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PART III

DEVELOPMENT OF GREASE LUBRICANTS FOR HIGH TEMPERATURE BALL AND ROLLER BEARINGS OF ELECTRICAL EQUIPMENT

TECHNICAL REPORT NO. WADD-TR-60-557, Part III
May 1963

Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 3044, Task No. 304403

(Prepared under Contract No. AF 33(616)-7597 by the American Oil Company, Research and Development Department, Whiting, Indiana; K. R. Bunting, R. G. Garst, F. K. Kawahara, H. M. Sellei, T. P. Traise, H. J. Liehe and R. S. Barnes, authors.)

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WADD TECHNICAL REPORT 60-557

PART III

DEVELOPMENT OF GREASE LUBRICANTS FOR HIGH TEMPERATURE BALL AND ROLLER BEARINGS OF ELECTRICAL EQUIPMENT

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American Oil Company
Research and Development Department
Whiting, Indiana

March 1963

Directorate of Materials and Processes
Contract No. AF33(616)-7597
Project No. 1(3-8128)

AERONAUTICAL SYSTEMS DIVISION
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by the Research and Development Department of the American Oil Company under USAF Contract No. AF33(616)-7597. The contract was initiated under Project No. 3044, "Aerospace Lubricants," Task No. 304403, "Grease Lubricants and Grease-Like Materials." This work was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, with Mr. J. Christian acting as project engineer.

This report covers work done from 1 November 1961 to 1 January 1963.

* were investigated.

ABSTRACT

The objective of this work is the development of grease systems capable of operating in loaded bearings over the temperature range of -65 to 900 F.* Current work was done on a 0 to 600 F grease system.

Most of the test work was done at 600 F under 5 lb. radial and 5 lb. axial load and 50 lb. radial and 25 lb. axial load. Some tests were carried out at 650 and 700 F under light load, also.

Greases made by blending F-6-7024 Silicone Fluid and one of several polyphenyl polysiloxanes and thickening with Ammeline have given the longest 600 F, high-load bearing tests to date. Bearing tests on a series of these greases range from 150 to 220 hours. Ammeline is the only thickener that gave these long bearing tests with these blends.

With other fluids and other additives in fluids ASU and modified ASU's gave results comparable to Ammeline. While several other experimental silicone fluids and additives gave results comparable to ASU or Ammeline thickened F-6-7024 Silicone Fluid, none were any better except titanium dioxide. When titanium dioxide was used at 2 to 3% in p-phenyl azoaniline modified ASU-F-6-7024 greases 600 F high-load bearing tests of 150 hours were obtained. This compound did not show any beneficial effect in other grease systems.

All the phosphonitrilic chloride-metal halide complexes tested showed hydrolytic instability.

This report has been reviewed and is approved.

R. L. Adamczak

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

Current and future Air Force weapons systems demand lubricants capable of performing satisfactorily at higher temperatures than those currently available. At the same time, the good low-temperature performance of currently used lubricants should be matched to permit operating at the low extreme of ground and airborne ambient temperature conditions. Greases are desirable lubricants for use in the ball and roller bearings of the electrical equipment of these weapons systems. Such greases should permit small bearings, rotating at high speeds under light loads, to be run for appreciable lengths of time at the high temperature without a drastic increase in power requirement. In addition, at low temperature, bearings filled with these greases should begin rotating and run without unusual power requirements.

The ultimate objective of this contract is the development of a grease system capable of operating in lightly-loaded ball and roller bearings over a temperature range of -65 to 900 F. This ultimate objective will be reached stepwise by development of grease systems for the following temperature ranges:

0 to 600 F
-40 to 700 F
0 to 800 F
40 to 900 F
-65 to 900 F

The specific performance criteria these greases must meet are:

At low temperature - the temperature at which a greased 20 mm bearing has a starting torque of not more than 5,000 g. - cm. and a running torque of not more than 500 g. cm. is defined as the lowest usable temperature of the grease.

At high temperature - the performance characteristics of greases are established by determining the number of continuous hours they will permit satisfactory operation of a 20 mm bearing, under fifty radial and twenty-five pounds axial load, rotating at 10,000 rpm, in air. A minimum of 200 hours at 600 and 700 F and 100 hours at 800 and 900 F are required for a grease to be considered satisfactory. Relubrication is not permissible at 600 and 700 F, but it is permitted every 50 hours at 800 and 900 F.

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Greases are two-phase system, a solid or thickener phase dispersed in a liquid or fluid phase. In greased ball bearings, the lubrication is provided primarily by the fluid, while the thickener gives the system enough body to remain in and around the bearing and act both as reservoir of fluid and as a seal against contamination. It is this combination of properties that make grease lubrication preferable over the other possible methods. The advantages of grease lubrication over fluid or mist lubrication are the simplicity and weight savings gained by the elimination of pumps, reservoirs, filters and lines and the protection against contamination. Dry film lubricants will not permit operating bearings at as high speeds or as high loads as greases because of the inability of the dry films to heal or reform their films.

The low-temperature behavior of a grease is dependent primarily on the low-temperature fluidity characteristics of the fluid and secondarily on the kind and amount and degree of dispersion of the thickener.

The high-temperature behavior of a grease is dependent on the thermal and oxidative stability and volatility of both the fluid and the thickener, the lubricating ability of the fluid, the phase stability of the thickener (i.e. not undergoing a phase change in the temperature range of interest), and the ability of the grease not to undergo a detrimental change in consistency as the result of exposure to high temperature and working in the bearing.

Extensive research and development has been done to extend the useful temperature range of greases, particularly for military aircraft use. The first significant progress was made with the development of high-melting soap thickeners and oxidation inhibitors to improve the stability of petroleum oils. These developments culminated in greases such as those meeting Specification MIL-G-3545, "Lubricating Grease, High Temperature," which are capable of operating over a range of 0 to 300 F. Greases of this type usable at temperatures below 0 F are attainable, but a major improvement in low temperature performance results in a drastic decrease in high temperature performance because of the increased volatility which necessarily accompanies improved low temperature properties in petroleum oils.

The next advances came with the use of synthetic fluids, such as esters and silicones. These synthetic fluids have wider liquid ranges, higher viscosity indexes and lower volatility characteristics than do comparable petroleum oils and their use led to diester fluid greases such as those meeting Specification MIL-G-3278, "Grease, Aircraft and Instrument (For Low and High Temperature)," which have a useful temperature range of -65 to 250 F. While these greases have improved low temperature properties they are not as effective at high temperatures as the best petroleum oil greases. This high temperature limitation is caused by the solubility of soap thickeners in synthetic fluids at elevated temperatures. Silicone fluid-soap thickened greases were not used extensively because of their poorer lubricity and lack of an appreciably wider temperature range.

Some of the synthetic fluids are stable enough to use in greases at temperatures much higher than 250 F. This prompted a search for higher temperature thickeners. A number of inorganic thickeners, such as clays, silicas and carbon blacks, have been used as grease thickeners for many years and they have excellent high-temperature stability. However, it is necessary to coat the surfaces of these inorganic thickeners with polar organic compounds to make them oleophilic enough to serve as thickeners for ball bearing greases. Without the proper kind and amount of surface coating the mechanical stability, age hardening, leakage and/or water resistance of these greases will be unsatisfactory. The currently used organic coatings all desorb or oxidize at relatively low temperatures and attempts to develop coatings which had stability at high temperatures were unsuccessful. A number of non-soap, high-melting organic compounds, which could be prepared in the desired small particle-size range and whose surfaces had the proper degree of oleophilic character, were developed as high temperature thickeners.

Using high temperature thickeners of this type and selected ester fluids, greases have been made which meet Specification MIL-G-25760, "Grease, Aircraft, Ball and Roller Bearing, Wide Temperature Range," and will lubricate heavily loaded bearings from -40 to 350 F. When one of these high-temperature thickeners is dispersed in the proper silicone fluid, greases are obtained that will lubricate lightly-loaded bearings from -65 to 450 F and that meet Specification MIL-G-25013, "Grease, Ball and Roller Bearing, Extreme High Temperature." Using different silicone fluids, greases are obtained that have a useful temperature range of -100 to 400 F and meet Specification MIL-G-27343, "Grease, Ball and Roller Bearing, for Temperatures Ranging From Minus 100 to Plus 400 F."

The upper temperature limits on these greases are primarily due to the high-temperature instability of the fluids used. These greases can be used at higher temperatures than those quoted as the upper limit, but the period of satisfactory operating time is drastically reduced. For example, in lightly-loaded, high-speed ball bearings over 500 hours of satisfactory operation are obtained at 350 F with MIL-G-25760 type greases and 450 F with MIL-G-25013 type greases. At 600 F, under similar conditions, operating times are less than 4 hours with MIL-G-25760 type greases and about 50 hours with MIL-G-25013 type greases.

The upper temperature limit of the high temperature thickeners used in these greases has not been definitely established but the best of them appear to be capable of serving at temperatures above 600 F, but probably below 700 F. Thus, the development of greases for use over the range of -65 to 900 F depends on finding both fluids and thickeners that are superior to those currently used in greases. However, for the 0 to 600 F range, the problem appears to be finding a better fluid and combining it with one of the superior currently available thickeners.

Under previous years' contracts, equipment was built or obtained for running dropping points, evaporations, roll stabilities, and loaded bearing tests or greases at temperatures up to 900 F. Most bearing tests, in the previous work, were run at 600 F under modified CRC L-35 conditions, using Pope type spindles and MRC S-17 bearings.

As discussed above, the methyl-phenyl silicone fluids were the most promising fluids known for high temperature greases at the outset of this work. Several of these fluids with any one of several high-melting organic thickeners gave bearing tests of 50 to 60 hours. Further testing of silicone fluids uncovered DC-QF-6-7024 fluid which when thickened with one of several thickeners gave bearing tests in excess of 100 hours. Many other silicones were tested, but none gave any better results. With all these silicone fluids, failures were attributed to the instability of the silicone fluids.

Other fluids and thickeners considered as potentially promising for use in high temperature greases were synthesized or obtained from available sources and screened for thermal stability and low volatility. Thickeners were also evaluated for thickening ability over the temperature range.

Phenoxyphenyl ethers and polyphenyls showed promise as high temperature fluids. Bearing failures of greases made from these fluids were interpreted as due to volatility of the fluid rather than instability.

Phosphonitrilic chloride polymers and complexes with metal halides exhibit fluidity over very wide temperature ranges. However, at high temperatures they evolve hydrogen chloride upon exposure to air, are acidic, and are corrosive to metals.

High melting inorganic and organic materials have been studied as potential thickeners for high temperature greases. Surface-treated, finely-divided inorganic solid thickeners such as silica, carbon black and glass fibers have shown promise. Some triazine derivatives and aryl substituted ureas have also shown promise as high temperature grease thickeners.

A number of different bearing designs and metallurgies were tested in the previous work. The MRC S-17 bearings have given the longest bearing tests.

The work reported here is a continuation and extension of the promising developments of the previous work. Emphasis has been on synthesizing less volatile and more stable fluids, improving current and developing new thickener systems, and evaluating greases under the higher load conditions specified by the contract.

The program was divided into four phases:

1) the synthesis or procurement of fluids.
2) the synthesis or procurement of thickeners.
3) the preparation and bench testing of greases made from phase 1 and 2 materials and
4) the evaluation of the performance characteristics of these greases in high temperature bearing tests.

II. SYNTHESIS OR PROCUREMENT OF FLUIDS

Most of the work on fluids concentrated on three classes of compounds: the phosphonitrilic chlorides, silicone fluids, and polyphenyl siloxanes.

a) Phosphonitrilic Chloride Polymers

The dichloro phosphinic nitride-metal halide complexes are liquids over extremely wide temperature ranges. However, they appear to lack hydrolytic stability, are acidic, and corrosive to metals. Several approaches were taken to overcome these deficiencies.

The preparation and properties of a number of these polymers and complexes were reported in ASD Technical Report 61-2. The zinc and ferric chloride complexes appeared to be the most promising. A sample of the zinc chloride complex was prepared in these laboratories and another obtained from the project engineer, ASD. The sample prepared here was fluid at room temperature and did not distill at 400 C (750 F) at 0.4 mm. The elemental analyses of the two samples appear somewhat different:

$(PNCl_2)_n/ZnCl_2$ Sample	% Zn	% P	% Cl	% N	% Unaccounted For
K-108-134	15.0	7.4	58.2	5.0	14.4
ASD	12.6	9.0	51.1	6.0	21.3

Both samples evolved hydrochloric acid on exposure to air. The sample prepared here was sent to Materials Central, ASD at the request of the project engineer. The sample obtained from ASD had a viscosity of 523 cs at 100 F and 46.11 at 210 F and a pour point of 25 F. A vapor pressure determination of this sample, using an isotensicope, was made and the data are shown in Figure I. The thermal decomposition temperature was 333 C (631 F), although some decomposition must occur as low as 250 C (482 F) because small bubbles kept appearing and growing in the fluid above this temperature. Below 333 C, the vapor pressure fits the integrated Clausius-Clopeyron equation:

$$\log_{10} P\,(mm) = -1503/T\,(°K) + 4.77$$

$$H_{vap} = 6.88 \text{ K cal/mole}$$

The low latent heat of vaporization suggests it may occur by depolymerization.

Dichloro phosphinic nitride-Ferric chloride n-mer

This material is reported to have a pour point of -46 C and not to polymerize at 670 C, although some decomposition did occur during heating. A sample of this material was prepared by heating a mixture of 104 g. (1 M) of phosphorous pentachloride, 27 g. (0.5 M) of ammonium chloride, 16.2 g.

