

1. Fluid stability and lubricity when subjected to high shear stresses at high temperatures;

2. Bulk modulus measurements at high temperatures and pressures;

3. Fluid lubricity studies at elevated temperatures in four-ball equipment;

4. Development and check-out of a high vacuum simulated bearing wear rig; and

5. High pressure effects on the viscosity of fluids;

Results of these activities are presented in the remainder of this report.

II.

STABILITY OF FLUIDS AT ELEVATED TEMPERATURES

A. Background

Two fluid circuits, the "homogenizer pump stand" and the "high temperature hydraulic circuit," are used to investigate the shear and thermal stability of fluids at elevated temperatures. The configurations of these circuits are shown schematically in Figures 1 and 2. Each system has a separate and distinct purpose, although they both have the common function of evaluating the resistance of fluids to degradation when they are sheared at high temperatures. Shearing is done mechanically in both circuits by forcing the experimental fluids through small and adjustable constrictions in splined plug valves. As the fluids flow through these throttling valves, the pressure drops from about 3,000 psig to about 100 psig. Fluid degradation is judged by examining samples of fluids periodically removed from the circuits during each experiment for changes in (1) flash point, (2) fire point, (3) viscosity at 100° and 210°F, (4) neutralization number, (5) molecular structure as indicated by infrared traces, and (6) amount of insolubles removed by centrifuging.

In the homogenizer pump stand, fluids are pumped at about 5 gpm at temperatures as high as 800°F. The pump, a modified Manton-Gaulin homogenizer, is capable of pumping fluids with poor lubricity. The circuit includes (1) a 440C stainless steel lacquer indicator to show the tendencies of the test fluids to deposit lacquer or other materials on close-fitting metal parts, (2) a corrosion indicator to give a measure of test fluid attack of materials generally used in aircraft hydraulic systems, and (3) a filter to give an indication, by means of pressure drop measurements and visual examination, of the sludging tendencies of the test fluids. Normally, experiments in the homogenizer pump stand are run continuously for 100 hr. with an average discharge pressure of 3,000 psig from the three-piston pump.

If a fluid is not appreciably degraded during pump stand experiments, it is then considered as a candidate for experiments in the high temperature hydraulic circuit. Information can be secured on fluid stability and the ability of fluids to prevent wear in various configurations of metallic contacts in the hydraulic pumps. The pumps normally used in this circuit are seven-piston New York Air Brake Model 69W03006-2 aircraft-type pumps and are generally driven at 3,750 rpm. Fluids used in this circuit must be capable of lubricating the close-fitting sliding metal contacts in these pumps. The pump discharge pressure is cycled at 1-min. intervals from about 2,700 psig, where the flow is approximately 9 gpm and maximum driving power is required,

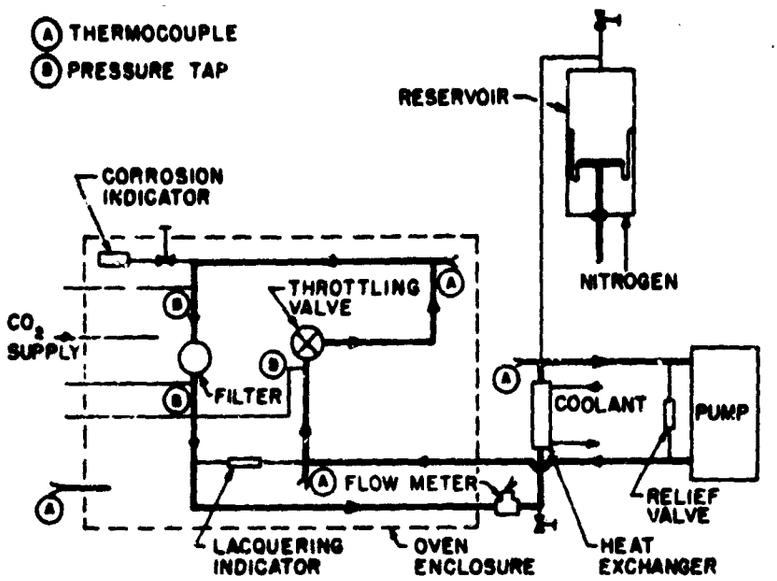

Figure 1 - Schematic Diagram of the Homogenizer Pump Stand

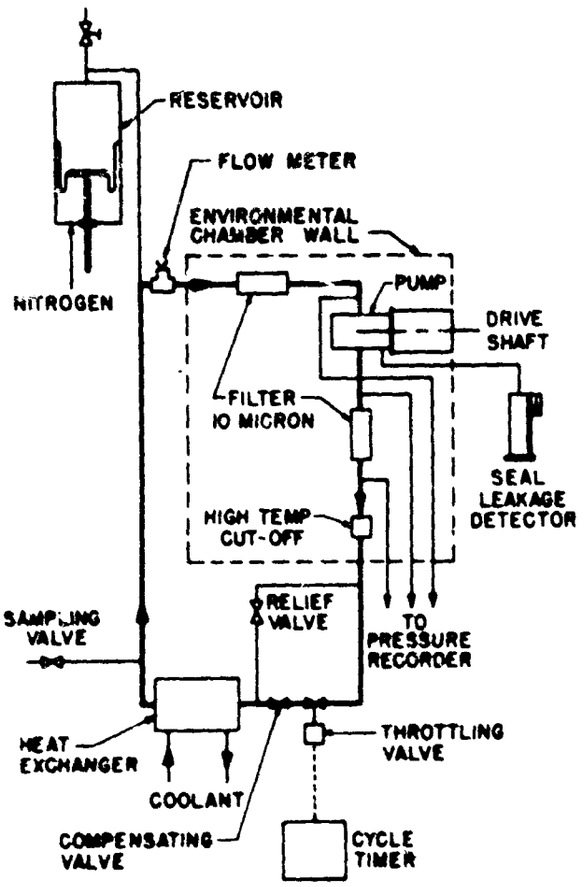

Figure 2 - Schematic Diagram of the High-Temperature Hydraulic Circuit

to about 3,000 psig, where a minimum flow of 0.5 to 1.0 gpm is delivered by these pressure-compensated pumps. Experiments in this high temperature hydraulic circuit are normally run continuously for 50 hr. at 400°F, 50 hr. at 500°F, and 50 hr. at each 50°F temperature increment above 500°F until the fluid degrades, the pump fails, or the procedure is altered. The pumps are disassembled and inspected initially and at the end of each 50-hr. run. The circuit is equipped with 10-micron filters in the pump inlet and pump discharge lines. Tendencies of the fluids to form sludge, insolubles, and/or permit excessive pump wear are indicated by pressure drop measurements across these filters and by visual inspection.

Four pump loop experiments were run with XF-1-0301, a fluorosilicone. This fluid, manufactured by Dow Corning, is based on a trifluoropropyl methyl substituted polysiloxane containing minor additives to improve boundary lubrication performance. The results of these investigations, conducted at 400°, 450°, 500° and 600°F, indicated that the XF-1-0301 fluid (1) is reasonably stable to 450°F, (2) will form solids at 500°F without appreciable changes in physical properties, and (3) will suffer decreases in viscosity and flash and fire points and an increase in neutralization number at 600°F. No gross molecular changes occurred during any of the experiments.

B. Homogenizer Pump Stand

Two experiments were conducted in the homogenizer pump stand circuit, shown schematically in Figure 1, with Dow Corning XF-1-0301, Lot No. 4. No significant change in fluid properties occurred as a result of pumping the fluid through the pump stand circuit for 100 hr. at 450°F. However, some shear and/or thermal degradation of the XF-1-0301 test fluid was indicated during a 58-hr. run at 600°F. The latter evaluation was stopped short of the planned 100-hr. duration because the pumping rate dropped below the sensitivity threshold of the flowmeter--less than 0.6 gpm.

1. Fluid: Samples of the XF-1-0301 test fluid were removed from the pump loop periodically while the experiments were in progress. Data determined from examinations of the samples are listed in Tables I and II. These fluid data are also shown graphically, along with some experiment operating data (pumping rate and filter pressure drop), on Figures 3 through 8.

TABLE I

FLUID DATA FOR THE 100-HR. 450°F HOMOGENIZER PUMP STAND EXPERIMENT WITH DOW CORNING XF-1-0301

Hours Pumped	Viscosity (cSt.) 100°F	210°F	Flash Point (°F)	Fire Point (°F)	Neut. No. (mg KOH/gm)	Insolubles
New	26.85	5.87	450	518	< 0.01	-
0	26.23	5.74	450	517	"	-
2	25.87	5.69	445	512	"	-
4	25.65	5.65	447	510	"	-
6	25.74	5.61	444	510	"	-
8	25.58	5.59	447	512	"	-
10	25.56	5.59	448	510	"	-
25	25.65	5.60	445	512	"	Trace
50	25.58	5.57	443	510	"	Trace
75	25.10	5.52	445	510	"	Trace
100	25.08	5.52	444	509	"	Trace

Note: Viscosity, flash and fire point, and neutralization number determined in acccrdance with ASTM D445-65, D92-66, and D664-58, respectively.

TABLE II

FLUID DATA FOR THE 58-HR. 600°F HOMOGENIZER PUMP STAND EXPERIMENT WITH DOW CORNING XF-1-0301

Hours Pumped	Viscosity (cSt.) 100°F	210°F	450°F	Flash Point (°F)	Fire Point (°F)	Neut. No. (mg KOH/gm)	Insolubles
New	26.85	5.87	1.36	450	518	0.01	-
0	26.05	5.73	1.33	445	518	0.05	-
2	25.61	5.70	1.32	445	515	0.06	-
4	25.18	5.61	1.31	430	510	0.08	-
6	25.17	5.61	1.32	430	505	0.08	-
8	24.99	5.61	1.31	425	500	0.08	-
10	25.03	5.56	1.32	419	495	0.09	-
25	24.32	5.50	1.31	390	485	0.13	-
35	24.23	5.41	1.31	395	490	0.18	Measurable
45	23.82	5.45	-	428	480	0.20	Measurable
50	23.62	5.42	-	400	482	0.21	Measurable
58	23.97	5.45	-	375	480	0.21	Measurable

Note: Viscosity, flash and fire points, and neutralization numbers determined in accordance with ASTM D445-65, D92-66, and D664-58, respectively.

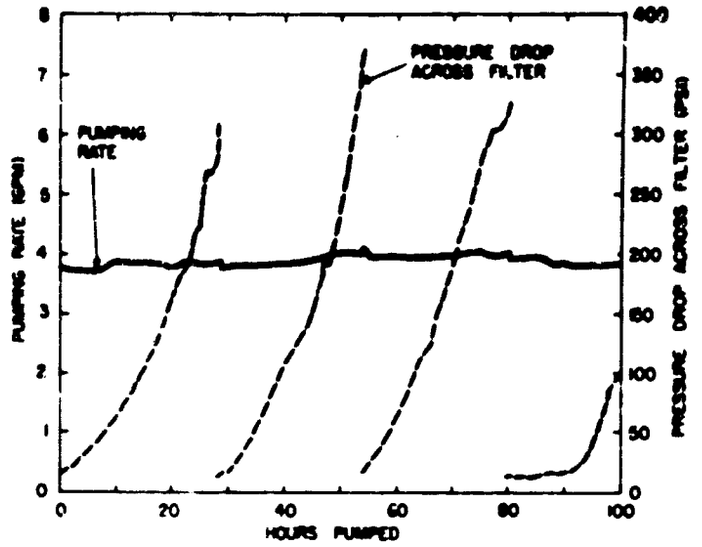

Figure 3 - Effect of Pumping Time at 450°F on Pumping Rate and Filter Pressure Drop - XF-1-0301

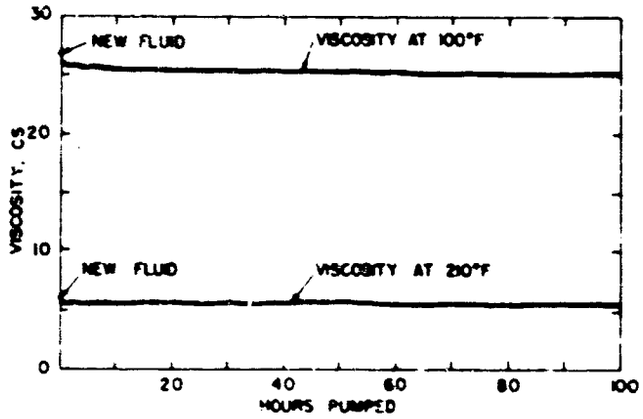

Figure 4 - Effect of Pumping Time at 450°F on Viscosity - XF-1-0301

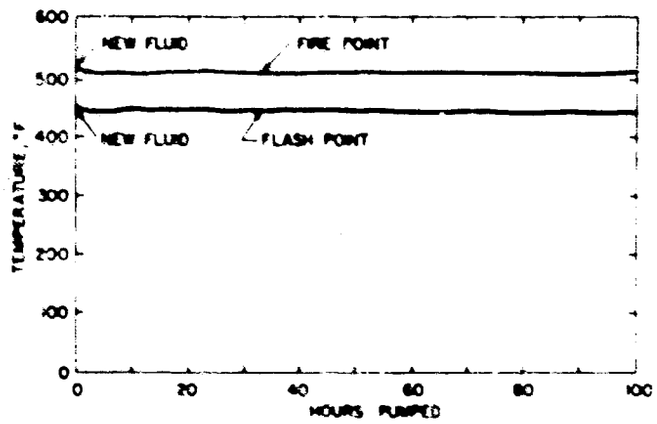

Figure 5 - Effect of Pumping Time at 450°F on Flash Point and Fire Point - XF-1-0301

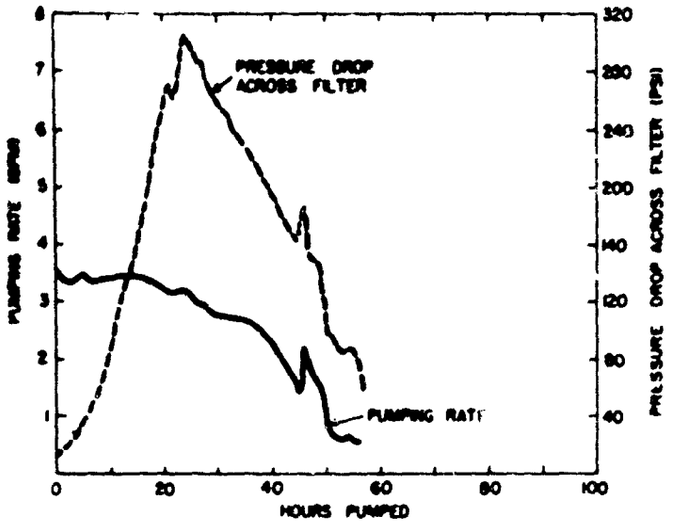

Figure 6 - Effect of Pumping Time at 600°F on Pumping Rate and Filter Pressure Drop - XF-1-0301

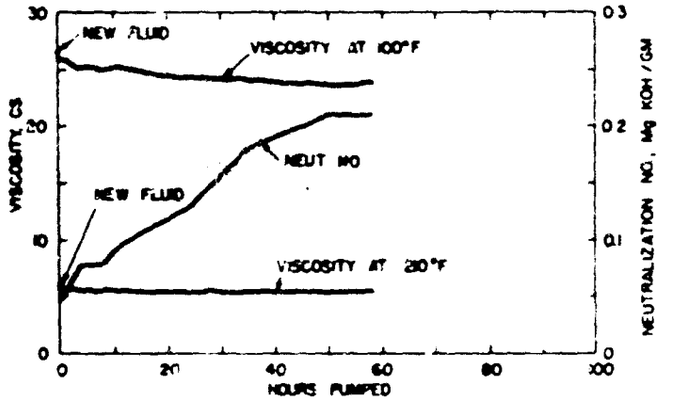

Figure 7 - Effect of Pumping Time at 600°F on Viscosity and Neutralization Number - XF-1-0301

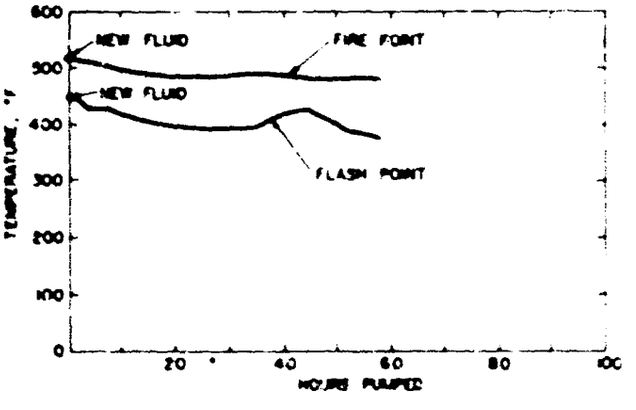

Figure 8 - Effect of Pumping Time at 600°F on Flash Point and Fire Point - XF-1-0301

The viscosity of the test fluid decreased as both experiments progressed. During the 100-hr. run at 450°F, the 100°F viscosity dropped from 26.85 cSt. for new XF-1-0301 fluid to 25.08 cSt. at the end of the run (6.6 percent loss) and during the 58-hr. run at 600°F, the 100°F viscosity dropped to 23.62 cSt. after 50 hr. of pumping (12.0 percent loss) and then rose to 23.97 cSt. at the end of the run. The final sample of the 58-hr./600°F run was taken after a seal ruptured and some new fluid from the reservoir was forced into the circuit. The 210°F viscosity dropped from 5.87 cSt. for new fluid to 5.52 cSt. after 100-hr. of pumping at 450°F (6.0 percent loss) and to 5.45 cSt. after 58 hr. of pumping at 600°F (7.2 percent loss). Because of the unexplained decrease in pumping rate during the 600°F run, approximate 450°F viscosities of the fluid samples from this run were determined. It was thought that a large decrease in viscosity may have occurred and permitted excessive pump slippage. The viscosity data in Table II do not indicate the drop in pumping rate was caused by any permanent viscosity loss. The viscosity values at 450°F listed in Table II should not be considered precise because the typical viscosity data published by Dow Corning in a New Product Information Sheet for XF-1-0301 were used in establishing a 450°F calibration constant for the capillary viscometer tube. Viscosities of new fluid published by Dow Corning (29.0 cSt. at 100°F, 6.2 cSt. at 210°F, and 1.45 cSt. at 450°F) were compared with those determined by MRI for new XF-1-0301, Lot No. 4 (26.85 cSt. at 100°F and 5.87 cSt. at 210°F). The value of 1.36 cSt. at 450°F for new Lot No. 4 test fluid was selected as being reasonable. The 450°F viscosity of the test fluid decreased to 1.3. cSt. after 4 hr. of pumping (3.7 percent loss) and remained fairly constant thereafter. Determinations for the last three fluid samples (45-, 50-, and 58-hr.) were not attempted at 450°F because of the influence of graphite debris from one of the secondary pump piston seals in the samples.

New fluid had a COC flash point of 450°F. The test fluid flash point after 100 hr. of pumping at 450°F was 444°F (1.3 percent loss) and after 58 hr. of pumping at 600°F it was 375°F (16.7 percent loss). New fluid had a COC fire point of 518°F. The test fluid fire point after 100 hr. of pumping at 450°F was 509°F (1.7 percent loss) and after 58 hr. of pumping at 600°F, it was 480°F (7.3 percent loss). Decreases in viscosity and flash and fire points for the two experiments with XF-1-0301 are summarized in the following tabulation:

Test Conditions	Viscosity 100°F	Viscosity 210°F	Flash Point	Fire Point
100 hr. at 450°F	-6.6%	-6.0%	-1.3%	-1.7%
58 hr. at 600°F	-12.0%	-7.2%	-16.7%	-7.3%

The neutralization number of new fluid is about 0.01 mg KOH/gm. No appreciable change occurred during the 100 hr. run at 450°F, but the neutralization number gradually rose to 0.21 mg KOH/gm after 58 hr. of pumping at 600°F.

Twenty-milliliter portions of the last four fluid samples from each experiment were centrifuged for 1 hr. on a 5.9 cm. mean radius arm at 20,000 rpm. Only a small trace of dark colored material was found in each of the four centrifuge tubes containing fluid from the 100 hr/450°F run. The four centrifuge tubes from the 58 hr/600°F run each contained about the same amount of dark colored material--on the order of 0.0063 gm/ml of fluid. It is probable that most of this material came from the disintegrating Grafoil* secondary piston seal and not from the fluid.

Infrared spectral traces were made of new XF-1-0301, Lot No. 4 fluid as installed in the circuit before each run and also of the 100-hr. sample from the 450°F run and the 58-hr. sample from the 600°F run. All traces are virtually identical, except for minor differences in transmittance, and indicate no gross molecular changes occurred during either pump loop experiment.

New XF-1-0301 is clear and almost colorless. Fluid from Lot No. 4 used in the pump stand experiments had a very slight yellow cast. The first samples from the 100-hr. run at 450°F appeared the same as new fluid but the test fluid became progressively more yellow-amber and slightly cloudy as pumping continued. The 100-hr. sample was very pale amber and somewhat cloudy although it remained relatively transparent. During the 58-hr. run at 600°F, the test fluid changed from the almost colorless appearance of new fluid through various deepening shades of yellow to opaque black after 35 hr. of pumping. The presence of fine graphite particles from the disintegrating pump piston secondary seal probably caused the fluid to become dark. After centrifuging, the fluid in the centrifuge tubes was yellow-amber in color and clear.

2. Corrosion data: Data regarding the seven types of metallic washer-shaped corrosion specimens are summarized in Table III for the 100 hr/450°F run and in Table IV for the 58 hr/600°F run. Two specimens of each type of metal are mounted in the corrosion indicator (location shown in Figure 1) during an experiment. Examination of the specimens at the end of the 100-hr. run at 450°F (Table III) revealed that all specimens were slightly darkened, none had a surface texture change, all the steel and titanium specimens gained weight, one aluminum specimen gained and one lost weight, and the beryllium copper specimens did not change weight. All of the weight changes were small. Examination of the corrosion specimens from the 58-hr. run at 600°F revealed a different, almost opposite effect. The aluminum specimens

* Trade name - Carbon Products Division, Union Carbide Corporation.

TABLE III

CORROSION DATA FOR THE 100-HR 450°F HOMOGENIZER PUMP STAND EXPERIMENT WITH DOW CORNING XF-1-0301

Material	Weight Change (mg/cm^2) Spec. No. 1	Spec. No. 2	Avg.	Change in Appearance Color	Texture
Aluminum, 2024-T4	-0.063	+0.063	0.0	Slightly Darkened	No Change
M-1 Tool steel	+0.183	+0.206	+0.195	Slightly Darkened	No Change
Chrome moly steel, 4140	+0.111	+0.109	+0.110	Slightly Darkened	No Change
302 Stainless steel	+0.152	+0.087	+0.120	Slightly Darkened	No Change
440 Stainless steel	+0.192	+0.190	+0.191	Slightly Darkened	No Change
Titanium, RC 130B	+0.151	+0.153	+0.152	Slightly Darkened	No Change
Beryllium copper QQ-C-530	0.0	0.0	0.0	Slightly Darkened	No Change

TABLE IV

CORROSION DATA FOR THE 58-HR. 600°F HOMOGENIZER PUMP STAND EXPERIMENT WITH DOW CORNING XF-1-0301

Material	Weight Change (mg/cm^2) Spec. No. 1	Spec. No. 2	Avg.	Change in Appearance Color	Texture
Aluminum, 2024-T4	-0.647	-0.161	-0.407	Slightly Darkened	No Change
M-1 Tool steel	+0.114	+0.023	+0.069	Dark--purple tint	No Change
Chrome moly steel	+0.067	0.0	+0.034	Dark--purple tint	No Change
302 Stainless steel	0.0	0.0	0.0	Slightly Darkened	No Change
440 Stainless steel	+0.043	0.0	+0.022	Dark--purple tint	No Change
Titanium, RC 130B	0.0	-0.066	-0.033	Slightly Darkened	No Change
Beryllium copper, QQ-C-530	+0.836	+0.764	+0.800	Dark	No Change

lost an appreciable amount of weight (0.41 mg/cm^2 average) and the beryllium copper specimens gained a considerable amount of weight (0.80 mg/cm^2 average). The other specimens did not have significant weight changes. All specimens were darkened and all steels, except 302 stainless, became quite dark and exhibited a purple tint. No specimens had any significant surface texture change.

3. Lacquer indicator: The lacquer indicator is made of 440C stainless steel and consists of a static 0.75 in. diameter piston and cylinder having a 0.0002 to 0.0004 in. diametral clearance. The piston has three lands, 0.312, 0.500 and 0.625 in. long, separated by 0.50 in. long relieved sections.

After the 100-hr. 450°F run, the piston required approximately 53-lb. force to break it loose from the cylinder. Once motion between the piston and cylinder was started, less than 1 lb. of force was required to remove the piston. There was no evidence of lacquer buildup on either the piston or cylinder. The high pressure end of the lacquer indicator contained some graphite material from the secondary piston seals. The filter removed virtually all of the graphite from the test fluid so there was no deposit of this material in the low pressure side of the indicator.

