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DEVELOPMENT AND EVALUATION OF A GREASE FOR –100°F. TO +350°F.

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FOREWORD

This report was prepared by Continental Oil Company under USAF Contract No. 33(616)-2364. This contract was initiated under Project No. 3044, "Aviation Lubricants," Task No. 73310, "Aircraft Lubricating Greases." The work was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. H. Schwenker acting as project engineer.

This report covers work conducted from December 1955 to December 1956.

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ABSTRACT

In the work directed toward the development of an aircraft grease suitable for use over a wide temperature range, emphasis has been placed on the utilization of certain hexa alkoxy disiloxane fluids as base oils. The thickener was limited to colloidal calcium carbonate coated through chemical interaction with the calcium salt of 3-methyl glutaric acid. Formulations using the base oil hexa (2-ethyl butoxy) disiloxane show promise of utility over a -100°F. to +325°F. temperature range. Formulations using the base oil hexa (2-ethyl hexoxy) disiloxane show promise of utility over -100°F. to +350°F. This fluid was not available in large quantities until the last quarter of the contract period. Time has permitted the preparation and evaluation of only a limited number of hexa (2-ethyl hexoxy) disiloxane greases. These greases perform in the range of 450 to 600 hours at 350°F. in the high speed testers, providing pure oxidation inhibitor (p,p'-dioctyldiphenylamine) is used. The pure grade is not commercially available at this time. The use of commercial grade inhibitor limits performance to around 150 hours at 350°F.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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

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INTRODUCTION

The purpose of this contract is the development of a wide temperature range grease operable from -100°F. to +350°F. The thickener for this grease development has been limited to a colloidal calcium carbonate coated with the calcium salts of aliphatic dicarboxylic acids.

The major difficulty encountered has been the selection of a suitable base oil. The aliphatic diesters were first studied as possible base oils. This work was described in WADC Technical Report 55-190. Although promising in many respects, their use was precluded because of high evaporation rates and lack of a sufficiently active oxidation inhibitor at 350°F. Greases were then prepared using certain hexa alkoxy disiloxanes as the base oil. The use of the disiloxane fluids was discussed in WADC Technical Report 55-190 Part 2. Formulations using the base oil hexa (2-ethyl hexoxy) disiloxane either surpassed or nearly met target requirements with the exception of high temperature performance. Here a maximum of 350 hours had been achieved. The major difficulty noted in the high temperature performance test was a rapid hardening of the grease due to oxidation and volatilization of the base oil. Several promising modifications designed to improve high temperature performance of disiloxane greases were in the developmental stage but had not been fully investigated because of the shortage of the base oil, hexa (2-ethyl hexoxy) disiloxane.

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SECTION I

MISCELLANEOUS STUDIES

Rapid hardening of the grease due to oxidation and volatilization of the base oil in the high temperature performance test appeared to be the major difficulty in the proposed grease development program. Several promising modifications designed to improve high temperature performance of disiloxane greases were reported in WADC Technical Report 55-190 Part 2 as being in the development stage. The results of these modifications as well as other miscellaneous studies are discussed in the following paragraphs. Because the available supply of the preferred hexa (2-ethyl hexoxy) disiloxane was extremely limited, most of these studies were made with the lower molecular weight hexa (2-ethyl butoxy) disiloxane analogue. Normal lifetime at 350°F. of control greases made with this fluid was, of course, very low, in the order of 60 to 90 hours.

New Coupling Agents

It was believed that the oil-holding ability of the grease structure might be improved by the use of substitute coupling agents for the 3-methyl glutaric acid normally used. Numerous acids had previously been screened (see WADC TR 55-190 Part 2). Of the acids screened, the most promising appeared to be 5,5'-methylene-disalicylic acid. Two additional acids, pyromellitic and isophthalic, were also found to be coupling agents for calcium carbonate. Pyromellitic acid and 5,5'-methylenedisalicylic acid were evaluated in the grease formulation. Exhibit A tests on greases coupled with these two acids showed no improvement over 3-methyl glutaric acid coupled greases. The use of these acids considerably reduced the high temperature life of the grease in addition to other shortcomings.

Sulfurized Alkyl Phenols

One possible cause for rapid hardening in the high temperature performance test other than volatilization of the base oil was thought to be due to the formation of insoluble polymeric oxidation products. This suggested the use of sulfurized alkyl phenols which are known to solubilize oxidation products as well as being dispersing agents for calcium carbonate. The initial work wherein sulfurized nonyl phenol was used in aliphatic diester grease formulations was described in WADC TR 55-190 Part 2. This work was continued in hexa (2-ethyl butoxy) disiloxane grease formulations.

The substitution of sulfurized alkyl phenols for a portion of the sulfonate dispersing agent was found to increase the high temperature performance life of hexa (2-ethyl butoxy) disiloxane greases as much as 50%. Two sulfurized alkyl phenols were evaluated. A grease containing sulfurized nonyl phenol substituted for 25% of the sulfonate dispersing agent ran 130 hours in the high temperature bearing. This was 44 hours longer than a similar grease containing no sulfurized phenol. The apparent viscosity of one sulfurized nonyl phenol containing grease was 7,920 poises at 16 $sec.^{-1}$ and -100°F. This is the lowest value obtained with any 3-methyl glutarate thickened disiloxane grease.

Similar greases were prepared using the sulfurized phenol of postdodecylbenzene overhead. The particular phenol used was prepared from the 20% overhead fraction distilled from postdodecylbenzene. The bottoms fraction or stripped postdodecylbenzene is the hydrocarbon presently used as the dispersing agent for the grease in the form of its sulfonate. The use of this sulfurized phenol resulted in an increase of 27 hours in the high temperature performance of the grease. There was no reduction in apparent viscosity at -100°F.

As can be seen, the above-described modifications did not approach Exhibit A requirements with respect to high temperature performance. The sulfurized phenols were therefore excluded from any further consideration this report period. They would be considered, however, in any future mixed base oil formulations which utilized high concentrations of silicone fluids (see WADC TR 55-190 Part 2, pages 12-13).

Mixed Base Oil Greases

Since extreme difficulty was being encountered in finding a supplier for the hexa (2-ethyl hexoxy) disiloxane fluid and since modifications of the hexa (2-ethyl butoxy) disiloxane greases were far from meeting target requirements, an attempt was again made to change base oils. As a first step, numerous oils were considered for replacing part of the hexa (2-ethyl butoxy) disiloxane fluid.

