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ASD-TDR-63-264 PART I

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DEVELOPMENT OF LUBRICANT SCREENING TESTS AND EVALUATION OF LUBRICANTS FOR GAS TURBINE ENGINES FOR COMMERCIAL SUPERSONIC TRANSPORT

PART I

TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-63-264

March 1963

Air Force Aero Propulsion Laboratory Aeronautical Systems Division Air Force Systems Command United States Air Force Wright-Patterson Air Force Base, Ohio

Project No. 648D

Supersonic Transport Research Program Sponsored by The Federal Aviation Agency

(Prepared under Contract No. AF 33(657)-9248 by the Southwest Research Institute, San Antonio, Texas: B. B. Baber, J. P. Cuellar, P. M. Ku, C. W. Lawler, C. M. Monita, H. E. Staph, authors.)

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March 1963

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ERRATA - August 1963

The following corrections are applicable to ASD-TDR-63-264, Part I, entitled "Development of Lubricant Screening Tests and Evaluation of Lubricants for Gas Turbine Engines for Commercial Supersonic Transport", and dated March 1963:

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FOREWORD

The work described in this report was performed at Southwest Research Institute, San Antonio, Texas, under USAF Contract 33(657)-9248. Project 648D, and administered by the Fuels and Lubricants Division, Air Force Aero Propulsion Laboratory, Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. Mr. G. A. Beane was the project engineer.

This report covers the work performed in the period of May 1, 1962 through February 28, 1963.

ABSTRACT

The present program was concerned with the development of lubricant screening tests and the evaluation of candidate lubricants for advanced gas turbine engines for the commercial supersonic transport. In an effort to develop the required lubricant screening tests, strong reliance was placed on the experience gained from lubricant screening tests developed for the past and current generations of gas turbine engines, but with due recognition of future requirements. These considerations led to the decision to develop the following tests: (1) lubricant oxidation-corrosion test, (2) lubricant deposits and degradation test, (3) gear load-carrying capacity test, and (4) rollingcontact fatigue test.

Under the lubricant oxidation-corrosion phase, a 425°F oxidationcorrosion test with good engine correlation was finalized, a high-temperature oxidation-corrosion test apparatus was developed, and the effect of test variables on lubricant oxidation-corrosion characteristics was studied at temperatures up to 600°F. In the lubricant deposits and degradation phase, considerable baseling data were obtained on candidate lubricants at bearing temperatures of 500 through 750°F. In the gear load-carrying capacity phase, dynamic calibrations and load-carrying capacity evaluations were conducted at gear temperatures of 400 through 700°F, and the correlation of several test methods studied. The rolling-contact fatigue work involved the development of a 3-ball/cone fatigue tester and the preparation for the performance of full bearing tests. Among the candidate lubricants evaluated, only a 5P4E polyphenyl ether appeared thermally and oxidatively stable at temperatures up to 600°F without causing significant metal corrosion. One candidate, MLO-62-1005, an ester, was stable up to 500°F, but was significantly corrosive to mild steel.

This technical documentary report has been reviewed and is approved.

Mare P Klumann

MARC P. DUNNAM Director, Fuels and Lubricants Division Air Force Aero Propulsion Laboratory

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I. INTRODUCTION

This report summarizes the work performed at Southwest Research Institute during the period of May 1, 1962 through February 28, 1963, on a program concerned with the development of lubricant screening tests and the evaluation of lubricants for advanced gas turbine engines for the commercial supersonic transport, under Contract AF 33(657)-9248, Project 648D. This program was initiated as part of a broad program of research and study to investigate the technical and economic problems related to the commercial supersonic transport being conducted with the financial support of the Federal Aviation Agency, Washington, D. C. Technical administration of this program was provided by the Nonmetallic Materials Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with the National Aeronautics and Space Administration lending basic research and technical support as needed.

The commercial supersonic transport under research study is anticipated to cruise at a speed in the neighborhood of Mach 3. At this speed, the lubricant in the sump of the lubrication system is expected to reach a temperature of approximately 500°F or higher. Further, as the lubricant flows through the engine and lubrication system, it is expected to be exposed to components made of different materials, operating at temperatures considerably in excess of 500°F. Finally, in its passage through the engine and lubrication system, the lubricant is expected to be brought into intimate contact with air. For these reasons, the oxidative, deposit-forming, and material-compatibility characteristics of lubricants subjected to high temperatures are of vital interest. In addition, due to considerations of weight and reliability, the capability of lubricants to sustain reasonably high gear and bearing loads over extended periods of high-temperature operation is also important.

Taking the problem as a whole, and drawing upon the experience on the lubricant screening tests for the current versions of aviation gas turbine engines, such as those employed in Military Specifications MIL-L-7808 and MIL-L-9236, it was decided that the aforementioned lubricant performance characteristics could be defined with reasonable adequacy by the following basic tests:

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- (1) Lubricant oxidation-corrosion test
- (2) Lubricant deposits and degradation test
- (3) Gear load-carrying capacity test
- (4) Rolling-contact fatigue tests:
 - (a) Three-ball/cone fatigue test
 - (b) Bearing fatigue test

It was realized that while the general nature of many of these tests was fairly well established for current applications, nevertheless, much needed to be done in projecting the basic concepts to meet the more severe requirements of the supersonic transport engine.

The details of the aforementioned tests and the work performed under the current program in developing such tests will be discussed in the succeeding chapters of this report. However, it is believed that some general comments will be helpful at this juncture.