After the 58-hr. 600°F run, the piston required approximately 430-lb. force to break it loose from the cylinder. Once motion between the piston and cylinder was started, 20- to 30-lb. force was required to remove the piston. There was no evidence of lacquer buildup on either the piston or cylinder although the indicator had a considerable amount of black material in it that presumably came from the secondary piston seals. Cleaning of the piston and cylinder with soft cloths restored sufficient clearance for the piston to fall through the cylinder without external force.

4. Filters: The pump loop is equipped with a 33-micron filter consisting of 12 discs having fine outer screens serving as the filtering media. The 100-hr. run at 450°F required four sets of filter discs because the Grafoil secondary piston seals had a tendency to add flakes of graphite to the test fluid. These flakes were caught by the filter media and caused excessive pressure drops to develop across the filter assembly. A total of 82.2 gm. of graphite was removed from the 4 sets of filters. One side of the last set of filter discs used is shown in Figure 9. These discs were in the circuit 20 hr. and yielded 19.0 gm. of residue.

One set of filter discs was used during the 58-hr. 600°F run. These discs, shown in Figure 10, had 29.5 gm. of residue on them. The residue coating was fairly thick and would have caused a large pressure drop across the filter assembly if the flow rate had not declined during the run.

5. Operation of experiments: Operating data are summarized in Table V for the 100 hr/450°F run and in Table VI for the 58-hr/600°F run.

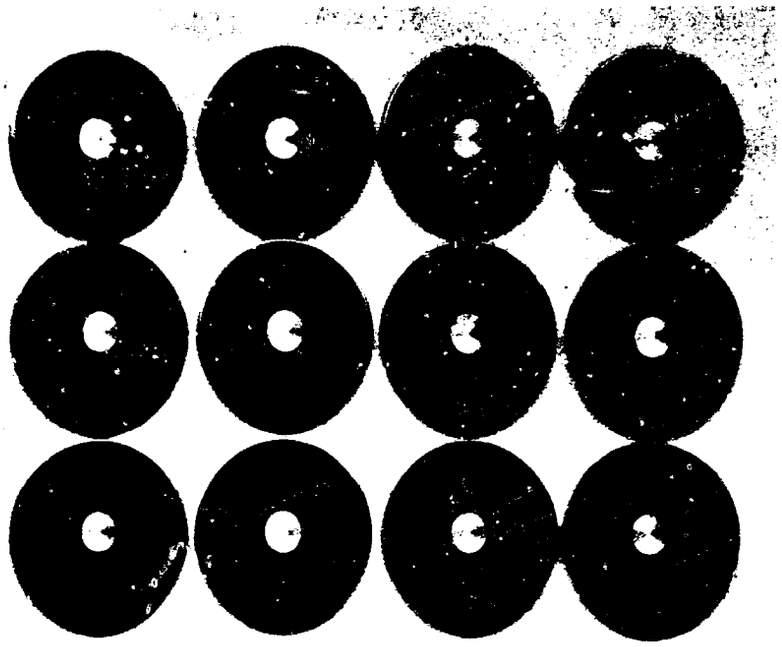

Figure 9 - Pump Stand Filter Discs After Use During Final 20 Hr. of 100-Hr. Experiment at 450°F With XF-1-0301

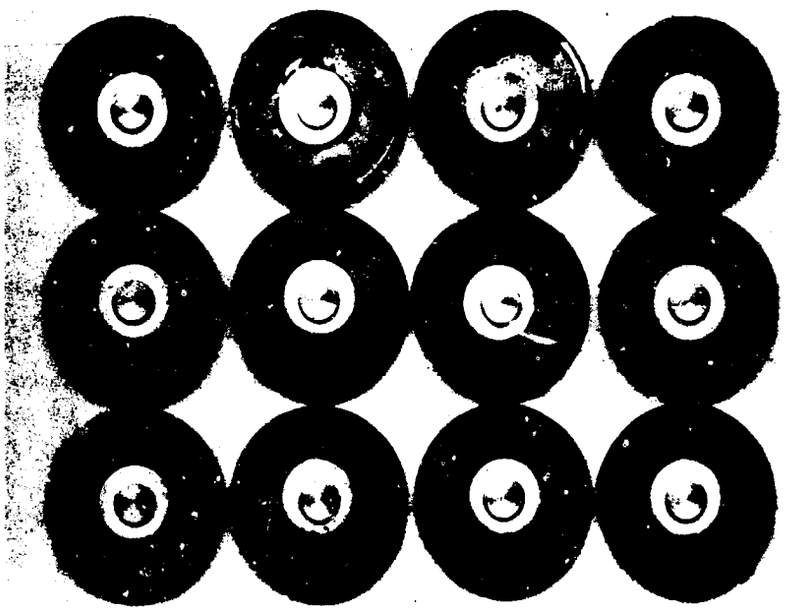

Figure 10 - Pump Stand Filter Discs After 58-Hr. Experiment at 600°F With XF-1-0301

TABLE V

OPERATING DATA FOR THE 100-HR. 450°F HOMOGENIZER PUMP STAND EXPERIMENT WITH DOW CORNING XF-1-0301

	Maximum	Minimum	Average*
Duration of test (hr.)	-	-	100
Pumping rate (gpm)	4.04	3.68	3.86
Reservoir pressure (psig)	60	60	60
Pump discharge pressure (psig)	3,075	2,950	3,003
Pressure before filter (psig)	425	70	184
Pressure after filter (psig)	60	55	57
Filter pressure drop (psi)	368	12	127
Pressure between piston seals (psig)	220	100	203
Temperatures (°F):			
Before throttling valve	450	435	444
After throttling valve	465	450	455
Oven	320	295	307
Shear cycles**	-	-	15,440
Fluid composition at end of run (%):			
Original charge	-	-	76.3
New fluid added	-	-	23.7

* Average of 66 readings taken at least 1 hr. apart.

** Total fluid quantity pumped divided by circuit volume of approximately 1.5 gal.

TABLE VI

OPERATING DATA FOR THE 58-HR. 600°F HOMOGENIZER PUMP STAND EXPERIMENT WITH DOW CORNING XF-1-0301

	Maximum	Minimum	Average*
Duration of test (hr.)	-	-	58
Pumping rate (gpm)	3.53	0.57	2.55
Reservoir pressure (psig)	65	60	61.2
Pump discharge pressure (psig)	3,000	2,825	2,979
Pressure before filter (psig)	360	70	203.8
Pressure after filter (psig)	60	45	53.4
Filter pressure drop (psi)	305	12	150.2
Pressure between piston seals (psig)	250	200	229
Temperatures (°F)			
Before throttling valve	593	556	583
After throttling valve	605	575	596
Oven	687	590	626
Shear cycles**	-	-	5,916
Fluid composition at end of run (%)***			
Original charge	-	-	60.7
New fluid added			39.3

* Average of 40 readings taken at least 1 hr. apart.

** Total fluid quantity pumped divided by circuit volume of approximately 1.5 gal.

*** Composition before piston seal failure at end of run.

a. 100-hr. Experiment at 450°F: During the 450°F run, the average test fluid temperature before the throttling valve was 444°F and the average temperature after the throttling valve, on the low pressure side, was 455°F. The pump discharge pressure averaged 3,003 psig. The fluid charge was pumped around the closed loop, and through the small orifice in the throttling valve, about 15,440 times.

A new type of material for piston secondary seals was used for this run. These seals, Union Carbide's Grafoil, contain 99.9 percent graphite, no resins or inorganic fillers, and are flexible. These seals were effective in reducing leakage past them, in this run, to essentially zero. Approximately 695 ml. of fluid leaked from the circuit during the 100-hr. test and probably most of this was lost through minor leaks in the circuit tubing inside the oven and then vaporized. It is estimated that the test fluid at the end of the run was composed of 76.3 percent of fluid initially placed in the circuit and 23.7 percent of fluid added from the reservoir during the run.

The Grafoil piston seals exhibited a tendency to add flakes of graphite to the test fluid. These flakes were effectively caught by the 33-micron circuit filter but the test had to be interrupted three times to change filter discs--a 3-hr. stop after 28.25 hr. of pumping, a 2-hr. stop after 54.25 hr. and a 1.25-hr. stop after 80 hr.

b. 58-hr. Experiment at 600°F: During the 600°F run (Table VI), the average test fluid temperature before the throttling valve was 583°F and the average temperature after the throttling valve was 596°F. The pump discharge pressure averaged 2,979 psig and dropped as low as 2,825 psig because of the decreasing pumping rate while the system was operating unattended. The fluid charge was pumped around the closed loop, and through the small orifice in the throttling valve, about 5,900 times.

The pressure drop across the filter was 12 psig at the start of the experiment when the pumping rate was 3.53 gpm. The pressure drop rose rapidly to 305 psig at 3.18 gpm after 24 hr. of pumping and then dropped almost as rapidly to 60 psig at the end of the run when the flow rate was somewhat less than 0.6 gpm. (See Figure 6.)

The reason for the large decrease in pumping rate during the evaluation is unknown. There is no question that the flow rate did decrease. Every instrument in the pump stand control console confirmed this fact. The temperature of the oven containing much of the circuit had to be raised repeatedly to maintain the test fluid at about 600°F, the size of the throttling valve orifice was decreased 41 times during the evaluation to keep the pump discharge pressure at about 3,000 psig, the pressure drop across the filter decreased rapidly during the last 34 hr. of the run, and the flowmeter indicated the flow rate was decreasing. Examination after the run showed that all filter discs were well loaded (Figure 10) but none were

ruptured. Inspection of the pump revealed only one problem - a considerable amount of one of the Grafoil secondary piston seals had eroded and the loosened material was carried away by the test fluid. Defective secondary piston seals would influence only the leakage rate and not the pumping rate. The pump valves (inlet, discharge and high pressure by-pass) were found to operate freely and to be in good condition. After the eroded pump piston secondary seal was replaced, the circuit was flushed with Stoddard Solvent. The pump flow rate was normal at about 5.25 gpm. Another possible cause of the decrease in pumping rate could be a large temporary viscosity loss of the test fluid which would permit excessive leakage past the ductile iron pump primary seals. It is not likely that there was enough permanent viscosity loss (Table II) to cause this large and unprecedented decrease in pumping rate. There is only slight evidence that a temporary viscosity loss occurred. Fluid leaking past the primary piston seals is ducted, through a needle valve, back to the pump inlet. The pressure up-stream of the needle valve was 200 psig at the start of the experiment and rose at nearly a constant rate to 250 psig at the end of the 58-hr. pumping period. No adjustments were made in the needle valve during this period. It is possible debris from the eroding secondary seal was constricting the needle valve but this same type of debris did not cause any rise in the pressure before the needle valve during the 100-hr. run at 450°F.

Leakage during the 600°F experiment totaled about 1,620 ml. Most of the fluid was lost through the Grafoil secondary piston seals. There was virtually no leakage during the early part of the run but the leakage at the end of the run, although erratic, averaged about 50 ml/hr.

After the flow rate dropped below 0.6 gpm at 58 hr., the decision was made to either increase the flow rate by increasing the pressure between the pump piston primary and secondary seals or to terminate the run. The pressure was increased from 250 to 600 psig by decreasing the needle valve orifice in the line connecting the interseal area to the pump inlet line. There was no indicated increase in pump flow rate but the pressure increase was sufficient to cause a complete failure of one of the Grafoil pump secondary piston seals after a few minutes at 600 psig. A major part of the test fluid charge was blown out through this broken seal in the few seconds required to stop the pump and relieve the reservoir pressure. The final fluid sample (58 hr.) included an appreciable amount of unworked fluid forced into the circuit when the seal failed.

C. High Temperature Hydraulic Circuit

Shear/thermal stability experiments were conducted with XF-1-0301, Lot 5 fluorosilicone at 400° and 500°F in the high temperature hydraulic circuit shown in Figure 2. The test fluid was not appreciably degraded during either run. Flash point, fire point and viscosity of the test fluid decreased only a relatively small amount during the runs. New fluid was slightly basic but

became essentially neutral as pumping time increased. The pressure drops across the filters increased only slightly during the 400°F run but increased a large amount during the 500°F run. Examination of the filters after the 500°F run showed they were covered with fine black material, which probably came from the test fluid as well as a few copper-colored particles, which came from the pump.

Both experiments were conducted with the same pump and the same fluid. The 400°F experiment was run for 50 hr., the pump was disassembled, inspected and reassembled; then the 500°F experiment was run for 30.45 hr.

Conduct of both experiments was almost routine. The fluid was initially heated to 400°F by using the environmental chamber heaters and by operating the NYAB pump. It was necessary to stop the pre-run work just prior to the zero-hour point in the test because of loss of end-clearance in one of the shafts in the pump drive system. Loading of the pump bearings was unaffected by this development. One of the couplings in the pump drive system was shortened, the test fluid was heated to 400°F again, and the first run was started. A 10-min. interruption of this 50-hr. run at 400°F occurred after 5.73 hr. of pumping because of a defective safety interlock switch. The remainder of the 400°F run proceeded without incident. Inspection of the pump between the 400°F and 500°F runs showed that it was in good condition but had a small amount of copper-colored material smeared onto some of the rubbing surfaces. The copper-colored material, probably from the pump bearings, was removed, the pump was reassembled, and the 500°F run was started. This run was interrupted after 7 hr. of pumping to permit changing of a leaking pump shaft seal. The run was again stopped after 30.45 hr. of pumping to change the discs in both filters because they were becoming plugged. Inspection of the pump at this time showed that the piston heads were severely worn so the run was terminated.

1. Fluid: Samples of the XF-1-0301 fluorosilicone test fluid were removed from the hydraulic circuit periodically while both the experiments were in progress. Data determined from examination of the samples are listed in Table VII. Viscosity, flash point, and fire point all decreased a small amount.

The decreases in viscosity during the experiments could well be partially attributed to differences in gas content of the specimens or to experimental variation. The 100°F viscosity changed from 29.11 cSt. for new XF-1-0301 to 28.66 cSt. after the 50 hr/400°F run (1.55 percent decrease) and then to 28.45 cSt. after the 30.45 hr/500°F run (2.27 percent decrease overall). The 210°F viscosity changed from 6.24 cSt. for new XF-1-0301 to 6.23 cSt. after the 50 hr/400°F run (0.16 percent decrease) and then to 6.09 cSt. after the 30.45/500°F run (2.40 percent decrease overall). By way of comparison, the viscosity of the XF-1-0301 (Lot 4) fluid pumped for 100 hr. at 450°F in the homogenizer pump stand decreased 6.59 percent at 100°F and 5.96 percent at 210°F.

TABLE VII

FLUID DATA FOR DOW CORNING XF-1-0301 LOT 5 FLUOROSILICONE AT 400°F AND 500°F IN THE HIGH TEMPERATURE HYDRAULIC CIRCUIT

Fluid Temp. (°F)	Hours Pumped	Viscosity (cSt.) 100°F	Viscosity (cSt.) 210°F	Flash Point (°F)	Fire Point (°F)	Neut. No. (mg KOH/gm.)	Insolubles
	New	29.11	6.24	465	523	0.112*	-
400	0	29.13	6.19	452	515	0.004*	-
	2	29.31	6.19	440	512	0.004*	-
	4	28.97	6.16	448	512	0.004*	-
	6	28.78	6.17	450	512	0.004*	-
	8	28.73	6.17	443	510	0.003*	Trace
	10	28.97	6.30	448	512	0.000	Trace
	25	28.93	6.21	452	512	0.000	Trace
	50	28.66	6.23	450	510	0.003**	Trace
500	0	29.28	6.27	450	516	0.000	-
	2	29.03	6.12	448	515	0.000	-
	4	28.76	6.13	450	515	0.000	-
	6	28.72	6.14	450	516	0.000	-
	8	28.78	6.12	451	515	0.000	-
	10	28.52	6.15	448	515	0.000	Trace
	25	28.33	6.10	448	516	0.000	Trace
	30.45	28.45	6.09	445	515	0.000	Trace

* Fluid basic.

** Fluid acidic.

Decreases in the flash and fire points were also relatively small. New XF-1-0301 (Lot 5) has a COC flash point of 465°F and a fire point of 523°F. After the 50-hr/400°F run, the test fluid had a flash point of 450°F (3.23 percent decrease) and a fire point of 510°F (2.49 percent decrease). After the 30.45-hr/500°F run, the test fluid had a flash point of 445°F (4.30 percent decrease overall) and a fire point of 515°F (1.55 percent decrease overall). These declines in flash and fire points are not large enough to be considered significant. In comparison, during the 100 hr/450°F run in the homogenizer pump stand with XF-1-0301 (Lot 4), the flash point dropped 1.35 percent and the fire point dropped 1.75 percent.

New XF-1-0301, Lot 5 is slightly basic and has an equivalent neutralization number of 0.112 mg KOH/gm. The test fluid became neutral after 10 hr. of pumping at 400°F and remained essentially neutral during the remainder of the 400°F run and during the 500°F run. The neutralization numbers listed on Table VII are quite small.

All XF-1-0301 specimens removed from the high temperature hydraulic circuit during both the 400° and 500°F runs contained some insoluble material that settled to the bottom of the glass specimen jars. The color of these insolubles ranged from an off-white to dark brown. There was no appreciable difference in the color of these insolubles as the pumping time increased. The majority of this material was fairly light in color and some of it appeared to remain suspended in several of the fluid samples to give them a slight haze. It is believed these insolubles came from the fluid and may well have been precipitated by thermal rather than shear effects because the zero-hour sample of the 400°F run had appreciably more of the insoluble material than did the samples taken later in either experiment.

It does not seem likely that the insolubles in the XF-1-0301 came from any contaminants in the pump loop. The circuit was thoroughly cleaned prior to the XF-1-0301 runs with Freon 113 (DuPont TF solvent) to remove the PR-143 polymeric perfluorinated fluid previously tested. The filters and the pump, which had been thoroughly cleaned, were then installed and the circuit was flushed at room temperature with new XF-1-0301 Lot 5. This fluid was subsequently drained before the test fluid charge of XF-1-0301 was installed. There was no precipitate on the bottom of the glass container used to store the flush XF-1-0301 and this fluid remained clear. The traces of insolubles noted in Table VII were centrifuged from 10 gm. of each of the seven fluid samples indicated. The centrifuge was operated at 22,000 rpm for 1 hr. on a 5.9 cm. mean radius arm. Insolubles on the bottom of each of the seven centrifuge tubes varied in color from a light to a dark tan. The liquid in the centrifuge tubes was clear. Fluid samples were colorless for the first 8 hr. of the 50 hr/400°F run but all samples for both runs taken after the 8 hr. point in the 400°F run had a yellow tint. There was no pronounced specimen color difference as pumping time increased beyond the 10 hr. point of the 400°F run. All specimens remained relatively clear although they all had a slight haze apparently from suspended insolubles. As noted above, the zero-hour specimen for the 50 hr/400°F run had more insolubles suspended in it than did any other specimen. In general, the haze in the fluid samples decreased as pumping time increased. This decrease occurred possibly because the insolubles were becoming caught on the filters.

Infrared traces were made of new XF-1-0301 Lot 5 and the final fluid sample from each experiment. No gross molecular changes in the fluid were indicated by the infrared traces.

2. Pump: The pump used during the 50-hr/400°F and 30.45-hr/500°F experiments with XF-1-0301 Lot 5 was Model 69W03006-2, Serial No. B2-13, manufactured by New York Air Brake Company. The manufacturer normally runs-in these pumps for 2 hr. at speeds to 6200 rpm with MIL-L-7808 and then flushes and fills them with MIL-C-8188.

The pump was prepared for use with XF-1-0301 by (1) completely dismantling it, (2) cleaning all parts in benzene, then chloroform, and then acetone, (3) visually inspecting all parts, (4) measuring the longitudinal movement between the piston heads and the piston bodies, (5) weighing the piston assemblies, collars and sleeves, (6) cleaning all parts again, (7) reassembling the pump with new Viton "A" seals and with all parts liberally coated with XF-1-0301, and (8) filling the pump with XF-1-0301 test fluid.

Inspections of the pump components were made before and after each of the two runs at 3,750 rpm with XF-1-0301. An initial inspection of the new pump revealed that it was one of the cleanest new pumps of this type that MRI has used. The only noteworthy irregularity in the new pump was the relatively rough appearance of part of the piston head balls of two of the piston heads. These balls operate in a socket in the piston bodies and the portion that can be seen during inspection is unloaded when a longitudinal compressive load is applied to the piston. All piston ball joints operated freely when rotated and rocked while under a compressive load.

a. 400°F Experiment: After the 50-hr/400°F run with XF-1-0301 Lot 5, inspection of the pump showed that it was still in very good condition but that a small amount of copper alloy material, primarily from the piston heads, had been deposited on some of the pump surfaces. There were no highly polished or worn areas on the copper alloy thrust bearing to indicate material loss from this bearing but the piston heads were changed in that:

(1) The central portion of the piston heads, defined roughly by the radius of the oil grooves, was rather dull. These surfaces were highly polished when the pump was new.

(2) The piston head areas beyond the oil grooves had highly polished bands about 1/8 in. wide that had been, when the pump was new, covered with the random light scratches normally seen on new piston heads.

(3) The remainder of the piston heads, narrow annular rings near the O.D. of the piston heads, were bright copper in color and rough.

(4) The two piston head balls, noted to have been rough when the pump was new, were still rough but somewhat smoother than they had been before the experiment. Operation under a longitudinal compressive load was still smooth.

Deposits of copper-colored material were noted on all rubbing surfaces of the camshaft, 6 of the 7 collars, the nutating plate, and the low pressure ends of the cylinders and pistons. All deposits were very thin and were, with only a few exceptions, a tarnished copper color. Small and bright copper deposits were noted on the low pressure ends of the pistons and cylinders, and on the surface of the camshaft contacted by the piston heads.

During the pump inspections before and after the 50-hr/400°F run with XF-1-0301, measurements were taken of some of the pump components. These measurements are tabulated on Tables VIII to XI. Weight changes of the piston assemblies (piston head plus piston) are listed in Table VIII. Three assemblies gained weight and four lost weight. All changes were 0.0006 gm. or less. In comparison, the average piston assembly weight loss during a similar 50-hr/400°F experiment with a deep dewaxed mineral oil was 0.0094 gm. The reason for weight gain shown by some of the piston assemblies listed in Table VIII is not known, but it is possible that some material was deposited on the interior piston surfaces that was not removed by solvent cleaning. Judging by the appearance of the piston heads, all of them lost some weight from these surfaces. The longitudinal movement between the pistons and the piston heads, listed in Table IX, increased an average of 0.0007 in. per piston. This increased movement could have been caused either by material loss or slight deformation of the ball joint material.

The collars, which slide on both the piston and the nutating plate, lost an average of 0.0083 gm. as shown on Table X. These losses are about 14 times as high as those of the 50-hr/400°F deep dewaxed mineral oil run and about 8 times as high as those of a 1,000-hr/400°F run with the polymeric perfluorinated PR-143. New collars usually have uniform scratches in the spherical portions that contact the pistons. When new, and after the 50-hr/400°F run, these collars were all highly polished on the spherical surfaces.

Six of the seven pump sleeve valves gained as much as 0.0011 gm. and had an average gain of 0.0005 gm. as shown in Table XI. These valves are essentially stationary with respect to the pump housing and the pistons slide through them for the entire stroke. Normally, as in the present case, there is very little weight loss from these components and often there is a small weight gain presumably because of the deposition of material from the test fluid. By way of comparison, the sleeves from the 50-hr/400°F run with a deep dewaxed mineral oil lost, on the average, about 0.0006 gm. each and the sleeves from the 1,000-hr/400°F run with PR-143 lost an average of about 0.0001 gm.