Silicone fluids could not be used as the base oil because of their lack of solvency for the sulfonate dispersing agent (see WADC TR 55-190). Silicone fluids were subsequently used in the grease formulation at around a 10% addition level to achieve work stability. The addition level was limited because of incompatibility in the system. It was later found that the use of sulfurized nonyl phenol in the grease system allowed the incorporation of larger amounts of silicone fluid. As much as 42% of DC510-50 silicone fluid could be used in the grease formulation (see WADC TR 55-190

Part 2, pages 12-13). This modification results in an increase in high temperature performance life which was attributed to the stability of the silicone fluid. This modification did not meet target requirements, however.

Since lubricity was one of the drawbacks in considering the utility of methyl-phenyl type silicone fluids (i.e., DC510-50), it was recommended by the Materials Laboratory, WADC, that DCF-60, a chlorinated silicone, be evaluated. It was thought that this substitution would enable the grease to pass the lubricity tests and also improve low temperature apparent viscosity and high temperature performance. Subsequent testing of greases containing DCF-60 showed no improvement over that of DC510 containing greases.

Emery Industries, Inc., supplied samples of two high temperature stable aliphatic polyesters. The two polyesters supplied were Emery 907R and Emery 999R. These esters did not have sufficiently low pour points to allow complete substitution for the hexa (2-ethyl butoxy) disiloxane base oil in a grease formulation. These were high flash point fluids, however; and it was thought that a blend of these esters and disiloxane fluid might be used as the base oil wherein some sacrifice would be made in low temperature properties of the grease in favor of a possible improvement in high temperature characteristics. Several greases were therefore prepared which utilized hexa (2-ethyl butoxy) disiloxane grease intermediates. The polyesters were used as the additional base oil necessary for the grease formulation. The formulations were adjusted so that the polyester would constitute about 50% of the base oil in the resulting grease. These greases required a higher active gelling agent level and were more thixotropic than disiloxane fluid greases. Higher concentrations of silicone fluid were required to work stabilize the finished formulations.

Greases prepared using Emery 907R fluid, which was the lower boiling of the two polyester fluids, exhibited very favorable low temperature properties at -100°F. Subsequent tests on mixed base oil greases which utilized these fluids proved them to be lacking in lubricity. These greases also failed within 10 hours in the 350°F. high speed bearing testers. Test results did not indicate any promise for mixed base oil greases of this type.

Dow Corning supplied a one-quart sample of DCXF-5830 fluid (a hexa-alkoxydisilphenane) for evaluation as a possible substitute for disiloxane fluids. This fluid had a flash point of 488°F. and a pour point of -47°F. and was found to have the necessary solvency for the sulfonate dispersing agent. A one-pound batch of grease was prepared using straight DCXF-5830 as a vehicle and subjected to Exhibit A specification tests. It was realized that the low temperature requirements could not be met with this grease. Apparent viscosity data were obtained, however, and were found to exceed 54,000 poises at 16 $sec.^{-1}$ and -100°F. Gear wear was borderline under a five-pound load and stripped under a ten-pound load. At 318 hours in the high temperature performance test, the micro safety switch cut out. The grease was a dry powder at this point, and the bearing was badly discolored.

Lubricity Additive Evaluation

A sample of MLO-55-499 (tin octoate) was supplied by WADC for evaluation as a lubricity additive in disiloxane greases. Attempts were made to incorporate this additive in the grease formulation at a 5% level. Unfortunately, MLO-55-499 acts as a catalyst for the decomposition of hexa (2-ethyl butoxy) disiloxane fluid. This was evident by the presence of large quantities of 2-ethyl butanol condensing from the effluent gas during the blending operation.

Corrosive Sulfur

The intermediate calcium carbonate dispersion utilized in preparing the subject grease was found to contain small amounts of colloidal sulfur. This sulfur either originated in the methanolic calcium sulfhydrate solution prior to carbonation or was formed in the intermediate during the carbonation step. It was sufficiently active in the intermediate as well as in grease formulations to blacken a copper strip within five minutes at 212°F. Several convenient methods for removing the bulk of the sulfur from the grease intermediate were evaluated. These methods included purging the intermediate with various gases (NH_3, H_2, CO_2, and N_2) at 302°F. and also contacting the ZnO at 302°F. The most practical of all the methods appeared to be the incorporation of a dry nitrogen blowing step in the intermediate preparation. To effect carbonation, the intermediate components are normally blown with CO_2 at ambient temperatures followed by stripping solvents to 302°F. Currently the intermediates are blown with CO_2 through 302°F. followed by purging with dry nitrogen at 302°F. for four hours. This removes the bulk of the colloidal corrosive sulfur. The grease formulations were found, however, to require the addition of a metal deactivator to completely inhibit copper corrosion. Several commercially available deactivators were evaluated of which certain metal alkyl carbamates appeared the most effective. Some difficulties were encountered in finding soluble metal alkyl carbamates. The only effective soluble carbamate found has been Vanlube 65 (cadmium di-2-ethyl hexyl dithiocarbamate in mineral oil).

Three hexa (2-ethyl butoxy) disiloxane greases containing varying amounts of Vanlube 65 and one grease containing quinizarin were evaluated in the MIL-G-3278 Bomb Copper Corrosion Test. The data from these tests are as follows:

TABLE I

BOMB COPPER CORROSION TEST

Grease Number		Per Cent	Pressure Drop	Copper Corrosion
9559	Quinizarin	0.1	None	Slight Tarnish
9560	Vanlube 65	5	None	Slight Tarnish
9570	Vanlube 65	2	None	Very Slight Tarnish
9572	Vanlube 65	1	2.5 p.s.i.	Very Slight Tarnish

As the MIL-G-3278 specification allows no more than a 0.5 p.s.i. pressure drop, the contract grease will require around 2% Vanlube 65.

There has been some indication that the addition of Vanlube 65 increases the high temperature performance life of disiloxane greases. This will be discussed in Section IV.

Oxidation Inhibitor

The oxidation inhibitor used in disiloxane greases is p,p'-dioctyldiphenylamine (DDA). The purified grade of this material is not commercially available at this time, and only small samples have been available for evaluation. A commercial grade of DDA, AgeRite Stalite S, is marketed by the R. T. Vanderbilt Company. There are, however, small amounts of impurities present in AgeRite Stalite S which are deleterious to the grease stability in the high temperature bearing testers. This was first detected with hexa (2-ethyl butoxy) disiloxane greases. A grease containing AgeRite Stalite S performed only 65 hours at 350°F. in the bearing tester. The same grease performed 105 hours when pure DDA was used.