The lubricant oxidation-corrosion test is a "glassware" test which exposes the test lubricant to controlled temperature conditions in the presence of selected metal specimens and in the presence of dry air. This test is used to determine the viscosity and acidity changes of the test lubricant, as well as the lubricant's corrosive effect on the test metals. The principal advantages of the glassware approach are its simplicity, economy, and the very modest quantity of test lubricant required. It is therefore ideally suited to quick and broad evaluations of experimental lubricants and test variables.

The lubricant deposits and degradation test is conducted in a roller bearing test machine, which subjects the test lubricant to a controlled temperature, in the presence of selected metal specimens and in the presence of humidified air. While the primary purpose of this test is to determine the deposit-forming tendency of the test lubricant, it also provides information or the lubricant's viscosity and acidity changes as well as its effect on metal orrosion. As this test is conducted under dynamic conditions using a complete bearing, the data obtained are generally considered to lend added confidence to those furnished by the oxidation-corrosion test. On the other hand, since this test is elaborate and expensive and requires a large quantity of test lubricant, it is usually conducted only on those lubricants that have shown promise in the oxidation-corrosion test. The gear load-carrying capacity test is conducted in a spur gear test machine, under controlled conditions. The purpose of the test is to determine the gear load-carrying capacity of the test lubricant—a property which cannot be determined with much confidence without resorting to the use of gears.

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Rolling-contact fatigue is a critical problem in extended engine operation, and there is evidence that lubricants exert a strong influence on rollingcontact fatigue. In the current program, the evaluation of lubricants with respect to rolling-contact fatigue is being accomplished in two ways. The 3-ball/cone fatigue test involves the use of a conical test specimen in rolling contact with three equally spaced balls. The design of the tester is such that it is particularly suitable for quick and broad evaluations of experimental lubricants and test variables. The bearing fatigue test employs complete ball bearings as the test specimens. However, it is elaborate and expensive, and is thus intended primarily to furnish baseline data with which to correlate the results from the 3-ball/cone fatigue test and other related fatigue programs.

In the development of the test methods, a consideration that must not be overlooked is the soundness of the basic concepts behind the tests. Clearly, the lubricant screening tests will not serve their purposes if they do not portray with substantial realism lubricant performance in the engine for which they are intended. Here, as the supersonic transport engine is not presently available, it may appear that there is no firm basis to judge the soundness of the basic concepts of the tests under development. Fortunately, this problem is not as serious as it may seem. The development of the jet aircraft and the gas turbine engine has been a process of continuous evolution. During this process of evolution, much valid experience has been gained. The evolution of Military Specifications MIL-L-7808 and MIL-L-9236 clearly shows that the basic concepts of the lubricant screening tests contained therein are essentially valid, even though the test devices and test conditions must be tailored to meet the specific applications. The test methods being developed in the current program draw heavily upon the basic concepts of the existing tests, but with due allowance being made for the supersonic transport engine requirements. As a matter of added precaution, a substantial amount of the initial experimental effort has been devoted to the 425°F temperature level, at which engine test data are available. Following this, tests are then made with temperature increased in steps to as high a level as the test lubricants permit, in order to gain an overall understanding of the effect of temperature which, it is felt, will aid materially in the test method development.

Another consideration which should be borne in mind is that, except for general guidelines such as the approximate temperature range and the presence of air and different metals, the specific design and operating details of the supersonic transport engine are not known at this time. Therefore,

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there is presently no way to tailor the screening tests to account for the specific details of the engine. The only logical approach is thus to develop tests of rather basic character, such as those already discussed, which will accommodate a broad spectrum of conditions likely to be encountered in the future engine. The performance of candidate lubricants will be established by such tests over a wide range of conditions. Later, as the engine becomes available and its specific details become known, the performance data of the candidate lubricants will then be examined, in the light of such specific details, to determine their suitability.

It was stated earlier that the objectives of the present program were to develop lubricant screening tests for the supersonic transport engine and to evaluate candidate lubricants with these tests. These two tasks are related at least in the sense that the validity of the tests must be established at the temperature level and under comparable operating conditions to be encountered in the supersonic transport engine. Quite apart from the availability of the engine itself as already discussed, the availablility of candidate lubricants for the supersonic transport engine is, at present, very limited. For this reason, much of the experimental work to date has been confined to establishing the baseline performance of a 5P4E polyphenyl ether, a fluid of known hightemperature stability, while the exploration of the effect of lubricants must, of necessity, be made with available production-type and experimental lubricants which may or may not prove suitable for the supersonic transport application. Evaluation of candidate lubricants will be undertaken as they become available. Meanwhile, it is believed that the data obtained on the available lubricants will aid as a whole in perfecting the test techniques. A listing of the test lubricants employed in this program to date, together with pertinent available information on them, is given in Appendix I. The properties of the 5P4E polyphenyl ether (F-1041) used to obtain baseline performance data are presented in Appendix II.

In closing these introductory remarks, it appears pertinent to state that considerable experience has been accumulated at Southwest Research Institute in the past nine years in lubrication research and lubricant test method development for aviation gas turbine engines, under Contracts AF 33(616)-498, AF 33(616)-2659, AF 33(616)-3820, AF 33(616)-6232, and AF 33(616)-7223. The work performed under these programs, already described in several summary technical reports $(1-5)^*$, has been of very material benefit to the program at hand. Further, although certain major test devices must be designed and constructed especially for the current

*Superscript numbers in parentheses refer to the List of References.

program, a large number of those previously developed and already available at SwRI have, with suitable modifications and refinements, been proven applicable. Major test devices that have been adapted for use in the present program include an oxidation-corrosion test apparatus with a liquid bath, two Erdco roller bearing machines, two WADD high-temperature gear machines, and two WADD ball bearing machines. Major test devices specifically developed under the present program include a high-temperature oxidationcorrosion test apparatus and a 3-ball/cone fatigue tester.