TABLE VIII

PUMP PISTON ASSEMBLY WEIGHT CHANGES DURING THE 400°F AND 500°F EXPERIMENTS WITH XF-1-0301

Cylinder No.	As Received	After 400°F Run	Change - 400°F Run	After 500°F Run	Change - 500°F Run	Total Change
1	87.8035	87.8041	+0.0006	87.7290	-0.0751	-0.0745
2	87.8614	87.8611	-0.0003	87.7736	-0.0875	-0.0878
3	87.5828	87.5823	-0.0005	87.5245	-0.0578	-0.0583
4	88.0938	88.0942	+0.0004	88.0449	-0.0493	-0.0489
5	88.1112	88.1108	-0.0004	88.0623	-0.0485	-0.0489
6	87.6159	87.6158	-0.0001	87.5466	-0.0692	-0.0693
7	87.6575	87.6581	+0.0006	87.5838	-0.0743	-0.0737
Total Change			+0.0003		-0.4617	-0.4614
Average Change			+0.00004		-0.0660	-0.0659

TABLE IX

CHANGE IN PUMP LONGITUDINAL PISTON BALL JOINT MOVEMENT DURING THE 400°F AND 500°F EXPERIMENTS WITH XF-1-0301**

Cylinder No.	As Received	After 400°F Run	Change - 400°F Run	After 500°F Run	Change - 500°F Run	Total Change
1	0.0290	0.0296	+0.0006	0.0315	+0.0019	+0.0025
2	0.0343	0.0348	+0.0005	0.0378	+0.0030	+0.0035
3	0.0348	0.0348	0.0	0.0383	+0.0035	+0.0035
4	0.0394	0.0406	+0.0012	0.0443	+0.0037	+0.0049
5	0.0240	0.0245	+0.0005	0.0255	+0.0010	+0.0015
6	0.0296	0.0288	-0.0008	0.0306	+0.0018	+0.0010
7	0.0314	0.0344	+0.0030	0.0360	+0.0016	+0.0046
Total Change			+0.0050		+0.0165	+0.0215
Average Change			+0.0007		+0.0024	+0.0031

TABLE X

PUMP COLLAR WEIGHT CHANGES DURING THE 400°F AND 500°F EXPERIMENTS WITH XF-1-0301*

Cylinder No.	AS Received	After 400°F Run	Change - 400°F Run	After 500°F Run	Change - 500°F Run	Total Change
1	5.6298	5.6226	-0.0072	5.6137	-0.0089	-0.0161
2	5.7208	5.7129	-0.0079	5.7006	-0.0123	-0.0202
3	5.7373	5.7254	-0.0119	5.7161	-0.0093	-0.0212
4	5.4925	5.4812	-0.0113	5.4702	-0.0110	-0.0223
5	5.6213	5.6150	-0.0063	5.6054	-0.0096	-0.0159
6	5.6924	5.6870	-0.0054	5.6784	-0.0086	-0.0140
7	5.5885	5.5805	-0.0080	5.5691	-0.0114	-0.0194
Total Change			-0.0580		-0.0711	-0.1291
Average Change			-0.0083		-0.0102	-0.0184

TABLE XI

PUMP SLEEVE WEIGHT CHANGES DURING THE 400°F AND 500°F EXPERIMENTS WITH XF-1-0301*

Cylinder No.	As Received	After 400°F Run	Change - 400°F Run	After 500°F Run	Change - 500°F Run	Total Change
1	9.8274	9.8281	+0.0007	9.8274	-0.0007	0.0
2	9.7779	9.7790	+0.0011	9.7787	-0.0003	+0.0008
3	9.7815	9.7825	+0.0010	9.7828	+0.0003	+0.0013
4	10.3443	10.3448	+0.0005	10.3448	0.0	+0.0005
5	9.8244	9.8246	+0.0002	9.8250	+0.0004	+0.0006
6	9.7622	9.7619	-0.0003	9.7628	+0.0009	+0.0006
7	9.8946	9.8948	+0.0002	9.8953	+0.0005	+0.0007
Total Change			+0.0034		+0.0011	+0.0045
Average Change			+0.0005		+0.0002	+0.0006

* All weights in grams. Pump Serial No. R2-13.
** All dimensions in inches. Pump Serial No. R2-13.

The component weight changes and the increases in ball joint movement that occurred during the 50-hr/400°F run with XF-1-0301 fluorosilicone were small and, with the exception of the collars, are considered normal. The high collar weight losses could be indicative of a problem. However, both the collar surfaces and the piston and nutating plate surfaces they contact were in good condition and showed no signs of excessive wear. It is interesting that, after the experiment, all collar surfaces that contacted the pistons were a bright copper color except the collar that ran against the piston that had the roughest piston head ball. This collar surface was polished but metallic silver in color. The copper-colored material was readily removed from the six collars with ammonium hydroxide.

b. 500°F Experiment: Preparation of this pump for use at 500°F with XF-1-0301 involved only three operations; removal of the copper deposits with ammonium hydroxide, thorough cleaning, and replacement of the Viton A seals which had taken a permanent set but which were still very flexible.

After 7 hr. of operation at 500°F, the test was interrupted because the pump shaft seal leakage was on the order of 50 ml/hr. A new shaft seal was installed and the test was continued to the 30.45 hr. point where it was ended because of a plugged pump inlet filter and excessive pump piston head wear (Figure 11). After solvent cleaning, the piston assemblies, collars, and sleeves were weighed and the changes in the longitudinal movement of the piston head ball joints were measured. These data are given on Tables VIII to XI. Although the changes in the sleeve weights were minor, the accelerated wear indicated by the other measurements is apparent. One comparison of pump wear during the 30.45 hr/500°F run with XF-1-0301 can be made by referring to the wear data from the 50 hr/400°F run with this same fluid also tabulated in Tables VIII to XI. Another comparison between the 30.45 hr/500°F run with XF-1-0301 fluorosilicone and a 47 hr/500°F run with MLO-60-294 deep dewaxed mineral oil, is shown in the following listing:

NEW YORK AIR BRAKE PUMP WEAR AT 500°F

	Average Component Weight Changes (gm.)			Longitudinal Ball
Fluid	Piston Assemblies	Collars	Sleeves	Joint Movement (in.)
XF-1-0301	-0.0660	-0.0102	+0.0002	+0.0024
MLO-60-294	-0.0090	-0.0010	-0.0007	+0.0014

The relatively rapid wear of the piston assemblies and collars is apparent. The piston assembly weight losses were obviously primarily from the copper

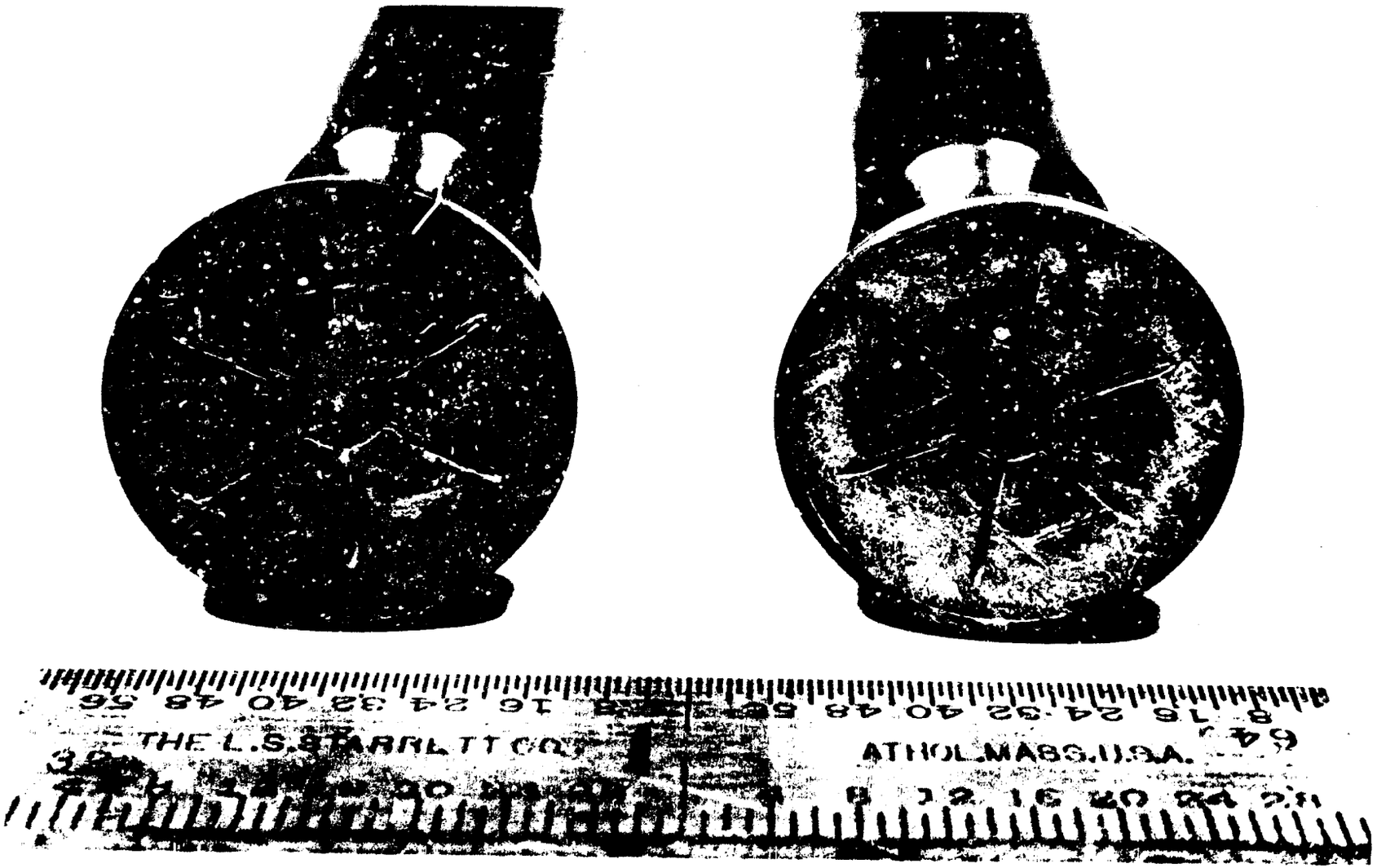

Figure 11 - Typical Pump Piston Heads After Pumping XF-1-0301 for 50 Hr. at 400°F and 30.45 Hr. at 500°F

alloy piston heads. The collar weight losses are not apparent - they do not look worn. The parts of the pistons and nutating plate that operate against the hardened steel collars also do not appear to be worn. Apparently collar wear occurred without gross damage to the surfaces.

Visual inspection of the pump after the 500°F run showed four outstanding features, any one of which would be cause for stopping the run with XF-1-0301: (1) all pis ea's were badly worn and rough (Figure 11), (2) there was a heavy smear o. copper-colored metal on the camshaft surface that runs against the piston heads (the darker area of Figure 12), (3) thin copper-colored deposits were present on several pump components including the low pressure ends of the pistons and cylinders (Figure 13), and (4) the pivot and nutating plate pivot seat (Figures 14 and 15) exhibited signs of metal displacement. Figure 14 is a top view of the hemispherical pivot, mounted in the pump cylinder block, showing some damage at the center, a groove near the maximum diameter, and relatively minor general surface roughness. Figure 15 shows the concave spherical seat in the nutating plate in which the pivot of Figure 14 operated. The cause of the relatively deep, and unprecedented, circular depressions in the nutating plate pivot seat (Figure 15) is not known. It is not believed that any large pieces of hard foreign material were in this area - there were none found in the circuit. However, a theory may be suggested. The excessive wear of the piston heads allowed a large end-clearance to develop. This large end-clearance then permitted the pivot to operate eccentrically in the pivot seat. Abrasive wear particles developed from this operation caused the pivot to become roughened, especially at the highly loaded center, which resulted in the wear patterns seen in the pivot seat (Figure 15). The groove near the edge of the pivot could have been caused by operation against the edge between the ground and unground portions of the pivot seat.

3. Experimental operation: Operating data for the two high temperature hydraulic circuit experiments with XF-1-0301 fluorosilicone are summarized in Table XII. The pump compensator was set at the mid-point of its adjustable range during both runs.

The general warm-up procedure followed prior to both runs consists of preheating the test fluid to about 125°F by using the environmental chamber heaters, starting the pump, and then bringing it up to 500 rpm within about 1 min. The pump is operated at 500 rpm for 10 to 15 min. with no restrictions in the circuit while the circuit and instrumentation are checked. The pump speed is then increased in 200-rpm increments to 3,750 rpm during the next 45 min. At this point, the pump discharge pressure was about 350 psig with XF-1-0301 and the fluid temperature was about 250°F.

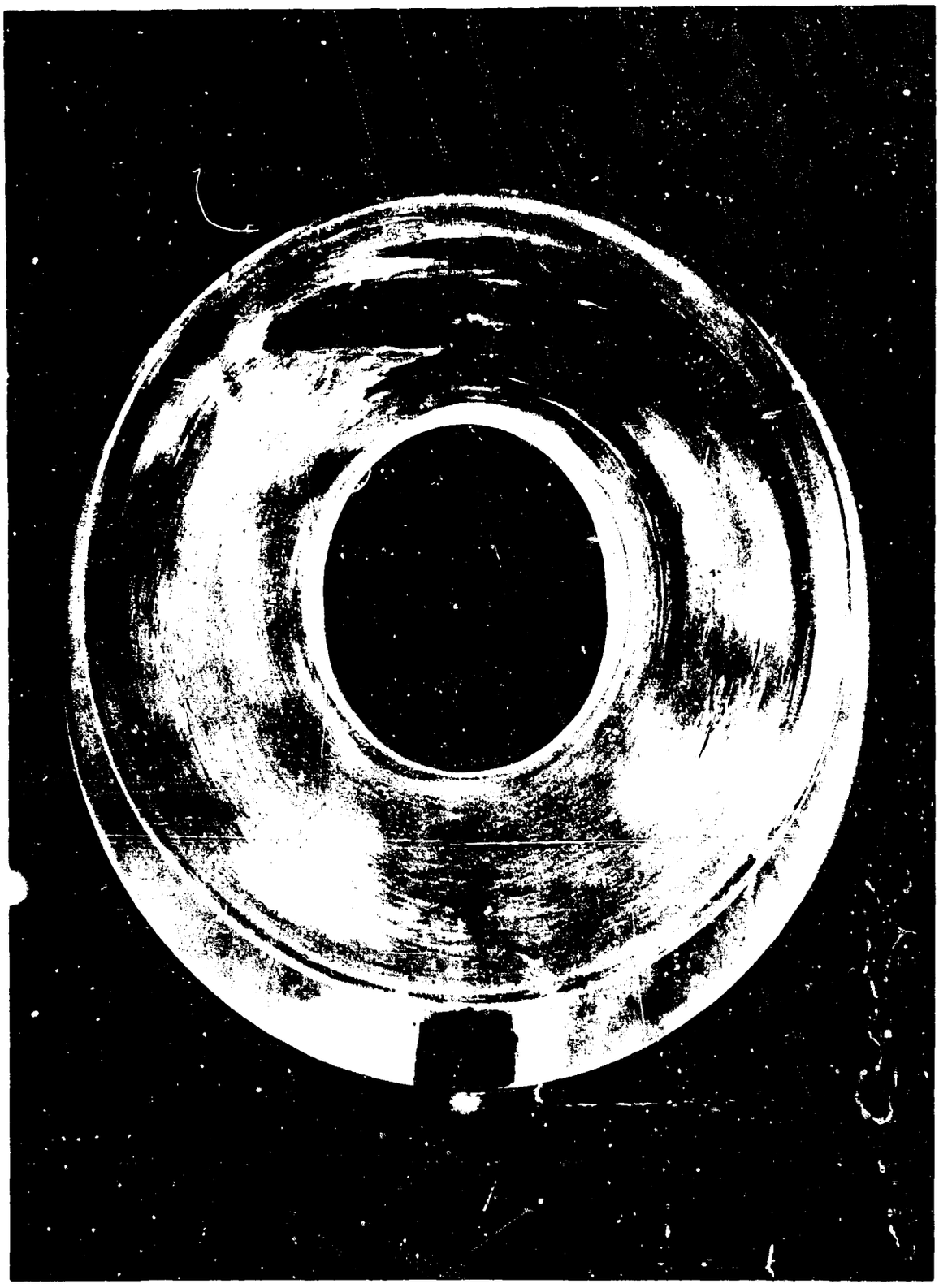

Figure 12 - Pump Camshaft Surface Contacted by the Piston Heads During the 30.45 Hr. Run at 500°F with XF-1-0301

Figure 13 - Low Pressure End of One Pump Cylinder After the 30.45 Hr. Run at 500°F with XF-1-0301

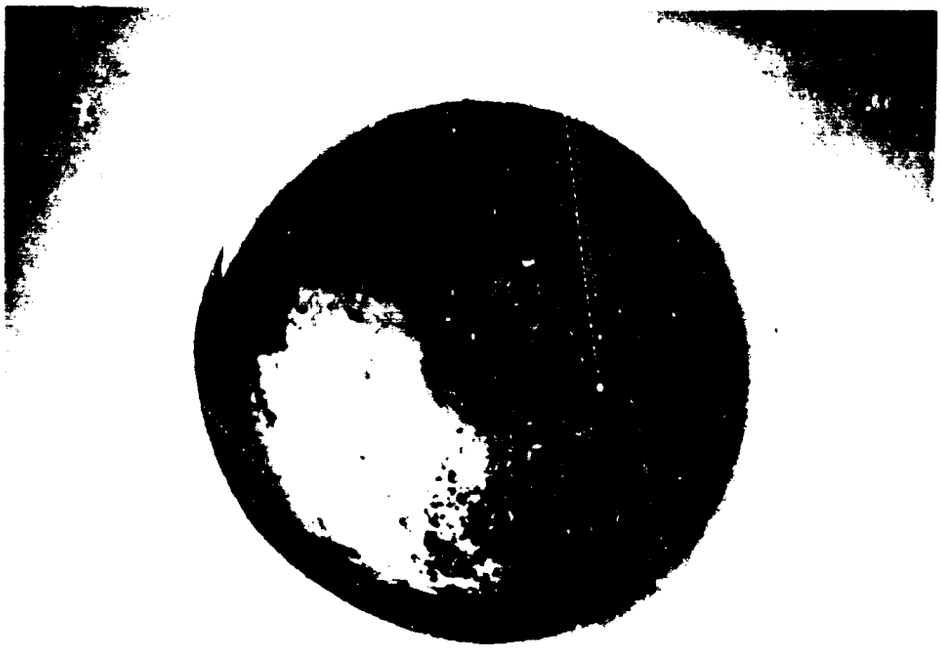

Figure 14 - Pump Nutating Plate Pivot After the 30.45 Hr. Run at 500°F with XF-1-0301

Figure 15 - Pump Nutating Plate Pivot Seat After the 30.45 Hr. Run at 500°F with XF-1-0301

TABLE XII

OPERATING DATA FOR 400°F AND 500°F HIGH TEMPERATURE HYDRAULIC CIRCUIT EXPERIMENTS WITH DOW CORNING XF-1-0301 LOT 5 FLUOROSILICONE

	Maximum		Minimum		Average	
	400	500	400	500	400	500
Nominal fluid temperature (°F)						
Duration of experiment (hr.)	-	-	-	-	50	30.45
Nominal pump speed (rpm)	-	-	-	-	3,750	3,750
Pumping rate (gpm)						
At maximum flow	8.52	8.23	8.43	8.14	8.48	8.22
At minimum flow	1.02	1.07	1.02	1.02	1.02	1.06
Shear cycles*	-	-	-	-	7,917	4,712
Reservoir pressure (psig)						
Initial	-	-	-	-	65	61
Final	-	-	-	-	65	92
Pump inlet pressure, initial (psig)						
At maximum flow	-	-	-	-	45	45
At minimum flow	-	-	-	-	60	58
Pump inlet pressure, final (psig)						
At maximum flow	-	-	-	-	43	32
At minimum flow	-	-	-	-	60	83
Pump discharge pressure (psig)						
At maximum flow	2,625	2,625	2,550	2,530	2,588	2,580
At minimum flow	2,920	2,920	2,900	2,810	2,906	2,866
Inlet filter ΔP at maximum flow (psi)						
Initial	-	-	-	-	20	16
Final	-	-	-	-	22	60
Temperatures (°F)						
Pump case	385	473	363	447	369	465
Pump inlet	404	**	381	**	384	**
Pump discharge	415	503	389	483	395	498
After throttling valve	415	504	399	485	402	500
Oven air	255	279	233	252	236	271
Shaft seal leakage (ml.)	-	-	-	-	93	336
Initial charge remaining in circuit (%)*	-	-	-	-	82.8	66.8

* Based on circuit volume of approximately 1.8 gal.

** Thermocouple inoperative.

The discharge pressure is then increased to 2,000 psig during the next 10 min. and held there until the test fluid reaches the test temperature. The test fluid is held at the test temperature while the compensator is checked for roughly the next 20 min. During the compensator check, the pump discharge pressure is raised to about 2,900 or 3,000 psig and the flow rate is dropped to about 1 gpm. If all parts of the circuit are operating satisfactorily, the cycling device on the throttling valve is started and the zero-hour test data are recorded. The compensator check is normally repeated at the end of a test run to get information regarding gross changes in pump condition. Data generated during compensator checks before and after the 400°F run are given in Figure 16. No check was made wher the 500°F run was stopped at the 30.45-hr. point because of the problem with the pump inlet filter pressure-drop. The data represented by Figure 16 indicate that the pump was not impaired by the 50 hr. of operation at 400°F with XF-1-0301.

a. Fifty-hour experiment at 400°F: Operating data for the 50-hr/400°F experiment with XF-1-0301 fluorosilicone are summarized in Table XII. The pump discharge conditions were cycled each minute between roughly 2590 psig at 8.5 gpm and 2900 psig at 1.0 gpm. The highest average fluid temperature was 402°F which existed after the fluid was forced through the splined plug throttling valve. The highest fluid temperature that existed at any time was 415°F. This condition occurred for a few minutes while the pump compensator was being checked just before the run was started. The maximum test fluid temperature was dropped to 400°F shortly after the test started and was held below 405°F for the remainder of the run.

The pressure drop between the reservoir and the pump inlet, which includes the large drop through the pump inlet filter, increased only 2 psig during the 50-hr. run. Apparently few particles from the pump or the fluid were being formed and then stopped by this 10-micron filter.

Only two minor operating problems occurred. After 2.33 hr. of initial circuit warm-up operation, end-clearance on the shaft between the pump and its drive motor went to zero. The pump bearings were not loaded because of this shaft length increase. The load was taken by the pump mounting plate. The length of one of the couplings was reduced and the run was started after another 1.55 hr. of warm-up operation. The second problem occurred after 5.73 hr. of 400°F operation. A faulty interlock switch opened and stopped the pump drive motor. The pump was restarted within 2 min. and the test conditions were reestablished 8 min. after the pump was restarted.

b. 30.45-Hour experiment at 500°F: Principal operating variables of the 30.45-hr/500°F run with XF-1-0301 are summarized in Table XII. The lower maximum flow rate of the 500°F run, compared with the 400°F run, could be expected because of the increased pump slippage with the lower viscosity

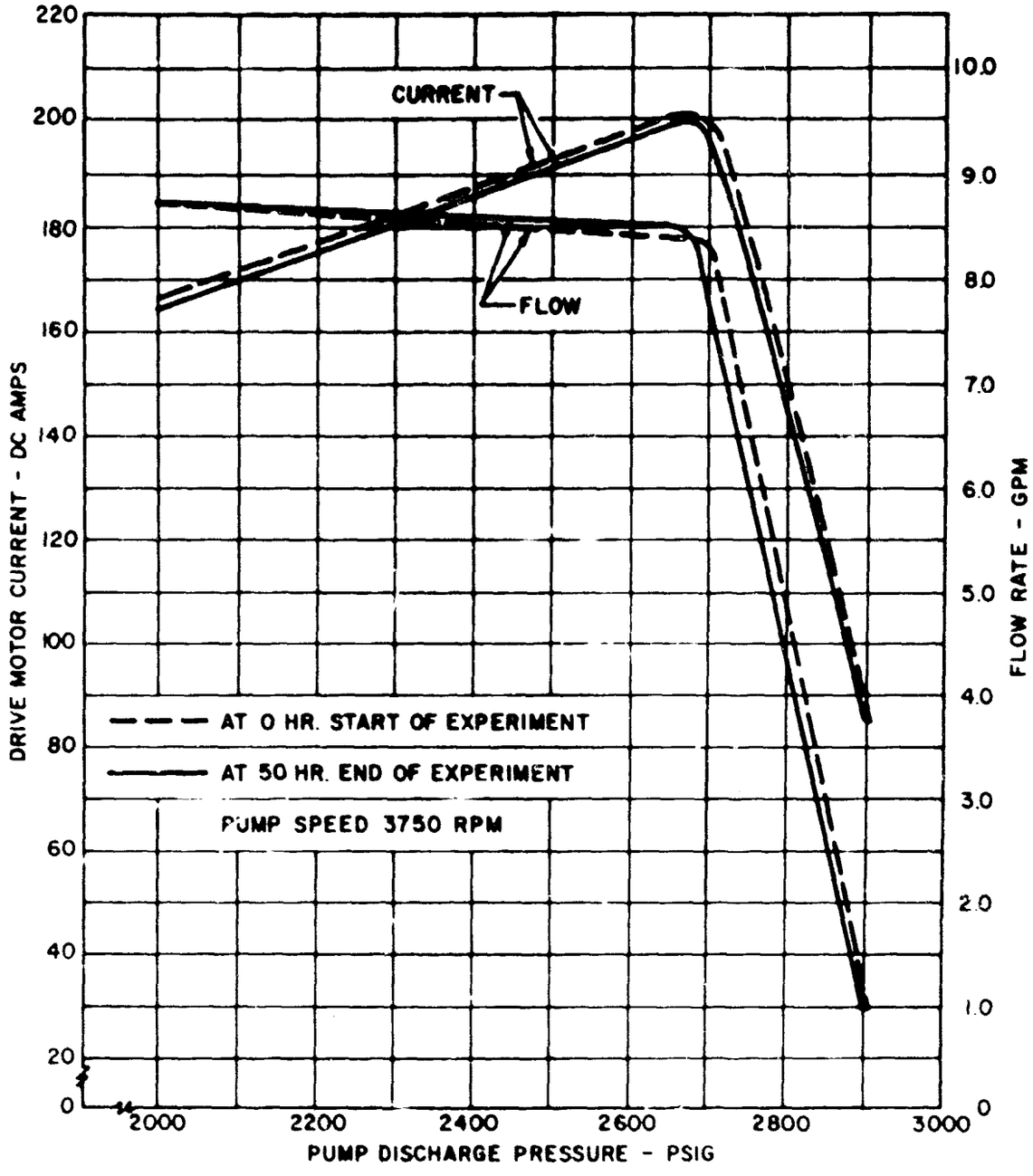

Figure 16 - Variation of Pumping Rate and Drive Motor Current With Pump Discharge Pressure Before and After the 50 Hr/400°F Experiment with XF-1-0301 Fluorosilicone

fluid and because of the decreased bulk modulus. The most significant information shown in Table XII is the filter pressure drop increase at maximum flow from 16 psi at the start of the run to 60 psi after 30.45 hr. of pumping at 500°F. Visual examination showed that both the pump discharge and inlet filters were covered with a thin layer of fine black particles with only a few small copper-colored particles. It is believed that the black material came from the fluid and the copper-colored material came primarily from the pump piston heads. As noted above, the pressure drop across the pump inlet filter increased only 2 psi during the 50-hr/400°F run with XF-1-0301. The filters were not removed from the circuit between the 400° and 500°F tests because pressure drop measurements indicated they were still reasonably clean. At maximum pumping rate, the pressure drops between the reservoir and the pump inlet, which includes tubing containing the pump inlet filter, was 22 psi at the end of the 400°F run and 16 psi at the beginning of the 500°F run. The ΔP decrease occurred because of the viscosity decrease with temperature increase.