More recent investigations with hexa (2-ethyl hexoxy) disiloxane greases have established the same trend (see Appendix IV). Greases which contained pure DDA approached the 600-hour requirement in the bearing testers (see greases Nos. 9586, 9588, and 9596). Two similar greases which contained AgeRite Stalite S only performed in the range of 150 hours (see greases Nos. 9605 and 9609).

SECTION II

FORMULATION VARIABLES INVOLVING GELLING AGENT

Considerable difficulties were experienced in preparing greases which utilized a freshly prepared batch of 3-methyl glutaric acid. The formulations resulted in cloudy fluids instead of greases. This difficulty was traced to the anhydrous nature of the acid. The initial work on this problem indicated that the addition of 7.5% water (by weight of 3-MGA used) during the grease formation was essential for optimum gelation using the normal procedure of grease preparation. The procedure used was to charge the grease mixer with the intermediate-oil-acid-solvent mixture and to start stripping immediately under slightly reduced pressure to prevent solvent leaching of grease from the mixer gears. Solvents are very steadily and rapidly removed to 356°F.

Using the above-mentioned water level (7.5%) and the customary preparation procedure, a lack of reproducibility was experienced from batch to batch. This lack of reproducibility was noticeable in the consistency and also in high temperature performance life of the grease. Since the disiloxane base oils used in the grease are subject to water hydrolysis, it was believed that varying amounts of water retained in the grease gel were responsible for this disparity. Subsequent preparations revealed that reproducibility with respect to consistency could be achieved by refluxing the volatile solvents for 30 to 45 minutes prior to stripping. At the previously determined optimum water level (7.5%), this modification resulted in excessive water retention in the gel structure, which reduced the high temperature performance life of the grease. It was found, however, that greases could be prepared by the reflux method without the addition of water. This resulted in a considerable improvement in high temperature performance life.

The effect of admixed water in the grease properties had not been considered in past formulation studies. It was, therefore, believed that further improvement in the grease might be accomplished by a thorough study of the grease formulation variables.

The first study was to establish the effect of various solvent systems on the reaction of 3-methyl glutaric acid with $CaCO_3$ in the grease intermediate and in the properties of the resultant gel. From these investigations, it was found that 3-MGA will react with the dispersed $CaCO_3$ in the intermediate with or without additional solvents other than the base oil. A stable gel or suitable grease was found to be dependent on a balanced solvent system, which normally includes benzene, acetone, and water. As previously pointed out, water appears to be the most critical component of the solvent system. The water must, however, be solubilized into the hydrocarbon system by a mutually miscible solvent (e.g., acetone).

The second study was to determine the effect of varying the ratio of active gelling agent to per cent conversion of the calcium carbonate at a constant water level. It was known from past experience that the conversion of 85% of the calcium carbonate to the calcium salt of 3-methyl glutaric acid was near optimum for the subject grease. For this study, the conversion ratio was limited to the 70 to 85% range. Admixed water was held at 7.5% by weight of 3-MGA used. Experimentally, this study consisted of preparing 3-pound batches of base grease for various active gelling agent levels and conversion ratios. One pound of each base grease was retained for testing. The remaining two pounds were blended with 10% DC510-50 silicone fluid (work stability additive) and 2% of AgeRite Stalite S (oxidation inhibitor). One pound of the resultant finished grease was used for work stability testing and miscellaneous Exhibit A tests, while the other pound was used for low temperature tests. The test results for these greases are graphically illustrated in figures 1, 2, and 3. There were no substantial differences noted in the greases as a result of varying the conversion ratio. All of the greases meet the consistency, work stability, and apparent viscosity requirements. Apparent viscosity data given in Figure 3 are at a 15.7 $sec.^{-1}$ shear rate. Contract specifications call for a 20 $sec.^{-1}$ shear rate. The values given would therefore be somewhat lower at a 20 $sec.^{-1}$ shear rate.

A very limited number of these greases were subjected to the high temperature bearing test. The performance life of the greases subjected to this test was very short because of excessive water retention in the gel structure. As previously stated, 7.5% admixed water was used to facilitate gelation. The reflux method was also used for these greases which would account for excessive water retention resulting in short performance lives at 350°F.

SECTION III

HEXA (2-ETHYL BUTOXY) DISILOXANE BASE OIL GREASES

High Temperature Performance

The majority of the experimental work performed this report period utilized hexa (2-ethyl butoxy) disiloxane fluid as the grease base oil. The properties of this fluid are given in Appendix I.

At the onset of this report period, the maximum life of hexa (2-ethyl butoxy) disiloxane base oil greases in the 350°F. bearing tester was in the range of 86 hours. When the optimum amount of water necessary for gelation was determined, as discussed in Section II, the performance life was increased to an average of 200 hours at 350°F. A series of tests were then made in an effort to establish the maximum temperature at which a hexa (2-ethyl butoxy) disiloxane grease formulation would satisfactorily perform for 600 hours in the bearing tester. The results of these tests are listed in the following table. A more thorough discussion is given in Appendix II (i.e., grease formulation, condition of test grease, bearing condition, weight loss, etc.).

TABLE II

Test Temperature	Hours Run, 10,000 r.p.m.
300°F.	600+
325°F.	600+
350°F.	200+
375°F.	193

These data were collected on a grease formulation which was coupled in the presence of 7.5% admixed water. The reflux method was not used. Subsequent test data at 350°F. on greases prepared by the reflux method at lower water levels showed performance equivalent to that in Table II. There appeared to be no difference between greases which were coupled at either a 0% or 2.5% water level.

Time has not permitted the evaluation of a "butoxy" fluid grease containing the copper corrosion inhibitor, Vanlube 65. There is some indication that this additive improves the high temperature life of disiloxane greases (see Section IV).

General Properties

Typical consistency, work stability, and apparent viscosity data are graphically illustrated in figures 1, 2, and 3. These properties are easily controlled to almost any desired value by adjusting the active gelling agent level. Additional typical properties of hexa (2-ethyl butoxy) disiloxane greases are given in Appendix III.