(0.1 M) of ferric chloride in 400 ml. of 1,2,4 trichloro benzene to 152 C for 11 hours. The mixture was filtered and solvents removed at 65 C/0.2 mm. There remained 65.5 g. of black liquid which was heated to 425 C/0.3 mm. for 2 hours. At 365 C/0.3 mm. about 10 g. of a yellow solid distilled over. After cooling, the dark liquid was filtered in a dry box and 45.5 g. recovered. Elemental analysis of the prefiltered product was: Fe - 7.69%, P - 17.03%, Cl - 65.4%, N - 5.95%, unaccounted for 3.93%. The filtered product is a mobile liquid at room temperature. One drop of this fluid in 3 ml. of water gave a solution with a pH of 1 in 10 seconds. A sample of this material solidified to a black solid when heated to 600 F for 24 hours in with an Almen pin.

A vapor pressure determination, using an isoteniscope, gave a broken curve. The data are shown in Figure II. No meaningful latent heat of vaporization could be calculated. Apparently, depolymerization began about 325 C (617 F) and then some repolymerization occurred about 355 C (671 F).

Diphenyl phosphinic nitride polymer

Diphenyl phosphinic nitride polymer was prepared by the following set of reactions:

$$\phi_2PCl + Cl_2 \longrightarrow \phi_2PCl_3$$

$$\phi_2PCl_3 + NH_3 \longrightarrow (\phi_2PN)_x + 3HCl$$

$$\Delta \qquad -\!\left(\overset{\phi}{\underset{\phi}{P}} = N\right)\!- \xrightarrow{\Delta} \text{trimer, tetramer, etc.}$$

12 g. of chlorine gas was added to 37.55 g. (0.17 M) of diphenyl chlorophosphine dissolved in 75 ml. of carbon tetra chloride and cooled to -15 C. This yellow solution was held overnight at -30 C and then warmed gradually to 25 C and stirred for two hours. The resultant diphenyl trichlorophosphorane was recrystallized from benzene to give 36 g. of product. This compound was then dissolved in liquid ammonia at -30 C and held overnight. The solution was then warmed to room temperature, which vaporized the excess ammonia. The resultant mixture was then extracted with chloroform to remove trimer and tetramer. There remained 8.6 g. of product which was heated for two hours at 270 C. Extraction with benzene yielded 5.5 g. of soluble material.

A portion of the diphenyl phosphinic nitride n-mer was placed in a test tube with an Almen pin and heated to 600 F. After only 2 hours, the white solid changed to a charred black mass.

Bis (triphenyl silanyl) phosphinic nitride

This compound was prepared by dropwise addition of 7.6 g. (0.05 M) of phosphorous oxy chloride to a mixture of 42.4 g. (0.15 M) of triphenyl silanol in 40 g. of pyridine and 50 g. of xylene. The reaction mixture was then heated to 220 F for several hours. Maceration in hot ethanol left 38 g. of product melting at 236-239 C (457-462 F).

A portion of this product was placed in a test tube with an Almen pin and heated to 600 F. After several days all of the product had volatilized and condensed around the cooler top of the tube. The condensed material was colorless and had the same melting point as the original product. This product appears to be stable, if ways could be found to lower the melting point and volatility this would be a promising class of compounds.

Perfluoroalkyl phosphonitrilates

Olin-Organics Division supplied samples of three perfluoroalkyl phosphonitrilates. These samples were placed in test tubes with Almen pins and heated to 600 F. The following results were obtained:

Fluid Formula	% Fluid Lost After 24 hrs.
$N_3P_3[OCH_2(CF_2)_4H]_6$	97.5
$N_4P_4[OCH_2(CF_2)_6H]_8$	99.0
$N_4P_4[OCH_2(CF_2)_4H]_4[OCH_2(CF_2)_8H]_4$	98.6

While the sample originally were colorless liquids, all that remained after the test were black powders.

b) Silicone Fluids

Because the best high temperature test results have been obtained with Dow Corning's F-6-7024 Silicone Fluid, a number of experimental silicone fluids from both General Electric and Dow Corning were screened and evaluated.

The first series of fluids were screened by thickening them with aryl substituted urea and testing the resultant grease under modified L-35 conditions at 600 F. Data on these experimental silicone fluids and the greases made from them were:

Fluid	Viscosity	Bearing Test - Hours
G.E. 189-114	690 c.s. at 100 F	190
189-159	1200 c.s. " "	130, 180
189-150	1220 c.s. " "	134
128-457	1430 c.s. " "	110
128-458	1640 c.s. " "	91
128-433	540 c.s. " "	45, 62
128-467	(128-433 inhibited)	70, 96
D.C. F-6-7024, lot 6	900 c.s. at 25 C	160
F-6-7024, lot 17	7180 c.s. " "	80
F-6-7039	(F-6-7024, lot 6 -inhibited	152
XF-6-7042	(F-6-7024, lot 6 - 4 times inhibitor	95, 107
XF-6-7043	(F-6-7024, lot 6-8 times inhibitor)	80

The second series of fluids were screened by thickening them with ASU and testing the resultant greases under modified L-35 conditions at 650 F. Data obtained were:

Fluid	Bearing Test - Hours
G.E. 128-534	35
128-557	28
128-558	27
128-559	70
128-565	35
114-1403	74, 62
D.C. F-6-7024, lot 6 - cut #1	3
F-6-7024, lot 6 - cut #2	26
F-6-7024, lot 6 - residue	78
F-6-7051	53
F-6-7068	57

Of the G.E. Silicone Fluids, 189-114, 189-159, 114-1403 and 128-559 (inhibited 114-1403) appear comparable but not superior to DC F-6-7024.

Additives in Silicone Fluids

The poor load carrying capacity of silicone fluids led to the consideration of a number of additives which might improve these fluids' performance in higher-load bearing tests. Knowledge of the effect of these additives on the stability of silicone fluids was desired and the shortage of bearing tester facilities dictated the use of the fluid screening test for this purpose. A vial containing 0.3 g. of sample and an Almen pin were placed in a test tube and heated to 600 F. The following results were obtained:

Fluid Composition	% Remaining 46 hrs.	64 hrs.	Observations at 64 hrs.
1. QF-6-7024, lot 6	21.8	12.3	Yellow liquid at 600 F. Solid at 70 F.
2. 1 + 4% Sulfur	-	58.4	Yellow solid at 600 F.
3. 1 + 15% Lead Oxide	44		Yellow solid at 600 F.
4. 1 + 10% Phenylpydrazine	-	16.8	Yellow liquid at 600 and 70 F.
5. 1 + 4% Phthalonitrile	-	7.4	Colorless liquid at 600 and 70 F.
6. 1 + 6% Urea		40.5	Liquid at 600 and 70 F.

Additives such as sulfur and lead oxide appear to increase the gelation tendencies of this silicone fluid, while urea appears to hold some promise for reducing the evaporation or fragmentation and evaporation tendencies.

Using a 5 g. sample of F-6-7024 Silicone Fluid instead of 0.3 g. sample gave a much slower evaporation rate:

Hours	22	44	139	226
% Remaining	97	94.8	86.8	78.3

The odor of formaldehyde was detected during this test and a positive aldehyde test was obtained using Fuchsin reagent for aldehydes. These results suggest oxygen attack on the methyl groups of the silicone fluid. Volatiles have been collected for NMR and molecular weight determinations which may shed further light on the mechanisms involved.

The lower evaporation rate of the larger sample may be due to the smaller surface to air ratio. Similar tests with nitrogen should give additional information and are planned.

c) Siloxanes

The excellent bearing test results obtained with methyl phenyl silicone fluids suggested the synthesis of a number of phenyl siloxanes.

<u>Hexaphenyl disiloxane</u>

29.45 g. (0.1 M) of triphenyl chlorosilane (mp. 98 C) was added dropwise to 27.6 g. (0.1 M) of triphenyl silanol (mp. 152 C) in one mole of pyridine. After addition was complete, the mixture was heated to 100 C for 3 hours. The solvents were then removed and the mixture extracted with ether. The extract was water washed until neutral and then dried and the solvent removed. Recrystallization from hexane ethanol gave a product with a mp. of 233-5 C. Mass spectrometry indicates this product is 99+ % pure. Elemental analysis: found - C-81.97%, H - 5.97%; calculated - C - 81.00%, H - 5.6%.

<u>Octaphenyl trisiloxane</u>

Redistilled dichloro diphenyl silane (0.5 M) in pyridine and benzene was reacted with triphenyl silanol (0.1 M). The reaction mixture was heated to 100 C for 3 hours, then cooled and stripped of solvents and extracted with ether. The ether extract was washed with water, dried, and the solvents removed. Maceration with hot ethanol gave 30 g. of a product melting at 130-140 C. Recrystallization from hexane ethanol gave 6.5 g. of a product melting at 210 C, which from its molecular weight is mostly hexaphenyl disiloxane. The mother liquor gave 8 g. of product with a mp. of 140-4 C and a MW of 694 ± 21 and 8 g. of another product with a mp. of 137-144 C and a MW of 756 ± 23.

Further work on these compositions will be done.

<u>Heptaphenyl methyl trisiloxane</u>

0.05 M of methyl phenyl dichlorosilane was added dropwise to a solution of 0.1 M triphenyl silanol in pyridine and benzene. Working the mixture up in the usual manner gave 21.3 g. of a white crystalline product with a mp. of 107-9 C.

Hexaphenyl dimethyl trisiloxane

The addition of 0.075 M of dimethyl dichlorosilane to 0.15 M of triphenyl silanol in pyridine and benzene and working up in the usual manner gave 33.5 g. of a crystalline product with a mp. of 135-7 C.

p-Phenoxyphenyl diphenyl silanol

p-Phenoxyphenyl magnesium bromide was prepared by reacting p-bromophenyl phenyl ether with magnesium in tetrahydrofuran.

This solution was then added dropwise to a solution of diphenyl dichloro silane in toluene. The reaction mixture was heated from 70-80 C for 10 hours. After cooling, the mixture was poured into ice water and stirred vigorously. Extraction with ether-chloro benzene, water-washing and drying, and subsequent removal of the solvents at 60 C/0.2 mm. left a product which contained only 0.29% chlorine.

Bis (phenoxyphenyl) hexaphenyl trisiloxane

p-Phenoxy phenyl diphenyl silanol was reacted with dichloro diphenyl silane in pyridine-benzene solution. After stripping of solvents at 50 C/0.3 mm., the product was dissolved in ether and washed until neutral. Removal of the solvents left a brown, viscous liquid that is not tacky.

Octaphenyl cyclotetra siloxane

40 g. of diphenyl dihydroxy silane was dissolved in 300 ml. of ethanol containing 6 drops of 5% sodium hydroxide and the mixture heated at 172 F for 4 hours. The reaction mixture was then cooled and the product filtered off and washed first with 50 ml. of 95% ethanol and then with 100 ml. of 90% ethanol. After drying overnight the product weighed 34 g. and had a mp. of 202-4 C. Mass spectrometry indicates this product is 99+ % pure.

Tris (triphenyl siloxyl) phenyl silane

The addition of 0.02 M of phenyl trichloro silane in benzene to 0.06 M of triphenyl silanol in pyridine and working up in the usual manner gave 21 g. of a crude product. Recrystallization from a mixture of 60 ml. chlorobenzene, 50 ml. acetone, and 100 ml. benzene gave 4 g. of product with a mp. of 227-232 C.

Tris (undecaphenyl penta siloxyl) phenyl silane

Tris (ω chloro octaphenphenyl tetra siloxyl) phenyl silane was prepared by beating 0.02 M octaphenyl cyclotetra siloxane and 0.007 M of phenyl trichloro silane in 60 g. of phenyl ether to 255 C for 6 hours. This compound was added to 0.04 M of triphenyl silanol in 79 g. of pyridine and this mixture heated to 200 F for 4 hours. Solvents were removed and the product taken up in ether and washed. Removal of the ether left 79.7 g. of crude product. Washing with hexane to remove the phenyl ether left 16 g. of product with a mp. of 138-162 C. Recrystallization from 50 ml. benzene and 100 ml. hexane left 13 g. of product with a mp of 141-166 C.

Polyphenyl polysiloxane

α -ω 0.01 M octyl cyclotetra siloxane was reacted with 0.005 M diphenyl dichloro silane by heating to 240 C for several hours. 8.28 g. of triphenyl silanol in 79 g. pyridine was added and this mixture heated and the solvents then removed. The crude product was dissolved in chlorobenzene and water washed. Stripping the solvent and macerating the product in ethanol left 6.7 g. of white crystals with a mp. of 175-188 C. Elemental analysis - C - 72-82%, H-5.44%, Cl 0.03%.

Penta deca phenyl methyl hepta siloxane

dichloro nonaphenyl methyl penta siloxane was prepared by heating 0.02 M octylphenyl cyclotetra siloxane and 0.02 M phenyl methyl dichloro silane in 40 g. of phenyl ether at 225-250 C for 7 hours. This mixture was then added to 0.08 M of triphenyl silanol in 79 g. pyridine and 79 g. benzene and the resultant mixture heated at 205 F for 6 hours. Removal of the solvent left 102 g. of crude product which was taken up in 400 ml. of ether and water washed. Removal of the solvent left 72.5 g. of product which was hexane washed to remove the phenyl ether. This left 21.5 g. of crystalline product. Recrystallization from 50 ml. benzene and 75 ml. hexane left 19 g. of product with a mp. of 110-114 C.

Dimerization of 5 Phenyl Ether

223 g. (0.5 M) of bis (m-phenoxy phenoxy) benzene was iodinated with 0.5 M iodine monochloride in acetic acid by refluxing 15 hrs. at 120 C. The reaction mixture was stirred into water and extracted with ether. The ether extract was washed free of acetic acid, dried and the solvent removed to give 270.5 g. of product. The iodine content of the product was 19.5%, and by titration 96.1% of the iodine was aryl iodide.