III. ISOTHERMAL BULK MODULUS OF EXPERIMENTAL FLUIDS

A. Background

As the maximum temperature and pressure limits of new hydraulic systems are increased, the bulk moduli of hydraulic fluids assume an increasingly important role. The apparatus shown schematically in Figure 17 has been used to determine isothermal bulk modulus values for a number of high-temperature hydraulic fluid candidates.

Bulk modulus is a measure of the compressibility, or elasticity, of a fluid. Isothermal secant bulk modulus ($\bar{B}_T$) is defined as

$$\bar{B}_T = -\left(\frac{\Delta P}{\Delta V/V_o}\right)_T$$

where ΔP = total change in fluid pressure,
ΔV = total change in fluid volume, and
V_o = initial fluid volume.

This value of $\bar{B}_T$, determined at constant temperature, is an average or mean value over a pressure range. If equipment is operating in a very narrow pressure range or essentially at a constant pressure, the tangent bulk modulus is more applicable. The definition of the isothermal tangent bulk modulus (B_T) is

$$B_T = -\left(V \frac{\partial P}{\partial V}\right)_T$$

where V = fluid volume under compression, and

$\frac{\partial P}{\partial V}$ = change of fluid pressure with respect to fluid volume at constant temperature.

The bulk modulus apparatus has been previously used at temperatures to 750°F and pressures to 10,000 psig in determining the isothermal bulk moduli of (1) a deep dewaxed mineral oil (MLO-60-294), (2) a phenyl methyl silicone (QF-258), (3) a chlorinated phenyl methyl silicone (MLO-56-843), (4) hexa-2-ethylbutoxydisiloxane (MLO-8200), (5) an ester of TMP (MLO-60-50), (6) di-phenyl-di-n-dodecyl silane (MLO-57-637), (7) bis-(phenoxyphenoxy) benzene (MLO-59-692), (8) a petroleum base hydraulic fluid (MIL-H-5606A), and a polymeric perfluorinated fluid (PR-143AC). Data for the first seven

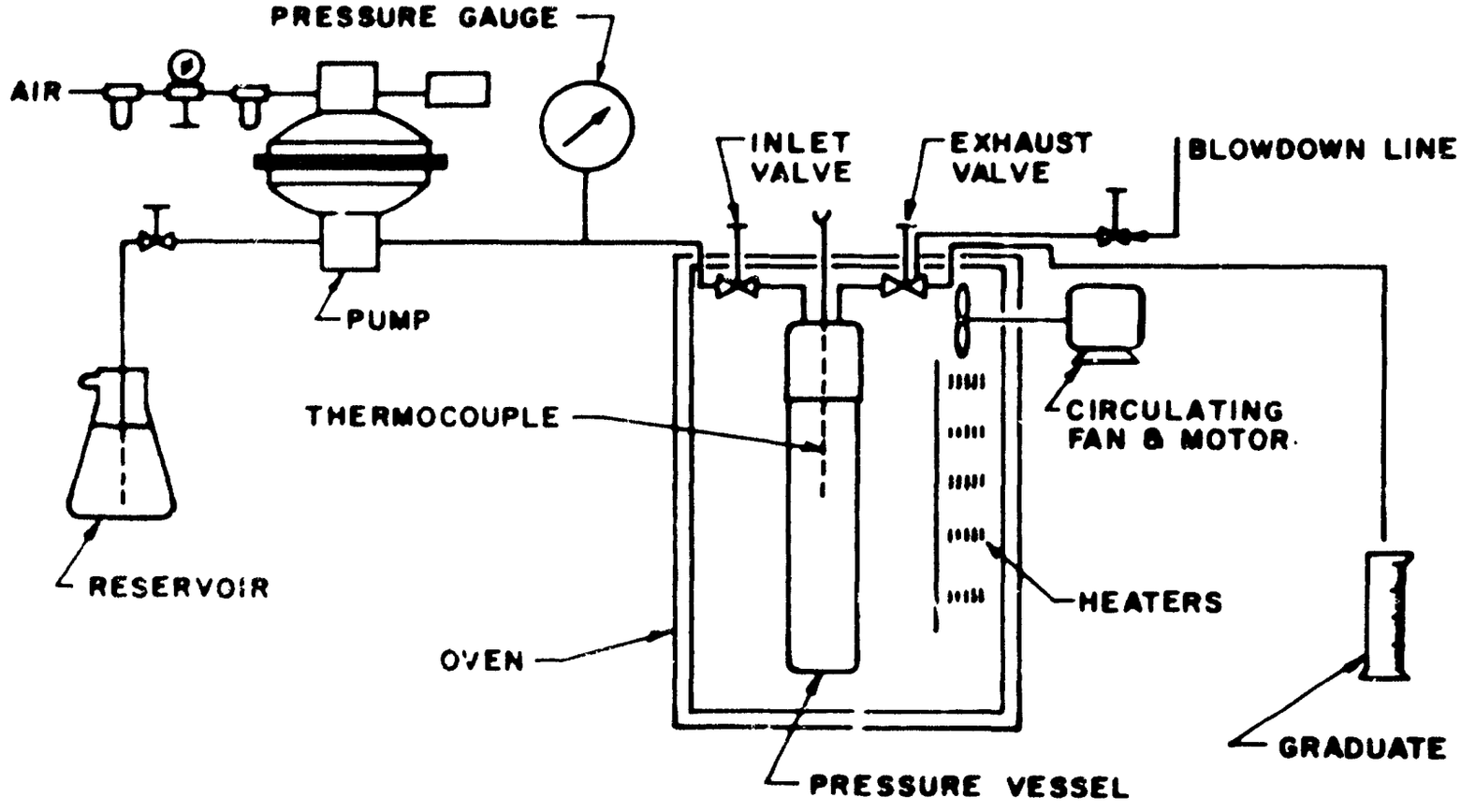

Figure 17 - Schematic Diagram of the Bulk Modulus Apparatus

fluids were reported in ML-TDR-64-12, Part I (Ref. 1); data for MIL-H-5606A were reported in ML-TDR-64-12, Part II (Ref. 2); and data for PR-143AC were reported in AFML-TR-67-8, Part I (Ref. 3).

In order to aid in the reduction of the experimental data to bulk modulus values, a computer program was written. The output of the program yields both isothermal secant and isothermal tangent bulk moduli as well as the standard error ($s_{\bar{B}_T}$) for each secant bulk modulus calculated. The computer is programmed to include all data points and produce the best values for the entire set of data without placing undue emphasis on single data points. Further descriptions of the apparatus and methods of operation and data reduction can be found in WADD-TR-60-855, Part III (Ref. 4), in ASD-TR-63-539 (Ref. 5) and in (Ref. 6).

B. Isothermal Bulk Modulus of XF-1-0301

The fluorosilicone fluid XF-1-0301 is manufactured by Dow Corning Corporation and is based on a trifluoropropyl methyl substituted polysiloxane with minor additives to improve boundary lubrication performance. Isothermal bulk modulus values for XF-1-0301, Lot No. 4, were measured at roughly 100°, 200°, 300°, 400°, and 450°F and at pressures, in 1,000 psi increments, between 1,000 and 10,000 psig. At least three replications of each data point were made. All data (164 values) were supplied to the computer program for reduction.

Density data for XF-1-0301 required in the computer program were measured and are listed in Table XIII. The density was determined in two different types of specific gravity bottles (Hubbard and Gay-Lussac) mounted in a common holder and immersed in a phenyl methyl silicone bath almost up to the ground glass joints between the bottles and the bottle tops. These bottles have small holes in their tops to allow the expanding fluid to escape. Density data were calculated from the known volume of the bottles, corrected for temperature, and the weight of the fluid they contained at specific temperatures.

TABLE XIII

DENSITY OF XF-1-0301 AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	1.142	1.140	0.002	1.141
100	1.134	1.133	0.001	1.134
200	1.086	1.085	0.001	1.086
300	1.036	1.035	0.001	1.036
400	0.983	0.982	0.001	0.983
450	0.960	0.958	0.002	0.959

The isothermal bulk modulus values for XF-1-0301 are presented in Table XIV. A comparison of some of these data with data for other fluids at 1,000 and 10,000 psig and 100° and 500°F is shown in Table XV. In general, the isothermal secant bulk modulus of XF-1-0301 is higher than the polymeric perfluorinated PR-143AC and chlorinated phenyl methyl silicone, about the same as MIL-S[illegible] disiloxane and QF-258 phenyl methyl silicone, and lower than the values for the petroleum base fluids, an ester of TMP, a silane, and a 5P4E polyphenyl ether.

TABLE XIV

ISOTHERMAL BULK MODULUS OF DOW CORNING XF-1-0301 FLUOROSILICONE

Temp. (°F)	Pressure (lb/in^2)	Secant ($\bar{B}_T$) (psi)	Std. Error ($s_{\bar{B}_T}$)	Tangent (B_T) (psi)
100.0	1000.0	188634.	852.	195116.
100.0	2000.0	195781.	799.	208852.
100.0	3000.0	202767.	756.	222518.
100.0	4000.0	209607.	722.	236118.
100.0	5000.0	216314.	697.	249656.
100.0	6000.0	222899.	682.	263136.
100.0	7000.0	229372.	675.	276562.
100.0	8000.0	235742.	676.	289935.
100.0	9000.0	242017.	684.	303258.
100.0	10000.0	248202.	699.	316535.
200.0	1000.0	136450.	502.	142955.
200.0	2000.0	143499.	539.	156648.
200.0	3000.0	150348.	487.	170248.
200.0	4000.0	156994.	447.	183762.
200.0	5000.0	163487.	419.	197197.
200.0	6000.0	169836.	402.	210559.
200.0	7000.0	176054.	398.	223853.
200.0	8000.0	182153.	406.	237083.
200.0	9000.0	188145.	422.	250254.
200.0	10000.0	194036.	447.	263368.
300.0	1000.0	97980.	452.	104515.
300.0	2000.0	104893.	380.	118148.
300.0	3000.0	111539.	318.	131658.
300.0	4000.0	117962.	269.	145057.
300.0	5000.0	124192.	232.	158358.
300.0	6000.0	130255.	212.	171568.
300.0	7000.0	136172.	210.	184696.
300.0	8000.0	141957.	224.	197747.
300.0	9000.0	147624.	251.	210727.
300.0	10000.0	153185.	285.	223641.
400.0	1000.0	68418.	382.	75000.
400.0	2000.0	75136.	311.	88543.
400.0	3000.0	81519.	249.	101922.
400.0	4000.0	87634.	197.	115160.
400.0	5000.0	93528.	156.	128275.
400.0	6000.0	99236.	131.	141280.
400.0	7000.0	104784.	127.	154187.
400.0	8000.0	110193.	143.	167003.
400.0	9000.0	115480.	172.	179736.
400.0	10000.0	120657.	208.	192394.
500.0	1000.0	44941.	376.	51601.
500.0	2000.0	51351.	320.	64993.
500.0	3000.0	57345.	276.	78164.
500.0	4000.0	63029.	242.	91154.
500.0	5000.0	68470.	218.	103991.
500.0	6000.0	73713.	203.	116695.
500.0	7000.0	78792.	198.	129282.
500.0	8000.0	83731.	202.	141764.
500.0	9000.0	88548.	213.	154151.
500.0	10000.0	93259.	231.	166451.

TABLE XV

COMPARISON OF THE ISOTHERMAL SECANT BULK MODULUS VALUES OF SEVERAL FLUIDS AT SELECTED TEMPERATURE AND PRESSURE LEVELS

	Isothermal Secant Bulk Modulus (psi) at			
	100°F and 1,000 psig	100°F and 10,000 psig	500°F and 1,000 psig	500°F and 10,000 psig
PR-143AC (MLO-65-32)	107,900	153,800	29,500	66,500
Chlorinated Phenyl Methyl Silicone (MLO-56-843)	155,000	202,700	43,000	82,900
Hexa-2-Ethylbutoxydisiloxane (MLO-8200)	186,000	238,500	48,400	92,300
Fluorosilicone (DC XF-1-0301)	188,600	248,200	44,900	93,300
Phenyl Methyl Silicone (QF-258)	179,700	236,600	53,900	102,100
Petroleum Base Hydraulic Fluid (MIL-H-5606A)	228,500	295,700	60,100	116,100
Deep Dewaxed Mineral Oil (MLO-60-294)	236,500	286,900	64,300	107,900
Ester of TMP (MLO-60-50)	242,700	297,300	63,900	110,800
Di-Phenyl-di-n-dodecylsilane (MLO-57-637)	267,200	316,700	78,200	122,000
m-Bis(m-phenoxyphenoxy)benzene (MLO-59-692)	414,100	466,600	143,300	192,100

IV. FOUR-BALL WEAR TESTER INVESTIGATIONS

A. Background

A Shell four-ball wear tester manufactured by Precision Scientific Company was modified to permit operation at high temperature and also to permit continuous recording of the torque transmitted from the spindle to the ball pot. These modifications are described in detail in Ref. 7, Part I, and in Ref. 8. The results of a comprehensive series of evaluations using this modified apparatus with a number of fluids were reported in Ref. 7, Parts I and II, and Ref 9.

A standard Precision Scientific Company four-ball EP lubricant tester was also used with several fluids (Ref. 9). This device differs from the Shell four-ball wear tester in that (1) the spindle ball is driven directly by the 1,735* rpm motor, (2) there are no lubricant heating provisions on the apparatus used, and (3) the maximum load is 800 kg. as compared to about 50 kg. for the Shell wear tester.

B. Experimental Results - Shell Apparatus

One-hundred four-ball wear tests were conducted with a bulk ball-pot temperature of 400°F for 1 hr. at 592 rpm using 0.5 in. diameter M-10 and 52100 steel ball specimens. Test fluids included new PR-143AC, Oronite 6294, XF-1-0301, Dow Corning 560, and QF-258. Tests were also conducted with PR-143AC, Oronite 6294, and XF-1-0301 that had previously been used in shear/thermal stability tests at 400°F or higher. Wear scar data are tabulated in Table XVI and the torque transmitted from the spindle ball to the balls clamped in the ball pot is listed in Table XVII.

There were two objectives sought in conducting these experiments. One was to determine if use of the fluids in the pump loops affected the four-ball tester data and the other was to secure some lubricity data for XF-1-0301 relative to the four other fluids which had been used in the high temperature hydraulic circuit at 400°F. Chronologically, these four-ball tests followed the XF-1-0301 runs at 450° and 600°F in the pump stand and preceded the 400° and 500°F runs in the high temperature hydraulic circuit. PR-143AC and a predecessor of Oronite 6294 deep dewaxed mineral oil operated satisfactorily while DC-560 and QF-258 resulted in pump failures.

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* Full load speed.

TABLE XVI

WEAR DATA FROM FOUR-BALL TESTS
(1 hr. at 592 rpm and 400°F)

Test Fluids[a]	Specimen Material	Wear Scars (in millimeters) Under Loads of: 40 kg	30 kg	20 kg	10 kg	4 kg	1 kg
PR-143AC (new)	M-10	0.56*	0.46	0.33	0.22	0.17	0.16
	52100	0.87	0.58	0.41	0.32	0.30	0.24
PR-143AC after 1,000 hr. at 400°F in pump loop[b]	M-10	0.52	0.44	0.28	0.21	0.16	0.13
	52100	1.75	0.71	0.39	0.33	0.29	0.25
Oronite 6294 (new)	M-10	0.99	0.99	0.76	0.22	0.18	0.20
	52100	0.98	1.29	0.52	0.40	0.37	0.32
Oronite 6294 after 100 hr. at 425°F in pump loop[c]	M-10	1.41	1.10	0.76	0.26	0.18	0.21
	52100	1.80	1.01	0.79	0.38	0.32	0.19
XF-1-0301 Lot 4 (new)	M-10	0.67*	0.60*	0.51	0.40	0.28	0.30
	52100	0.57	0.52	0.45	0.30	0.40	0.27
XF-1-0301 Lot 4 after 100 hr. at 450°F in pump loop[c]	M-10	0.63	0.63	0.53	0.26	0.28	0.25
	52100	0.80	0.67	0.45	0.29	0.28	0.24
Dow Corning 560 (new)	M-10	1.84	1.69	1.36	0.98*	0.54	0.41
	52100	2.17	1.80	1.34	0.63	0.50	0.44
QF-258 (new)	M-10	2.45	2.37	2.23	1.81	1.18	0.58
	52100	3.42	3.38	2.69	2.17	1.59	0.80

a/ PR-143AC DuPont polymeric perfluorinated fluid.
Oronite 6294 deep-dewaxed mineral oil (MIL-H-27601).
XF-1-0301 Dow Corning fluorosilicone.
Dow Corning 560 chlorinated phenyl methyl silicone.
QF-258 phenyl methyl silicone.

b/ 1,000-Hr. experiment conducted in the high temperature hydraulic circuit using a New York Air Brake piston pump.

c/ 100-Hr. experiment conducted in the pump stand using a modified Manton-Gaulin triplex pump.

* Average data for 2 experiments.

TABLE XVII

TORQUE DATA FROM FOUR-BALL TESTS
(1 hr. at 592 rpm and 400°F)

Test Fluid[a]	Specimen Material	Torque (in-oz) under loads of: 40 kg	30 kg	20 kg	10 kg	4 kg	1 kg
PR-143AC (new)	M-10	22.96*	18.96	14.29	-	-	-
	52100	29.12	20.00	13.05	7.03	2.53	0.68
PR-143AC after 1,000 hr. at 400°F in pump loop[b]	M-10	23.53	18.70	11.77	5.23	2.66	1.06
	52100	29.05	14.41	12.83	6.40	3.18	1.06
Oronite 6294 (new)	M-10	39.85	30.81	17.56	6.76	2.94	-
	52100	35.35	27.81	14.06	6.82	4.89	1.06
Oronite 6294 after 100 hr. at 625°F in pump loop[c]	M-10	42.65	32.00	20.63	7.06	2.29	2.33
	52100	37.95	27.97	17.51	8.57	3.04	1.76
XF-1-0301 Lot 4 (new)	M-10	28.23*	24.81*	16.04	7.55	3.53	-
	52100	30.80	23.17	14.85	9.00	1.08	1.41
XF-1-0301 Lot 4 after 100 hr. at 450°F in pump loop[c]	M-10	32.37	24.61	15.28	7.41	2.35	1.06
	52100	36.47	21.72	13.61	8.22	2.12	-
Dow Corning 560 (new)	M-10	79.45	65.49	44.38	12.09*	2.60	0.68
	52100	75.38	60.97	39.21	10.97	2.83	1.06
QF-258 (new)	M-10	85+	64.28	45.57	21.79	7.82	1.06
	52100	85+	69.29	46.72	22.59	9.05	1.06

a/ PR-143AC DuPont polymeric perfluorinated fluid.
Oronite 6294 deep-dewaxed mineral oil (MIL-H-27601).
XF-1-0301 Dow Corning fluorosilicone.
Dow Corning 560 chlorinated phenyl methyl silicone.
QF-258 phenyl methyl silicone.

b/ 1,000-Hr. experiment conducted in the high temperature hydraulic circuit using a New York Air Brake piston pump.

c/ 100-Hr. experiment conducted in the pump stand using a modified Manton-Gaulin triplex pump.

* Average data for 2 experiments.

Some general remarks can be made regarding the effects of new and used fluids on the four-ball data:

1. Wear scars on M-10 specimens were roughly the same size for both new and used fluids except for Oronite 6294 at the higher loads. The used Oronite 6294 permitted greater M-10 wear under 30 and 40 kg. loads than did new Oronite 6294.

2. Wear scars on 52100 specimens were generally higher under 30 and 40 kg. loads when run in used fluid rather than new fluid. Differences at lower loads were not appreciable.

3. The transmitted torque generally was not appreciably different for comparable experiments run in new and used test fluid nor did the torque appear to be greatly influenced by differences in the two test specimen materials--M-10 and 52100 in this case.

Judging from the wear scar data of Table XVI, it would appear that XF-1-0301 has lubricating characteristics that are much better than the fluids which permitted pump failure and roughly equivalent to the fluids that did provide satisfactory pump operation at 400°F. The torque data in Table XVII strongly reinforce this observation. As noted in a previous section of this report, a New York Air Brake Model 69W03006-2 did perform well at 400°F with XF-1-0301.

C. Experimental Results - EP Four-Ball Apparatus

Four experiments were conducted with new XF-1-0301 fluorosilicone fluid and M-10 steel balls in the EP apparatus. Two runs were made for 5 min. under 100-kg loads with a room temperature start. The results of these runs have been added, in Table XVIII, to a tabulation of previous work that originally appeared in Ref. 9. Although the wear scars were lower than those found with a deep dewaxed mineral oil, a polyphenyl ether, and an ester of TMP, the maximum torque transmitted from the spindle to the ball pot was relatively high.

Two additional EP four-ball tests were run for 1 hr. with M-10 steel balls in XF-1-0301 fluid. During the first test under 100-kg. load, the fluid heated from room temperature to about 275°F, and a 3.07 mm. wear scar was produced. This scar is roughly twice as large as that produced under identical test conditions except with PR-143AC fluid (Ref. 9). The second EP test with XF-1-0301 was under a 200-kg load. After 1 hr., all the fluid had left the ball pot in the form of smoke. No wear scar could be measured because of the gross metal transfer between the spindle ball and the balls clamped in the ball pot. Under these same conditions, PR-143AC produced a wear scar of about 2.6 mm.

TABLE XVIII

SUMMARY OF EP FOUR-BALL TESTS

Specimen Material - M-10
Load - 100 kg
Duration - 5 min.
Temperature - room temperature start
Speed - approximately 1,800 rpm

Lubricant a/	Avg. Scar (mm.)	Torque (in-oz) Max.	Torque (in-oz) Time (sec.)	Torque (in-oz) Final Value
PR-143	0.950	b/		-
PR-143	0.998	b/		-
MLO 60-294	3.071	130	17	70
MLO 60-294	3.186	100	18	70
MLO 63-15	3.274	130	6	100
MLO 63-15	3.366	70	0	70
MLO 60-50	3.098	140	20	140
MLO 60-50	3.330	160	50	150
DC-560	2.362	90	5	90
DC-560	2.408	90	1	90
XF-1-0301	2.217	156	1	96
XF-1-0301	2.635	202	6	108

a/ PR-143 - DuPont polymeric perfluorinated fluid.
MLO 60-294 - deep-dewaxed mineral oil.
MLO 63-15 - mmm-5P4E with 5 percent tricresyl phosphate.
MLO 60-50 - ester of trimethylolpropane.
DC-560 - chlorinated phenyl methyl silicone.
XF-1-0301 - Dow Corning fluorosilicone.

b/ No torque indicated on chart. Torque estimated to be less than 20 in-oz.