In general, hexa (2-ethyl butoxy) disiloxane greases meet all of the contract target specifications with the exception of Navy Gear Wear and high temperature performance at 350°F.

SECTION IV

HEXA (2-ETHYL HEXOXY) DISILOXANE BASE OIL GREASES

High Temperature Performance

As previously stated, hexa (2-ethyl hexoxy) disiloxane fluid appeared to be the most promising base oil for the subject grease. The properties of this fluid are given in Appendix I. This material has a flash point of 480° to 485°F., which is considerably higher than that of hexa (2-ethyl butoxy) disiloxane (410°F.). Its properties at -100°F. are also favorable.

Several greases have been prepared and evaluated which utilized various small supplies of hexa (2-ethyl hexoxy) disiloxane fluid as the base oil. The original supply was a one-quart sample supplied by Oronite Chemical Company. Oronite's fluid appeared to have the desired physical properties; however Oronite could not supply additional quantities. A grease prepared from this fluid ran 350 hours in our bearing testers at 350°F. (see WADC TR 55-190 Part 2). This grease was evaluated at both the WADC and Navy test laboratories. The performance life of the grease in their test machines was considerably less than that achieved in our testers. The performance life in their testers varied from 98 to 220 hours at 350°F. It was found that the discrepancy in test data at Continental and WADC was due to bearing slippage in our testers. This was corrected with some minor modifications in the test equipment and is described in Section V.

Several companies were contacted in an effort to find a commercial supplier for this fluid. Although a commercial supplier could not be found, a number of the companies contacted had an interest in synthesizing the fluid. The interested companies were Anderson Laboratories, Dow Corning, Stauffer Chemical, and Royal Lubricants.

Anderson laboratories supplied several quarts of hexa (2-ethyl hexoxy) disiloxane fluid having similar properties to Oronite's fluid. Anderson's attempts to pilot larger quantities failed, however, resulting in a polymeric material which exhibited unsatisfactory properties. It was Anderson Laboratories' belief that they could not supply a fluid comparable with Oronite's.

Stauffer Chemical Company supplied two quarts of hexa (2-ethyl hexoxy) disiloxane. Stauffer's fluid had a low flash point (i.e., 345°F. versus 485°F. for Oronite's). A grease prepared with this fluid performed 216 hours in the 350°F. tester. This grease was prepared before the optimum water level and preparational technique were established (see Section II). It is, therefore, possible that greases prepared with this fluid using the present known formulation technique would perform somewhat longer at 350°F. Stauffer Chemical Company continued their research and have made progress in the attempts to raise the flash point but have not achieved the 485°F. flash exhibited by Oronite's fluid.

Dow Corning synthesized and supplied one quart of hexa (2-ethyl hexoxy) disiloxane. Like Stauffer's fluid the flash point was somewhat low. A grease prepared from this fluid performed 258 hours in the 350°F. tester. There was evidence of bearing slippage in the test shaft, however; and the grease has not been tested on the reconditioned machines.

Royal Lubricants Company supplied a quart sample of fluid for our examination and reported that they could supply any desired quantity. The quart sample supplied for examination was found to be essentially the same as that supplied by Oronite. A grease prepared from this fluid performed for 531 hours in one test run. Subsequent testing revealed a complete lack of correlation between the various bearing test machines. It was at this time, the last quarter of the present contract period, that the test machines were completely modified (see Section V).

A larger supply of fluid was received from Royal Lubricants which enabled a thorough examination of hexa (2-ethyl hexoxy) disiloxane greases. Formulation and Exhibit A test data for these greases are given in Appendix IV. The first grease (No. 9582) prepared from this new supply of fluid was somewhat deficient with respect to yield and Exhibit A test results. Grease 9582 was prepared at a zero additional water level during the coupling step. From previous experience, it was believed that the yield could be increased by adjusting the water level during the grease preparation. Grease 9584 was prepared at the same active gelling agent level as grease 9582, but the water level was adjusted to 2.5% by weight of 3-methyl glutaric acid used during the coupling step. This adjustment in formulation did not improve the yield but did show an improvement in high temperature performance life over that of grease 9582. In order to improve the consistency of the formulation, grease 9588 was prepared at a 1% higher active gelling agent level. Grease 9588 was prepared before the high temperature bearing test results for grease 9584 were available and was, therefore, prepared at a zero water level. The increase in thickener was rewarding, however, in that the oil holding ability of the grease was improved. This is evident by the 550-hour performance life of grease 9588 at 350°F. as compared with an average performance life of 54 hours with grease 9582. Detailed results of the high temperature test on grease 9588 are given in Appendix V.

The greases discussed in the above paragraph (Nos. 9582, 9584, and 9588) did not contain a metal deactivator to inhibit copper corrosion. These greases were blended with Vanlube 65 and again subjected to the high temperature bearing test. The respective blends are Nos. 9585, 9586, and 9596 (see Appendix IV). Grease 9582 which performed 54 hours at 350°F. performed 245 hours after adding 1% Vanlube 65 (grease 9586). Grease 9588 which previously performed 550 hours only performed 448 hours after the addition of 2% Vanlube 65 (grease 9596). Detailed results of the high temperature test on grease 9586 are given in Appendix VI and on grease 9596 in Appendix VII.

The above-described greases which approached high temperature target requirements utilized pure p,p'dioctyldiphenylamine (DDA). Grease Nos. 9605 and 9609, which performed 158 and 151 hours, respectively, utilized AgeRite Stalite S. Hexa (2-ethyl hexoxy) disiloxane greases will probably not meet high temperature target requirements unless a source of pure DDA can be found.

General Properties

Hexa (2-ethyl hexoxy) disiloxane base oil greases appear to approach all of the contract target requirements with the exception of Navy Gear Wear. Typical properties are given in Appendix III.

SECTION V

TEST EQUIPMENT

Navy Gear Wear

A new Navy Gear Wear tester was built and put into operation near the beginning of this contract period. Correlation between the new tester and the tester which was already in operation was found to be excellent.

High Speed and Temperature Bearing Machines

All high temperature performance tests were carried out under specification VV-L-791, method 33.1. One test machine was in operation prior to this contract period. Two additional machines were built and put into service at the onset of this contract period. The original design was unchanged. In the original design, the test bearing was held on the shaft by a tight press fit. Some difficulties were experienced in getting correlation between the three testers in service. This was traced to test bearing slippage on the shaft and was temporarily corrected by chrome plating the shafts. This modification was found, however, to be an unrealiable means of holding the bearing.