0.05 M of this product in 30 ml. of tetrahydrofuran was slowly added to 0.1 M of magnesium in 100 ml. of tetrahydrofuran. At 150 F after 2 hours reaction began and continued for 4 hours. 13 g. of anhydrous cupric chloride was added and heating continued 4 more hours. The reaction mixture was poured into ice water and the product then taken up in ether. The ether solution was washed with ammonium hydroxide, potassium iodide and water. Solvents were removed at 65 C/0.2 mm. and left 24.4 g. of a brown viscous liquid. Copper and potassium were absent and the iodine content was 1.95 %.

Bis (triphenyl silyl) glutarate

This compound was prepared by reacting 0.1 M of triphenyl silanol in 150 g. of pyridine and 0.05 M of perfluoro glutaroyl chloride in 20 ml. of benzene at 106 C for several hours. Solvents were removed under reduced pressure and the residue macerated with absolute ethanol. The 16 g. of product had a mp of 230-4 C and an analysis of C-825, H - 6.2 while the calculated values are C-7-.0, H - 4.28.

Siloxane - Hydroquinone n-mers.

0.1 M of hydroquinone and 0.12 M of diphenyl dichloro silane were reacted in 79 g of pyridine by heating to 193 F for one hour. Then 0.08 M of triphenyl silanol were added and the mixture heated to 223 F for 3 hours. Working up the product in the usual way gave 53 g. of product.

Evaporation Testing of Siloxanes

A number of these materials were screened by placing 0.3 g. sample and an Almen pin in a vial and placing the vial in a test tube and heating the tube to 600 F for various periods of time. The data obtained were:

Compound	% Remaining after 64 hours	Observations at 64 hours
F-6-7024	12.3	Liquid at 600 F solid at 70 F
Polyphenyl polysiloxane	52.4	Colorless liquid at 600 F
Octylphenyl trisiloxane	57.6	Yellow liquid
Five phenyl ether	2.6	Solid
Dimerized five phenyl ether	71.4[1]	Black liquid
Bis(triphenyl silyl) glutarate	2.1	Solid
Hydroquinone siloxane polymer	59.4	Solid tar
Bis(phenoxy phenyl) hexaphenyl trisiloxane	65.2[1]	Black liquid

[1] After 24 hours.

While the octylphenyl trisiloxane and the polyphenyl polysiloxane appear to have about the same rate of evaporation after 64 hours and even longer times, the trisiloxane gelled at 296 hours with about 20% remaining but the polysiloxane was still liquid at 434 hours with about 9% remaining.

The solidifying of the hydroquinone polymer was disappointing. While most of the glutarate evaporated, the volatile portion was identical with the starting material, suggesting a higher molecular weight product might have promise.

III. SYNTHESIS OR PROCUREMENT OF THICKENERS

The search for improved high temperature thickeners continued and study was concentrated on:

1) Modified ASU thickeners
2) High melting organic polymers
3) Surface modified inorganic thickeners

1) Modified ASU Thickeners

Loaded bearing test results with both ASU and Ammeline thickened F-6-7024 Silicone Fluid varied from about 50 to 100 hours at 600 F. While most of the variation appears to be due to differences in bearings and testers, even under the best conditions results are still a long way from the desired 200 hours. Further improvements were sought in fluids, thickeners and additives to reach the desired life.

A number of different isocyanates and amines were used to make aryl substituted urea thickeners, but none were any better than those currently used (TODI and p-toluidine and p-chloroaniline) and most were not as good. However, the use of p-aminobenzoic acid (p-ABA) as the amine component offered the opportunity to further react with the benzoic acid groups and possibly effect further improvements. p-Bromoaniline was more effective than either tribromo or trichloroaniline and p-phenyl azoaniline gave excellent results. The following results illustrate this point:

	Grease Thickener	Acid No.	Penetration	Bearing Test, Hrs.
1.	5% p-ABA + 5% TODI	440	26.0	48
2.	1 + 4% p-bromoaniline	19	300-310	85
3.	1 + 6% p-bromoaniline	8	fluid	-
4.	1 + 6% p-bromoaniline	5.5	fluid	-
5.	1 + 13% tribromoaniline	2.8	fluid	-
6.	1 + 8% trichloroaniline	3.7	335	60
7.	1 + 4% p-azoaniline	15.3	284	121
8.	1 + 6% p-azoaniline	13.6	292	67-136(6 tests)
9.	1 + 8% p-azoaniline	14.5	304	115
10.	1 + 10% p-azoaniline	11.4	288	79

All of the greases in this table had been heat-treated for 4 hours at 450 F. The neutralization numbers were then determined and bearing performance tests run. No difference in performance was found for greases containing from 0.6 to 1.1 moles of p-phenyl azoaniline per mole of p-amino-benzoic acid, however, grease containing excess p-Azo A resulted in shorter bearing tests.

Longer heat treating times tended to lower the neutralization number and bearing performance life of these greases. For example, a grease containing 6% p-Azo A heat-treated for 96 hours, had an acid number of 0.67 and a bearing life of 31 hours. Addition of 1% chlorendic anhydride to this grease raised the acid number to 14.5 and the bearing performance to 72 hours. It is not clear whether the moderate acidity imparted by this additive or its chlorine content was responsible for the improved bearing performance, however, 1% of this additive in the base grease (5% p-amino benzoic acid +5% TODI) gave no improvement.

Replacement of TODI with 1,5 naphthalene diisocyanate or diphenyl methane 4,4'diisocyanate resulted in greases that gave inferior bearing performance test results.

A number of other variations in processing these greases were also tried without obtaining significant improvement in bearing performance tests. Prepolymerizing the TODI used for the base grease gave no improvement. Elimination of solvents in base grease preparation, gave poorer results. Isopropanol was the solvent which gave greases with the best bearing performances. Making heavier base greases with higher thickener contents gave no improvement.

The greases tended to be thixotropic and a number of additives were investigated as auxiliary thickeners. Among the additives tested were: benzoguanamine, cymel 300 (hexamethoxy methyl melamine), azo dyes, carbon black, chlorendic anhydride, benzyl alcohol, Baymal (colloidal alumina), ironphthalocyanine, iodobenzoic acid, titanium dioxide and various combinations of some of these. While many of these auxiliary thickeners in the 2 to 5% range improved the consistency of the greases, none improved bearing performance, except titanium dioxide. Using 2 or more percent titanium dioxide increased the penetration to harder than 200 and the bearing performance to about 150 hours at 600 F. However, when 10% titanium dioxide was added to an Ammeline thickened grease with excellent bearing performance, the resultant grease gave a very poor bearing test.

2) High Melting Organic Polymers

The reaction product of methylene bis 4-phenyl isocyanate and trimellitic anhydride is a "no-melt" polymer which ought to be a good grease thickener if it could be produced in or made into small particle sizes. However, all attempts to get this polymer into the proper particle size range failed.

The polybenzimidoles made by reacting a phenylene diamine and esters of aromatic acids reported by C. S. Marvel and co-workers, also could not be obtained in or micronized into the proper particle size range for grease thickeners. A coordination complex formed by reacting benzimidazole and cobaltous nitrate had a melting point above 480 C (896 F). However, this material would not form a stable grease with F-6-7024 fluid even at 50% concentration.

An azo dye formed by the reaction of 5-chloro salicylic acid and p-phenyl azoaniline had a melting point above 480 C(896 F) and gave good grease when 38% was milled into F-6-7024 Silicone Fluid. However, unloaded bearing performance at 600 F of this grease and similar ones with calcium hydroxide and with calcium hydroxide and Alon C added only lasted from 8 to 32 hours.

Permansa Red 10363, 1(2-chloro-4-nitrophenyl azo)-2-naphthol, a high melting dye from Sherwin-Williams gave a No. 2 grade grease when 40% was milled into F-6-7024 fluid. However, these greases became fluid after 4 hours heat treating at 450 F.

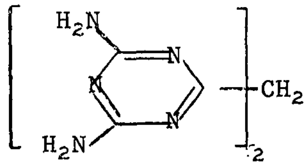
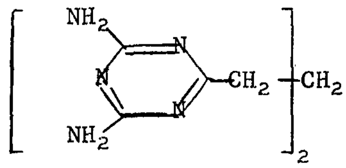
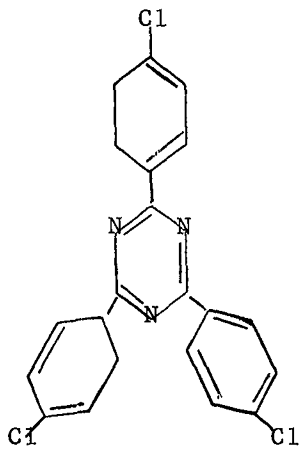
Calcium fluoride thickened F-6-7024 Silicone Fluid at concentrations of 40 to 60%. However, all these greases became fluid after 4 hours heat treating at 450 F.

A sample of purified asbestos, C-9-12M-500 ES, was received from ASD. 4.5% in F-6-7024 Silicone Fluid was adequate to make a No. 2 grade grease, but the bearing performance of this grease was only fair.

Our testing of inhibited silicone fluids has never shown any advantage for the inhibited fluid over the uninhibited. It was suggested that this may be due to the inhibitor coating out on the thickener. A sample of dry, preformed ASU thickener was sent to Dow-Corning and coated with inhibitor and returned to us. This sample, DC(X2-8-3062), was used to thicken F-6-7024 Silicone Fluid and the bearing performance test was so poor that nothing further was done with this thickener.

TL 126 Teflon from Liquid Nitrogen Processing and DC 410 gum from Dow Corning both gave greases that had poor bearing performance tests.

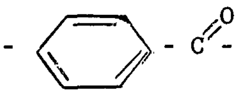
Because Ammeline is such a satisfactory thickener, several s-triazine compounds were prepared and evaluated.

Compound	Structural Formula	m.p.
Methylene Bis-2,4 diamino-s-triazine		410 C(770 F)
1,3 Bis- [2,4 diamino-6-S-triazinyl] propane		340C(642 F)
Tri-2,4,6-P-chlorophenyl-S-triazine		330-335 C(626-635 F)

The first two compounds were effective thickeners for F-6-7024 fluid, while the third was not. However, greases made with the first two compounds were mechanically unstable after they had been heated to 600 F.

3) Surface Modified Inorganic Thickeners

Alizarin-diamine and its calcium complex were deposited over several finely divided inorganic solids but greases made from these thickeners failed to show promise. The solids coated were Cabosil HS-5, Baragel 24 and Battery Fluff Carbon. The bearing performance of greases made from these thickeners were all disappointingly low.

The surface of specially dried Cabosil HS-5 was treated with several reagents and the products are still undergoing evaluation. The surface was physically coated with methyl red, $(CH_3)_2$ N -  - OH, and the resultant solid milled into F-6-7024 Silicone Fluid. At 9.25% a grease of 241 penetration was obtained. In static testing this grease was unchanged after 100 hours at 600 F, but it gave a relatively short bearing performance test.

The silica surface was chemically reacted with diphenyl dichloro silane, toluene 2,4 diisocyanate, benzene phosphonic dichloride, and titanium tetrachloride and phenol. Only the product from diphenyl dichlorosilane has been partially evaluated. Dispersing this reaction product in F-6-7024 Silicone Fluid resulted in a thixatropic grease, but the addition of pentane 1,5 diol resulted in a grease that was heat and mechanically stable. After 10 days at 600 F in a static test this grease appears unchanged.

IV. PREPARATION AND BENCH TESTING OF EXPERIMENTAL GREASES

Experimental thickeners were evaluated by milling them into F-6-7024 Silicone Fluid and testing the resultant grease.

Experimental fluids were evaluated by thickening with ASU or Ammeline thickener and testing the resultant grease. Whenever feasible, the experimental fluid was used as the only fluid component; however, because of the high melting points of the polyphenyl siloxanes, they were generally blended with F-6-7024 fluid.

Experimental additives, to improve oxidation stability or extreme pressure properties, were added to base greases by warming the grease to 150-200 F adding the desired amount of additive and milling the resultant mixture.

The development of meaningful high temperature grease bench tests remains a major problem. A number of bench tests have been investigated. None correlate well with bearing test results, but a few are able to pick out greases that will not run an appreciable time in the bearing performance test. Because of the time, expense and large back-log of greases to be bearing performance tested, efforts continue to be put on finding more meaningful bench tests.

After preparation of the greases they are heat-treated at 450 F for 4 hours and remilled. Those greases that become very soft are eliminated from further consideration. Those greases that become harder, have more fluid milled into them until they are back to the desired 300 penetration range and they are then heat-treated for an additional 4 hours. Further hardening after this additional heat treating, is considered cause for elimination of the material.

Those greases that survive heat-treating were then evaluated in high temperature bearing performance tests. Screening was done in the modified L-35 test, under 5 pounds radial and 5 pounds thrust load. The more promising greases were then tested in bearing tests employing 50 pounds radial and 25 pounds thrust load.

Roll stability tests at 600 F were run on several greases. Greases thickened with regular ASU and p-Azo Aniline modified p-amino benzoic acid ASU thickeners gave soft, non-uniform greases after 4 hours in this test. Based on these roll stability tests these greases would be considered unsatisfactory for use at 600 F, but these greases performed quite well in bearing performance tests at 600 F.