V. HIGH VACUUM SIMULATED BEARING WEAR RIG

A. Introduction

An apparatus was developed for use in evaluating friction and wear characteristics of bearing lubricants from atmospheric pressure to absolute pressures lower than 10^{-6} torr and at temperatures ranging from room temperature to 1500°F. A conventional contact configuration utilizing a lubricated test ring and two rub shoes, 180° apart, was selected primarily to permit direct comparison of data from this new rig with the large amount of data previously collected on other test devices using similar test specimens but not capable of the extreme environmental conditions of the new apparatus. The rub-shoe normal load can be varied from 0 to 600 lb/shoe and the sliding speed can be set to any level between 15 and 215 ft/min. This new rig was built on a 6-in. vacuum flange which will fit the 6-in. Ultek crosses of the vacuum systems at the Air Force Materials Laboratory and at Midwest Research Institute.

B. Description

The apparatus consists basically of a drive motor, a magnetic coupling, and the test fixture mounted on a 6-in. vacuum flange. The assembly drawing, shown in Figure 18, includes a frameless drive motor attached to the permanent magnet driving member of a magnetic coupling. The driven member of the coupling is attached to one end of a shaft within the vacuum chamber. The test ring is mounted on the other end of this shaft. Two pneumatic bellows apply loading forces to the diametrically opposed rub-shoes through sapphire balls. All mechanical components are mounted on a 6-in. Ultek vacuum flange. The test fixture is bakeable to 300°C (572°F) after the drive motor and the permanent magnet of the magnetic coupling have been removed.

The operation of the apparatus can be readily visualized by referring to the schematic diagram shown in Figure 19. The drive motor is a frameless DC torque motor rated at 132 in-lb torque at speeds to 1,800 rpm although use above 600 rpm is not contemplated. In this application, the current drawn by the motor is a linear function of the load torque: 1 ampere = 7.0 in-lb. At 600 rpm, the current drawn by the motor to drive the unloaded test fixture is approximately 1 ampere. The motor control panel contains instrumentation to maintain constant speed, adjustable from 40 to 600 rpm, over the full load range. Motor current (motor torque) is monitored by a single set point meter which can be adjusted to stop the motor when the load torque exceeds a predetermined value. A 0-50 mv. DC signal, proportional to the motor current, is available at recorder terminals on the control panel. A frameless motor was utilized so that the motor

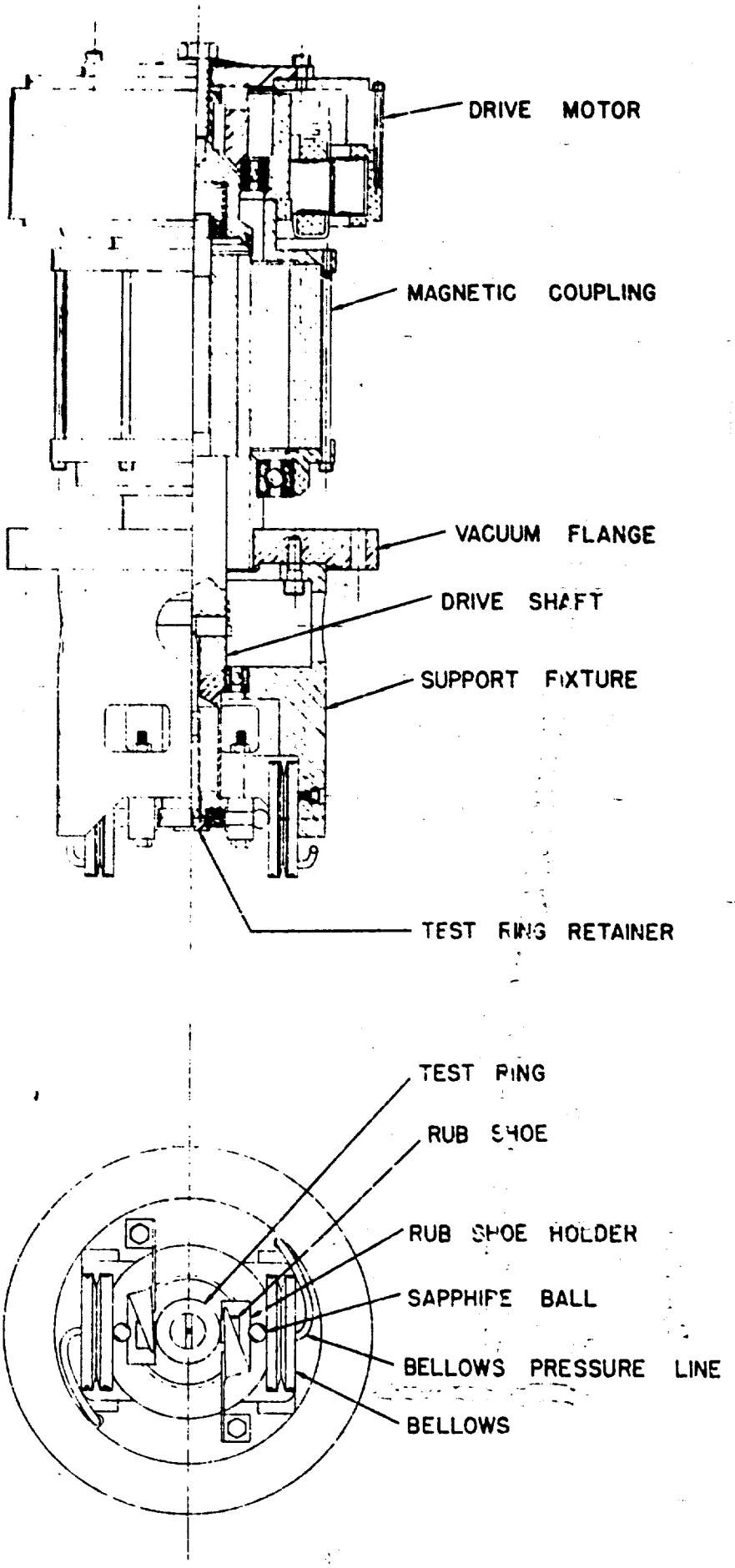


Figure 18 - High Vacuum Simulated Bearing Wear Rig

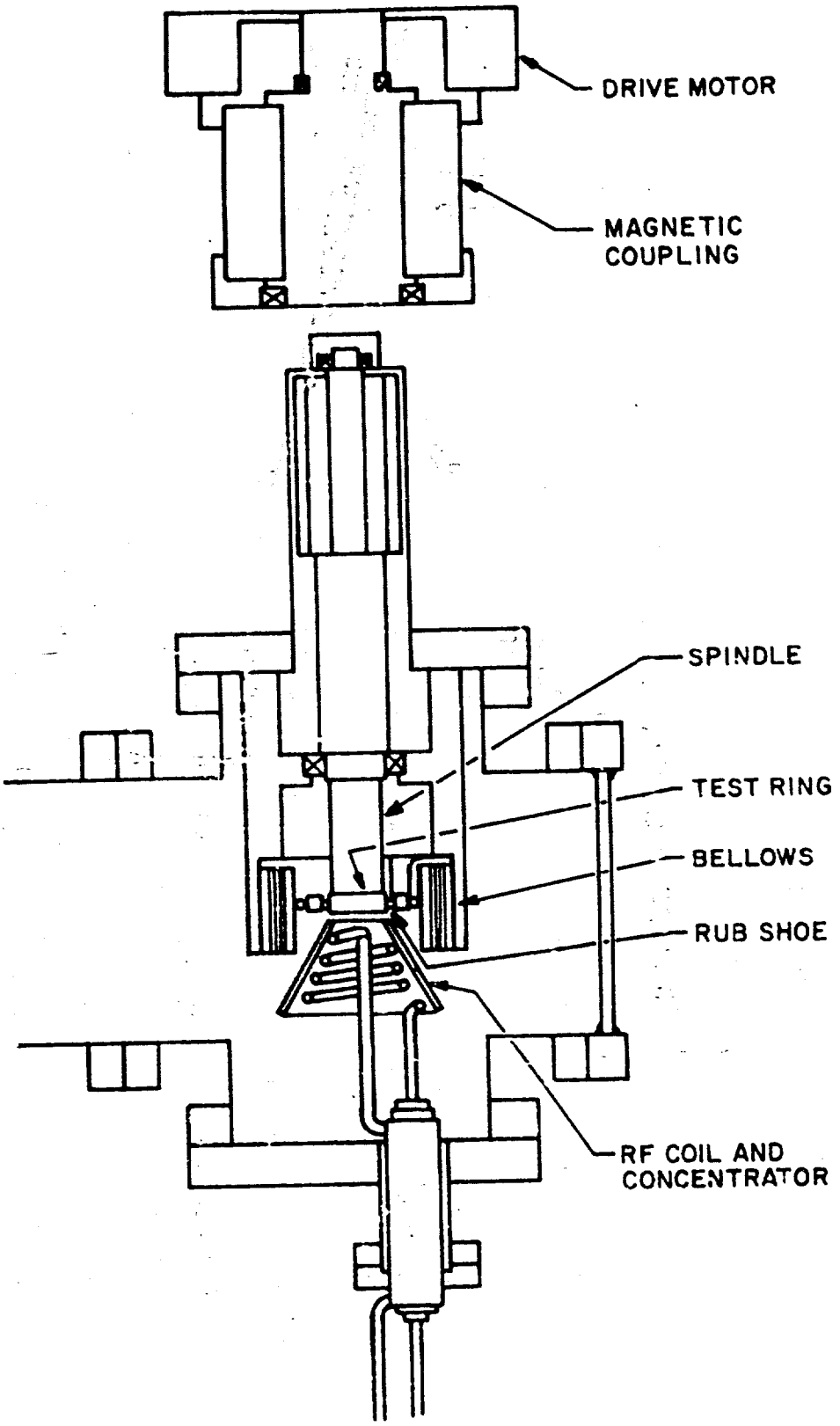


Figure 19 - Schematic of Vacuum Bearing Wear Rig

could be attached to the permanent magnet driving member of the coupling. This arrangement permits the use of only two bearings for alignment and support of both the magnet and the motor. The magnetic coupling has been found to have a maximum torque capability of 61 in-lb.

The driven member of the coupling is attached to the René 41 spindle within the vacuum chamber to rotate the test ring which is 1.375 in. O.D. by 0.625 I.D. by 0.375 in. thick and is held in place by a René 41 bolt. Two Inconel X pneumatic bellows, which have an effective area of 3.48/sq in and can withstand up to 200 psig, apply loading forces through sapphire balls to the diametrically opposed rub shoes. These shoes measure 0.250 in. along the ring axis by 0.500 in. long by 0.400 in. wide. The rub shoes are held in place by René 41 holders which utilize spring forces to retain the shoes. A water-cooled RF coil and concentrator heats the test ring and rub shoes when powered by an induction generator. Specimen temperatures are monitored and controlled by an optical pyrometer sighted on the test ring through a view-port in the vacuum chamber.

All bearings are of the angular contact type and are spring loaded for stability. The bearings initially installed in the vacuum chamber were stainless steel lubricated with powdered MoS_2, but, as discussed later, these bearings were replaced because one of them failed. The bearings used outside the vacuum chamber are grease lubricated.

With the exception of the soft iron armature of the magnetic coupling, the bearings, and the René 41 and Inconel X components previously mentioned, all components inside the vacuum chamber were made of Type 316 stainless steel.

The apparatus can be operated in any position. However, it is anticipated that the specimens will usually face downward to prevent wear debris from damaging the spindle bearings. Photographs of the apparatus mounted in a vacuum system in this position and out of the vacuum system, in an inverted position, are shown in Figures 20 and 21. The RF coil and concentrator used in heating the specimens is shown in Figure 22.

C. Initial Operation

The wear tester was initially operated in air without heat being added. Test rings were coated with dry film lubricants; rub-shoes were not coated. Duplicate tests with identical specimens were run on the Mark VB rub-shoe tester. In the new wear tester at 500 rpm and 100-lb. normal load, surface temperatures of the test ring were observed to rise at the rate of 2.35°F/min. to a maximum temperature of approximately 450°F; whereas, surface temperatures of the test ring in the Mark VB rose only to 200°F maximum.

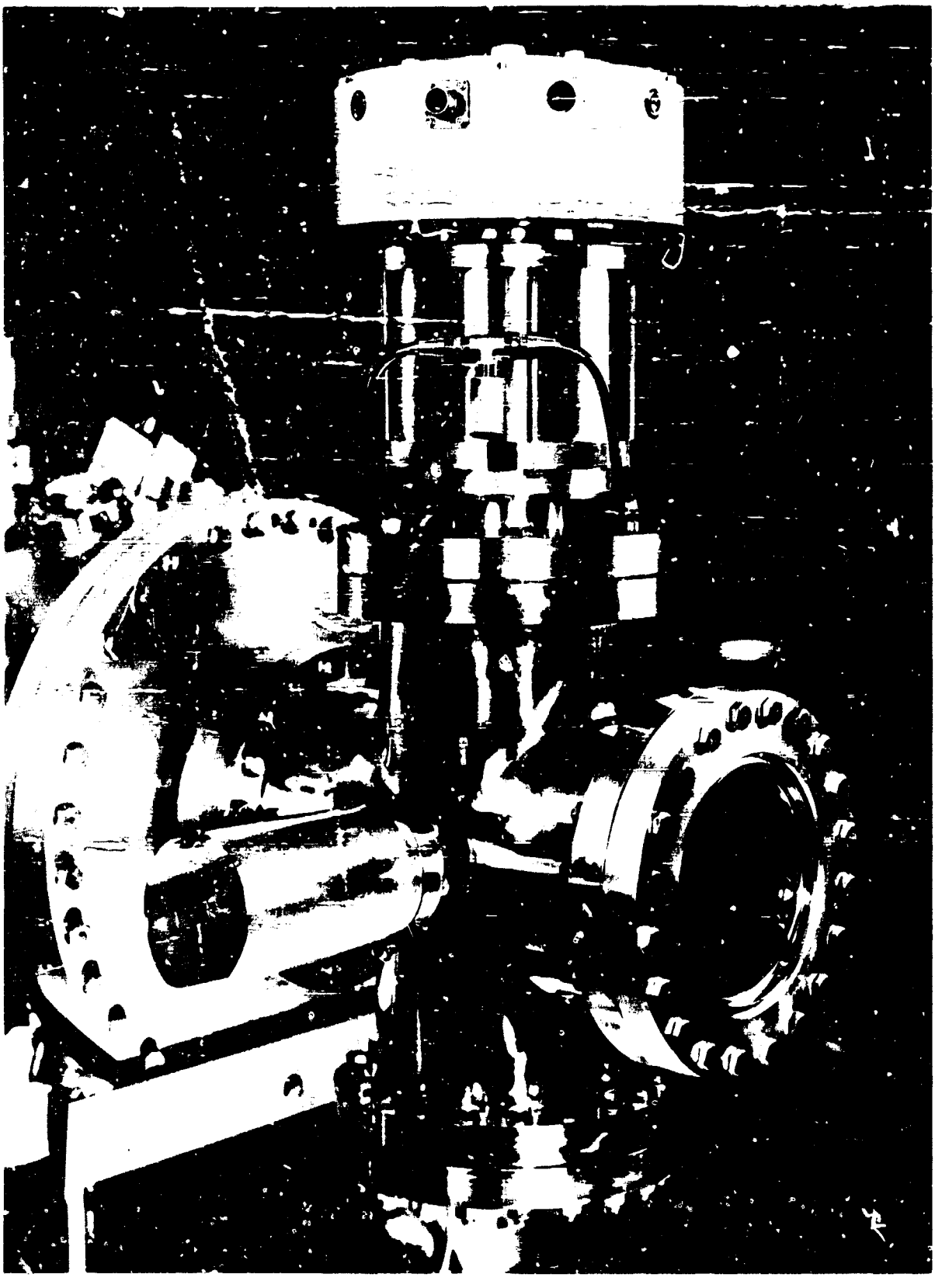


Figure 20 - Vacuum Bearing Wear Rig and Drive Motor Mounted on Vacuum Chamber

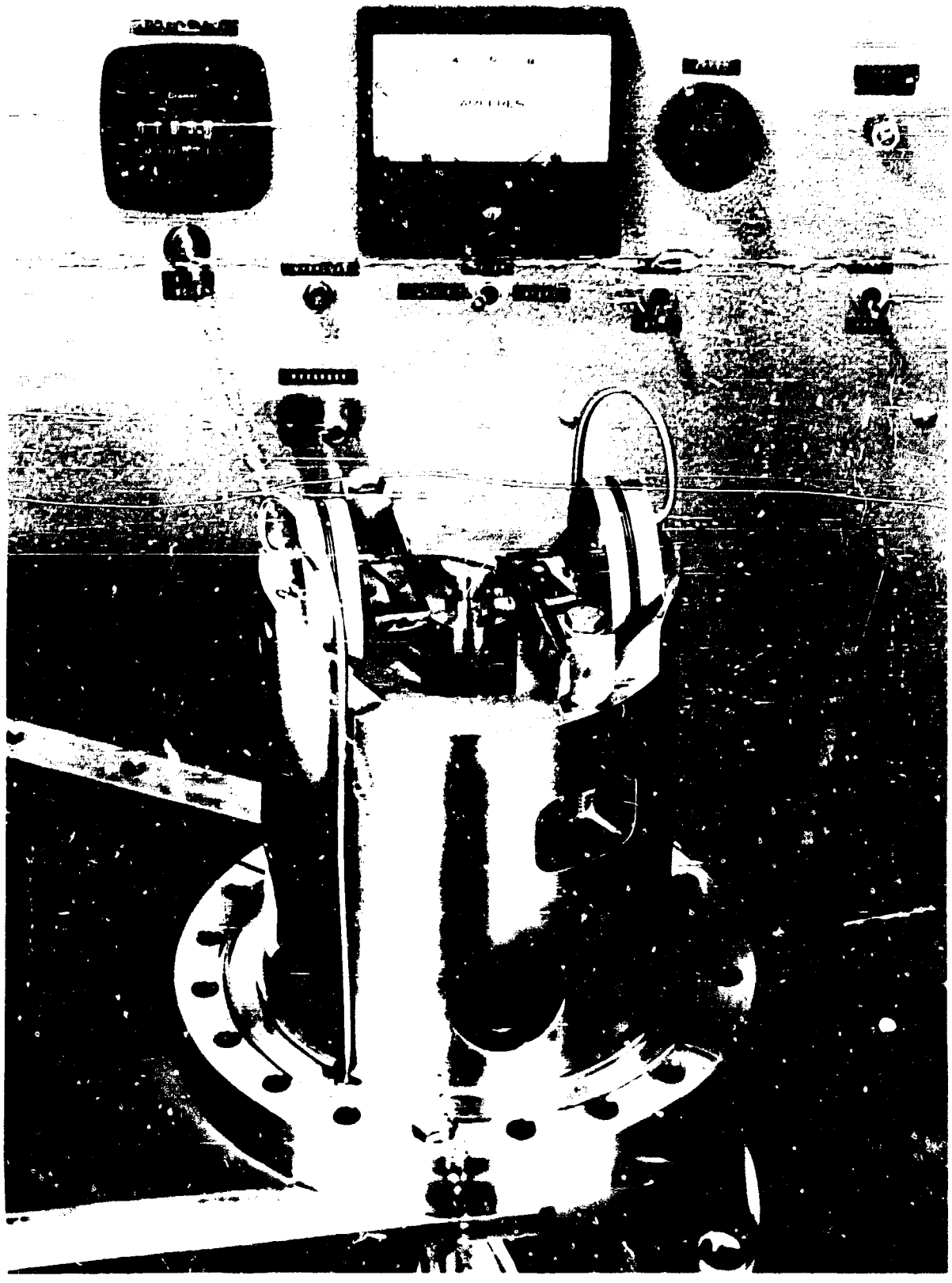


Figure 21 - Vacuum Bearing Wear Rig

Figure 22 - RF Coil and Concentrator for Vacuum Bearing Wear Tester

The difference in heating characteristics is attributed to the relatively good thermal isolation of the shaft and rub-shoe holders in the new tester. When designing the tester, thermal isolation was considered necessary for satisfactory operation of an RF induction heater in the system. The coefficient of friction measured on the two testers was identical for all films measured when the specimen temperatures were comparable. When heat was added to the Mark VB tester during one test to cause a temperature rise similar to that of the new tester, the wear life of the MLR 17-2 films on the two machines compared favorably. Table XIX shows the data taken for comparative tests with the two wear testers.

TABLE XIX

COMPARATIVE WEAR TESTS IN RUB-SHOE DEVICES

Vacuum Bearing Wear Rig

Film type	MLR17-1	MLR17-1	MLR17-2	MLR17-2	MLR17-2
Film thickness (in.)	0.0004	0.0002	0.0003	0.0005	0.0003
Coefficient of friction	0.02	0.02	0.03	0.02	0.02
Wear life (min.)	248	227	277	202	183
Maximum temperature (°F)	-	450	-	475	430

Mark VB Wear Tester

Film type	MLR17-1	MLR17-1	MLR17-2
Film thickness (in.)	0.0003	0.0002	0.0005
Coefficient of friction	0.02	0.02	0.02
Wear life (min.)	466	520	145
Maximum temperature (°F)	205	195	475

Note: The MLR17 films are bonded with polyimide resin and contain MoS_2 as the primary lubricant.

In the first vacuum tests, 10^{-6} torr was attained without bake-out and 10^{-5} torr was maintained while the specimen was heated to 1500°F and run at 100-lb. normal load, 180-fpm rubbing speed. In a second test, 10^{-8} torr was attained after a short bake-out at 275°F. Helium leak tests disclosed no leaks in the system. The vacuum system was able to maintain 10^{-7} torr when the specimen was heated to 1500°F and run at 50-lb. normal load, 180 fpm for 1 min. then stopped and cooled to room temperature. The cyclic heating, running and cooling was repeated five times before it was noticed that the retaining bolt for the ring specimen had loosened.

The nut for the hold-down bolt was modified to provide a spring locking action. In a third test, 10^{-8} torr was attained without bake-out and 10^{-7} torr maintained under operating conditions. The specimen was heated to 1500°F, cooled and heated to 1500°F again. The specimen was rotated for 1 min., stopped, rotated for 11 min., stopped, rotated for 1 min., stopped, and rotated for 1 min. At no time was there evidence of the hold-down bolt loosening.

D. Bearing Modification

One of the stainless steel bearings in the vacuum chamber failed after only a few hours of operation. Both bearings were equipped with metal ball separators and lubricated with molybdenum disulfide. Debris from the bearing that failed, which was in the upper part of the apparatus, may have entered the other bearing so replacements were secured for both.

Bearings of the sizes needed (03 and 07) having phenolic separators were readily available only in 52100 steel. Bearings of this type were secured and subjected to the following operations:

1. They were disassembled by heating the outer race with a heater tape.

2. The balls were cleaned and then burnished with MoS_2 to furnish initial lubrication.

3. The races were cleaned and plated with gold less than 0.00001 in. thick to inhibit corrosion.

4. The phenolic separators were discarded and replaced with Duroid separators. Duroid is Teflon filled with asbestos fiber and MoS_2 manufactured by the Rogers Corporation of Rogers, Connecticut. The ball complement of the 07 bearing was reduced from 15 to 12 and the ball complement of the 03 bearing was reduced from 10 to 8.

5. After assembly of the bearings, again using a heater tape on the outer race, they were cleaned prior to installation with DuPont TF solvent (Freon 113) in a vapor degreaser. Initial operation has been satisfactory.

VI. FLUID VISCOSITIES AT HIGH PRESSURES

A. Background

A high-pressure, high-temperauure falling weight viscometer system, similar to that described in the 1953 ASME pressure-viscosity report (Ref. 10), is being used to determine the viscosity and compressibility of lubricating and hydraulic fluids. It includes a high pressure test chamber which will hold either a falling weight viscometer or a fluid compressibility measuring assembly in the desired high temperature (to 400°F) and high pressure (to 250,000 psig) environment. Density, as a function of temperature and pressure, is derived from the compressibility data and used with corresponding viscometer data to determine absolute viscosities and related information.

Modifications are frequently made to the apparatus or its instrumentation to increase reliability and accuracy and to facilitate the operating procedures. The appearance of the high pressure viscometer is shown in Figure 23 and a schematic diagram of the hydraulic control system is shown in Figure 24.

B. Apparatus Modifications and Maintenance

1. Electrical feedthroughs: The replacing of the numerous seals and insulators for the electrical feedthroughs from the manganin coil pressure sensor and the high pressure test chamber, both in the highest pressure parts of the apparatus, was discussed in AFML-TR-67-8, Part II (Ref. 11). The swaged sheath conductors secured for this purpose, two conductors in one sheath for the pressure sensor and six conductors in one sheath for the test chamber, were brazed into plugs made of the same type of tool steel used for the original type of feedthrough. Excessive softening of the tool steel occurred and allowed wear and eventual leakage of the hexane hydraulic fluid. New plugs were fabricated using an AISI type A6 steel manufactured by Crucible Steel Company under the trade name of Orbit. The advantage of using this air quench tool steel is that it allows silver brazing temperatures to be used in brazing the swaged sheath conductors into the plugs without appreciably reducing the hardness of the plug. A silver brazing temperature of 1300°F reduces the hardness below 45 Rockwell C, but during air quench, the material retempers to approximately 47 Rockwell C as opposed to 36 for the AISI-4340 or 6140 tool steels formerly used. An additional advantage is also realized in that the brazing temperature can be distributed along a greater length of the sheathed conductors and permits a better capillary fill of the void between the plug I.D. and the O.D. of the swaged sheath.