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II. LUBRICANT OXIDATION-CORROSION (500°F AND BELOW)

A. General Remarks

The overall objectives of the lubricant oxidation-corrosion phase of the program were to develop apparatus and procedures for determining the oxidation and corrosion characteristics of high-temperature gas turbine lubricants and to evaluate the oxidation and corrosion characteristics of candidate lubricants under environmental conditions representative on Mach 3 class gas turbine engine designs.

For the sake of convenience, this phase will be discussed in two parts: Part I pertaining to studies in the 425 through 500°F region and Part II (see the next chapter) concerned with studies at temperatures at and above 500°F. This division is made mainly because of the differences in the test apparatus used. The oxidation-corrosion apparatus dealt with in this chapter was developed earlier under Contract AF 33(616)-7223 primarily for operation at 425°F, in connection with the development of screening tests for MIL-L-9236 lubricants⁽³⁾. For that work, a liquid bath with a maximum temperature capability of 500°F was considered adequate. With the initiation of the current program for supersonic transport engine application, it was clear that another test apparatus with higher temperature capability would be required; thus the design and construction of the high-temperature apparatus was immediately initiated. Meanwhile, it was felt that much useful background information, of direct benefit to the current program, could be gained by completing a cooperative test program already started on an 18-hr 425°F oxidation-corrosion test and by studying the behavior of selected lubricants in the 425 to 500°F temperature range with the existing apparatus. Such data, it was felt, would verlap the data to be obtained in the lower temperature range of the hightemperature investigations, and would therefore be valuable in comparing with and in interpreting the high-temperature results.

This chapter will deal primarily with the work performed under the current program in the 425 to 500 °F range using the oxidation-corrosion apparatus with the liquid bath. However, for the sake of continuity, important aspects of the work previously conducted under Contract AF 33(616)-7223 will also be discussed.

Twenty lubricants were evaluated with the 18-hr 425°F oxidationcorrection tests. Twelve of these lubricants had previously been engine-tested at 425 F by ASD – A good correlation between the oxidation-correction test and the engine test was shown. A cooperative test program on the 18-hr 425°F exidation-correction test was undertaken by five laboratories. Good reproductibility was obtained



Eleven lubricants were evaluated to determine their breakpoints with respect to oxidation stability by increasing the test temperature from 425° F, in 25° F increments, until the viscosity measured at 100° F was increased by 100 percent. Seven of the lubricants proved to be unsatisfactory at 450° F, two failed at 475° F, and one failed at 500° F. Only one of the lubricants showed very good oxidation stability up through 500° F; however, this lubricant exhibited significant corrosion of the mild steel specimen at the higher test temperatures.

B. <u>Test Apparatus</u>

A detailed description of the test apparatus used in this phase of the oxidation-corrosion work is presented in test method form in Appendix III. Briefly, a 350-ml lubricant sample is placed in a 64-mm glass sample tube containing five 1 in. X 1 in. square metal specimens of aluminum, silver, titanium, steel, and stainless steel. The tube is equipped with a sidearm head and placed in a stirred and thermostated oil bath. The bath is normally maintained at 425°F; however, bath temperatures of 450, 475, and 500°F have also been used in this program. Provision is made for bubbling clean, dry air through the lubricant sample. The overhead vapors leave the tube through the head sidearm and are condensed. The condensate is not returned to the sample tube but is collected separately.

C. Test Procedures

1. 18-Hr 425°F Oxidation-Corrosion Test

The procedure for the 18-hr 425° F oxidation-corrosion test is described in detail in Appendix III. Briefly, a 350-ml sample of the test lubricant is placed in the sample tube containing the five specified metal specimens. The bath temperature is maintained at 425° F, and clean, dry air is continuously bubbled through the lubricant sample at a rate of 236 g/hr (equivalent to 197 liters/hr measured at 70° F and 760 mm) during the entire 18-hr test period. Terminal viscosity and neutralization number determinations are made on the lubricant sample, and weight changes of the metal specimens are noted. The overhead condensate, which is not returned to the sample tube, is collected, weighed, and evaluated for neutralization number. All viscosity determinations are made at 100° F.

2. 18-Hr Modified Oxidation-Corrosion Test

The 18-hr modified exidation-corresion test is conducted in identical manner as the 18-hr 425°F exidation-corresion test, except that the bath temperature is maintained at 450, 475, or 500°F, as required.

D. Development of 18-Hr 425" F Oxidation-Corrosion Test

The 18-hr 425° F oxidation-corrosion test is an evolution of a test originally developed by the Celanese Chemical Company⁽⁶⁾. From engine tests conducted at 425° F at ASD, it was found that a major limiting factor in lubricant service was oxidative degradation, especially viscosity increase In the course of development of a 425° F oxidation-corrosion test that would correlate with the 425° F engine test in oxidative degradation, it was necessary to conduct a study of the effect of several test variables. This study led to the finalization of the 18-hr 425° F oxidation-corrosion test in its present form which, except for the use of the same test apparatus, differs drastically from the Celanese test in test conditions and procedure. Much of the initial exploratory work on this test has already been reported⁽⁵⁾ and will not be repeated here This report will only relate the results of the study on the effect of test variables. since they form the background for the development of the 18-hr 425° F oxidation-corrosion test.

Tests were first conducted at 425°F bath temperature, at 96 liters/hr air flow. The metal specimens were either aluminum, silver, titanium, steel, and copper, or with copper omitted. The initial lubricant sample volume was varied from 250 to 350 ml. The test duration was 50 hr, with intermediate sampling (20 ml) every 5 hr (after 15 hr) for viscosity and neutralization number determinations. Different oil makeup quantities were introduced during the sampling periods: no makeup, makeup for only the overhead losses, and makeup for both overhead losses and the 20-ml samples.