Among the factors that can cause failure of a grease in the bearing performance test are high leakage, high evaporation and drastic changes in consistency. Bench tests designed to determine these properties of greases were run on a number of greases. One version of this test was a "whirled disk," in which a 20-mesh stainless steel screen, 31 mm. in diameter was coated with a 2.5 x 24 mm. disk of grease and then the screen was impaled on a glass rod. The test specimen was then placed in a test tube housed in a 600 F aluminum block. After the test grease reached 600 F, the disk was rotated at 7500 rpm for one minute. Glass thickened grease and a Teflon TFE-Baymal thickened grease showed very poor adhesion in this test, although both of these greases gave very respectable bearing performance test results. A grease, thickened with Phosphatherm RN and Teflon FEP 120, adhered to the disk extremely well, but could not even be started in the bearing performance test because of its extremely high torque requirements.

In another version of this test the stainless steel screen is heated to 600 F in either an oven or an aluminum block and the greases evaporation and leakage tendencies studied. When the screens were heated in an oven, they were placed on watch glasses and when they were heated in a block they were placed in test tubes. The following data were obtained:

Oven Heated.

Sample	% Evaporated in 1 Day	Comments
F-6-7024, lot 6	30	-
Dry ASU	69, 61	Residue chlorine content nil.
LG-0677, B-417	42	No leakage.
ASU-Arochlor 1254	9	No leakage.
10% Glass Fiber-F-6-7024, lot 6	55	Remaining grease still soft.
ASU-5 ϕ ether (MLO-59-692)	53	Residue was brittle flakes.
ASU-Silphenylene	60	Residue rubbery.
Ammeline-F-6-7024, lot 6	31	Residue soft, greaselike.
Block Heated		
LG-0677, B-565	20 10[1]	Residue black and crusted - chlorine content nil - no leakage.
LG-0677 + 5% PAN	25	Residue black and hard. PAN on glass wool.
LG-0677 + 5% PAN + 1% excise amine reactants	17[2]	" " " "
LG-0677 + 5% PAN + 1% MHS	26	" " " "
LG-0677 + 5% TFE Item 6	20	Residue black and crusted with some yellowish particles.
TL-126 Teflon-F-6-7024, lot 6 + 3% PAN	33	High oil leakage residue hard.
40% Permansa Red + 60% F-7024, lot 6	-	All grease had leaked out.

[1] 2 days - 15%, 5 days - 40%.
[2] 2 days - 29%, 5 days - 44%.

Although these tests were run for screening purposes only, it was hoped that some conclution might be drawn with bearing performance results. Disappointingly, the ASU thickened greases containing excess amine appeared poorer in this test than did the regular ASU greases, which is just the opposite of the results in the bearing performance test. Repeatability was not very good, but trends were consistent. The disappearance of chlorine from the residue of ASU greases in this test substantiates results obtained on residues of greases from bearing performance tests. While this test may be useful in screening out very poor greases, it can not be used to detect which of several good greases is the best.

ASD expressed interest in obtaining low temperature data on LG-0677 (ASU thickened F-6-7024 Silicone Fluid). Previous reports indicated this grease is extremely brittle at -65 F. Torque and apparent viscosity tests were run which indicated that a temperature of about +15 F is the lowest temperature at which this grease can satisfy the contract specified requirements.

Apparent viscosity determinations were made on this grease at 20 and 32 F at a shear rate of 20 reciprocal seconds. The data obtained were:

Temperature	Apparent Viscosity Poises at 20^{-1} sec.
32	5,600
20	14,000

Torque determinations were made by the ASTM D 1478-57 T procedure. These data showed:

Temperature	Torque, g - cm.	
	Starting	After 10 min. Running
15	10,300	8,100
10	24,500	17,700

V. EVALUATION OF GREASES IN HIGH TEMPERATURE BEARING TESTS

A total of 347 high temperature bearing tests were run with 204 bearings at 10,000 rpm during this reporting period. Sixty-five tests were run at 600 F under modified L-35 conditions. The composition of the greases and results of their tests are shown in Table I. Sixty-two tests were run at 650 or 700 F under modified L-35 conditions. The results of these tests are shown in Table II. Two hundred and twenty tests were run at 600 F with 50 pounds radial and 25 pounds axial load. The results of these tests are shown in Table III.

Because of the large back-log of greases for bearing testing, the lightly loaded testers were used as a screening test to select greases to run under higher loads. As work progressed and better performing greases were developed, these screening runs began to run over 200 hours. As a means of accelerating these screening tests the temperature was raised to 650 F in many tests and to 700 F in a few tests. It was realized that no correlation has been established between running times at 600 F and 650 F, but for ASU greases it was found that 650 F tests lasted approximately 0.3 to 0.4 as long as 600 F tests. A typical 600 F test on an ASU thickened F-6-7024 Silicone Fluid containing phenyl alpha naphthylamine is about 200 hours and at 650 F the average of 17 tests on this type of grease was 65 hours. There are real dangers in accelerating tests by raising the temperature. Changes in composition or consistency can occur or the stability temperature of one or more component may be exceeded which would make any comparison between results at two temperatures very misleading. The correlation found for one grease system certainly may not hold for a different system. However, because the ultimate goal is for systems suitable for much higher temperatures and the pressing need for a screening test, data at higher temperatures were considered worthwhile to obtain.

A method was developed for measuring belt tension, as a part of the program to eliminate as many bearing performance test variables as possible. All of the high-temperature, high speed bearing testers use the weight of the motor, partly counterbalanced, to apply tension to the drive belt. Tension was measured and adjusted to fifteen pounds with a spring scale and a dial indicator. The dial indicator was mounted against the motor pulley to detect movement; the spring scale was connected to the center of the motor pulley to pull in the direction of the test spindle. Gradually increasing tension was applied to the scale. The scale reading was taken just as the motor and pulley started to move. This method was adopted as routine for use with all bearing testers.

About mid-year, a series of low bearing performance test results caused concern. Careful rechecking of conditions, procedures and tester dimensions failed to suggest any cause for these low results. A group of ninety-eight MRC S-17 bearings and two New Departure X-14047 bearings were analyzed by Bearing Inspection, Inc. on their Model BA-20-2 Electronic Bearing Analyzer. The bearings were also measured for bore, O.D., width and eccentricity. All of the bearings met ABEC Class 3 inspections. Both of the X-14047 bearings were smooth and quiet on the dynamic analyzer. Of the 98 MRC S-17 bearings, 67 were rated good on relative noise and vibration, 19 were marginal and 14 were rated rejected. Most of the rejects were due to inner race faults.

As these bearings were used for performance testing, an attempt was made to correlate bearing performance with dynamic analysis rating. However, no detectable pattern could be found. Long and short tests were obtained with each class of rated bearings.

An experimental bearing from the Marlin-Rockwell Corporation with a Mo_2S cage, failed after 18 hours with an ASU-F-6-7024 grease. Cage breakage was found after the test.

A special packing and start-up technique was developed for use with the dry, fibrous greases resulting when high percentages of polyphenyl polysiloxanes were tested. Quite short tests were obtained when several of these materials were tested in the usual way. However, much longer tests were obtained when the grease was packed firmly into one side of the bearing; the bearing turned over and four drops of F-6-7024 fluid was distributed over the bearing and this side firmly packed with grease. The test unit was then assembled and heated to 500 F, and the bearing rotated 12 times by hand before the high speed test was started.

VI. DISCUSSION

The development of longer-lived, 600 F greases continues to depend primarily on finding more satisfactory fluids. While better bearings, thickeners and additives are also important and desirable the most dramatic increases in bearing performance result when more stable, less volatile fluids are used. The phenyl siloxanes fluid systems offer real promise of meeting the requirements for a 600 F. grease.

At the beginning of this reporting period ASU or Ammeline thickened F-6-7024 Silicone Fluid were the most satisfactory greases that had been developed. At 600 F under light load the best of these greases run satisfactorily for about 200 hours. Increasing to 50 pounds radial and 25 pounds axial load reduces the performance life of these greases to about 80-120 hours.

A number of other thickener systems were tested and none were found to give better results than ASU or Ammeline. Most of them were not nearly as good. However, the search for better and higher temperature thickeners must continue because both ASU and Ammeline will not be stable enough for use at 700 F. Also, there is always the hope that a really good thickener will give better performance at 600 F also.

The best bearing test results were obtained with MRC S-17 bearings. Twenty-five tests were run under higher load conditions with New Departure X-14047 bearings. In general, there is some indication that the X-14047 bearings may be less sensitive to E.P. additives in some formulations than the MRC S-17 bearings. However, only one test was greater than 90 hours and six were in the 75 to 85-hour range. On the average, this is not as long test lives as obtained with the S-17 bearings. Two tests were run with Barden BJH-204 bearings. Both tests were shorter than those obtained on the same grease on S-17 or X-14047 bearings. These results on the different bearings is in line with results obtained in previous years at lower loads.

A large number of additives were tested in varying percentages in a number of grease formulations but only a few were found to be effective.

In previous work it was found that methyl hydrogen silicone added to ASU-F-6-7024 grease gave the best bearing performance tests obtainable. Further testing this reporting period gave variable results, due possibly to variations from lot to lot of MHS. It was most effective in extending test times under high load conditions at 600 F. The optimum amount appeared to be between 1 and 2%:

Batch of MHS	% in Grease	Bearing Test Hours
1043, lot 301	1	106
"	2	95
"	4	4
1043, lot 302	1	58, 56

The effect of MHS as a load carrying additive is quite limited and falls far short of the goal of 200 hours.

An excess of amine in ASU-F-6-7024 greases show a definite improvement in loaded and unloaded bearing tests. The amines may be either excess amine reactants or phenyl alpha naphtha amine in the range of 1%. For example, 1% PANA raises these greases' high-load test performance from about fifty hours to about ninety hours.

Unfortunately combining both MHS and PANA in the same grease, results in shorter tests than those obtained on the grease without any additives.

Titanium dioxide used as an auxiliary thickener to overcome the thixotropy of p-phenyl Azo aniline modified ASU grease gave greases with excellent bearing performance tests when enough TiO_2 was used to result in a very hard grease, i.e. penetration less than 200. Tests of 150 hours were obtained with greases of this type containing 3% TiO_2. Lesser or greater amounts of TiO_2 resulted in shorter tests and use of a number of other auxiliary thickeners in this grease system showed no improvement in bearing test performance. Also, use of TiO_2 with ASU or Ammeline resulted in no improvement in bearing performance.

A number of different silicone fluids and variations of F-6-7024 Silicone Fluid were tested without uncovering a significantly better fluid. A number of experimental silicone fluids from General Electric and Dow Corning were made into ASU greases and tested. Most gave shorter bearing tests than F-6-7024, but several from G.E. appeared about equal in performance. These fluids were numbered 189-114, 118-159, 189-150, 114-1403 and 128-559. Because they did not offer significantly better performance they were not studied too extensively. Various viscosity grades, distillation fractions and inhibited samples of F-6-7024 were made into ASU greases and bearing tested. Higher viscosity grades and the lower boiling fractions of F-6-7024 gave greases with poorer bearing performances than regular F-6-7024. Use of small amounts of inhibitor in F-6-7024 resulted in ASU grease that gave bearing tests comparable to uninhibited F-6-7024, while the use of large amounts of inhibitor gave greases with significantly poorer bearing tests. These poorer results were not due to the inhibitor interfering with the thickener reaction, because similar poor results were obtained when the preformed thickener was treated with the inhibitor and then this modified thickener used to make a grease with F-6-7024 Silicone Fluid.

The evaporation and thermal screening tests seem to indicate definite limitations to the best of the present grease systems for temperatures of 600 F and higher. Primarily this is a problem of the fluid rather than the thickener. The two classes of fluids which have been used with the most success, the polyphenyl ethers and high phenyl content methyl phenyl silicones, both show high evaporation losses at these elevated temperatures. There are also indications of oxidation in these tests with F-6-7024. The odor of formaldehyde and positive Fuchsin tests for aldehydes were noted and the fluid tended to darken. Presumably, this oxidation is on the methyl groups.

The polyphenyl polysiloxanes showed low volatility and good thermal and oxidation stability in these screening tests, which suggests they are a promising class of materials for high temperature greases. Unfortunately, they have rather high melting points and as a result they were tested in greases blended with F-6-7024 Silicone Fluid. Even when using 60% F-6-7024 and 40% polyphenyl polysiloxane the resultant greases were so hard it was necessary to preheat the bearing to 500 F to obtain a good bearing test.

ASU, p-phenyl azoaniline modified ASU, and silica thickened blends of F-6-7024 and polyphenyl polysiloxane fluids gave bearing test results comparable to the best results obtained with straight F-6-7024 fluid. However, Ammeline thickened blends gave significantly better bearing tests, several of these tests running in excess of 200 hours at 600 F under high load conditions. The following bearing test results were obtained at 600 F, under 50 pounds radial load and 25 pounds axial load, at 10,000 rpm, and using a full pack of grease made by thickening the fluid with 35% Ammeline:

Fluid	Grease Penetration	Test No.	Bearing Test, Hours
F-6-7024	296	744	120
		724	113
40% $\phi_6\ Si_2O$ 60% F-6-7024	100	781	133
		739	148
40% $\phi_8\ Si_3\ O_2$ 60% F-6-7024	-	725	216
40% $\phi_{16}\ Si_7O_6$ 60% F-6-7024	-	732	171
40% $\phi_8\ Si_4O_4$ 60% F-6-7024	192	754	179
		777	164
		779	169
40% $\phi_7\ CH_3Si_3O_2$ 60% F-6-7024	170	774	202
		747	168
40% $\phi_{15}\ CH_3Si_7O_6$ 60% F-6-7024		751	164

While these results are sketchy they indicate a very promising advance in bearing test life. Obviously, other compounds and concentrations need to be explored to optimize these results.