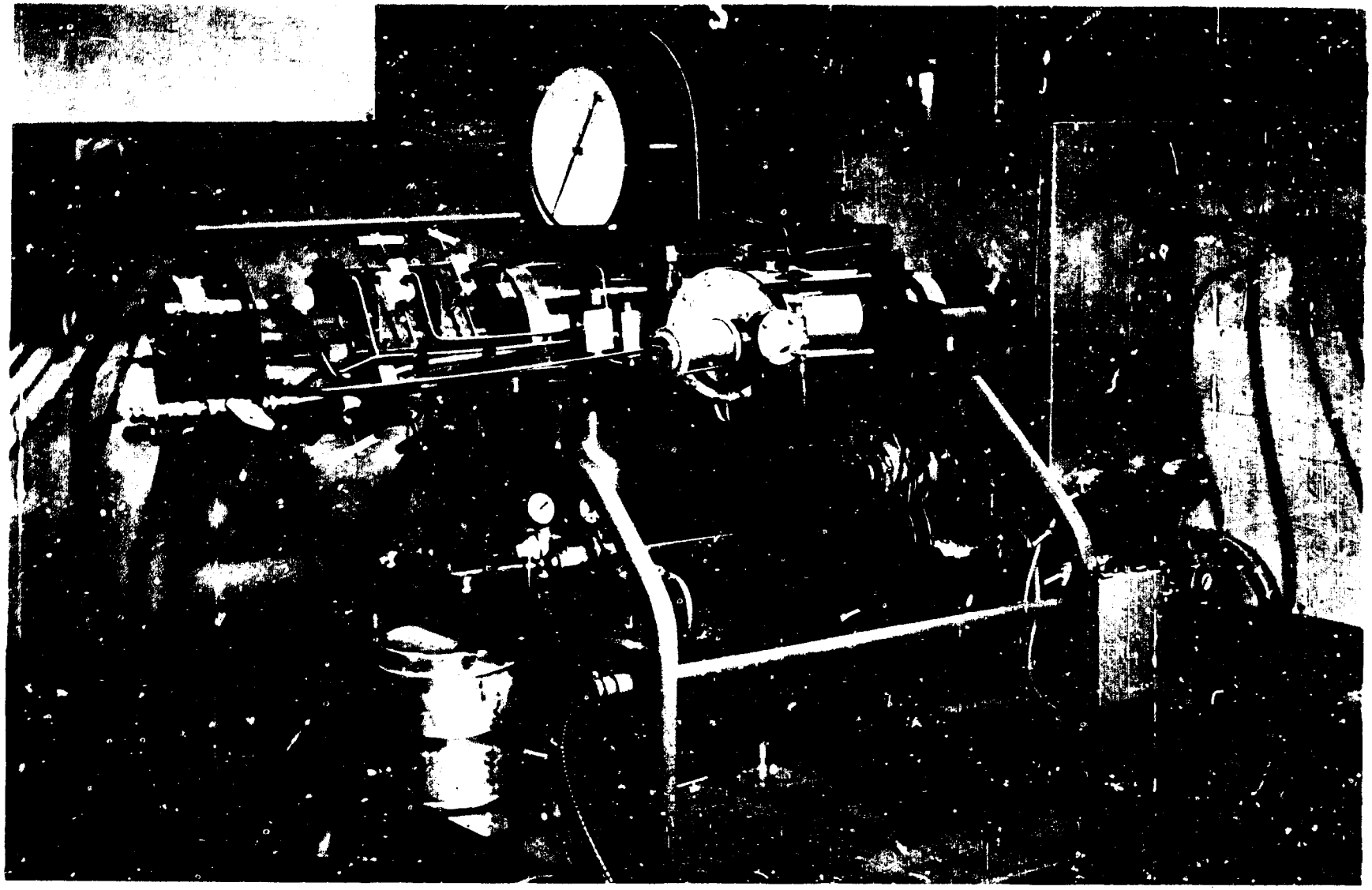
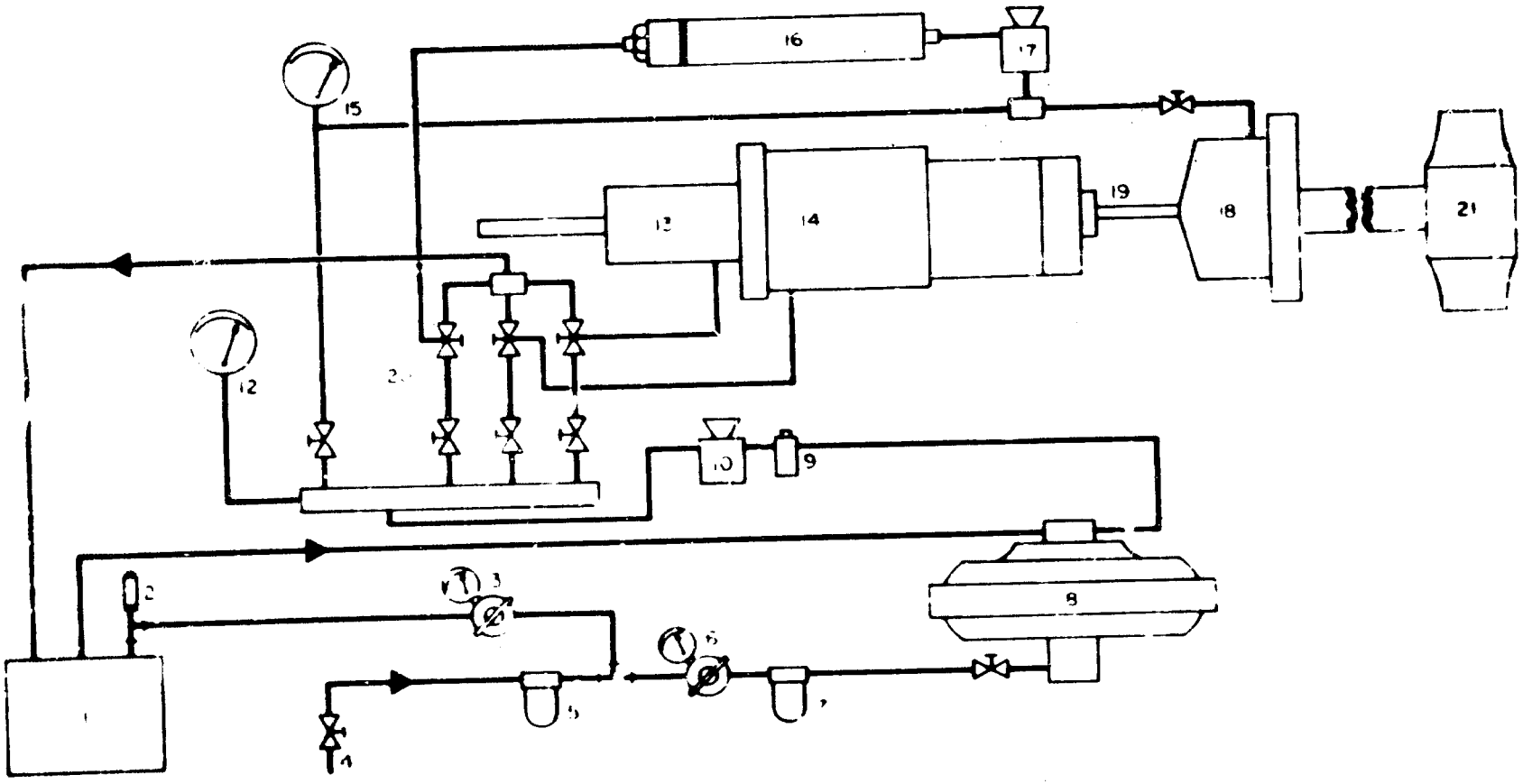


Figure 23 - High Pressure Viscometer



1 - RESERVOIR
2 - RESERVOIR RELIEF VALVE
3 - RESERVOIR REGULATOR
4 - UTILITY AIR
5 - WATER TRAP
6 - PUMP - REGULATOR
7 - OILER
8 - PUMP
9 - BLOW OUT SNUBBER
10 - 10,000 PSIG RUPTURE DISC
11 - VALVE MANIFOLD
12 - PUMP DISCHARGE PRESSURE
13 - BACK-UP CYLINDER
14 - LOW PRESSURE CYLINDER
15 - HIGH PRES. 50,000 PSIG HEISE GAGE
16 - INTENSIFIER
17 - 30,000 PSIG RUPTURE DISC
18 - HIGH PRES. CYLINDER & TRANSITION TUBE
19 - HIGH PRESSURE PISTON
20 - SYSTEM CONTROL VALVES
21 - HIGH PRESSURE CHAMBER

Figure 24 - Viscometer Hydraulic Control System

2. High pressure cylinder piston: The piston for the high pressure cylinder developed a longitudinal crack. A replacement was fabricated from centerless ground Carpenter Air Hard No. 484 tool steel (AISI A-2). Operation of the apparatus with this piston has been satisfactory.

3. Manganin coil pressure sensors: The pressure in the test chamber is monitored by a small coil made of linen-insulated manganin wire installed in the high pressure cylinder. At atmospheric pressure, this coil will have a resistance on the order of 100 ohm but the resistance will change linearly with pressure increases. Roughly a 3 percent increase will occur between atmospheric pressure and 100,000 psig. New coils must be stabilized to prevent resistance drift both by holding at elevated temperatures and at elevated pressures. The coils are calibrated by using an accurate Bourdon tube pressure gauge manufactured by the Heise Company as a secondary standard.

Sudden changes in pressure of the hexane fluid surrounding these coils will cause relative movement of the wires in the coil and eventually cause intermittent shorting to occur. Two failures occurred during the 1968 calendar year operations. Partial temperature and pressure stabilization of two to four coils was required before a suitable coil could be found each time a replacement coil was required.

4. Test fluid temperature: The temperature of the test fluid within the high pressure chamber is dependent upon the temperature of the liquid bath surrounding the chamber. Three steps have been taken to provide the high pressure viscometer with more accurate test fluid temperature measurement and more rapid changes in temperature between test conditions.

a. Test fluid temperature measurement: Initially, the test fluid temperature was assumed to be the same as that of the bath fluid surrounding the test chamber after an extended period at a constant temperature. However, test fluid temperature measurements can now be made with an internal test chamber thermocouple. Recent modifications to the test chamber terminal plug have eliminated the original three separately sealed electrical conductors and substituted six magnesium oxide insulated conductors (four iron and two constantan) in a single swaged sheath which is brazed into the terminal plug. The effects of pressure on the thermocouple, made with one each of the iron and constantan leads, were found to be negligible. The thermocouple EMF responds rapidly during the introduction of cooler hydraulic fluid to the test chamber while the pressure is being increased and also during the essentially adiabatic cooling during a pressure decrease. These temperature variations are small and normal readings are obtained after a short equalization period.

The primary advantage of this thermocouple in the interior of the test chamber is the elimination of prolonged periods of soaking to assure equalization of the temperatures of the bath outside of the chamber and the test specimen inside the chamber.

b. Heating control of high pressure chamber bath: Instrumentation to provide steady state temperature control of the test chamber bath has been added to the high pressure viscometer system. This instrumentation includes (1) a potentiometric control system with a thermocouple input and a retransmitting slidewire, (2) a current adjusting transformer (CAT) which provides adjustable proportional band control with rate and reset action, and (3) a single phase solid state power supply. Bath temperature control of $\pm 0.1°F$ has been repeatedly demonstrated over the desired elevated temperature range.

c. Cooling control of high pressure chamber bath: A refrigeration system has been installed in the chamber bath to provide control below room temperature. The system includes: (1) a freon compressor and condenser, (2) an expansion coil, (3) a calibrated temperature control hand valve, and (4) a sensitive suction pressure switch. Temperature control, demonstrated to be on the order of $\pm 0.05°F$, is achieved by adjusting the compressor suction pressure. The refrigeration system is now capable of maintaining constant bath temperatures down to about 45°F. Lower temperatures could be reached with the addition of insulation to the bath.

C. Test Fluid Turbulence

The possibility of turbulence occurring in the viscometer tube as the weight falls through the tube was considered. The basic relationship between the viscosity of the fluid and time-of-fall of a weight through the fluid is a logarithmic function and these functions were plotted (Figure 25). The resulting "curve" is a straight line and indicates no departure from laminar flow. A step in the curve would be expected if turbulent flow developed. Data plotted included times-of-fall to 1,329 sec. and known fluid viscosities of the calibrating fluids from 3.18 to 7,880 centipoise.

D. Computer-Assisted Data Reduction

Reduction of the experimental data to viscosities, densities, and related information is not complex but it is repetitive and if done by hand, rather cumbersome and time-consuming. The chances of human error are high. For these reasons, three computer programs were prepared.

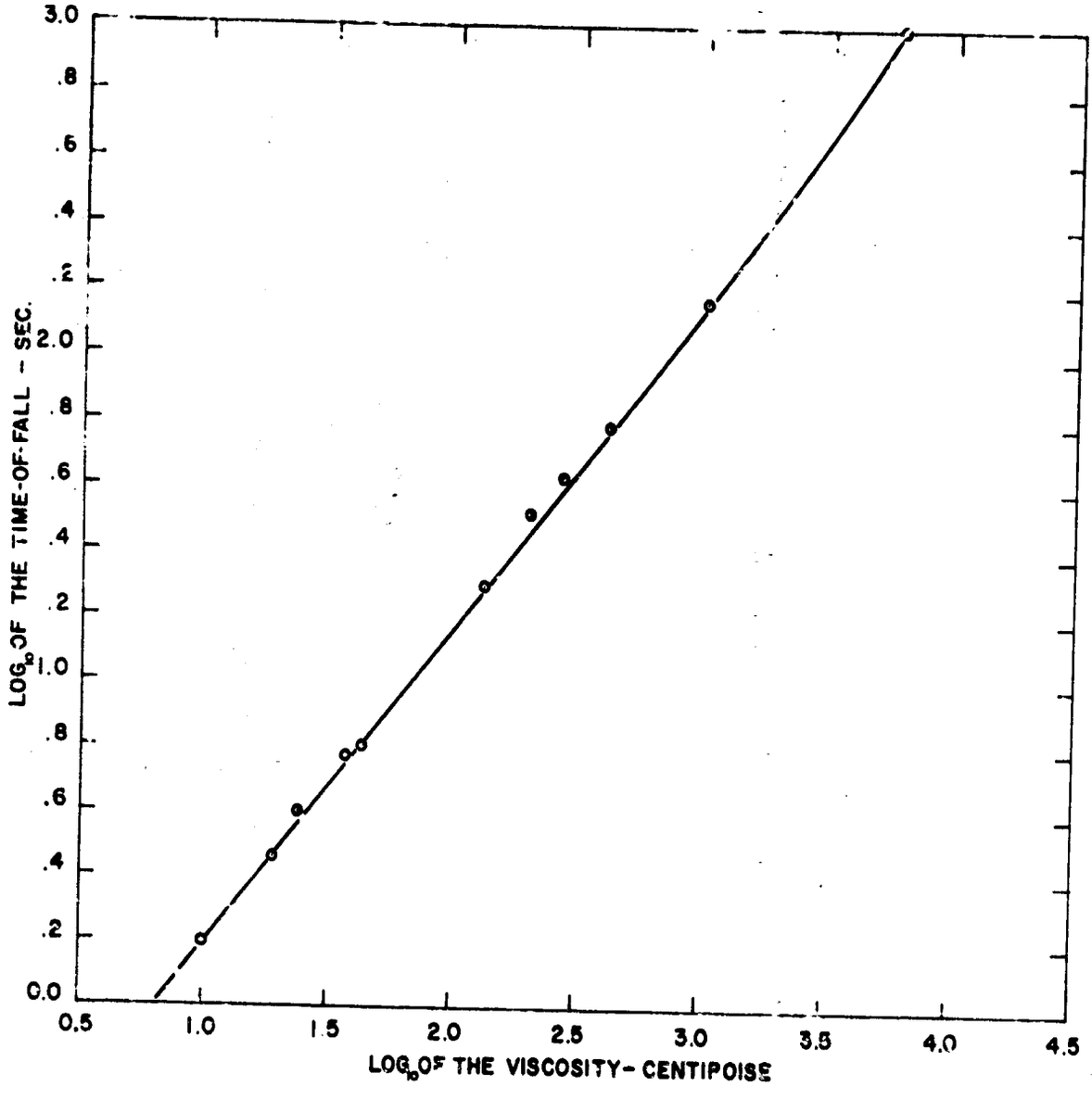


Figure 25 - Visccaity vs. Time-of-Fall of Weight
Through Calibrating Fluids

One of these programs, a relatively simple one, is used with a Wang calculator to rapidly reduce the experimental data to arrive at rough values for the form factors of the viscometer weight. These calculations can be done while the experimental work is in progress and provide a constant check on accuracy and repeatability.

A second program, also relatively simple, is used for precise and rapid calculation of the test fluid density values. This work is done on a time-sharing computer for which direct access by way of a keyboard is available. The resulting density-temperature-pressure data are plotted. The density data reported and used for the viscosity calculations are read from these graphs. Variations of the density data from the curves normally are less than 0.3 percent.

The third program, written in FORTRAN IV for an IBM 360 computer, is used to reduce all other experimental data. All operations, including conversion to proper units, are internal to the program. This program was initially described in Ref. 11 but has been modified to make it more versatile. Minor changes in the program logic, as originally presented in Ref. 11, have been made. The logic of the current program is presented in Appendix I.

E. Fluids to be Studied

Ten fluids have been scheduled for investigation in the high pressure viscometer. Initial investigations will cover up to 300°F and 150,000 psig if the fluid does not become too viscous at 150,000 psig. Subsequent investigations will be conducted with the less viscous fluids to 300°F and 250,000 psig. The fluids scheduled are as follows:

Fluid Code	Fluid Type	Fluid Code	Fluid Type
F-1041	5P4E	O-67-7	MIL-L-7808F
O-64-4	F-50	O-67-20	MIL-L-7808G
O-64-15	SAE 20	H-1001	MIL-L-23699
H-1026	MIL-L-7808C	O-64-25	MIL-L-23699
GTO-885	MIL-L-9236	O-66-25	

In addition, study of an additive-free bis(2-ethyl hexyl)sebacate* at 100° and 210°F was scheduled to get data for comparison with data for a similar fluid reported in Volume II of the ASME Pressure-Viscosity Report (Ref. 10) and with data on the same fluid reported by Novak and Winer of the University

* Plexol 201H, Lot 21-3614, PL-5159 manufactured by Rohm and Haas.

of Michigan (Refs. 12 and 13). The density of these 11 fluids at one atmosphere as a function of temperature is one of the inputs to the computer programs used to reduce the experimental data. These density data have been determined for all fluids except H-1001, which is not yet available, and are reported in Tables XX to XXIX. The procedures used to measure density are described briefly in this report in the section on bulk modulus (page 35). All planned pressure-viscosity work with the diester bis(2-ethyl hexyl)sebacate, the 5P4E polyphenyl ether (F-1041), and the F-50 chlorinated phenyl methyl silicone (0-64-4) has been completed and is reported in the following section.

TABLE XX

DENSITY OF F-1041 POLYPHENYL ETHER AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	1.194	1.195	-0.001	1.195
100	1.187	1.187	0	1.187
200	1.141	1.141	0	1.141
300	1.103	1.103	0	1.103
400	1.062	1.063	+0.001	1.062
450	1.041	1.039	+0.002	1.040

TABLE XXI

DENSITY OF 0-64-1 TYPE F-50 FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	1.038	1.038	0	1.038
100	1.030	1.029	+0.001	1.030
200	0.982	0.982	0	0.982
300	0.934	0.936	-0.002	0.935
400	0.888	0.889	-0.001	0.889
450	0.871	0.869	+0.002	0.870

TABLE XXII

DENSITY OF O-64-15 TYPE SAE 20 OIL AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.866	0.866	0	0.866
100	0.860	0.860	0	0.860
200	0.827	0.826	+0.001	0.827
300	0.794	0.793	+0.001	0.794
400	0.759	0.757	+0.002	0.758
450	0.736	0.739	-0.003	0.738

TABLE XXIII

DENSITY OF H-1026 TYPE MIL-L-7808C FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.920	0.920	0	0.920
100	0.916	0.915	+0.001	0.916
200	0.873	0.873	0	0.873
300	0.835	0.834	+0.001	0.835
400	0.798	0.796	+0.002	0.797
450	0.780	0.777	+0.003	0.779

TABLE XXIV

DENSITY OF GTO-885 TYPE MIL-L-9236 FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.959	0.959	0	0.959
100	0.953	0.953	0	0.953
200	0.912	0.912	0	0.912
300	0.871	0.871	0	0.871
400	0.831	0.829	+0.002	0.830
450	0.811	0.808	+0.003	0.810

TABLE XXV

DENSITY OF O-67-7 TYPE MIL-L-7808F FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.921	0.921	0	0.921
100	0.912	0.913	-0.001	0.913
200	0.875	0.870	+0.005	0.873
300	0.838	0.838	0	0.838
400	0.797	0.795	+0.002	0.796
450	0.774	0.772	+0.002	0.773

TABLE XXVI

DENSITY OF O-67-20 TYPE MIL-L-7808G FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.945	0.945	0	0.945
100	0.937	0.938	-0.001	0.938
200	0.899	0.899	0	0.899
300	0.858	0.857	+0.001	0.858
400	0.817	0.816	+0.001	0.817
450	0.796	0.793	+0.003	0.795

TABLE XXVII

DENSITY OF O-64-25 TYPE MIL-L-23699 FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	1.000	0.997	+0.003	0.999
100	0.992	0.991	+0.001	0.992
200	0.952	0.951	+0.001	0.952
300	0.911	0.910	+0.001	0.911
400	0.867	0.866	+0.001	0.867
450	0.847	0.846	+0.001	0.847

TABLE XXVIII

DENSITY OF O-66-25 FLUID AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.933	0.933	0	0.933
100	0.925	0.924	+0.001	0.925
200	0.886	0.886	0	0.886
300	0.849	0.847	+0.002	0.848
400	0.809	0.806	+0.003	0.808
450	0.787	0.784	+0.003	0.786

TABLE XXIX

DENSITY OF BIS(2-ETHYL HEXYL)SEBACATE* AT ONE ATMOSPHERE AS A FUNCTION OF TEMPERATURE

Temperature (°F)	Density, gm/ml			
	Hubbard Bottle	Gay-Lussac Bottle	Difference	Average
76	0.911	0.911	0.0	0.911
100	0.904	0.903	+0.001	0.904
200	0.864	0.864	0.0	0.864
300	0.826	0.825	+0.001	0.826
400	0.785	0.784	+0.001	0.765
450	0.766	0.763	+0.003	0.765

* Plexol 201 H, Lot [illegible]-3614, PL-5159.

F. Results of Experimental and Analytical Work

All planned pressure-viscosity work with the diester bis(2-ethyl hexyl)sebacate, the 5P4E polyphenyl ether (F-1041), and the F-50 chlorinated phenyl methyl silicone (0-64-4) has been completed. In addition, the data previously reported in AFML-TR-67-8, Part II (Ref. 11) for an ester of TMP (MLO-60-50) have been recompiled to take advantage of the latest analytical techniques.

The measured values of the densities of the four test fluids at elevated temperatures and pressures are listed in Table XXX. The three separate determinations for bis(2-ethyl hexyl)sebacate at 100°F demonstrated good repeatability. Data listed on Table XXX were plotted, best fit working curves were drawn, and the density values used in subsequent calculations and tabulations were read from these curves.

The values of bulk modulus tabulated in the tables of properties for each test fluid should be considered as approximations only. The equation used in calculating the isothermal secant bulk modulus using data from the high pressure viscometer reduces to

$$\bar{B}_T = \frac{P}{1-(\rho_o/\rho)}$$

when atmospheric pressure (0 psig) is taken as the initial pressure, P_o, and ρ is the density of the test fluid. As can be seen, bulk modulus calculations will be extremely sensitive to the accuracy of the density determinations. Pressure, P, is a very large number (magnitude of 10^4 to 10^5) and the term $[1-(\rho_o/\rho)]$ will be very small (magnitude of 10^{-1} to 10^{-3}). For example, an error of 0.2 percent (roughly ± 0.002 gm/ml) in the determination of ρ_o could be expected to result in an error larger than 10 percent in bulk modulus at 10,000 psi.