The effect of several test modifications on oil viscosity change is shown in Figure 1 for oil O-60-23. When no oil makeup was provided, severe deterioration occurred after approximately 25 hr, as illustrated by curve 7. In general, the effect of the other factors investigated was negligible for this particular oil. The presence of copper caused a slight reduction in the rate of viscosity increase (curves 1, 2, 3 vs curves 4, 5, 6). Some minor improvement in performance was also evident for O-60-23 when using the larger starting sample volume and/or extending the oil makeup procedure to include the 20-ml samples. A study of Figure 1 reveals that a terminal viscosity that would approximate the 19.7 cs shown by the engine test could be obtained either by a shorter test with no oil makeup, or by the 50-hr test with oil makeup. In the latter case, some improvement in correlation would be obtained by eliminating the copper specimen.

The results of a similar g: oup of tests conducted on oil Q-60-27 are shown in Figure 2. Final viscosity on O-60-27 in the engine test was 27.7 cs. The 50-hr oxidation-corrosion test consistently gave higher terminal values of about 43 cs (curve 4). Comparison of the test with and without copper



FIGURE 1. EFFECT OF TEST VARIABLES ON 0-60-23

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. . . .



FIGURE 2. EFFECT OF TEST VARIA, LES ON 0-60-27

(curve 4 vs curve 1) reveals the extreme effect exhibited by the metal on this particular oil. Terminal viscosity without copper was approximately 21 cs. From the data presented, several additional observations on oil performance can be made. A larger starting sample or the addition of sample makeup served to depress the viscosity curve (this effect was also noted with O-60-23). A marked increase in oil deterioration resulted when no makeup for losses or samples was accomplished even though copper was not present (curve 7 vs curve 2). Thus, it was apparent that the major factor contributing to poor correlation of the earlier tests on $O-60-27^{(5)}$ was the copper specimen. In this case, the catalytic action of the metal completely overshadowed all other performance characteristics of the lubricant.

The results of similar tests conducted on oil O-60-12 are presented in Figure 3. Although some depression of curves 2 and 3 was noted as a result of copper elimination and sample makeup, the oil was quite insensitive to the test changes.

Figures 4 and 5 present data on oils O-60-13 and O-58-24, respectively, showing the effect of omitting copper and using complete makeup for losses and samples, in comparison with the normal procedure. The modified test on O-60-13 exhibited a gradual rise in viscosity with no tendency toward accelerated deterioration such as that shown by the normal test procedure, in which a sharp increase was noted after 30 hr. Final viscosity for the latter test at 50 hr was 62 cs. This value was considerably lower than the 91 cs reported in the engine test on O-60-13. For O-58-24, both procedures gave a continuous and rapid viscosity increase, with the modified procedure slightly less severe.

The general effect of the various test modifications on neutralization number were essentially identical in nature with those observed for viscosity increase for all of the oils investigated. For the sake of brevity, 'the detailed results will not be presented here.

At this point in the program, an evaluation was made of the effects of various procedure modifications in order that conditions could be applied to improve correlation with engine test results. As previously discussed, O-60-27 could not be suitably aligned with engine data so long as a copper specimen was included in the test. Another change in procedure was the decision to eliminate the makeup of oil. Results had indicated that the oil makeup in the oxidation-corrosion test had a drastic influence on some lubricants, and it was hoped that by elimination of oil makeup, O-60-13 and O-58-24 might be brought more in line with the engine test results. These two oils, particularly O-60-13, had not shown the severe extent of deterioration using the earlier 50-hr test as was reported in the engine test.



FIGURE 3. EFFECT OF TEST VARIABLES ON 0-60-12



FIGURE 4. EFFECT OF TEST VARIABLES ON 0-60-13



FIGURE 5. EFFECT OF TEST VARIABLES ON 0-53-24

Excluding the oil makeup and the copper specimen necessitated a third change in test conditions. O-60-23 would be moved considerably out of correlation with the engine test as a consequence of these procedure modifications (see curve 7, Fig. 1). As a result, the test duration was shortened to 24 hr to accomodate the sharp increase in viscosity of O-60-23 under such conditions. Exploratory runs under these conditions confirmed the improvement in engine correlation for all oils except O-60-13, which was moved farther out of line with regard to engine data. A terminal viscosity of 34 cs was obtained, whereas engine test showed 91 cs.

At this time, an investigation was made of the effect of air flow rate. The test oil volume was increased from 250 to 350 ml to offset the increased oil losses expected at the higher air flows. Oil O-60-13 was tested at 48_{i} 96 (standard air rate at that time), 144, and 197 liters/hr. The results obtained are graphically illustrated in Figure 6. Examination of the figure shows that this oil was very sensitive to air flow rate, with both neutralization number and viscosity rising rapidly at air flow rates in excess of about 120 liters/hr.

The data obtained on O-60-13 at 197 liters/hr air flow and 24 hr test time very closely approximated the level of deterioration shown by the engine. A similar test on five other oils under investigation was run at 197 liters/hr. None of the lubricants included in these tests showed the sensitivity to air demonstrated by O-60-13. Examination of the results indicated that a 24-hr test at an air rate of 197 liters/hr would give a suitable lineup of the oils, and several tests were run on the eleven oils in this program using these conditions. However, these data will not be discussed, as later developments necessitated reducing the test time to 18 hr.