The test machines were then completely renovated and modified to incorporate a positive lock on the test bearing. The newer design incorporates a push fit test bearing held by a retainer which locks the inner race of the test bearing more precisely (see Figure 4). This modification has eliminated bearing slippage on the shaft which, in turn, increased the repeatability and reproducibility of test results.

At the present time, the three test rigs have logged a total of 17,000 hours. The rigs are checked daily for possible out-or-round test bearing housings, misalignment of shafts, bearing slippage, reduction in r.p.m., temperature changes, etc. In addition to the daily inspections, the main bearings are lubricated every 1,000 hours.

SECTION VI

CONCLUSIONS AND PLANS

From recent fragmentary data, it appears that hexa (2-ethyl hexoxy) disiloxane base oil greases made with purified oxidation inhibitor will meet all of the contract target requirements with the possible exception of Navy Gear Wear. Present plans are to pilot 25 pounds of a grease utilizing this fluid for WADC specification tests.

Greases have been prepared utilizing hexa (2-ethyl butoxy) disiloxane as the base oil which are operational over a -100°F. through +325°F. temperature range. These greases will also meet target requirements with the exception of gear wear. Present plans are to pilot 50 pounds of a grease utilizing this fluid for WADC specification tests.

At the request of the contract engineer, a 25-pound batch of grease will be piloted, which will utilize as the base oil Hercoflex 600. This grease will also be supplied to WADC for specification tests.

APPENDIX I

PROPERTIES OF DISILOXANE FLUIDS

	Hexa(2-Ethyl Butoxy) Disiloxane	Hexa(2-Ethyl Hexoxy) Disiloxane
Density 20/20°C.	0.9075	0.9047
Flash, COC, °F.	410	490
Fire, COC, °F.	465	540
Boiling Point, °F. 0.1 mm.	428	500
Vapor Pressure, mm. at 400°F.	1.2	0.09
Kinematic Viscosity		
at 100°F. CS	11.12	15.19
at 120°F. CS	3.94	4.96
Pour Point, °F.	-100	-100

APPENDIX II

BEARING PERFORMANCE DATA OF A HEXA (2-ETHYL BUTOXY) DISILOXANE GREASE AT VARIOUS TEMPERATURES

Grease Formulation No. 9384

Dispersion Ratio	0.4
Conversion of $CaCO_3$	78. %
Hexa (2-Ethyl Butoxy) Disiloxane	73.1%
DC510-50 cs. Silicone Fluid	10. %
AgeRite Stalite S	2. %
Active Gelling Agent	
Calcium 3-Methyl Glutarate	5.1%
Calcium Carbonate	0.8%
Calcium PDB Sulfonate	9.0%

300°F. Test

No difficulty was encountered in meeting the specifications for the high temperature performance test with grease 9384 at 300°F. as indicated by the following results:

Conditions of Bearing After 600 Hours at 300°F.

(204 Conrad - Type 8 - Ball Bearing)

Separator	Good - Slight control surface wear, no ball pocket wear.
Raceways	Good - Slight nonuniform ball path.
Balls	Very good
Weight Loss	.004 mg.
Deposits	Light deposits of varnish on separator, raceways, and balls.

Condition of Grease after 600 Hours at 300°F.

Consistency	Good
Texture	Smooth and buttery
Bleeding	A small amount
Color	Straw color to light brown
Oiliness	Good

325°F. Test

No difficulty was encountered in running 600 hours for the high temperature performance test with grease 9384 at 325°F.; however the formulation was only a borderline pass at this temperature. These results can be summarized as follows:

General Observation:

1. The adherence of this grease is undoubtedly affected by the increase in temperature.
2. The rate and amount of oil loss increase with increasing temperature.

Condition of Bearing After 600 Hours at 325°F.

(204 Conrad - Type 8 - Ball Bearing)

Separator	Good - Slight control surface wear, no ball pocket wear.
Raceways	Good - Slight nonuniform ball path, trace of discoloration on outer race.
Balls	Good - Slight moderate bands.
Weight Loss	.032 mg.
Deposits	Moderate deposits of varnish on separator, raceways, and balls.

Condition of Grease After 600 Hours at 325°F.

Consistency	Putty-like
Texture	Dry
Bleeding	Poor
Color	Dark brown

350°F. Test

Grease 9384 had performed an average of 204 hours at 350°F. Weight loss and condition of bearing were not determined.

375°F. Test

A maximum of 196 hours at 375°F. was achieved in the high temperature performance test with grease 9384. Listed below are the bearing results:

Condition of Bearing after 196 Hours at 375°F.

(204 Conrad - Type 8 - Ball Bearing)

Separator	Good - Discoloration with slight control surface wear.
Raceways	Good - Discoloration.
Balls	Good - Discoloration.
Weight Loss	.100 mg.
Deposits	Heavy varnish deposits on separator, raceways, and balls.

Condition of Grease 9384 after 196 Hours at 375°F.

Consistency	Putty-like
Texture	Dry
Color	Dark brown
Oiliness	Poor

APPENDIX III

TYPICAL PROPERTIES OF DISILOXANE GREASES

	Air Force Requirements	BASE OIL USED: Hexa(2-Ethyl Butoxy) Disiloxane	BASE OIL USED: Hexa(2-Ethyl Hexoxy) Disiloxane
Penetration			
60 strokes	260-340	Pass	Pass
100,000 strokes	375 max.	Pass	Pass
Oil Separation	5% max.	2	2
Water Washout	20% max.	20	15
Low Temperature Torque	5 sec. max.	2	2
Apparent Viscosity Poises at 20 sec.$^{-1}$ -100°F.	15,000	Pass	Pass
Navy Gear Wear			
5-lb. Load	2.5 mg./1,000 cycles	4	4
10-lb. Load	3.5 mg./1,000 cycles	11	11
Bearing Test, 10,000 r.p.m. 350°F.	600 hours	200+	450-600+