Even more promising results were obtained when small percentage of bis(triphenyl silyl) perfluoro glutarate were added to Ammeline thickened F-6-7024 Silicone Fluid greases. The following results were obtained at 600 F, under 50 pounds radial and 25 pounds axial load, at 10,000 rpm and using a full pack of grease:

% Additive in Ammeline Thickened F-6-7024	Grease Penetration	Test No.	Bearing Test Hours
0	296	{774, 724}	120, 113
1.5	300	775	150
3.0	311	763	183
		770	208
5.0	300	778	165
10	311	771	133

These results are comparable to those obtained with the polyphenyl polysiloxanes, but at much lower percentages. This lower percentage enables the grease to maintain its low temperature properties. Bearing tests on these greases were started in the usual way at room temperature.

The chemical analysis of the bis(triphenyl silyl) perfluoro glutarate does not correspond to the calculated composition. This raises the question as to just what compound this is. Preliminary results indicate it may be hexaphenyl disiloxane with some small amount of fluorine impurity. This composition needs to be investigated further to follow up this promising lead.

VII. CONCLUSION

Greases made by blending F-6-7024 Silicone Fluid and one of several polyphenyl polysiloxanes and thickening with Ammeline have given the longest 600 F, high-load bearing tests to date. Bearing test results on a series of these greases range from 150 to 220 hours. Further exploitation of this approach offers hope of meeting the desired 200 hours at 600 F.

A number of experimental silicone fluids and variations of F-6-7024 Silicone Fluid were tested and none were better than F-6-7024 Silicone Fluid itself.

Ammeline thickener is the only thickener that gave the exceptionally high bearing tests with blends of F-6-7024 Silicone Fluid and polyphenyl polysiloxanes. With other additives in F-6-7024 Silicone Fluid ASU and p-phenyl azoaniline modified ASU were equally as good as Ammeline. For use at 700 F new thickeners will have to be developed.

Among the many additives investigated, only titanium dioxide, used as an auxiliary thickener in p-phenyl azoaniline modified ASU-F-6-7024 grease gave significantly better performance.

All of the phosphonitrilic chloride-metal halide complexes studies were hydrolytically unstable.

VIII. SUGGESTIONS FOR FUTURE WORK

The promising initial results obtained with the polyphenyl polysiloxanes should be followed up and optimized. This should enable meeting the requirements for a 600 F grease.

To develop greases for 700 F, new thickeners will have to be developed. Triazine systems and silicas appear the most promising candidates. New or improved fluids will also be required. More stable phosphonitrilic chlorides and the polyphenyl polysiloxanes appear attractive approaches.

TABLE I

HIGH TEMPERATURE BEARING TESTS
600 F, 10,000 RPM, MRC-S-17 Bearings
5 lb. radial, 5 lb. axial loads, 3.2 g grease

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Residue, g	Hours	Test Variations and Remarks
424	p-ABA-TODI-p-Phenylene diamine	9.5	F-6-7024 Lot 6	292	0.6	114	
425	Oxidized Carbon Black	14	F-6-7024 Lot 6	300	1.0	80	
426	JM Glass Fiber (100)	4	F-6-7024 Lot 6	-	0.2	21	Grease ran out
427	ASU	14	5-Phenyl Ether MLO-59-692	312	1.0	10	
428	p-ABA-TODI-Melamine	15	F-6-7024 Lot 6	337	-	45	
430	p-ABA-TODI-o-Tolidine	11	F-6-7024 Lot 6	280	0.6	36	
431	ASU	16	F-6-7024-6 + 1% MHS	328	0.8	137	
432	JM Glass Fiber (Code 100)	20	F-6-7024 Lot 6	130	0.2	47	
433	15% JM-100 5% Estersil GT	20	F-6-7024 Lot 6	-	0.6	14	
434	ASU	13	F-6-7024 Lot 15	310	0.9	140	
435	Ammeline	33	F-6-7024 Lot 6	-	0.8	103	
437	Laminar 5	40	F-6-7024	296	2.1	6	
439	ASU (Preformed)	18	F-6-7024 Lot 6	329	-	143	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
440	ASU	13	F-6-7024 Lot 6	308	0.6	136	Full packed brg. and end cap
443	p-ABA-TODI-Sulfonamide	10	F-6-7024 Lot 6	280	0.0	12	
444	p-ABA-TODI-1-Amino Anthraquinone	10	F-6-7024 Lot 6	321	0.4	106	
449	ASU (Preformed)	19	F-6-7024 Lot 6	329	0.9	159	
450	JM-100-ASU	12	F-6-7024 Lot 6	-			Heater burned out
452	ASU	19	F-6-7024 Lot 17	325	0.7	80	7000 cs. vis. oil
453	Teflon (Item 6)	30	F-6-7024 Lot 6	286	-	53	Black debris
455	1-Naphthyl Isocyanate-p-Phenylene diamine	13	F-6-7024 Lot 6	304	0.4	106	
456	5% JM-100 + 7% ASU	12	F-6-7024 Lot 6	-	0.6	21	
457	ASU (Preformed)	19	G.E.-128-467	333	2.4	9	Cold end failure?
459	ASU (Preformed)	17	DC-XF-7043	317	0.9	80	
460	ASU (Preformed)	19	F-6-7024 Lot 6	329	0.6	152	
462	ASU (Preformed)	19	G.E.-128-433	329	1.3	45	
464	ASU (B-514)	13	F-6-7024 Lot 6	312	1.1	69	
465	ASU (Preformed)	19	XF-6-7042	317	0.8	95	
468	ASU (Preformed)	19	G.E.-128-467	333	0.9	96	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
469	ASU (Preformed)	19	XF-6-7042	317	1.1	107	
471	ASU (Preformed)	19	G.E.-128-458	321	1.1	91	
472	ASU (Preformed)	19	G.E.-128-433	329	1.0	62	
475	ASU (Preformed)	19	G.E.-128-467	333	0.9	70	
476	ASU (Preformed)	19	G.E.-189-150	321	0.7	134	
478	ASU (Preformed)	19	G.E.-128-457	313	1.2	111	
479	ASU (Preformed)	19	G.E.-189-159	333	1.2	82+	
482	JM-100 Glass Fiber	10	F-6-7024 Lot 17	272	0.5	6	
483	ASU (Preformed)	19	G.E.-189-159	333	0.8	178	
484	ASU (Preformed)	19	G.E.-189-159	333	0.7	127	Same as test No. 483 but different spindle used
485	ASU (Preformed)	19	G.E.-189-114	331	1.0	189	
491	ASU + 1/2% TODI	19	F-6-7024 (6)	-	0.8	82	
493	MLC-60-263 Ammeline	35		-	4.2	150	Full packed bearing end cap
495	ASU + 2% TODI	19	F-6-7024 (6)	325	1.2	98	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Residue, g	Hours	Test Variations and Remarks
503	ASU + 1/2% of each amine	19	F-6-7024 (6)	320	1.0	128	
507	(ASU from end cap of test No. 140 at 136 hrs. at 600°)			330	0.6	132	
512	ASU	13	F-6-7024 + 1/2% of each of amine reactants	310	0.4	259	
518	p-ABA-TODI-p-Br Aniline	13.5	F-6-7024	304	0.6	89	
523	ASU	13	F-6-7024 + 5% PAN	-	0.5	254	
526	ASU	13	F-6-7024 + 1% of amine reactants	310±	-	67	
527	ASU	13	F-6-7024 + 1% of each amine reactant	310±	0.6	224	
531	p-ABA-TODI-p-Ø azoaniline	13.5	F-6-7024 Lot 6	280	2.7	189	Full packed bearing end cap
542	p-ABA-TODI (1:1)	17	F-6-7024 Lot 6	308	1.6	192	Full pack
552	(No melt foam) MDI tetrachlorophthalic acid	40	F-6-7024, 6-Ø ether	300	0.3	34	Very gritty residue
554	ASU	13	F-6-7024 + 1% amine reactants	-	0.2	110±	
560	Ca Benzoate	50	F-6-7024	296	0.1	5	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
566	Azo dye, Alon C $Ca(OH)_2$ & Cl-Salicylic acid	38	F-6-7024	317	-	8	Full pack
572	p-ABA	5	F-6-7024	345	0.5	51	Full pack
598	DC 410 Gum	75	F-6-7024 + 5% PAN	230	2.9	2	Full pack
600	p-ABA TODI 1,5 diamino naphthalene	4 4 3	F-6-7024	305	1.1	175	Full pack
602	p-ABA MDI	5 10	F-6-7024	345	0.7	27	Full pack
728	ASU	13	DC-XF-6-7052	284	0.4	128	0.9 vibration rating
733	ASU	20	Hercoflux 600 + 1/2% PANA	296	0.3	1	0.9 vibration rating
735	ASU	13	DC-XF-6-7068	278	0.6	113	

TABLE II

HIGH TEMPERATURE BEARING TESTS
650 and 700 F, 10,000 RPM, MRC-S-17 Bearings
5 lb. radial, 5 lb. axial loads, 3.2 g grease

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
536	ASU	13	F-6-7024 + 5% PANA	-	1.2	25	700 F, full packed bearing
538	p-Aminobenzoic acid TODI p-Phenylazoaniline	5 5 4	F-6-7024 + 5% PANA	-	-	36	700 F, full packed bearing
539	ASU	13	F-6-7024 + 1% X's amine reactants	-	-	102	650 F
544	ASU	13	F-6-7024 + 5% PANA	-	0.5	109	650 F
551	TODI + p-chloroaniline	13	F-6-7024 + 1% X's p-Cl aniline	304	0.1	90	650 F
555	ASU	13	F-6-7024 + 2% Di-β-naphthyl p-phenylene-diamine	-	0.1	56	650 F
559	1,5-diisocyanatonaphthalene + p-phenylazoaniline	13	F-6-7024 + 5% PANA	-	0.2	53	650 F
562	ASU	13	F-6-7024 + 1% X's amine reactants	-	0.2	73	650 F
563	ASU	13	F-6-7024 + 1% X's amine reactants + 5% PANA	-	0.8	83	650 F

TABLE II

HIGH TEMPERATURE BEARING TESTS
650 and 700 F, 10,000 RPM, MRC-S-17 Bearings
5 lb. radial, 5 lb. axial loads, 3.2 g grease

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
536	ASU	13	F-6-7024 + 5% PANA	-	1.2	25	700 F, full packed bearing
538	p-Aminobenzoic acid TODI p-Phenylazoaniline	5 5 4	F-6-7024 + 5% PANA	-	-	36	700 F, full packed bearing
539	ASU	13	F-6-7024 + 1% X's amine reactants	-	-	102	650 F
544	ASU	13	F-6-7024 + 5% PANA	-	0.5	109	650 F
551	TODI + p-chloroaniline	13	F-6-7024 + 1% X's p-Cl aniline	304	0.1	90	650 F
555	ASU	13	F-6-7024 + 2% Di-β-naphthyl p-phenylene-diamine	-	0.1	56	650 F
559	1,5-diisocyanatonaphthalene + p-phenylazoaniline	13	F-6-7024 + 5% PANA	-	0.2	53	650 F
562	ASU	13	F-6-7024 + 1% X's amine reactants	-	0.2	73	650 F
563	ASU	13	F-6-7024 + 1% X's amine reactants + 5% PANA	-	0.8	83	650 F

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
567	ASU (B-565)	13	F-6-7024 + 1.5% PAN	330	0.4	105	650 F
569	p-P Azo A	8.4	F-6-7024	296	1.1	7	650 F (?) Instrument battery dead
573	ASU (B-565)	13	F-6-7024 + 5% Diphenylamine	330	0.6	62	650 F
575	ASU (B-417)	13	F-6-7024 + 1.0% Amine reactants	310	0.1	77	650 F
576	Coated ASU (DC-XZ-803062)	20	F-6-7024	300	3.3	3	Full pack, 650 F, (0.3 g crystals on cold end of spindle)
578	ASU (B-417)	13	F-6-7024 + 1% Amine reactants	310	0.6	65	650 F (new grease shields, tight fit)
579	ASU (B-565)	13	F-6-7024 + 2% PAN	330	0.3	82	650 F
583	ASU (B-565)	13	F-6-7024 + 5% PAN	330	0.4	87	650 F
584	p-ABA	5 5	F-6-7024	296	1.1	43	650 F (Ran 16 hrs. more after re-starting)
585	1,5-Naphthalene di-isocyanate	5	F-6-7024 + 5% PAN	330	1.1	92	650 F and full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
587	ASU (B-565)	13	F-6-7024 + 5% PAN	330	0.9	42	650 F
591	ASU (B-565)	13	F-6-7024 + 5% PAN + 1% Cerous Stearate	330	0.6	49	650 F
592	ASU (B-565)	13	F-6-7024 + 5% PAN	330	-	77	650 F (New housing)
593	ASU (B-622)	15	F-6-7024 (10) + 5% PAN	321	0.3	52	650 F
595	ASU (B-622)	15	F-6-7024 (10 + 5% PAN	321	0.6	59	650 F
605	ASU (B-565)	13	F-6-7024 + 2% PAN + 2% DSPD	308	0.4	49	650 F
607	ASU (B-628)	15	F-6-7024 + 5% PAN	276	-	58	650 F
609	ASU	13	GE 128-565	296	1.0	35	650 F (Ag flaking from cage)
610	ASU	12	GE 128-534	304	0.5	35	650 F
616	ASU	13	GE 128-557	292	0.5	28	650 F
618	ASU	13	DC-s Lot 6 Residue of 7024 + 5% PAN	304	0.2	78	650 F
623	ASU	12	GE 128-558	315	0.0	27	650 F