Since earlier work had indicated the extreme sensitivity of some oils to the presence of copper, all of the 24-hr tests were conducted with only four metal specimens; aluminum, titanium, silver, and steel. With four specimens, the absence of a "diagonal" in the wired assembly resulted in a very loose and unstable configuration. In an effort to provide a positive specimen mounting method, two tests were run using stainless steel wire racks which held the specimens vertically in the oil sample. The specimens were arranged in the form of a square, each specimen contacting only the stainless steel rack. The entire assembly slipped over the glass air delivery tube and was positioned about 1.5 in. from the bottom of the test tube. Specimen assembly was considerably easier when using the rack, in comparison with the wire-tying method. However, several of the oils tested showed more severe deterioration when using the rack than when using the wired specimens. In addition, metal specimen corrosion was quite severe when using the rack. Even titanium and aluminum were attacked, which had never been observed in any previous tests. It was concluded that the most probable cause for the test irregularities noted was the presence of crevices in the spot-welded joints of the rack, which would trap contaminants during cleanup of the apparatus.



NEUT NO., MG KOH/G, After 25 hr test time

17
In view of more urgent problems in test standardization, the use of a rack was discontinued. All subsequent tests were then run with a wired fivespecimen set, adding a stainless steel specimen to the assembly as illustrated in Figure 53 in Appendix III.

Upon adding the stainless steel specimen, a rigorous check of all operating methods was made in order to finalize the test procedure. A discrepancy was noted in the bath temperature, due partially to a significant error introduced in the thermometer reading by the relatively high temperature of the air space between the bath oil and the bath cover. It was found necessary to apply a <u>partial</u> emergent-stem correction to the thermometer readings. This correction resulted in a net increase in bath temperature of about 2°F; i. e., the bath previously had been running low. Subsequent tests at the verified bath temperature of 425°F indicated a higher level of severity. O-60-23 was most seriously affected by the temperature change, and was moved badly out of correlation with engine data. O-60-23 has consistently been the most critical oil of the group, and its performance has been the limiting factor in severity of the tests developed.

A series of tests was run to establish the reduction in test time required to bring O-60-23 back into line; it was determined that at 18 hr, the viscosity was still at a reasonable level. This series also served to illustrate the accelerated deterioration characteristic of O-60-23 once its induction period had been exceeded:

0-60-23	<u>Vis, cs/100°F</u>
New Oil	16. 0
18 hours	20.4
20 hours	31, 7
22 hours	39.4
24 hours	61.9
100- hr engine test	19. 7

A limited study was also made to determine whether any effect in test severity was contributed by the stainless steel specimen, which had been added at the time of the bath temperature correction. Based on very limited data from 24-hr tests on O-60-23 and O-59-26 with and without stainless steel, no significant effect of the metal could be noted.

E. 18-Hr 425° F Oxidation-Corrosion Test Results

1. Test Results

Using the 18-hr 425° F oxidation-corrosion test procedure, twenty lubricants submitted by ASD were evaluated. A summary of the results obtained is presented in Tables 1 and 2. Table 1 presents a summary of the test data on oil deterioration. Although no more than four tests were run on any of the oils, it is apparent from this table that the test repeatability was excellent, particularly with respect to oil viscosity change. O-58-24 and MLO-61-1014 exhibited the largest variance in viscosity between individual tests, however, this is not considered excessive in view of the very high level of deterioration in both cases.

Three oils listed in Table 1 were each represented by two or more different batches of the same nominal formulation. Test results on O-61-17 and O-60-12 checked each other closely, as did MLO-61-1011 and O-60-23. However, O-61-19 and O-60-27 gave very dissimilar results. Terminal values for O-61-19 in viscosity and neutralization number were considerably higher. The differences were so large as to be far beyond the normal accuracy of the test as exhibited by all other lubricants evaluated; thus it is felt that the only explanation lies in some batch-to-batch variation for this one lubricant.

Table 2 lists the average of all results obtained on metal specimen corrosion. As shown, all of the oils gave little or no corrosion; the only appreciable attack was on silver by O-60-13, O-61-19, and MLO-61-1014.

2. <u>Test Reproducibility</u>

An evaluation of the reproducibility of the 18-hr 425°F oxidationcorrosion test was made by SwRI in cooperation with four other laboratories. Tests were performed on three selected lubricants (O-60-13, O-60-23, and O-60-27) using the test method described.

Two determinations on each fluid were initially made by the participating laboratories. All laboratories showed very good test repeatability. The average results from the duplicate tests are given in Table 3. As to test reproducibility, note that Laboratories C and D were considerably less severe in the results reported for O-60-13, which showed rather extensive deterioration in tests conducted by the other three organizations. Test reproducibility for oils O-60-23 and O-60-27 was satisfactory, although Laboratories C and D again obtained a lower level of deterioration. TABLE 1. SUMMARY OF 18-HOUR 425°F OXIDATION-CORROSION TEST RESULTS