APPENDIX IV

FORMULATION AND EXHIBIT A TEST DATA FOR

HEXA(2-ETHYL HEXOXY) DISILOXANE GREASES

Exp. No.	Per Cent AGA	Per Cent Conversion	Per Cent H_2O to Couple	Per Cent DC510	Per Cent Oxidation Inhibition	PENETRATION STROKES 60	PENETRATION STROKES 3,000	Dropping Point °F.	Per Cent Oil Sep.	GEAR WEAR 5-Lb. Load	GEAR WEAR 10-Lb. Load	BEARING TEST Mach. No.	BEARING TEST Hours Run
9582	6	85	0	10	2 DDA	360				4.0	14.1	1	50
												2	50
												3	62
9584	6	85	2.5	10	2 DDA	350	350	450+	1.5	4.5	8.2	1	290
												2	280
												3	351(1)
9585(2)	6	85	0	10	2 DDA					3.1	11.6	2	245
9586(3)	6	85	2.5	10	2 DDA					3.0	11.4	1	600+
9587	7	85	0	(Base grease for 9588)		300				1.7	5.0		
9588	7	85	0	10	2 DDA	300				2.8	7.5	3	550
9596(4)	7	85	0	10	2 DDA	300				5.2	Strip	2	448
9605(5)	7	85	2.5	10	2 Stalite S	340	350			2.7	7.2	1	158
9609	7	85	2.5	10	2 Stalite S	345	350			3.4	Strip	3	151

(1) Stopped test due to difficulties with temperature controller. At times temperature was 30° too low. This trouble developed after 300 hours performance. All other tests were stopped due to micro switch cutoff or after completing 600 hours.
(2) Grease 9582 plus 1% of Vanlube 65 (Cadmium di-2-ethyl hexyl dithiocarbonate).
(3) Grease 9584 plus 1% of Vanlube 65.
(4) Grease 9588 plus 2% of Vanlube 65.
(5) 1.5% Vanlube 65.

APPENDIX V

GREASE 9588 - BEARING PERFORMANCE AT 350°F.

The high temperature performance test was run 550 hours at 350°F. with grease formulation 9588 before micro switch cutoff. Listed below are the bearing results:

Condition of Bearing After 550 Hours at 350°F.

(204 Conrad - Type 8 - Ball Bearing)

Separator	Good - A slight control surface and ball pocket wear.
Raceways	Good - Discoloration of outer race.
Balls	Good - Slight moderate bands
Weight Loss	.042 mg.
Deposits	Light deposits of varnish on separator, raceways, and balls.

Condition of Grease After 550 Hours at 350°F.

Consistency	Crumbly
Texture	Dry
Bleeding	Trace
Color	Straw brown
Oiliness	Trace

General Information

At the time of failure, the temperature did not increase over the preset of 350°F.; however the slippage of the test bearing was 200 r.p.m.

APPENDIX VI

GREASE 9586 - BEARING PERFORMANCE AT 350°F.

No difficulty was encountered in running 600 hours for the high temperature performance test with grease 9586 at 350°F. as indicated by the following results:

Condition of Bearing After 600 Hours at 350°F.

(204 Conrad - Type 8 - Ball Bearing)

Separator	Excellent - A very slight control surface wear, no ball pocket wear.
Raceways	Good - Slight discoloration on outer race.
Balls	Good - Very slight moderate varnish bands.
Weight Loss	.024 mg.
Deposits	Light deposits of varnish on separator, raceways, and balls.

Condition of Grease After 600 Hours at 350°F.

Consistency	Putty-like
Texture	Somewhat dry
Bleeding	A small amount
Color	Straw brown
Oiliness	Fair

General Observations

1. Lubricity appears to be no problem, inasmuch as the test bearings reveal little or no weight loss or other characteristics indicating a lack of lubricity.

2. Its life at 350°F. would probably be in the range of from 700 to no more than 800 hours.

APPENDIX VII

GREASE 9596 - BEARING PERFORMANCE AT 350°F.

A maximum of 448 hours at 350°F. was achieved in the high temperature performance test with grease 9596. Listed below are the bearing results:

Condition of Bearing After 448 Hours at 350°F.

(204 Conrad - Type 8 - Ball Bearing)

Separator	Good - A slight control surface and ball pocket wear.
Raceways	Good - Discoloration of outer and inner race.
Balls	Good - Very slight moderate bands.
Weight Loss	.050 mg.
Deposits	Light varnish deposits on separator, raceways, and balls.

Condition of Grease After 448 Hours at 350°F.

Consistency	Crumbly
Texture	Dry
Bleeding	None
Color	Dark brown
Oiliness	None

General Observations

At the time of failure, the temperature increased 75° over the preset 350°F. with slippage of 300 r.p.m.

APPENDIX VIII

EXHIBIT A

Development and evaluation of a grease with high load carrying capacity that is operational over a -100° to 350°F. temperature range. There is no restriction on the materials to be used in making this grease. An oil must be found with good lubricating properties that is stable over this temperature range and with low volatility at the upper temperature limit. A good gelling agent stable over the temperature range is also needed. The grease must possess oxidation stability, permit operation of low torque aircraft equipment at -100°F., provide adequate lubrication over the temperature range of -100°F. to 350°F., and supply adequate protection against corrosion.

Requirements for a -100° to 350°F. Grease

Test and Method	Requirement
1. Oxidation Stability Spec. VV-L-791, Method 345.3	Pressure drop shall not exceed: 5 p.s.i. in 100 hours 25 p.s.i. in 500 hours
2. Dropping Point Spec. VV-L-791, Method 142.1	Above 375°F.
3. Worked Penetration Spec. VV-L-791, Method 31.1	Above 260, Below 340
4. Dirt Spec. VV-L-791, Method 300.5	The grease shall not contain dirt or other foreign particles exceeding the following limits: 7,500 per cc. of 25-micron diameter or above 1,600 per cc. of 75-micron diameter or above None of 125-micron diameter or above
5. Bomb Copper Corrosion Spec. MIL-G-3278, 4.2.2.3.	Pressure drop shall not exceed 0.5 p.s.i. in 20 hours. No decomposition of the grease as evidenced by discoloration in the vicinity of the copper strip or appreciable stain or corrosion beyond that obtained in a blank test under the same conditions without grease. A slight change in the color of the grease, not green or dark brown, is acceptable.