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
624	ASU	13	DC's Cut #2 of F-6-7024 (6)	311	0.8	26	650 F
627	ASU	13	GE 128-559	304	0.4	70	650 F
629	ASU	13	GE 114-1403	300	0.8	74	650 F
632	ASU	13	DC's Cut #1, F-6-7024 (6)	296	1.1	3	650 F
634	Purified Asbestos C9-12M-500 ES	4.7	F-6-7024	296	1.2	26	650 F
636	ASU (B-565)	13	F-6-7024 + 1% NH_4HCO_3	320	-	59	650 F and full pack
637	ASU (B-565)	13	F-6-7024 + 2% PAN + 2% N,N'-disalicylidene O-phenylene diamine	320	0.5	29	650 F
641	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	292	-	35	650 F, Full pack + end cap and special shield
645	ASU	12	GE 128-565 + 5% PAN	325	0.8	36	650 F, full pack
646	ASU	10	F-6-7024 + 2% quinoline	290	-	48	650 F, full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
652	ASU (B-688)	13	F-6-7024 + 5% PAN	340	0.6	72	650 F, full pack
655	ASU (B-688)	13	F-6-7024 + 1% Primene 81 R	321	-	-	650 F, full pack
660	ASU (B-688)	13	F-6-7024 + 2% PAN + 5% Perylene	340	1.0±	68	650 F, full pack
662	ASU (B-688)	13	F-6-7024 + 0.1% Primene 81 R	324	-	57	650 F
666	ASU (B-688)	13	F-6-7024 + 1.5% PAN	328	0.5	27	650 F
671	ASU (B-490)	23	DC-F-6-7051	300	0.3	53	650 F
672	ASU (B-688)		F-6-7024 + 5% Urea	308	0.4	62	650 F
677	ASU (B-688)	13	F-6-7024 + 1% Phenothiazine	320	0.9	95	650 F
680	ASU (B-688)	13	F-6-7024 + 1% Phenothiazine	320	0.5	9	650 F
681	ASU	13	F-6-7024 + 5% PAN	340	0.3	65	650 F
683	ASU	12	Hexa deca phenyl hepta siloxane + 5% PAN	Too Hard	-	2	650 F (Preheated)
686	None		Hexa deca ∅ hepta-siloxane-F-6-7024	400+	0.1	2	650 F

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
688	ASU	13	Hexa deca Ø hepta-siloxane F-6-7024 (40:60)	Too Hard	0.4	37	650 F (Preheated)
689	ASU	13	15-Ø-1 Methyl hepta-siloxane-F-6-7024 (50:50)	Too Hard	0.4	32	650 F (Preheated)
692	None		F-6-7024	Fluid	0.0	4	650 F
694	ASU	13	Hepta Ø methyl tri-siloxane F-6-7024 (40:60) + 5% PAN	300	0.1	18	650 F
698	ASU (B-688)	13	F-6-7024 + 5% PAN	340	-	43	650 F
700	ASU (B-688)	13	F-6-7024 + 5% PAN	340	0.6	49	650 F
703	ASU (B-688)	13	F-6-7024 + 5% PAN	340	0.4	63	650 F
704	(ASU in commercial silicone oil grease)			300	0.6	127	550 F & L-35 cycling (To check assembly vs. 135 hrs. of a previous test)
709	ASU	13	Tris undeca Ø penta-siloxyl Ø silane F-6-7024 (50:50) + 5% PAN	Too Hard	1.4	44	650 F
712	ASU	13	F-6-7024 + 5% PAN	340	0.6	53	650 F (Bearing E 672 of 0.7 Vibration)

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
716	ASU (B-417)	13	F-6-7024 + 1% ASU amine reactants	320	0.7	32	650 F

TABLE III

HIGH TEMPERATURE BEARING TESTS
600 F, 10,000 RPM, MRC-S-17 Bearings
50 lb. radial, 25 lb. axial loads, 3.2 g grease

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
429	ASU	13	F-6-7024 + 1% MHS	308	1.7	38	ND X-14047 bearing
436	ASU	13	F-6-7024 + 1% Zn dibutyl dithiocarbamate	308	0.7	60	ND X-14047 bearing
438	ASU	13	Percolated F-6-7024	317	1.3	64	
441	ASU	13	F-6-7024	308	-	83	Full pack plus end cap ND X-14047 bearing
442	ASU	13	F-6-7024 Lot 17	308	0.8	6	
445	ASU	13	DC-XF-6-7043	308	0.6	40	(Eight times normal inhib.)
446	ASU	17	DC XF-6-7039	329	1.5	74	
447	p-ABA-TODI plus Ammeline & Ammelide	15	F-6-7024	347	0.6	69	
448	Naphthyl isocyanate plus phenylene diamine	13	F-6-7024	304	1.1	23±	Belt broke
451	ASU	13	F-6-7024 + 2% MHS 1040	308	-	61	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
454	ASU	13	F-6-7024 + 2% MHS 1043	308	0.7	95	
458	ASU	13	F-6-7024 + 1/2% p-iodo benzoic acid + 1/2% DAAQ	308	1.1	94	ND X-14047 bearing
461	ASU	13	F-6-7024 + 2% old MHS	308	0.1	56	Barden BJH 204 bearing
463	ASU	13	F-6-7024 + 1/2% IBA + 1/2% DAAQ	308	0.5	41	Barden BJH
466	ASU	13	F-6-7024 + 1/2% IBA + 1% DAAQ	308	0.9	75	ND X-14047 bearing
467	ASU	13	F-6-7024 + 5% Tri Ø phosphite	312	1.0	13	ND X-14047 bearing
470	p-ABA-TODI plus 5% DAAQ + 1% IBA	15	F-6-7024	317	1.0	76	ND X-14047 bearing
473	p-ABA-TODI-DAAQ	14	F-6-7024	329	0.7	39	NX X-14047 bearing
474	p-ABA-TODI + 3% IBA + Ammeline	17	F-6-7024	290	0.8	66	ND X-14047 bearing
477	p-ABA-TODI-Ammeline	15	F-6-7024	296	0.7	68	ND X-14047 bearing
480	p-ABA-TODI-1-AAQ	16	F-6-7024	317	0.5	27	ND X-14047 bearing

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
481	p-ABA-TODI-Ammeline	15	F-6-7024 3% Pb $(Et)_4$	-	1.0	47	ND X-14047 bearing
486	p-ABA-TODI-DAAQ + 1% IBA	15	F-6-7024	329	0.8	68	
487	p-ABA-TODI-Ammeline	12	F-6-7024 + 5% Pb $(OAc)_2$	321	0.7	10	
488	p-ABA-TODI-Ammeline	12	F-6-7024	321	0.2	19	ND X-14047
489	p-ABA-TODI-Ammeline	12	F-6-7024 + 5% MoS_2	317	1.0	5	ND X-14047
490	p-ABA-TODI-Ammeline + 1% IBA	13	F-6-7024	317	-	54	ND X-14047
492	ASU	13	F-6-7024	308	0.5	83	ND X-14047
494	ASU	13	G.E.-114-1403 + 2% MHS	300	0.9	54	ND X-14047 Full pack
496	ASU	13	F-6-7024	308	0.8	81	ND X-14047 bearing
497	p-ABA-TODI-DAAQ	15	F-6-7024 + 1% benzyl disulfide + 2% Arochlor 2565	330	0.7	32	ND X-14047 Full pack
498	Ca Alizerin-Diamine on Carbon Black	20	F-6-7024	330	0.7	16	ND X-14047 Full pack
499	ASU	13	F-6-7024	308	1.1	51	
500	ASU	19	F-6-7024 + 2% MHS 1043	-	0.9	66	ND X-14047

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
501	p-ABA-TODI-DAAQ (5) (5) (5) + 2% IBA	17	F-6-7024	-	0.3	48	
502	ASU	13	F-6-7024 + 2% MHS 1043	308	9.7	79	ND X-14047
504	p-ABA-TODI-DAAQ (5) (5) (6) + 2% IBA	18	F-6-7024	286	0.6	79	
505	ASU	13	F-6-7024 + 4% MHS 1043	308	0.4	4	
506	ASU	13	F-6-7024 + 5% TFE Teflon 6	300	1.1	99	
508	p-ABA-TODI-DAAQ + 1% IBA	17	F-6-7024	312	0.9	75	
509	p-ABA-TODI-Br Aniline	14	F-6-7024	304	0.7	89	
510	p-ABA-TODI-Br Aniline + 2% IBA + 1% DAAQ	14	F-6-7024	308	0.5	47	
511	ASU	13	F-6-7024 + 1% MHS 1043	-	0.4	106	
513	Same as 509 + 5% Teflon	19	F-6-7024	-	0.3	85	
514	Ca Alizarin on Carbon	21	F-6-7024	240	1.6	17	
515	ASU	13	F-6-7024 + 5% Laminar $CaCO_3$	-	0.5	90	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
516	p-ABA-TODI + 1.5 B_2O	12.5	F-6-7024	325	0.3	22	
517	p-ABA-TODI- Br Aniline	13.5	F-6-7024	-	0.3	48	
519	p-ABA-TODI + Tri-Cl-Aniline	17	F-6-7024	333	1.9	60	
520	ASU	13	F-6-7024 + 8% Fe powder	-	1.1	34	
521	p-ABA-TODI + 4% p-phenylazoaniline	14	F-6-7024	-	0.4	86	
522	ASU	13	F-6-7024 + 1% Amine reactants 5% TFE Teflon	-	0.3	77	
524	Same as 521 + 1% DAAQ & Tetrachlorphthalic anhydride	16	F-6-7024	-	0.2	90	
525	ASU	13	F-6-7024 + 1% MHS 1043 and 5% TFE Teflon 6	-	0.4	98	(One restart after 69 hrs.)
528	p-ABA-TODI-Azoaniline	14	MLO-60-231 Phenyl Ether + 18% F-6-7024	333	0.6	14	
529	ASU	13	F-6-7024 + 5% PAN	-	1.0	112	
530	p-ABA-TODI-Azoaniline + 2% Tribrom aniline	16	F-6-7024 + 5% PAN	276	0.3	68	(After restart at 22 hrs.)
532	ASU	13	F-6-7024 + 5% DPPD	-	0.9	58	

Test No.	Thickeners	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
533	ASU	13	F-6-7024 + 5% PAN & 1% MHS	-	-	3	Full pack and end cap
534	ASU	13	F-6-7024 + 5% PAN	-	1.5	90	Full pack and end cap
535	ASU	13	F-6-7024 + 2% Ag° powder	-	1.5	17	Full pack and end cap
537	p-ABA-TODI-PDA (p-Phenylene diamine)	17	F-6-7024 + 5% PAN & 1% Tri Br Aniline	-	0.6	67	Full pack
540	ASI	13	G.E.-189-114 + 5% PAN	-	0.8	43	
541	6 Ø Ether, MDI and tetra-Cl-phthalic anhydride	38	F-6-7024	300	0.3	14	
543	ASU	13	F-6-7024 + 5% Quinquephenyl	-	1.2	78	
545	Alizarin on Barogel	14	F-6-7024	-	0.2	8	Full pack
546	p-ABA-TODI + Diamino-naphthalene	12	F-6-7024	309	0.5	1	Full pack
547	ASU	13	F-6-7024 + 1% lead di-methyl dithiocarbamate	-	1.0	60	
548	ASU	13	F-6-7024 (Lot 10) + 1% MHS	-	-	58	