Acidity, mg KOH/g(a) Overhead 29.8 9.7 3.7 27.1 5.6 80.4 4.3 55.7 20.9 24.9 12.5 9.8 123.4 2.0 2.7 4.4 28.2 0.7 1.5 2.9 Loss, g^(a) Total Oil 71.2 91.5 169.3 84.3 112.5 137.5 73.5 82.6 86.2 32.2 116.3 82.2 90.5 118.5 114.7 143.7 141.0 164.0 122.0 124.0 17. 3, 16. 6, 16. 95 1.1,0.9,1.3,0.9 0.4,0.6,0.4,0.4 Neut. No., mg KOH/g 21. 3, 20. 6, 15. 5 19.8, 29.8, 13.3 9.9,9.3,13.0 7.4, 6.8, 6.2 3. 6, 3. 6, 3. 0 1.8, 2.1, 1.2 5.2,4.5,4.5 0.8,0,9,0.7 1.4,0.6,1.0 9.3,9.3,9.9 Final 0.10,0.10 0.4,0.5 5.27 5.97 0.12 0.16 5.0 Initial 0.14 0.05 0.09 0.14 4.12 0.03 0.02 0.14 0.04 0.05 0.07 0.05 0.06 0.09 0.02 0.04 0, 14 0.14 0.03 0.11 19.4, 19.6, 19.8, 19.8 17.5, 17.4, 17.5, 17.8 189. 5, 158. 2, 204. 4 493. 5, 472. 1, 445. 5 Viscosity, cs at 100°F 91.5,100.4,92.5 44.9,40.0,39.7 28.0, 28.0, 29.9 20.4, 20.3, 20.4 22. 6, 22. 6, 22. 8 24. 3, 23. 4, 23. 7 19.3, 19.5, 19.4 20.0, 20.0, 20.2 13. 6, 49. 8, 44. 8 Final 17.7, 17.8 15.4, 15.1 28.0 29.7 25.6 15.9 15.6 Initial 34.7 18.7 18.7 17.2 21.2 25.7 20.8 15.9 15.6 16.1 16.0 15.0 16.1 5. 7 16. 2 15.0 15.3 24.4 14.7 ŝ Tests No. of MLO-61-1011(d) MLO-61-1012 MLO-62.1006 MLO-61-1013 MLO-61-1014 Oil Code Lot 231-C Lat 261-C Lot 291-G (9)21-19-0 (>)61-19-0 0-59-15 0-59-26 0-60-23 O-60-28 0-60-12 C-60-13 61-09-0 0-60-27 0-58-24 0-60-11 0-60-3

(a) Average values

(b) Same formulation as 0-60-12.

(c) Same formulation as O-60-27.

(d) Same formulation as O-60-23.

		M	letal Weight	: Change	mg/cm ²	
Oil	Number				Stainless	
Code	of Tests	Aluminum	Titanium	Silver	Steel	Steel
O-58-24	3	+0.02	+0.01	-0.01	+0.01	+0.02
O-59-15	4	0.00	-0.01	-0.02	+0.03	+0.03
O-59-26	3	0.00	+0.01	-0.10	+0.01	+0.04
O-60-3	3	+0.01	0.00	-0.01	+0.01	+0.02
O-60-11	3	0.00	+0.01	-0.01	+0.02	+0.02
O-60-12	4	-0.01	-0.01	-0.06	-0.01	0.00
O-60-13	3	+0.01	0.00	-0.24	+0.01	+0.02
0-60-19	3	+0.01	+0.01	-0.01	0.00	+0.03
O-60-23	3	0.00	-0.01	-0.06	0.00	+0.01
O-60-27	3	0.00	+0.01	-0.02	+0.02	+0.04
O-60-28	3	-0.01	0.00	-0.04	+0.01	+0.02
O-61-17(a)	2	0.00	+0.02	-0.02	+0.02	+0.04
O-61-19 ^(b)						
Lot 231-G	3	-0.01	-0.01	-0.41	-0.02	+0.03
Lot 261-G	1	-0.05	-0.02	-0.09	+0.05	+0.02
Lot 291-G	1	-0.02	-0.01	-0.16	0.00	+0.01
MLO-61-1011(c)	1	+0.02	-0.02	-0.09	-0.05	-0.05
MLO-61-1012	1	-0.01	0.00	-0.13	-0.02	-0.02
MLO-61-1013	1	+0.02	+0.02	-0.05	-0.01	0.00
MLO-61-1014	3	+0.03	+0.03	-0.37	+0.06	+0.04
MLO-62-1006	2	-0.01	-0.02	-0.09	-0.02	-0.01

TABLE 2. AVERAGE METAL SPECIMEN WEIGHT CHANGE FOR18-HOUR 425°F OXIDATION-CORROSION TESTS

(a) Same formulation as O-60-12.

(b) Same formulation as O-60-27.

(c) Same formulation as O-60-23.

a second conversion

	Sam	ple	Samp	ole	Overhead	
	Vis, cs	/100°F	NN, mg	KOH/g	NN, mg	Oil
Laboratory	Initial	Final	Initial	Final	KOH/g	Loss, g
			0-60-13			
SwRI	25.7	94 . 8	0. 02	10.7	80.3	71
Α	25.5	72.4	0. 02	10.5	101.1	59
В	25.6	97.1	0.01	13.8	9 9. 5	73
С	25.0	29.2	0.12(1)	0.9	6. 7	36
$C (rerun)^{(2)}$		85.3		10.8	85.4	75
D	25.5	30.0	0. 02	0.6	10.6	28
D (rerun)	21.8	24.6	0. 02	0.4	3.5	42
			0-60-23			
SwRI	16.0	23.8	0. 05	4.7	20. 9	86
А	16.1	20.2	0.07	4.3	13.2	75
В	16.1	178	0.02	0.5	4.4	70
C	15.7	17.4	0.13(1)	0.4	3. 7	51
$C (rerun)^{(3)}$		16.9		0.5	2.0	72
D	16.0	17.4	0.05	0.3	5.7	60
D (rerun)	16.0	16.9	0. 03	0.6	6.8	58
			0-60-27			
SwRI	15 0	19.4	0. 07	0.8	2.0	144
А	15.0	19.2	0.12	1.8	2.3	139
В	15.0	18.9	0. 02	0.8	2.0	140
Ĉ	14.7	17.5	0.19(1)	0.7	2.0	117
C(rerun)(3)	* *	17.8	. .	0.8	3. 5	135
D	15.0	17.9	0.10	0.6	2.8	119
D (rerun)	14.9	17.0	0.07	0.6	1. 7	114

TABLE 3. REPRODUCIBILITY OF 18-HOUR 425°F OXIDATION-CORROSION TEST

(1) ASTM D 974 Colorimetric Method (all others by potentiometric method).