Test and Method	Requirement
6. Water Resistance a. Water Washing Spec. VV-L-791, Method 325.2	Not more than 20 per cent of the grease shall be washed from the bearing when tested.
b. Water Immersion Spec. MIL-G-3278, 4.2.2.4	No cloudiness or other evidence of emulsification of the grease.
7. Evaporation Spec. VV-L-791, Method 35.1	Grease shall not lose more than 2.0 per cent of its weight in 22 hours.
8. Oil Separation Spec. VV-L-791, Method 32.1	Grease shall not lose more than 5 per cent of its weight in 30 hours.
9. Apparent Viscosity Spec. VV-L-791, Method 30.6	Viscosity of the grease shall not exceed 15,000 poises at a shear rate of 20 reciprocal seconds at -100°F.
10. Gear Wear Test Spec. MIL-G-3278, 4.2.2.8	The brass gear of a helical gear set shall not wear more than 2.5 mg. per 1,000 cycles under a 5-pound load nor more than 3.5 mg. per 1,000 cycles under a 10-pound load.
11. Worked Stability Spec. VV-L-791, Method 31.3	375 after working 100,000 double strokes.
12. Low Temperature Torque Spec. MIL-G-7421, 4.4.2.1	A 204 Conrad-type 8-ball bearing lubricated with the grease shall turn its first complete revolution in each direction in not more than 5 sec. at -100°F.
13. High Temperature Performance Spec. VV-L-791, Method 33.1	The grease shall lubricate a 204 Conrad-type 8-ball bearing satisfactorily for a minimum of 600 hours at 350°F.
14. Humidity Corrosion Test Spec. MIL-G-4343, 4.3.2.3	Each of the two tested bearings must have a numerical rating of 2 or less.