Test No.	Thickeners	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
549	ASU	13	F-6-7024 + 1% Azo-aniline & 1% chlor-endic anhydride	-	-	80	
550	ASU	13	F-6-7024 + 3% Dilauryl selenide	-	-	49	
553	ASU	13	F-6-7024 + 5% 6 Ø Ether 1% MHS 1043	-	1.3	92	
556	p-ABA-TODI-Ø Azoaniline	14	F-6-7024 + 5% PAN	-	0.1	42	Full pack
557	p-ABA-TODI-Ø Azoaniline (5) (5) (6)	16	F-6-7024	300	0.5	136	Full pack
558	ASU	13	F-6-7024 + 1% PAN +1% MHS 1043	-	0.2	51	
561	ASU	13	F-6-7024 + 1% MHS 1043 (Lot 302)	-	1.7	56	
564							
565							
570	ASU (B-565)	13	F-6-7024 + 5% 6 Ø ether + 2% TODI	330	1.6	47	Full pack
571	p-ABA TODI	5 5	F-6-7024	284	1.4	115	Full pack - Ag flaking off cage
574	p-ABA TODI	5 12	F-6-7024	308	0.9	87	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
577	p-ABA TODI p-P Azo A	5 5 10	F-6-7024	247	0.1	79	Full pack (Grease not milled)
580	ASU (B-565)	13	F-6-7024 + 4% p-P Azo A	330	0.3	54	Full pack
581	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	312	1.0	87	Full pack (p-P Azo A added in absence of alcohol)
582	TODI p-P Azo A	5 13	F-6-7024	300	1.1	99	Full pack
586	p-ABA TODI p-P Azo A	5 8	F-6-7024 + 5% Laminar $CaCO_3$	280	1.0	45	Full pack
588	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	300	0.3	22	Full pack (ND X-14047 bearing)
589	ASU (B-417)	13	F-6-7024	310	1.6	93	
590	ASU (B-565)	13	F-6-7024 + 5% PAN	330	1.0	106	Full pack
594	p-ABA TODI	5 5	F-6-7024	308	0.4	3	Full pack (Grease ran out to housing)
596	p-ABA TODI	5 5	F-6-7024	308	0.9	96	Full pack (Ag flaking on cage)
597	ASU (B-565)	13	F-6-7024 + 5% PAN + 1% Tocopherol	330	0.9	46	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
599	TL-126 Teflon	44	F-6-7024 + 3% PAN	321	1.4	45	
601	ASU (B-565) TL-126 Teflon	12 10	F-6-7024 + 5% PAN	317	1.0	39	
603	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	308	0.9	105	Full pack and end cap
604	ASU (B-565)	13	F-6-7024 + 2% $CaCO_3$ + 2% Amine reactants	330	1.4	26	
606	Ammeline	33	F-6-7024 + 3% PAN	317	1.4	99	Full pack and end cap
608	MRC's E g 520 Grease			$>$350	0.5	18	Full pack with end cap
611	p-ABA TODI p-P Azo A Benzoguanamine	5 5 8 8	F-6-7024	304	0.5	105	Full pack
612	ASU (B-565)	13	F-6-7024 + 5% PAN	320	-	18	Full pack MRC's MoS_2 Retainer Brg. (Cage broken) Full pack less than 3 g.
613	ASU (B-628)	12	F-6-7024 + 1% DC-1107 MHS	311	1.4	74	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
614	ASU (B-628)	12	F-6-7024 + 5% PAN + 5% Di Ø Selenide	320	0.7	39	
615	p-ABA TODI	5 5	F-6-7024	300	1.0	79	Full pack (Grease aged 91 hrs. at 450 F)
617	ASU	13	GE 114-1403 + 5% PAN	300	1.5	29	
619	p-ABA TODI p-P Azo A Azo Dye	5 5 4 5	F-6-7024	284	1.1	(63+)	Full pack (Axial load cable broke)
620	p-ABA TODI p-Br Aniline	4 4 4	F-6-7024	300	1.5	59	Full pack
621	ASU (B-565)	13	F-6-7024	320	1.3	22+	Cabe broke
622	ASU	13	40% Octa Ø Trisiloxane 60% F-6-7024	-	1.0	106	Full pack
625	p-ABA TODI	5 5	F-6-7024 + 2.5% Benzoguanamine, 1%	317	0.7	(115)	Full pack 350 F (Instr. failure)
626	ASU (B-628)	12	F-6-7024 + 1% DC 1107 MHS	311	1.5	86	Full pack
628	(Same as Test #625 above)			317	0.7	1+	Full pack plus end cap. Ran out
630	ASU (B-628)	12	GE 114-1403 + 5% PAN	304	0.9	71	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
631	p-ABA TODI p-P Azo A Azo Dye	5 5 4 3	F-6-7024	284	1.0	57+	Full pack (Thermostat failed)
633	ASU (B-565)	13	F-6-7024 + 5% PAN	321	1.5	26+	(Cold end failure)
635	(Same as Test No. 631)			284	-	1+	Full pack
638	(Same as for Test No. 631)			284	0.5	87	Full pack
639	ASU (B-565)	13	F-6-7024 + 5% PAN	320	1.3	(39+)	(Difficulty with new thermostat)
640	ASU (B-565)		F-6-7024 + 5% PAN	320	0.6	48	Full pack
642	(Same as for Test No. 641)		F-6-7024 + 1% Hex Acid (Chlorendic Anhydride)	284	0.8	72	Full pack
643	p-ABA TODI	8 8	F-6-7024	220	1.1	77	Full pack
644	ASU (B-565)	13	F-6-7024 + 1.5% PAN	320	1.0	85	Full pack
647	ASU	14	Tris-tri Ø silyl phosphate-F-6-7024 (40:60) + 5% PAN	Too Hard	1.3	1.3	Full pack (Preheated)
648	p-ABA TODI	8 8	F-6-7024	220	1.1	68	Full pack
649	ASU	13	Poly Ø polysiloxane F-6-7024 (40:60)	Too Hard	2.3	3	Full pack (Preheated)

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
650	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	284	0.8	31	Full pack (Grease aged 96 hrs. at 450 F)
651	ASU (B-565)	13	F-6-7024 + 3% TL-126 Teflon + 5% MLO-60-231 Phenyl ether + 5% PAN	320	0.8	63	Full pack
653	TODI p-Aminophenol	14 5	F-6-7024	337	1.1	39	Full pack
654	TODI 4-0-tolylazo-0-toluidine	6	F-6-7024	311	0.8	45	
656	TODI-Prepolymerized p-ABA p-P Azo A	8 8 4	F-6-7024	284	1.0	91	Full pack
657	ASU (B-688)	13	F-6-7024 + 5% PAN	340	0.5	80	Full pack
658	ABA TODI p-P Azo A	5 5 6	F-6-7024	292	1.0	79	Full pack (Incorrectly assembled)
659	p-ABA TODI p-P Azo A	5 5 10	F-6-7024	268	1.0	55	Full pack
661	ASU (B-565)	13	GE 114-1403 + 0.5% MHS-1107 Lot 843	300	1.0	79	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
663	p-ABA TODI p-P Azo A Cymel 300	5 5 6	F-6-7024	280	0.5	54	Full pack
664	ASU (B-688)	13	F-6-7024 + 0.5% MHS 1107 + 0.5% PAN	324	0.5	12	
665	ASU (B-628)	10	F-6-7024 + 10% F-1265 (1000 cs) Fluoro-silicone + 1% Toluidine	301	0.6	67	
667	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	317	0.9	66	Full pack
668	ASU (B-417)	13	F-6-7024	321	1.3	65+	(Test interrupted)
669	ASU	11	Octa Ø Tri siloxane-F-6-7024	300	1.4	73	Full pack
670	p-ABA	5	F-6-7024 + 1% Chlorendic anhydride	333	1.0	55	Full pack
673	ASU (B-628)		F-6-8024 + 5% PAN	276	0.3	83	Brg. rotated in 0.5% MHS 1107 in 7024 at 600 F - Drained at 450 F
674	p-ABA TODI p-P Azo A 1-Phenylazo-2,4-diaminobenzene mono HCl	6.6 6.6 4.0	F-6-7024	300	0.9	48	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
675	p-ABA TODI p-P Azo A Benzoguanamine	5 5 6 8	F-6-7024	284	0.3	45	Full pack plus end cap. Yellow crystals at cold end
676	ASU (B-688)	13	F-6-7024 + 5% PAN	340	0.8	55	ND X-14047 Brg. (Secondary seal with Teflon-7024 Grease)
678	ASU (B-688)	13	GE-114-1403	300	1.4	62	
679	p-ABA TODI p-P Azo A	5 5 8	F-6-7024	284	0.6	85	
682	ASU	13	F-6-7024 + 1% PAN	320	1.1	68	ND X-14047 Brg.
684	Cobalt benzimidazole complex + ethylene diamine	35	F-6-7024	264	0.3	2	
685	ASU	13	DC-XF-6-7068 + 5% PAN	298	1.0	57	
687	p-ABA TODI Br-Benzoic Acid DAAQ	5 5 7 9					
690	p-ABA TODI p-P Azo A	5 5 8	F-6-7024	284	0.3	53	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
691	ASU (B-688)	13	F-6-7024 + 1% PAN	320	1.7	88	
693	TODI p-Amino benzanilide	4 6.5	F-6-7024	280	0.1	22	Full pack
695	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	292	2.0	1+	Full pack (Mech. failure. Chlorinated solvent used in prep.)
696	ASU (B-565)	13	F-6-7024	320	0.9	32	(Bearing pretreated with MoS_2 Dri Film)
697	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	292	1.2	40	Full pack
699	ASU (B-628)	15	F-6-7024 + 0.5% MHS-1107	268	1.2	59	ND X-14047 Brg.
701	ASU	13	F-6-7024	320	1.0	51	
702	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	292	0.4	77	Full pack plus end cap
705	ASU (B-688)	13	F-6-7024 + 0.5% MHS-1107 + 0.5 % TPSA	320	1.2	57	
706	p-ABA TODI p-P Azo A	5 5 6	F-6-7024 + 1% chlorendic anhyd. + 0.3% Benzyl Alc.	304	0.6	16	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
707	Phosphotherm RN FEP 120-Washed	20 30	F-6-7024	260	3.0	0	(Unable to start. Torque too high)
708	p-ABA TODI p-P Azo A	5 5 6	F-6-7024 + 1% Chlor-endic Anhyd.	304	0.5	75	Full pack
710	Benzidine p-Cl Ø isocyanate p-Biphenylisocyanate	4.9 4.1 5.2	F-6-7024 + 1% PAN	310	0.7	37	
711	p-ABA TODI p-P Azo A Baymal	5 5 10 5	F-6-7024	288	1.0	34	Full pack
713	ASU	13	F-6-7024 + 2% 1-Amino anthracene	330	0.8	46	(Bearing E 607 of 0.7 Vibration)
714	ASU	13	F-6-7024 + 1% PAN	320	1.0	41	(Bearing run previously from 83 hrs. in #673. Cleaned and repacked)
715	p-ABA TODI p-P Azo A	5 5 6	F-6-7024	317	0.6	107	Full pack (Bearing E 659 0.4 Vibration)
717	ASU (B-688)	13	F-6-7024 + 2% PAN + 1% β-Naphthol	330	0.8	37	
718	ASU		F-6-7024 + 5% PAN	330	1.0	76	

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
719	ASU	13	F-6-7024 + 1% Tri Ø amine + 1% MHS-1107	320	1.3	73	Yellow crystals at cold end
720	p-ABA TODI p-P Azo A Battery Fluff Carbon	5 5 6 2.5	F-6-7024	306	0.6	79	Bearing of 0.4 vibration
721	ASU	12	Octaphenyl trisiloxane F-6-7024 (40:60) + 5% PAN	Too Hard	0.6	96	(Preheated)
723	p-aminobenzoic acid TODI p-phenylazoaniline Barogel	5 5 6 1	F-6-7024	296	0.5	85	Full pack, 0.8 vibration
724	Ammeline	35	F-6-7024, Lot 10		2.0	113	Full pack, 1.3 vibration
725	Ammeline	33	60% F-6-7024 40% Octaphenyl tri-siloxane	Too Hard at R.T.	-	216	Full pack
726	p-aminobenzoic acid TODI p-phenylazoaniline	5 5 8	F-6-7024	284	0.6	102	Full pack, 1.0 vibration
727	Ammeline	33	F-6-7024	296	0.5	161	Full pack, bearing gold plated
729	Ammeline	35	F-6-7024, Lot 10	-	-	121	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
730	p-aminobenzoic acid TODI p-phenylazoaniline	5 5 6	F-6-7024 + 1% chlorendic anhydride	304	0.4	78	Full pack, 1.0 vibration, 203 pen. grease outside of 304 pen. grease
731	Ammeline	33	60% F-6-7024 40% octylphenyltrisiloxane	-	1.5	127	Grease unmilled, full packed, 0.3 vibration
732	Ammeline	35	60% F-6-7024 40% $\emptyset_6$ Si_7O_6		1.8	171	Full packed, bearing preheated before start
734	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	5 5 6	F-6-7024	304	0.6	80	Full pack, 1.0 vibration
736	ASU	13	F-6-7024 + 2% encapsulated MHS	320	1.2	78	
737	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	10 10 3 2	F-6-7024	<200	0.2	136	Full pack, 1.0 vibration
738	Ammeline	35	60% F-6-7024 40% $\emptyset_8$ Si_3O_2	-	>2	28	Full pack, preheated, 1.2 vibration
739	Ammeline	35	60% F-6-7024 40% $\emptyset_6$ Si_2O	-	1.0	148	Full pack, preheated

Test No.	Thickener	%	[illegible] and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
740	p-aminobenzoic acid TODI p-phenylazoaniline DADI	5 5 6 6	F-6-7024	200	0.4	93	Full pack, 1.0 vibration
741	Ammeline	33	60% F-6-7024 40% Me $\emptyset_7$ Si_3O_2	170	-	(4)	Mechanical failure
742	Ammeline	33	F-6-7024	296	1.3	(33)	Mechanical failure
743	Ammeline	33	60% F-6-7024 40% Me $\emptyset_7$ Si_3O_2	170	-	202	Full pack, preheated, 1.0 vibration
744	Ammeline	33	F-6-7024	296	-	120	Full pack
745	p-aminobenzoic acid TODI p-phenylazoaniline	10 10 3	F-6-7024	154	0.6	95	Full pack, 1.0 vibration
746	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	10 10 3 2	F-6-7024	160	-	81	Full pack, gold plated bearing, 0.4 vibration
747	Ammeline	33	60% F-6-7024 40% Me $\emptyset_7$ Si_3O_2	170	1.2	173	Full pack, 1.0 vibration
748	Ammeline	35	$(\emptyset\text{-}O\text{-}\emptyset)_2$ $\emptyset_6$ Si_3O_2	-	0.2	70	Full pack, preheated, 1.0 vibration
749	p-aminobenzoic acid TODI p-phenylazoaniline	5 10 4	F-6-7024	160	0.3	108	Full pack, 1.0 vibration

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
750	Ammeline	36	60% F-6-7024 40% $(\emptyset\text{-O-}\emptyset)_2\ \emptyset_6\ Si_3O_2$	-	0.8	132	Full pack, preheated, 1.0 vibration
751	Ammeline	35	60% F-6-7024 40% $\emptyset_{16}\ MeSi_7O_6$	-	1.5	164	Full pack, preheated
752	Ammeline TiO_2	30 10	F-6-7024	302	1.7	31	End cavity filled, 1.0 vibration
753	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	10 10 3	F-6-7024	162	0.7	157	Full pack
755	Cab O Sil	4	60% F-6-7024 40% $\emptyset_8Si_4O_4$	284	0.1	30	Full pack, preheated
756	Benzidine p-chlorophenylisocyanate p-phenylisocyanate	4.9 4.1 5.2	F-6-7024 + 1% MHS 1107	300	1.5	103	Full pack, 1.2 vibration
757	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	8 8 2.4 2.4	75% F-6-7024 25% $\emptyset_8\ Si_4O_4$	-	0.4	89	Full pack, preheated
758	Ammeline	33	60% F-6-7024 40%	166	0.9	219	Full pack, preheated, 1.1 vibration
759	ASU	13	F-6-7024 + 1% PANA + 1% 5,10,10	327	0.6	44	Full pack, 1.1 vibration