(2) Test run in dry-well aluminum block.

(3) Test run in wet-weil aluminum block.

Details concerning the conduct of the tests were solicited from all participating laboratories and, although several minor deviations in procedure were noted, no explanation was apparent for the low values obtained by Laboratories C and D The most probable cause of differences between laboratories appeared to be variations in sample operating temperature; however, in this work, the highest sample temperature reported was by Laboratory C.

The three lubricants were subsequently retested by Laboratories C and D. These results are also given in Table 3. Duplicate determinations were made on O-60-23 and O-60-27; however, only single det rminations were run on O-60-13 due to the paucity of oil sample on hand. It will be noted that the rerun values obtained by Laboratory C indicated a much higher level of severity than previously reported for O-60-13. These results showed very good agreement for all laboratories except D. Laboratory C reported that a close investigation of operating temperatures revealed that the tests performed initially had been run at a sample temperature estimated to have been approximately 10 to 15° F lower than originally reported.

It should be noted that, following the usual lubricant testing practice: the test procedure called for control of the bath temperature but not the sample temperature. However, the cooperating laboratories were requested to measure this temperature on a "dummy" oil sample at the specified bath temperature of 425°F and air flow rate of 197 liters/hr. Laboratory C, which used a dry-well aluminum-block heat medium in this work, found that the lubricant selected for making the temperature check was some 10°F higher due to the heat of oxidation. To overcome this complication, nitrogen was used in measuring the sample temperature for the rerun test instead of air. The range of sample temperatures reported at the specified air flow of 197 liters/hr is given by the following tabulation for all participants:

Labora ory	Heat Medium	Temperature. *F
SwRI	Ori bath	422-423
· A	Oil bath	421-422
B	Aluminum block	415-416
С	Aluminum block	410-415 (estimate)
C (rerun)	Aluminum block	425-426
D	Oil bath	422-423

These values were obtained with the heat medians at 425°F except in the case of Laboratory C which elected to operate the aluminum block at 428°F in the initial tests and at 433°F in the rerun tests. Although not reported, it is assumed that the sample temperature initially obtained by Laboratory D applied also for the rerun tests.

While the lack of test reproducibility shown by the original data of Laboratory C could be attributed to the low sample temperatures, no clarification of results was forthcoming in the case of Laboratory D. Repeat tests by this laboratory were in good agreement with its previous tests for oils O-60-23 and O-60-27. For the lubricant of most interest, O-60-13, the results were again much less severe than those obtained by the other participants. However, there was some doubt concerning the purity of the oil sample used in this repeat test. Laboratory D reported an initial viscosity for O-60-13 of 21.8 cs at 100°F, which was considerably lower than all reported values for the new oil. A portion of the sample was returned to SwRI and a viscosity measurement also gave a value of 21.8 cs. A sample was then taken from the original container in which the oil had been stored at SwRI. A value of 25.7 cs was obtained, which was in agreement with previous experience. Unfortunately, Laboratory C did not obtain initial oil properties in their repeat rests and, upon request, reported that insufficient oil was available to perform a viscosity check on O-60-13. Thus, it appears that the integrity of the oil sample used by Laboratory D in the repeat test had in some way been compromised, and the test data must be considered suspect.

In an effort to obtain additional indications of the repeatability and reproducibility which could be expected from the 18-hr 425°F oxidationcorrosion test, tests were performed to determine the effect of relatively small changes of air rate and temperature, such as might be encountered with poor control of test conditions. Table 4 gives a comparison of standard 425' F data with those obtained in tests 5" F above and below the normal bath temperature. It will be noted that most of the oils were very sensitive to minor temperature changes at this level. Of the six oils selected for this work, O+60-11 was the only one indicating little or no effect of bath temperature within the ±5°F range studied, while the other oils were all affected significantly by the $\pm 5'F$ temperature change. It is evident that temperature must be controlled to a much closer tolerance than #5"F in order to achieve reasonable reproducibility of results. For the most sensitive oil, 0-60-23, a $\pm 5^{\circ}$ F change in bath temperature would destroy completely the correlation. with engine test results (discussed in the next section). Although this pronounced temperature sensitivity of the test is unfortunate, it is believed to be inherent in the nature of the oils and the temperature level.

The effect of a 10 percent variation in air flow rate was also investigated. In this case, as illustrated by the data in Table 5, the effect on test results was negligible. O-38-24 and O-50-13 gave a measurable change in viscosity at the higher air rate of 217 liters/hr; however, the level of the change is not considered serious.

	OXIDATION-CORROSION TI	EST RESULT	rs	
	Final Viscosity, cs at 100°F,	Final Net	it. No., m	g KOH/g,
Oil	with Indicated Bath Temp, °F	with Indi	cated Bath	Temp, °F
Code	420 425(a) 430	420	425(a)	430

270

81

34

162

46

20.4

20

10

15

6.2

3.6

0.5

TABLE 4. EFFECT OF BATH TEMPERATURE ON 18-HOUR

(a) Average of three standard tests.

161

31

19

61

18

20.5

184

42

29

95

24

20.3

0-58-24

0-59-26

0-60-3

0-60-11

O-60-13

0-60-23

°F

27

13

20

20

4.8

9.4

17

19

11

6.8

3.4

4.7

TABLE 5. EFFECT OF AIR FLOW RATE ON 18-HOUROXIDATION-CORROSION TEST RESULTS

Oil Code	Final Vi with Indica	scosity, cs ated Air Rat	at 100°F, te, liters/hr	Final Neut with Indicate	. No., n ed Air Ra	ng KOH/g, ate, liters/hr
	177	197(a)	217	177	<u>197(a)</u>	217
O-58-24	174	184	205	18	17	17
O-59-26	42	42	54	8.8	6.8	8.2
O-60-3	27	29	31	24	19	26
O-60-11	20	20	21	(b)	3.4	3.0
O-60-13	91	95	134	22	11	15
O-60-23	23	24	24	6.4	4.7	5.1

(a) Average of three standard tests.