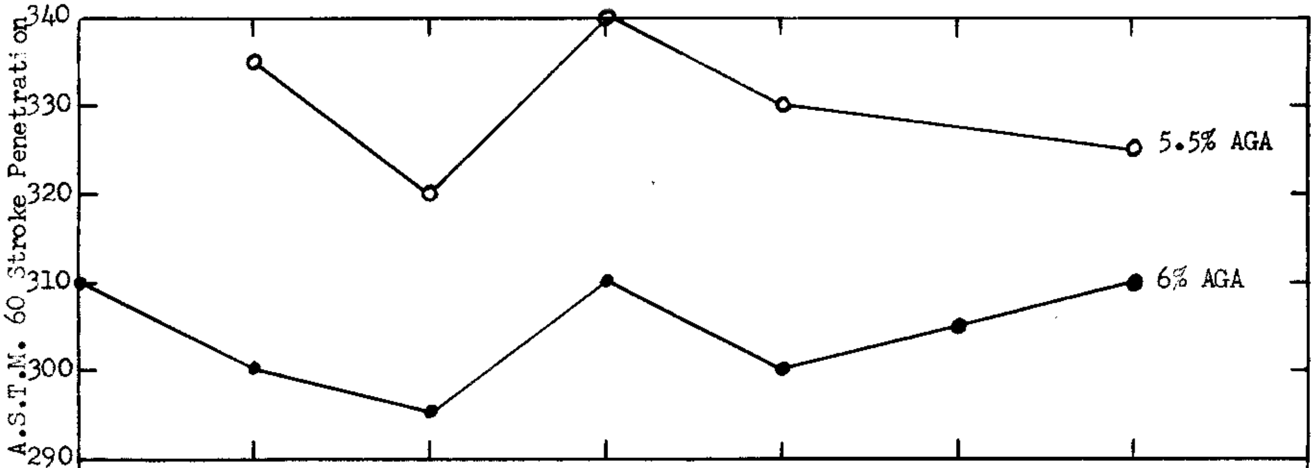

FIGURE 1 EFFECT OF AGA VS. CONVERSION ON PENETRATION

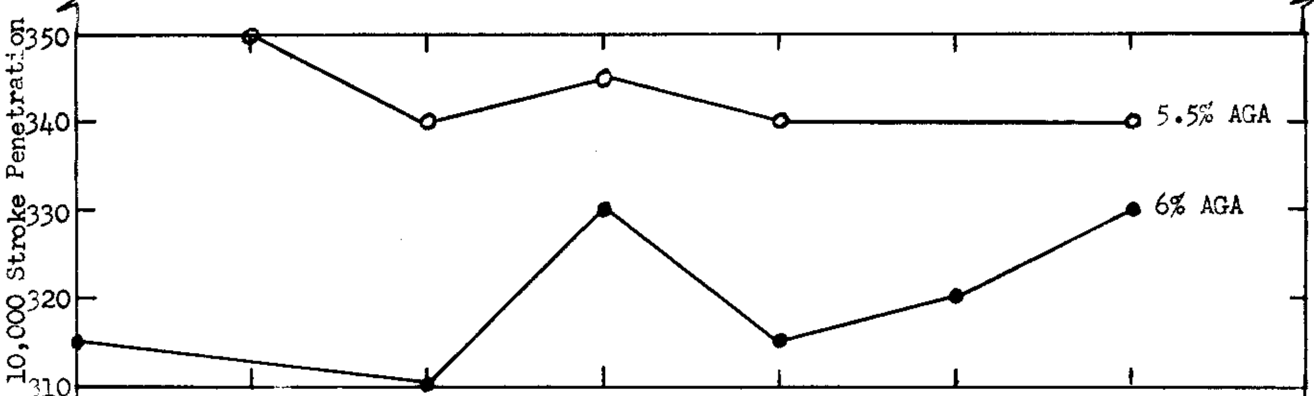

FIGURE 2 EFFECT OF AGA VS. CONVERSION ON WORK STABILITY

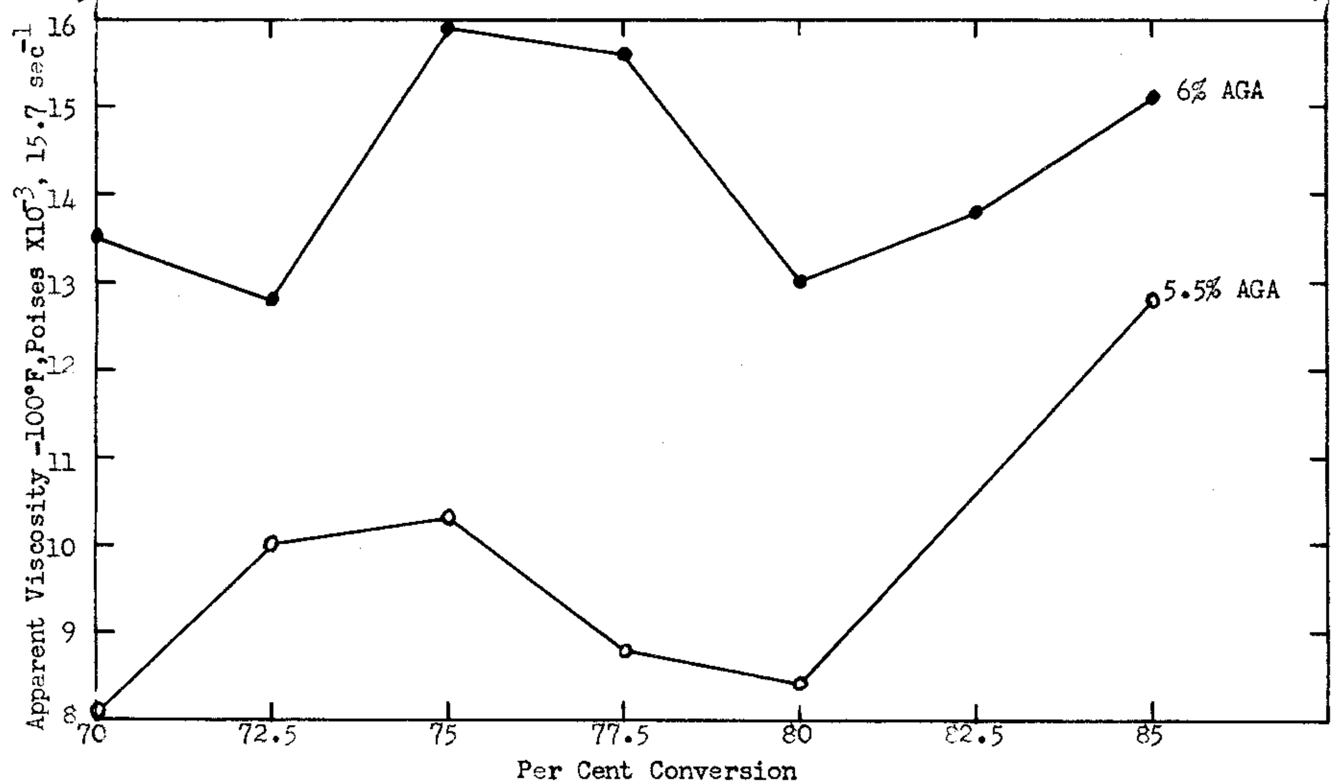

FIGURE 3 EFFECT OF AGA VS. CONVERSION ON APPARENT VISCOSITY

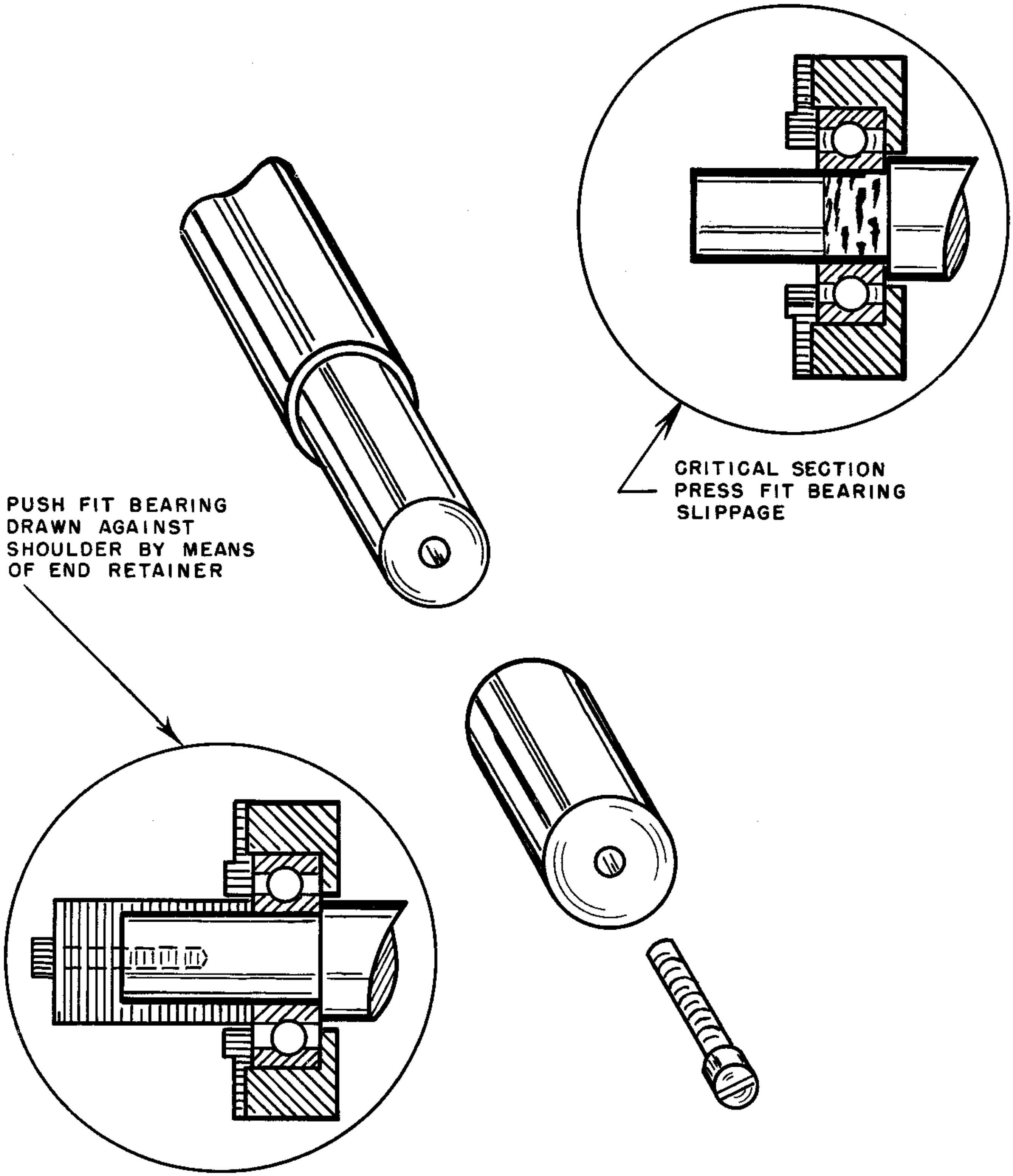

FIGURE 4 IMPROVED METHOD OF RETAINING TEST BEARING