1. Bis(2-ethyl hexyl)sebacate: The good agreement of data generated at 100° and 210°F for an additive-free bis(2-ethyl hexyl)sebacate with data available in the literature (Refs. 10, 12 and 13) tends to establish the validity of pressure-viscosity data reported in subsequent paragraphs for other fluids. This diester is one of the few fluids for which comparative data from more than one previous investigation are available.

The time-of-fall of the viscometer weight through this fluid at 100° and 210°F is shown in Figure 26 as a function of pressure. Experimental work was not carried to the freezing pressure of this diester in order to conserve the apparatus. The computed data are tabulated on Table XXXI.

TABLE XXX

MEASURED DENSITIES AT ELEVATED PRESSURES OF FLUIDS STUDIED IN THE HIGH-PRESSURE VISCOMETER

Temperature (°F)	Pressure (psig/1,000)	Bis(2-ethyl hexyl)sebacate			P-1041	0-64-4	MLO-60-50
		1st Run	2nd Run	3rd Run			
32	0	-	-	-	-	-	1.0560
68	0	-	-	-	-	1.0452	-
	5	-	-	-	-	1.0711	-
	10	-	-	-	-	1.0991	-
	15	-	-	-	-	1.1226	-
	20	-	-	-	-	1.1378	-
	25	-	-	-	-	1.1566	-
	30	-	-	-	-	1.1694	-
77	0	-	-	-	-	1.0391	1.0550
	5	-	-	-	-	1.0709	-
	10	-	-	-	-	1.0960	-
	15	-	-	-	-	1.1119	-
	20	-	-	-	-	1.1375	-
	25	-	-	-	-	1.1551	-
	30	-	-	-	-	1.1675	-
	35	-	-	-	-	1.1790	-
100	0	0.9040	0.9040	0.9040	1.1870	1.1294	1.0260
	5	0.9206	0.9181	0.9222	-	1.0612	-
	10	0.9365	0.9353	0.9368	-	1.0867	1.0588
	15	0.9534	0.9504	0.9505	-	1.1081	-
	20	0.9654	0.9667	0.9697	-	1.1289	1 0927
	25	0.9724	0.9719	0.9747	-	1.1405	-
	30	0.9848	0.9826	0.9853	-	1.1537	1.1225
	35	0.9945	-	-	-	1.1657	-
	40	1.0018	-	1.0036	-	1.1765	1.1474
	45	1.0104	-	-	-	-	-
	50	1.0182	-	-	-	-	1.1707
	55	1.0260	-	-	-	-	-
	60	1.0328	-	1.0322	-	-	1.1930
	65	1.0395	-	-	-	-	-
	70	1.0458	-	-	-	-	1.2120
	75	1.0533	-	-	-	-	-
	80	1.0585	-	-	-	-	1.2316
	85	1.0638	-	-	-	-	-
	90	1.0685	-	-	-	-	1.2445
	100	-	-	-	-	-	1.2569
210	0	0.8600	-	-	1.1386	0.9775	0.9770
	5	0.8776	-	-	1.1557	1.0170	-
	10	0.8958	-	-	1.1739	1.0432	-
	15	0.9082	-	-	1.1880	1.0621	-
	20	0.9238	-	-	1.2042	1.0858	-
	25	0.9348	-	-	1.2167	1.1052	-
	30	0.9481	-	-	1.2311	1.1197	-
	35	-	-	-	1.2459	1.1326	-
	40	0.9664	-	-	1.2563	1.1429	-
	45	-	-	-	1 2645	1.1535	
	50	0.9846	-	-	1.2768	1.1573	-
	55	-	-	-	-	1.1613	-
	60	1.0205	-	-	-	1.1708	-
	65		-	-	-	1.1721	-
	70	1.0326	-	-	-	1.1749	-
	80	1.0600	-	-	-	-	-
	90	1.0464	-	-	-	-	-
300	0	-	-	-	1.0606	0.9380	0.9380
	5	-	-	-	1.084	0.9570	-
	10	-	-	-	1.0980	0.9771	-
	15	-	-	-	1.1137	-	-
	20	-	-	-	1.1298	1.0175	-
	25	-	-	-	1.1437	-	-
	30	-	-	-	1.1579	1.0516	-
	35	-	-	-	1.1698	-	-
	40	-	-	-	1.1799	1.0789	-
	45	-	-	-	1.1874	-	-
	50	-	-	-	1.1988	1.0997	-
	55	-	-	-	1.2081	-	-
	60	-	-	-	1.2182	1.1206	-
	65	-	-	-	1.2264	-	-
	70	-	-	-	1.2312	1.1372	-
	80	-	-	-	-	1.1538	-
	90	-	-	-	-	1.1682	-

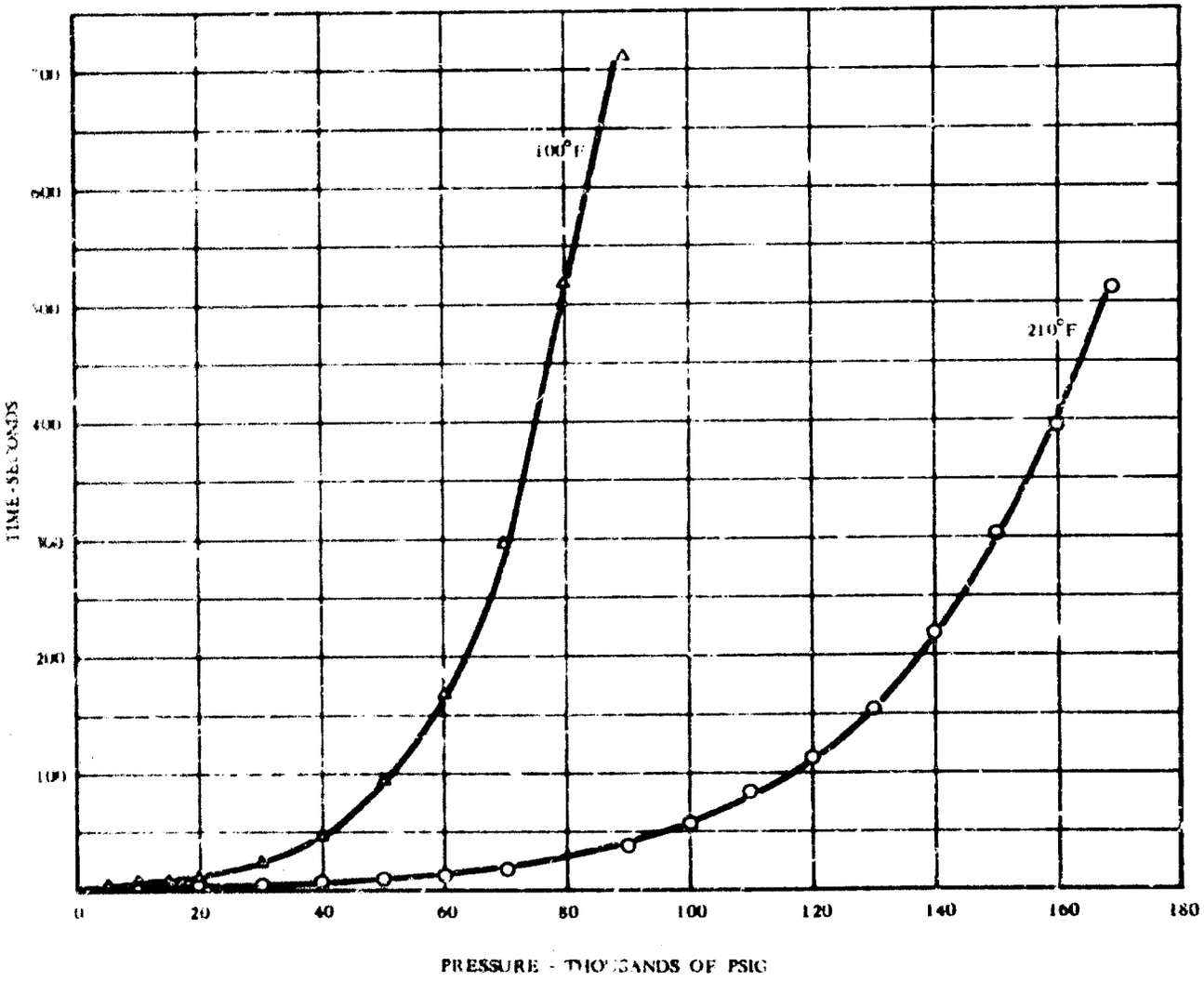


Figure 28 - Viscometer Weight Fall-Time as a Function of Temperature and Pressure for Bis(2-ethyl hexyl)sebacate

TABLE XXXI

PRESSURE-VISCOSITY DATA FOR BIS(2-ETHYL HEXYL)SEBACATE

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
100.	14.7	2.0959	13.66	12.35	12.54	12.40	-	0.9040	1.1062
100.	5000.0	3.4613	22.24	20.45	20.75	20.51	296612.	0.9195	1.0875
100.	10000.0	5.3336	33.65	31.45	31.75	31.39	306393.	0.9345	1.0701
100.	15000.0	7.7572	48.16	45.65	45.94	45.42	323182.	0.9480	1.0549
100.	20000.0	11.3795	69.55	66.85	67.46	66.70	337193.	0.9610	1.0406
100.	30000.0	24.5560	146.23	143.81	144.89	143.26	371132.	0.9835	1.0168
100.	40000.0	47.9143	279.19	279.89	282.24	279.06	407138.	1.0025	0.9975
100.	50000.0	94.2951	539.18	549.59	552.56	546.34	442022.	1.0193	0.9811
100.	60000.0	169.0153	955.90	993.40	999.95	978.81	477232.	1.0340	0.9671
100.	70000.0	297.1799	1648.29	1725.76	1724.85	1705.44	512517.	1.0470	0.9551
100.	80000.0	516.7263	2932.26	2996.54	3030.25	2996.16	549611.	1.0583	0.9452
100.	90000.0	799.7629	3849.11	4110.84	4142.35	4095.75	586198.	1.0680	0.9363
210.	14.7	0.7325	5.07	4.36	4.41	4.36	-	0.8600	1.1628

TABLE XXXI (Concluded)

TEST TEMP (F)	TEST PRESSURE (PSI)	STINGER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
210.	10000.0	1.2349	9.19	7.32	7.35	7.27	270606.	0.9930	1.1198
210.	20000.0	2.0160	12.89	11.89	11.91	11.78	293016.	0.9230	1.0834
210.	30000.0	3.3136	20.54	19.48	19.58	19.35	321525.	0.9485	1.0543
210.	40000.0	5.2070	31.43	30.51	30.61	30.27	349910.	0.9710	1.0299
210.	50000.0	8.3375	49.20	48.73	49.02	48.46	379502.	0.9905	1.0096
210.	60000.0	12.7417	73.75	74.30	74.77	73.93	409831.	1.0075	0.9926
210.	70000.0	18.9704	108.04	110.41	110.94	109.69	441606.	1.0220	0.9785
210.	80000.0	30.2529	217.67	227.75	229.57	226.99	505450.	1.0463	0.9557
210.	100000.0	57.6283	316.08	333.94	335.06	331.29	537660.	1.0565	0.9465
210.	110000.0	82.1568	446.32	475.56	475.65	470.30	570342.	1.0655	0.9385
210.	120000.0	113.5083	611.47	656.41	659.40	651.98	603373.	1.0735	0.9315
210.	130000.0	150.4524	804.60	869.37	871.60	861.79	637030.	1.0805	0.9255
210.	140000.0	218.5002	1160.69	1261.68	1268.36	1254.09	670396.	1.0870	0.9200

This table includes all the property data available from the pressure-viscosity studies--kinematic viscosity in centistokes, absolute viscosity in centipoises, isothermal secant bulk modulus in psi, and density and specific volume in cgs units. These data are tabulated as functions of temperature and pressure. The time-of-fall of the weight through the viscometer tube is also listed. Two extra absolute viscosity columns, "roll left" and "roll right", were included to indicate the minor differences in measured viscosity that were observed as the falling weight traveled from one end of the viscometer tube to the other and then back to the original position. The column listing "absolute viscosity" is not the average of these two "roll left" and "roll right" viscosity values because slightly different falling weight "form factors" were used in computing the data in the three "absolute viscosity" columns--a roll left, a roll right, and an average form factor.

Comparisons of the absolute viscosity and density data determined in this work with those reported in 1953 by the ASME (Ref. 10) and in 1968 by Novak and Winer of the University of Michigan (Refs. 12 and 13) are shown graphically in Figures 27 and 28. The curves on these figures were drawn to fit the ASME data points. The fluid for the ASME study was supplied by the Naval Research Laboratory and the fluid for the MRI and University of Michigan determinations was supplied by Rohm and Haas under the trade name Plexol 201H (Lot 21-3614, PL-5159).

The absolute viscosity data for all three studies are in excellent agreement. There are some differences in density data. Novak reported (Ref. 13) that the density "at all temperatures and pressures was determined from bulk modulus correlations" and referenced, without elaboration, a paper by Wright and another by Tichy and Winer (Refs. 14 and 15). These papers develop methods for predicting the bulk moduli and densities of petroleum oils (Ref. 14) and silicone fluids (Ref. 15) at very high pressures and a wide range of temperatures. Both used, in part, data from the ASME Pressure-Viscosity Report (Ref. 10) in developing their predictions. The only data generally required by these methods to estimate bulk moduli and density are the density of the fluids of interest at atmospheric pressure and 77°F. Details of the adaptation of these procedures for petroleum oils and silicones to density determinations for the synthetic fluid bis(2-ethyl hexyl)sebacate are not clear. At any rate, the absolute viscosity data of Novak and Winer were not influenced because the density data were used by them only to determine kinematic viscosity.

2. F-1041 polyphenyl ether (5P4E): Scheduled pressure-viscosity work with F-1041 has been completed. The time-of-fall of the viscometer weight through this fluid at 100°, 210°, and 300°F is shown in Figure 29 as a function of pressure. It will be noted that only one point appears at 100°F.

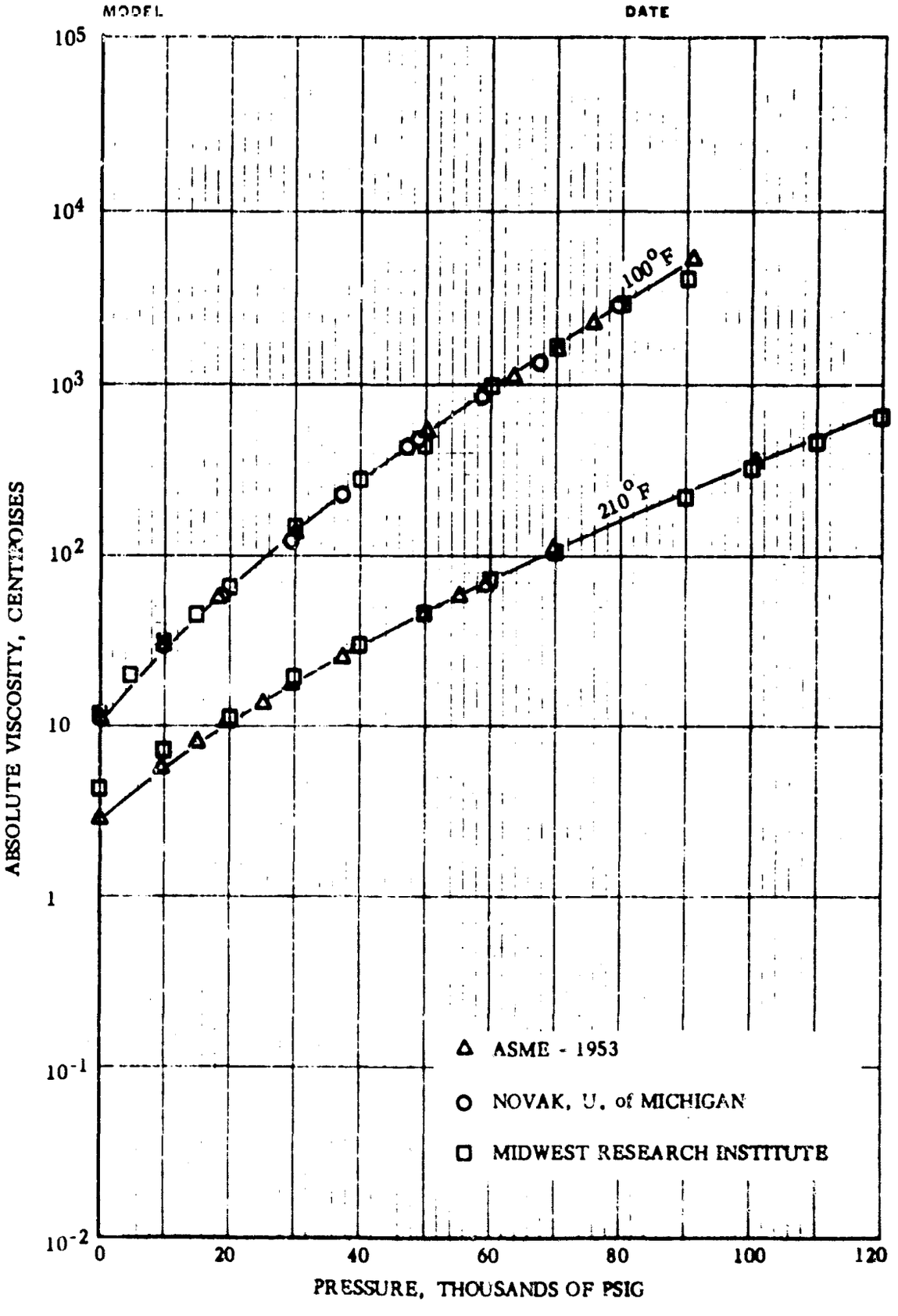


Figure 27 - Comparison of Absolute Viscosity-Pressure Data for Bis(2-ethyl hexyl)sebacate

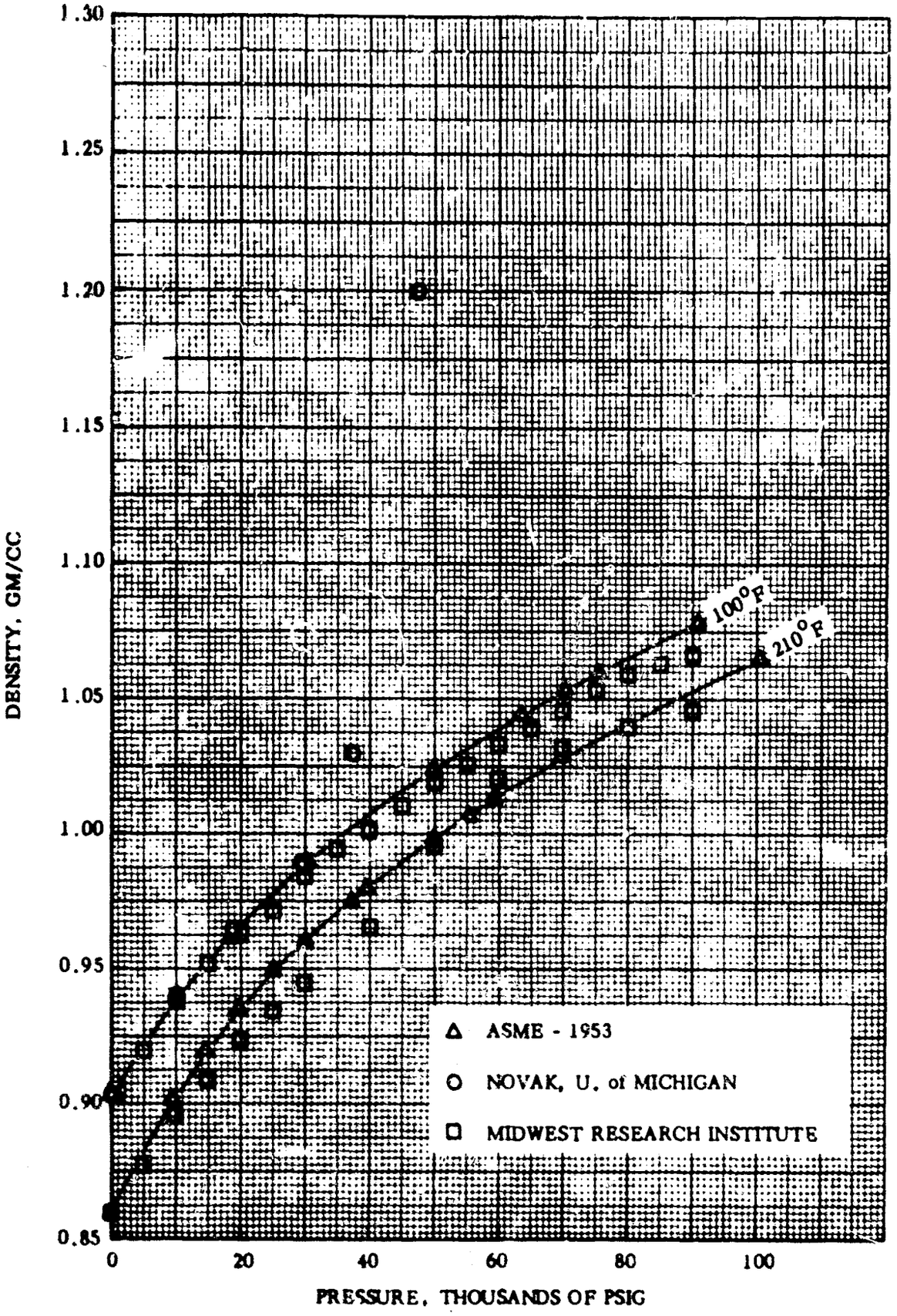


Figure 28 - Comparison of Density-Pressure Data for Bis(2-ethyl hexyl)sebacate

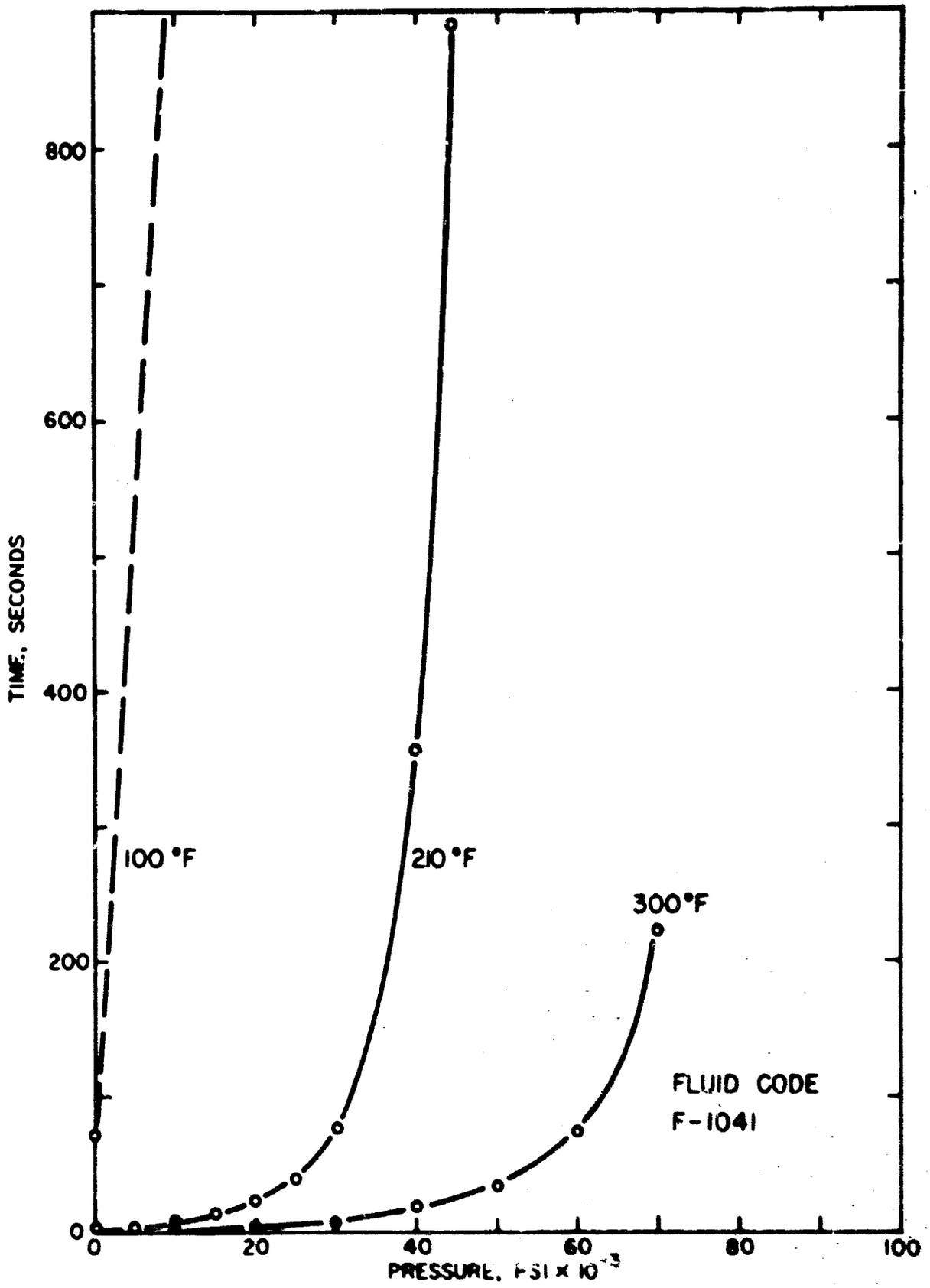


Figure 29 - Viscometer Weight Fall-Time as a Function of Temperature and Pressure for F-1041 (a 5P4E Polyphenyl Ether)

At 10,000 psig and 100°F, the F-1041 became very viscous and the time-of-fall noted (979.2 sec.) was longer than is practical to measure. The data shown graphically in Figure 29 were used, with data generated with the compressibility apparatus and several parameters of the high pressure viscometer, to compute the data shown in Table XXXII. It will be noted that, at each temperature level, experimental work was stopped before the test fluid became a solid or reached the probable pour point.