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
760	Ammeline	35	60% F-6-7024 40% $\emptyset_2$ $Si(OH)_2$	160	2.1	37	Full pack, preheated
761	Ammeline	35	60% F-6-7024 40% $\emptyset_3$ SiOH	-	2.0	120	Full pack, preheated, 0.8 vibration
762	p-aminobenzoic acid TODI p-phenylazoaniline p-phenylphenylisocyanate	5 5 3 2	F-6-7024	284	0.3	120	Full pack, 0.8 vibration
763	Ammeline	35	F-6-7024 + 3% $(\emptyset_3\ SiOC)_2\ (CF_2)_3$ (C with =O)	311	1.2	183	Full pack
764	Ammeline	35	60% F-6-7024 40% $\emptyset_8$ Si_4O_4	223	0.9	95	Full pack - not preheated
765	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	10 10 3 5	F-6-7024	146	0.5	114	Full pack
766	Ammeline	35	60% F-6-7024 20% $\emptyset_3$ SiOH 20% $\emptyset_2$ $Si(OH)_2$	243	0.7	170	Full pack, preheated
767	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	9 9 3 3	75% F-6-7024 25% $\emptyset_8$ Si_4O_4	154	0.3	127	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
768	Ammeline	35	60% F-6-7024 40% $(\emptyset_3 SiO)_3 PO$	113	1.6	117	Full pack, preheated
769	Ray Dean's Rilube #63	33	F-6-7024 + 0.5% PANA	300	-	67	Full pack
770	Ammeline	35	F-6-7024 + 3% $(\emptyset_3 SiO\underset{O}{\overset{\|}{C}})_2(CF_2)_3$	311	1.1	208	Full pack
771	Ammeline	33	F-6-7024 + 10% $(\emptyset_3 SiO\underset{O}{\overset{\|}{C}})_2(CF_2)_3$	311	1.0	133	Full pack
772	TODI + H_2O + alcohol	8.5	F-6-7024 + 1.5% PANA	288	1.7	102	Full pack
773	Ammeline	34	60% F-6-7024 40% $\emptyset_8 Si_4O_4$ + 3% $(\emptyset_3 SiO\underset{O}{\overset{\|}{C}})_2 + (CF_2)_3$	210	1.2	145	Full pack, preheated
774	ASU	13	F-6-7024 + 1% PANA + 3% $(\emptyset_3 SiO\underset{O}{\overset{\|}{C}})_2(CF_2)_3$	304	0.9	109	Full pack
775	Ammeline	35	F-6-7024 + 1.5% $(\emptyset_3 SiO\underset{O}{\overset{\|}{C}})_2(CF_2)_3$	300	1.2	150	Full pack

Test No.	Thickener	%	Fluid and Additives	Grease Pen.	Grease Residue, g	Hours	Test Variations and Remarks
776	p-aminobenzoic acid TODI p-phenylazoaniline TiO_2	9 9 3 3	F-6-7024 + 3% $(\not{0}_3\ SiOC)_2\ (CF_2)_3$ O	146	0.6	148	Full pack
777	Ammeline	35	60% F-6-7024 40% $\not{0}_8\ Si_4O_4$	192	1.8	164	Full pack, preheated
778	Ammeline	35	F-6-7024 + 5% $(\not{0}_3\ SiOC)_2\ (CF_2)_3$ O	300	1.9	165	Full pack
779	Ammeline	33	40% F-6-7024 60% $\not{0}_8\ Si_4O_4$	60	1.0	169	Full pack, preheated
780	ASU	13	F-6-7024 + 1% PANA + 1% $F(CF_2)_{10}\ CH_2OH$	320	0.8	64	Full pack
781	Ammeline	35	60% F-6-7024 40% $\not{0}_6\ SiO_2$	-	2.5	133	Full pack, preheated
782	Ammeline Rilube	35 10	F-6-7024	312	1.4	97	Full pack

FIGURE 1

VAPOR PRESSURE OF $(PNCl_2)_n/ZnCl_2$

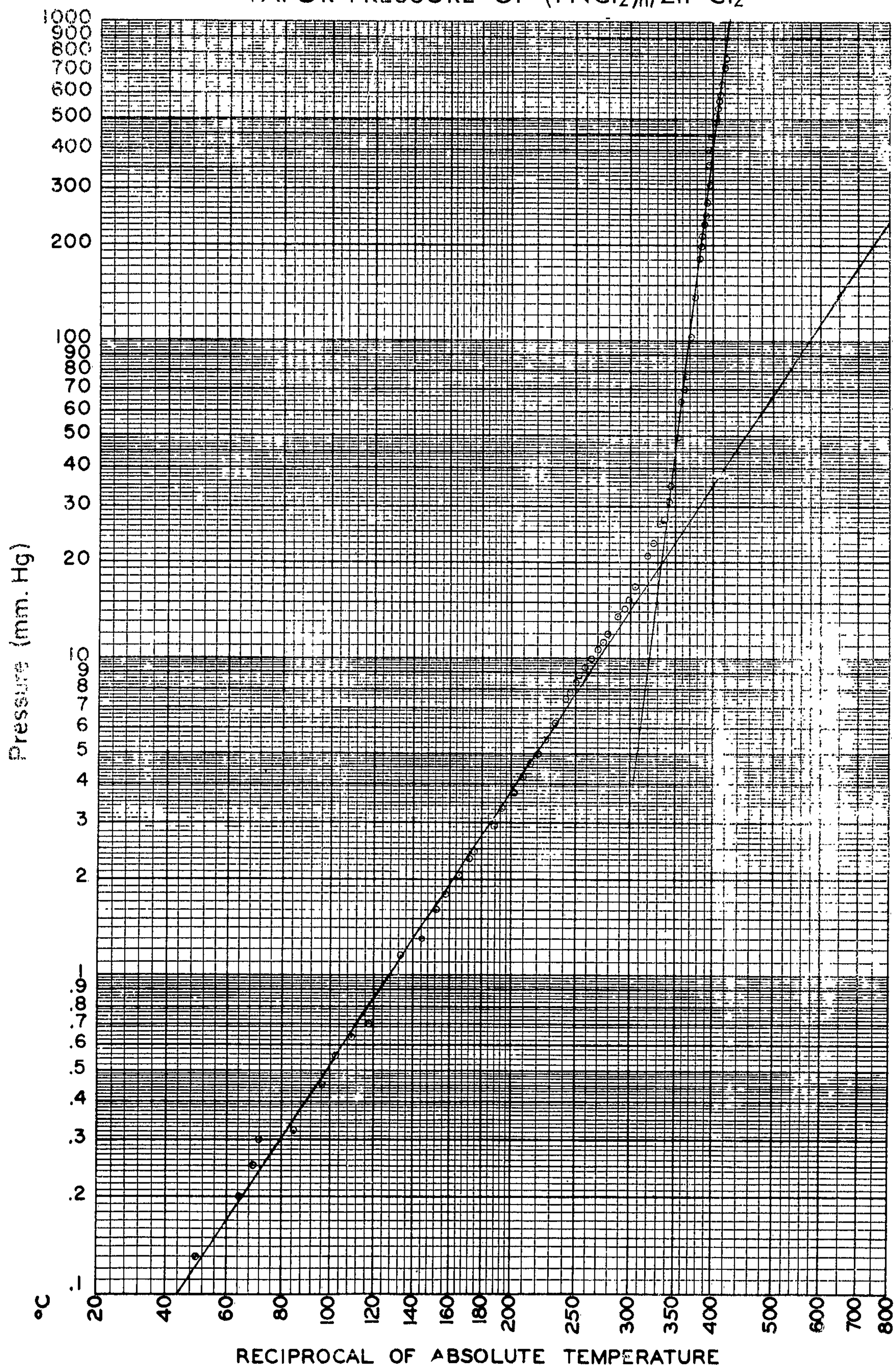
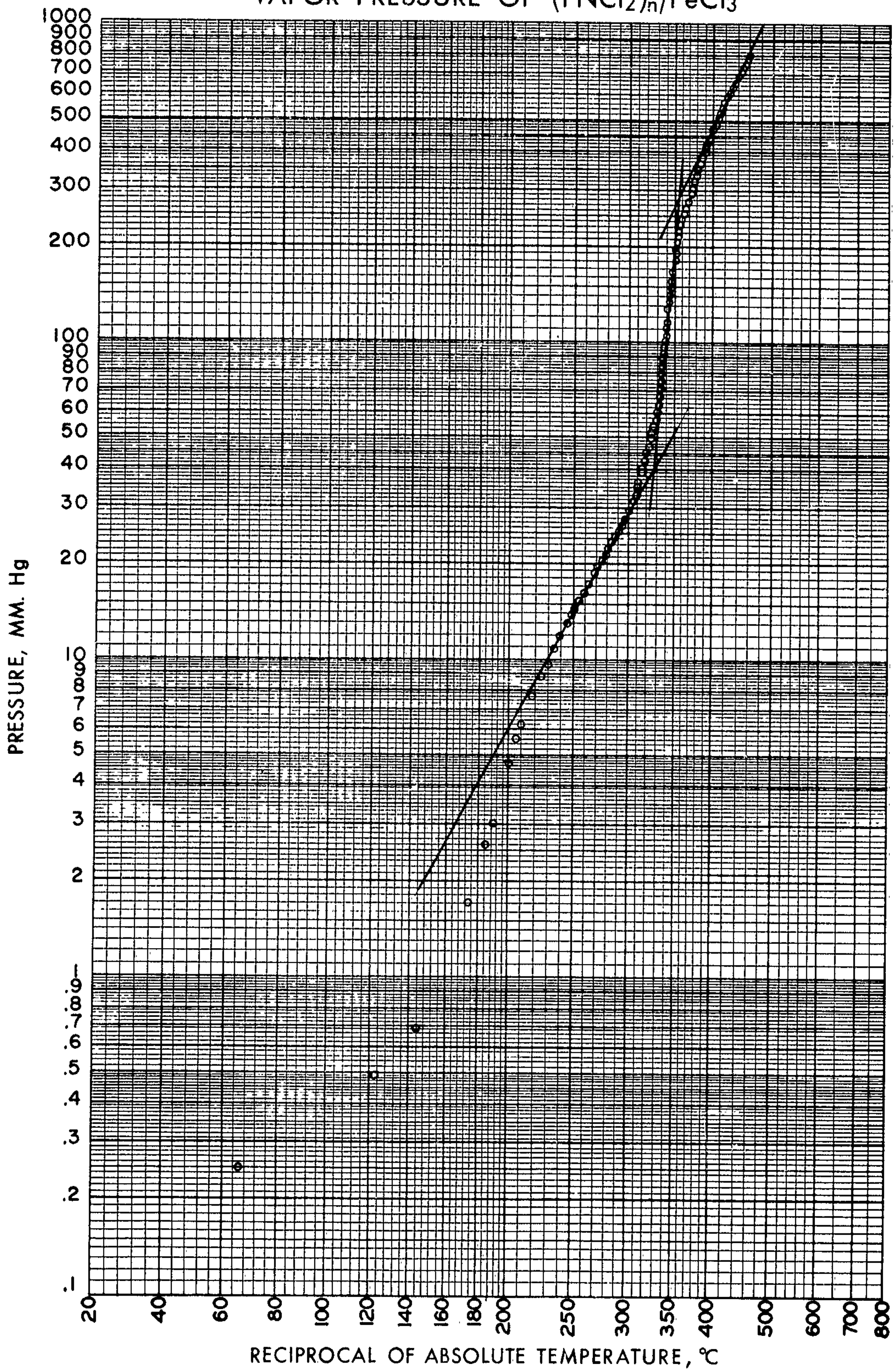


FIGURE 2

VAPOR PRESSURE OF $(PNCl_2)_n/FeCl_3$



Aeronautical Systems Division, Dir/Materials and Processes, Nonmetallic Materials Lab, Wright-Patterson AFB, Ohio.
Rpt Nr WADD-TR-60-557, Part III. Progress report, May 63, 67p. incl illus., tables.

Unclassified Report

The objective of this work is the development of grease systems capable of operating in loaded bearings over the temperature range of -65 to 900 F. Current work done on a 0 to 600 F grease system. Most of the test work was done at 600 F under 5 lb. radial and 5 lb. axial load and 50 lb. radial and 25 lb. axial load. Some tests were carried out at 650 and 700 F under light load also. Greases made by blending F-6-7024 Silicone Fluid and one of several polyphenyl polysiloxanes and thickening with Ammeline have given the longest 600 F, high-load bearing tests to date. Bearing tests on a

(over)

1. Lubricants (Lubrication & bearings)
2. Greases
3. Additives
I. AFSC Project 3044, Task 304403
II. Contract AF 33(616)-7597
III. American Oil Co. Whiting, Indiana
IV. K. R. Bunting, et al.
V. Not aval fr OTS
VI. In ASTIA collection

series of these greases range from 150 to 220 hours. Ammeline is the only thickener that gave these long bearing tests with these blends. With other fluids and other additives in fluids ASU and modified ASU's gave results comparable to Ammeline. While several other experimental silicone fluids and additives gave results comparable to ASU or Ammeline thickened F-6-7024 Silicone Fluid, none were any better except titanium dioxide. When titanium dioxide was used at 2 to 3% in p-phenyl azoaniline modified ASU-F-6-7024 greases 600 F high-load bearing tests of 150 hours were obtained. This compound did not show any beneficial effect in other grease systems. All the phosphonitrilic chloride-metal halide complexes tested showed hydrolytic instability.