More work is planned regarding the comparison of the data for F-1041 with any available data for fluids of similar composition.

3. 0-64-4 Type F-50 chlorinated phenyl methyl silicone: All scheduled pressure viscosity work with 0-64-4 has been completed. The time-of-fall of the viscometer weight through this fluid at 68°, 77°, 100°, 210°, and 300°F is shown in Figure 30 as a function of pressure.

Following the same procedures indicated in the preceding discussions, the output of the experimental pressure-viscosity studies of 0-64-4 silicone was computed and is tabulated on Table XXXIII. These data have not yet been compared with other data available for similar fluids.

4. MLO-60-50 ester of TMP: Viscosity and density data for MLO-60-50 at high pressures were previously reported in AFML-TR-67-8, Part II (Ref. 11). The experimental data for this fluid were reduced again using the improved and expanded data handling techniques developed during the past year. Results of the recalculations are tabulated in Table XXXIV. Comparison of the absolute viscosity data of Table VII on page 25 of Ref. 11 with that of Table XXXIV shows minor differences (3.9 percent average). A similar comparison of density data of Table V, page 20 of Ref. 11, with that of Table XXXIV also shows no major discrepancies (1.2 percent average). The primary reasons these differences exist are that (1) viscometer weight fall-times of less than 1 sec. through calibrating fluids are no longer considered in calculating the weight form factors because of the relatively large value of the corrections that must be applied, and (2) the density data are now smoothed before analytical viscosity work is started. The MLO-60-50 data reported here are considered to be of greater accuracy than those reported in AFML-TR-67-8, Part II (Ref. 11). However, the overall precision of these data is probably less than that for the three other test fluids discussed in this report because (1) experimental procedures were still being refined when the MLO-60-50 data were taken, and (2) density data at elevated pressures and temperatures other than 100°F were estimated (pages 18 and 19 of Ref. 11).

TABLE XXXII

PRESSURE-VISCOSITY DATA FOR F-1041
(a 5P4E polyphenyl ether)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
100.	14.7	73.8173	352.80	418.78	420.14	415.41	-	1.1870	0.8425
100.	10000.0	980.4832	4537.33	5535.55	5554.09	5491.60	366372.	1.2200	0.8197
210.	14.7	3.1332	15.69	17.88	18.31	18.10	-	1.1395	0.8776
210.	5000.0	4.9199	24.24	28.01	28.09	27.78	361097.	1.1555	0.8654
210.	10000.0	8.1490	39.66	46.50	46.14	45.62	355303.	1.1725	0.8529
210.	15000.0	12.0364	57.41	68.20	68.51	67.74	367422.	1.1880	0.8418
210.	20000.0	22.7015	106.64	128.34	129.04	127.58	376093.	1.2035	0.8309
210.	25000.0	38.4332	178.01	219.21	219.83	217.35	387899.	1.2180	0.8210
210.	30000.0	75.1681	343.65	423.21	428.30	423.49	401576.	1.2315	0.8120
210.	40000.0	356.8572	1555.50	2002.35	2004.60	1982.05	434634.	1.2550	0.7968
210.	45000.0	885.1404	3918.71	4959.12	4916.40	4861.09	451966.	1.2655	0.7902
210.	50000.0	2726.3398	11969.60	15255.25	15237.04	15065.61	472036.	1.2745	0.7846
300.	10000.0	1.9175	10.02	11.00	11.39	11.26	297421.	1.0975	0.9112

TABLE XXXII (Concluded)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
300.	20000.0	3.6015	18.22	20.57	20.82	20.59	330115.	1.1290	0.8857
300.	30000.0	7.2776	35.82	41.40	41.79	41.32	363522.	1.1560	0.8651
300.	40000.0	15.1704	72.93	86.02	86.65	85.67	396803.	1.1795	0.8478
300.	50000.0	34.5242	162.74	195.21	197.52	195.30	431786.	1.1995	0.8337
300.	60000.0	74.0045	342.49	417.32	420.08	415.35	463015.	1.2185	0.8207
300.	70000.0	222.5785	1013.02	1252.09	1218.49	1204.78	493273.	1.2360	0.8091

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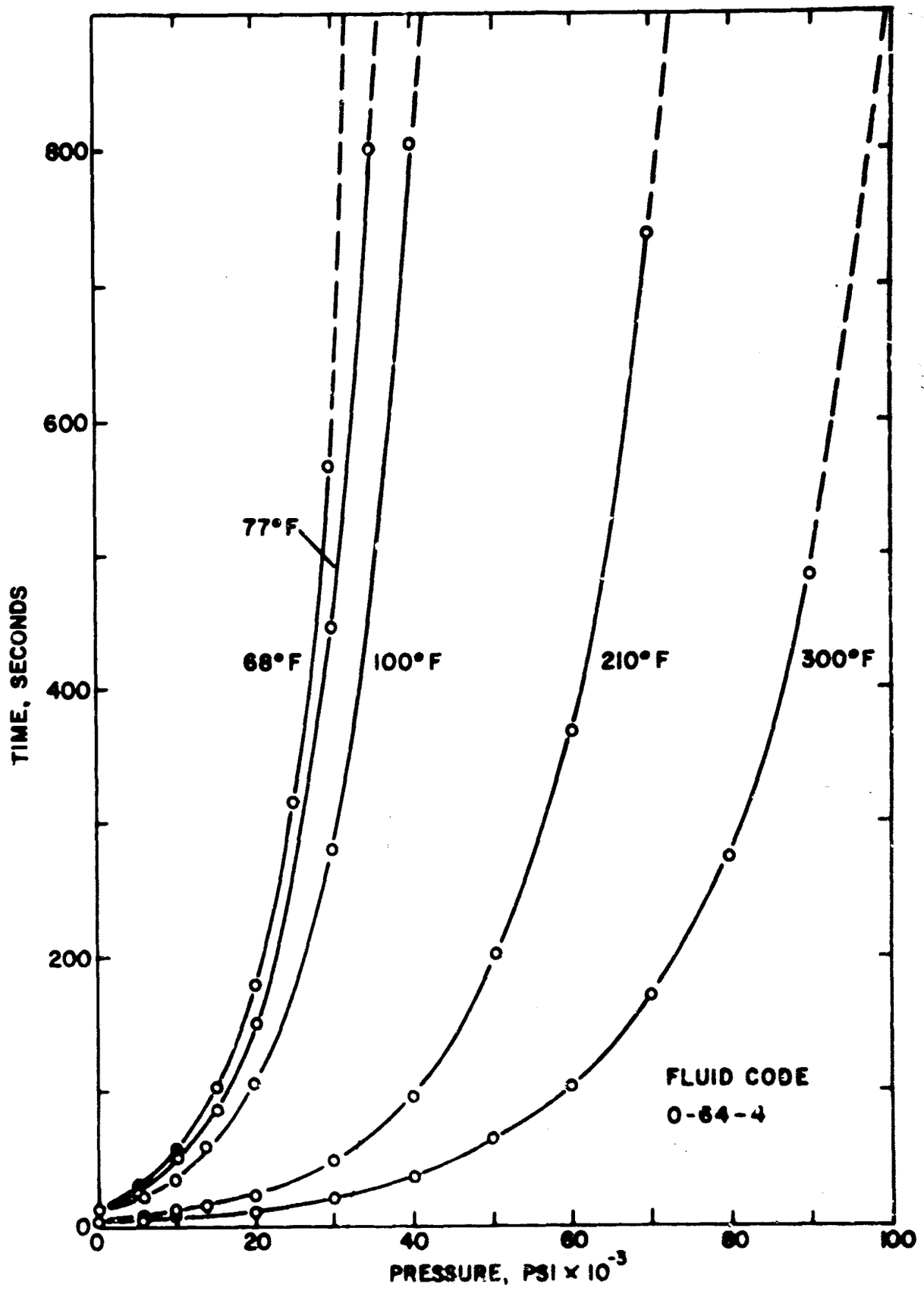

Figure 30 - Viscometer Weight Fall-Time as a Function of Temperature and Pressure for 0-64-4 (F-50 Chlorinated Phenyl Methyl Silicone)

TABLE XXXIII

PRESSURE-VISCOSITY DATA FOR 0-64-4
(F-50 chlorinated phenyl methyl silicone)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
68.	14.7	14.0361	89.19	93.04	93.44	92.39	-	1.0432	0.9586
68.	5000.0	32.1333	173.12	185.64	188.34	186.22	184241.	1.0723	0.9326
68.	10000.0	61.2094	320.50	352.23	361.20	357.13	196952.	1.0990	0.9099
68.	15000.0	105.2118	538.07	603.45	605.58	598.77	214848.	1.1215	0.8917
68.	20000.0	183.2711	919.58	1048.32	1050.96	1039.13	235537.	1.1400	0.8772
68.	25000.0	321.8474	1588.53	1836.66	1854.87	1834.00	255796.	1.1562	0.8649
68.	30000.0	579.9553	2795.28	3269.08	3284.46	3247.50	277791.	1.1695	0.8551
77.	14.7	13.8591	77.42	80.45	81.06	80.14	-	1.0391	0.9624
77.	5000.0	26.9803	146.26	155.99	157.13	155.36	194616.	1.0665	0.9376
77.	10000.0	50.1502	264.43	286.89	289.53	286.27	204589.	1.0925	0.9153
77.	15000.0	89.3158	459.36	512.64	514.51	508.73	217686.	1.1160	0.8961
77.	20000.0	153.5134	773.36	878.54	880.35	870.45	234468.	1.1360	0.8803
77.	30000.0	454.2410	2219.81	2588.30	2597.55	2568.32	275650.	1.1660	0.8576

TABLE XXXIII (Continued)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
77.	35000.0	814.4917	3934.99	4633.44	4661.82	4609.37	297778.	1.1775	0.8493
100.	14.7	10.2106	57.64	59.34	59.59	58.92	-	1.0294	0.9714
100.	5000.0	20.7947	113.81	120.35	120.44	119.08	188167.	1.0575	0.9456
100.	10000.0	36.9454	196.28	212.96	213.89	211.48	195146.	1.0850	0.9217
100.	15000.0	63.0272	326.96	362.11	362.01	357.93	212710.	1.1075	0.9029
100.	20000.0	106.6228	542.29	610.88	613.63	606.73	232030.	1.1265	0.8877
100.	30000.0	280.5461	1383.84	1600.41	1601.69	1583.68	272974.	1.1565	0.8647
100.	40000.0	806.5073	3901.57	4588.25	4613.43	4561.53	320874.	1.1760	0.8503
210.	14.7	3.3552	20.07	19.62	19.75	19.53	-	0.9775	1.0230
210.	5000.0	6.1238	35.21	35.63	35.82	35.41	146670.	1.0120	0.9881
210.	10000.0	10.4909	60.36	60.78	60.74	60.05	162736.	1.0415	0.9602
210.	15000.0	15.7303	95.14	90.80	91.10	90.07	179748.	1.0665	0.9376
210.	20000.0	23.7818	125.59	136.88	137.39	135.85	199359.	1.0865	0.9204
210.	30000.0	48.2522	247.16	276.45	277.47	274.35	237979.	1.1165	0.8961

TABLE XXXIII (Concluded)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
210.	40000.0	96.1105	481.32	548.94	551.18	544.97	279878.	1.1405	0.8768
210.	51000.0	201.6407	993.18	1149.11	1154.89	1141.89	328730.	1.1570	0.8643
210.	60000.0	369.0049	1797.80	2099.84	2111.48	2087.73	367875.	1.1680	0.8562
210.	70000.0	733.0935	3576.55	4202.44	4223.63	4176.11	416457.	1.1750	0.8511
100.	14.7	1.8098	11.37	10.64	10.67	10.55	-	0.9358	1.0686
100.	5000.0	3.4257	20.98	20.07	20.14	19.91	225707.	0.9570	1.0449
100.	10000.0	5.6283	33.64	32.88	33.02	32.65	234412.	0.9775	1.0230
100.	20000.0	11.6788	66.68	67.84	68.05	67.28	249082.	1.0175	0.9828
100.	30000.0	21.6022	118.96	124.91	125.57	124.15	275833.	1.0500	0.9524
100.	40000.0	37.4715	201.06	215.94	217.59	215.08	310855.	1.0740	0.9311
100.	50000.0	63.0392	331.14	362.27	365.16	361.06	345765.	1.0940	0.9141
100.	60000.0	102.3349	529.95	586.86	591.40	584.74	383247.	1.1095	0.9013
100.	70000.0	170.4221	869.63	975.73	983.85	972.78	421805.	1.1220	0.8913
100.	80000.0	273.7485	1394.24	1565.57	1580.01	1562.23	463525.	1.1310	0.8842
100.	90000.0	454.9998	2302.48	2622.52	2640.09	2610.39	504478.	1.1390	0.8780

TABLE XXXIV

PRESSURE-VISCOSITY DATA FOR MLO-60-50
(an ester of trimethylolpropane)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
32.	14.7	11.4871	57.96	61.20	60.99	61.42	-	1.0560	0.9470
32.	5000.0	20.8598	103.51	111.02	111.20	110.83	325009.	1.0725	0.9324
32.	10000.0	37.6855	184.36	200.39	201.45	199.33	350643.	1.0870	0.9200
32.	15000.0	66.1840	318.56	351.53	353.08	349.97	368476.	1.1035	0.9062
32.	20000.0	106.4264	504.65	564.71	565.31	564.09	355239.	1.1190	0.8937
32.	25000.0	145.2119	678.82	769.78	773.72	765.83	363461.	1.1340	0.8818
32.	30000.0	-	-	-	-	-	376133.	1.1470	0.8718
32.	35000.0	250.3832	1142.77	1325.61	1332.61	1318.57	390384.	1.1600	0.8621
77.	14.7	4.6115	23.60	24.63	24.54	24.73	-	1.0350	0.9662
77.	10000.0	12.7136	63.38	67.75	67.55	67.95	314416.	1.0690	0.9355
77.	20000.0	31.1265	150.41	165.52	165.34	165.71	336034.	1.1005	0.9087
77.	30000.0	69.4817	326.23	368.80	368.18	369.43	355131.	1.1305	0.8846
77.	40000.0	147.0157	675.20	779.22	779.02	779.42	377961.	1.1575	0.8639

TABLE XXXIV (Continued)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
77.	50000.0	297.4414	1333.68	1575.07	1567.50	1582.66	404452.	1.1810	0.8467
100.	14.7	2.8619	14.92	15.30	15.22	15.38	-	1.0260	0.9747
100.	10000.0	7.4546	37.56	39.77	39.53	40.02	320908.	1.0590	0.9443
100.	20000.0	17.2292	84.16	91.74	91.13	92.35	340624.	1.0900	0.9174
100.	30000.0	36.6200	173.77	194.63	193.01	196.25	357443.	1.1200	0.8929
100.	40000.0	70.6707	326.82	375.03	371.64	378.44	377777.	1.1475	0.8715
100.	50000.0	112.0000	506.59	593.72	592.34	595.10	401367.	1.1720	0.8532
100.	60000.0	230.6682	1024.41	1222.12	1219.57	1224.67	428621.	1.1930	0.8382
100.	70000.0	440.2236	1923.92	2331.79	2404.84	2258.37	456127.	1.2120	0.8251
100.	80000.0	-	-	-	-	-	484334.	1.2290	0.8137
100.	90000.0	1337.1941	5698.53	7086.12	7200.43	6971.20	514550.	1.2435	0.8042
100.	100000.0	-	-	-	-	-	545118.	1.2565	0.7959
210.	14.7	0.8293	4.57	4.46	4.59	4.33	-	0.9770	1.0235
210.	10000.0	1.5096	8.03	8.10	8.20	8.00	315314.	1.0090	0.9911

TABLE XXXIV (Continued)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
210.	20000.0	2.6111	13.46	13.99	14.01	13.96	332643.	1.0395	0.9620
210.	30000.0	4.3327	21.68	23.17	23.20	23.14	348589.	1.0690	0.9355
210.	40000.0	6.8685	33.38	36.67	36.67	36.67	361648.	1.0985	0.9103
210.	50000.0	10.0083	51.17	57.61	57.61	57.62	377853.	1.1260	0.8881
210.	60000.0	17.0000	78.68	90.52	90.31	90.73	397869.	1.1505	0.8692
210.	70000.0	23.4765	106.49	124.91	126.04	123.78	418930.	1.1730	0.8525
210.	80000.0	36.0000	160.50	191.47	191.03	191.92	441852.	1.1930	0.8382
210.	90000.0	54.0079	237.29	287.24	291.62	282.83	466574.	1.2105	0.8261
210.	100000.0	74.5780	323.29	396.68	400.24	393.10	490801.	1.2270	0.8150
210.	110000.0	105.2478	451.53	560.15	563.01	557.23	517856.	1.2405	0.8061
210.	120000.0	154.1249	655.09	820.83	820.22	821.43	544784.	1.2530	0.7981
210.	130000.0	220.6450	930.53	1176.19	1175.91	1176.46	572544.	1.2640	0.7911
210.	140000.0	310.2000	1299.34	1655.36	1651.51	-	600540.	1.2740	0.7849
300.	14.7	0.5282	3.04	2.85	2.76	2.95	-	0.9380	1.0661

TABLE XXXIV (Concluded)

TEST TEMP (F)	TEST PRESSURE (PSI)	SINKER FALL TIME (SEC.)	KINEMATIC VISCOSITY (CST)	ABSOLUTE VISCOSITY (CPS)	ROLL LEFT ABSOLUTE VISCOSITY (CPS)	ROLL RIGHT ABSOLUTE VISCOSITY (CPS)	BULK MODULUS (PSI)	DENSITY (GM/CC)	SPECIFIC VOLUME (ML/GM)
300.	10000.0	-	-	-	-	-	333448.	0.9670	1.0341
300.	20000.0	1.1832	6.41	6.37	6.33	6.42	349123.	0.9950	1.0050
300.	30000.0	1.7431	9.17	9.38	9.32	9.44	363021.	1.0225	0.9780
300.	40000.0	2.1358	10.93	11.47	11.39	11.55	376504.	1.0495	0.9528
300.	50000.0	2.9870	14.91	16.03	15.93	16.12	392375.	1.0750	0.9302
300.	60000.0	3.8637	18.82	20.71	20.67	20.74	406340.	1.1005	0.9087
300.	70000.0	5.2048	24.77	27.86	27.80	27.92	421123.	1.1250	0.8889
300.	80000.0	9.0603	42.26	48.47	47.97	48.97	439044.	1.1470	0.8718
300.	90000.0	12.1606	55.67	65.02	64.77	65.27	457045.	1.1680	0.8562
300.	100000.0	16.1171	72.65	86.17	85.86	86.48	478226.	1.1860	0.8432

G. Bibliography of Pressure-Viscosity Work

During the process of determining that the diester bis(2-ethyl hexyl) sebacate would be one of the better fluids for us to study in order to compare our pressure-viscosity data with that produced by previous investigators, numerous pertinent references were found. These references are listed in Appendix II.

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APPENDIX I

LOGIC OF HIGH PRESSURE VISCOMETER COMPUTER PROGRAM

The main computer program for reduction of the data obtained from the High Pressure Viscometer is written in FORTRAN IV programming language for use on an IBM Series 360 Computer with Disk Operating System. The program is actually a combination of three programs; (a) a statistical subprogram for averaging weight fall-times, (b) a calibration program for calculating falling weight form factors, and (c) a computation program for calculating viscosity and other data for each combination of pressure and temperature.

1. Statistical subprogram: The statistical subprogram converts the weight fall counts to seconds and computes the average. The program is constructed to read 11 data cards each time it is called upon. Each data card has two fall counts--one for roll left and one for roll right. At least one value of each must be nonzero, must be the first value read, and zero's must be entered for missing fall counts. After reading the fall counts, the subprogram operates in the following manner:

a. All nonzero fall counts are converted to seconds and averaged.

b. The fall-time with the greatest deviation from the average is determined and compared to a preselected time limit (± 5 percent of the average).

c. If the deviation does not exceed the limit, the average fall-time is returned to the main program.

d. If the deviation exceeds the limit, that fall-time is discarded and steps a and b are repeated until a maximum deviation is found which does not exceed the time limit.

e. In step d, if the number of discarded fall-times exceeds one-third the number of original nonzero fall-times, the data are considered scattered and a fall-time of 0 sec. is returned to the main program.

2. Form factor calibration: The form factor calibration section computes an average falling weight form factor for roll left and for roll right and also an average of the two. The density-viscosity properties of the NBS calibration fluids are known and read into the program. Roll left

and roll right fall-times are averaged and the average of the averages is taken. The three form factors (roll left, roll right, average) are then computed, stored for further use, and are also printed out in a form suitable for reproduction.

3. Pressure-temperature data: This portion of the program calculates the weight fall-time, absolute viscosity (roll right, roll left, average), kinematic viscosity, isothermal secant bulk modulus, and specific volume at given temperatures and pressures. The program operates in the following manner:

a. A given temperature, pressure, and density are read.

b. Fall counts at the given temperature and pressure are read, converted, averaged, and fall-time roll left, fall time roll right, and fall-time average are calculated.

c. Absolute viscosity for roll right, roll left, and average are calculated for the given temperature and pressure.

d. Kinematic viscosity is calculated for the given temperature and pressure.

e. Specific volume and bulk modulus are calculated for the given temperature and pressure.

Steps a - e are repeated for each combination of temperature and pressure.

f. Pressure, temperature, fall-time, density, the three absolute viscosities, kinematic viscosity, bulk modulus, and specific volume are printed out in a form suitable for reproduction.

g. Pressure, temperature, fall-time, density, absolute viscosity average, kinematic viscosity, and bulk modulus are punched onto data cards.

APPENDIX II

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13. ABSTRACT

Four shear/thermal stability pump loop experiments were conducted between 400° and 600°F with XF-1-0301 fluorosilicone. This fluid is reasonably stable up to 450°F, will form solids without appreciable property changes at 500°F, and will undergo physical property changes when used at 600°F. XF-1-0301 satisfactorily lubricated an aircraft-type piston pump at 400°F but permitted excessive wear at 500°F.

The bulk modulus of XF-1-0301 was determined to 450°F and 10,000 psig. In general, the isothermal secant bulk modulus for this fluid is higher than a polymeric perfluorinated fluid and a chlorinated phenyl methyl silicone, about the same as MLO-8200 disiloxane and a phenyl methyl silicone, and lower than the bulk moduli of petroleum base fluids, an ester of TMP, a silane, and a 5P4E polyphenyl ether.

Comparative four-ball tests were run at 400°F with XF-1-0301, a polymeric perfluorinated fluid, a deep dewaxed mineral oil, and chlorinated and unchlorinated phenyl methyl silicones. In addition, new and used fluids, other than the silicones, were also studied. The tests indicated that at 400°F the XF-1-0301 fluid will have lubricating characteristics similar to the polymeric perfluorinated fluid and the deep dewaxed mineral oil and superior to the silicones. There was no great difference in the results of the tests run in new fluids and those run in fluids which had been used in pump loop experiments.

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Lubricity						
Hydraulic Pump Loops						
High Vacuum Bearing Wear						
Bulk Modulus						

13. ABSTRACT (Concluded)

Febrication of a high vacuum simulated bearing wear rig was completed. This rig will be used in existing vacuum systems to evaluate, primarily, solid lubricants to 1500°F.

Pressure-viscosity data for the diester bis(2-ethyl hexyl)sebacate were determined to 90,000 psig at 100°F and to 140,000 psig at 210°F. Agreement of these data with those of previous investigators is good. In addition, the viscosity and density of two fluids, a 5P4E polyphenyl ether and a chlorinated phenyl methyl silicone, were determined to 300°F. The maximum pressure of the work with the polyphenyl ether was 70,000 psig and the maximum pressure of the work with the silicone was 90,000 psig. Both fluids became too viscous for higher pressure work.

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