(b) Sample lost in handling.

3. Correlation with Engine Test Results

Engine tests were conducted by ASD at 425°F sump temperature on twelve of the twenty lubricants evaluated in the 18-hr 425°F oxidationcorrosion test. Using the engine test data supplied by ASD, an evaluation was made of the correlation between engine and the oxidation-corrosion test results. A summary of all test results used in this correlation is given in Table 6. It should be noted that of the engine tests listed in the table, only three oils completed the 100-hr test.

The correlation of viscosities between the engine test and the oxidation-corrosion test is given in Figure 7. Data points are plotted for percent viscosity increase in each case. As evidenced by the figure, the correlation for the three oils that completed the 100-hr engine test was good. As for the remaining oils, most should yield better correlation than indicated, if the engine tests were carried to completion thus giving higher final viscosities from the engine tests. Judged in this light, the only instances of poor correlation were O-58-24 and O-60-28. However, considering the nature of the engine test, it is felt that the overall correlation was quite satisfactory.

The correlation of neutralization numbers between the oxidationcorrosion test and the engine test is shown in Figure 8. The scatter of data points was somewhat greater than that shown in the viscosity correlation. Applying similar reasoning as before, the major instances of poor correlation were O-58-24, O-60-19, and O-60-28. Even so, considering the nature of the engine test, the overall correlation was quite reasonable.

4. Effect of Copper Specimen

It was shown in the study of test variables that the use of a copper specimen adversely affected the correlation between the oxidation-corrosion test and the engine test. However, this effect was noted before the 18-hr 425° F oxidation-corrosion test was finalized, and needed to be verified under the test conditions as finalized. For this purpose, six of the oils already evaluated by the standard test were run under otherwise identical conditions except for the use of a copper specimen in place of the stainless steel specimen normally used. Table 7 compares the data so obtained with those from the standard oxidation-corrosion test and the engine test. Note that the final viscosity results substantiated the sarlier findings. The correlation with engine test was, on the whole, less satisfactory with the copper specimen present. In particular, the test with copper did not properly separate O-60-27and O-61-19, whereas both the engine test and the standard oxidation-corrosion test indicated that O-61-19 deteriorated much more than O-60-27.

 TABLE 6. COMPARISON OF 18-HOUR 425°F OXIDATION-CORROSION

 TEST RESULTS WITH 425°F ENGINE TEST DATA

	Initial F	roperties	18-hr 425°	F O-C Test(a)	42	5°F Engine 1	lest
Oil Code	Vis, cs at 100°F	Neut. No. , mg KOH/g	% Vis Increase	Neut. No. , mg KOH/g	Test Time, hr	% Vis Increase	Neut. No. , mg KOH/g
0-58-24	34.7	0.14	431	16.9	20 (b)	541	61.3
0-59-15	18.7	0.05	S	1.1	50	3	I.5
0-59-26	18. 7	0.09	123	6.8	40	102	6.3
0-60-3	17.2	0.14	67	19.2	60	14	10.7
O-60-11	21.2	4.12	-4	3.4	30	-13	5.7
O-60-12	16.1	0.03	6	0.5	100	48	2.8
0-60-13	25.7	0.02	269	10.7	20 (b)	226	12.9
0-60-19	20.8	0.14	6	1.7	54	-5	9.8
0-60-23	16.0	0.05	48	4.7	100	26	1.2 (c)
0-60-27	15.0	0.07	29	0.8	100	76	3.1
O-60-28	16.1	0.05	25	1.0	60	126	5.6
O-61-19	15.7	0.09	197	9.5	50	85	5.0

(a) Average of several determinations in Table 1.
(b) 400°F engine test
(c) 90-hr sample



FIGURE 7. 18-HR 425" F OXIDATION-CORROSION TEST VISCOSITY CORRELATION WITH 425" F ENGINE TEST DATA



FIGURE 8. 18-HR 425° F OXIDATION-CORROSION TEST NEUTRALIZATION NUMBER CORRELATION WITH 425° F ENGINE TEST DATA

TABLE 7. EFFECT OF COPPER ON 18-HOUR 425°FOXIDATION-CORROSION TEST RESULTS

Oil Code	Initial	Standard Test(a)	Test with Cu <u>Replacing S.S.</u>	Engine <u>Test</u> (b)
	2	liscosity, cs at	100°F	
O-58-24	34.7	184	130	220(20)
O-59-26	18.7	41.5	27.4	37.8(40)
O-60-13	25. 7	95	56.6	91(20)
O-60-23	16.0	23.8	17.8	19.7(100)
O-60-27	15.0	19.5	36.2	27.7(100)
0-61-19 ^(c)	15.7	46.7	40.1	29(50)
		Neut. No., mg	KOH/ 5	
O-58-24	0.14	16.9	11.4	61. 3(20)
O-5 9-26	0.09	6.8	5.2	6. 3(40)
O-60-13	0.02	10.7	8.7	12.9(20)
O-60-23	0.05	4.7	0.6	1.2(100)
O-60-27	0.07	0.8	8.1	3.1(100)
0-61-19(c)	0.09	9.3	9.4	5.0(50)

(a) Average of several determination 3 in Table 1.

(b) Numbers of parenthesis indicate hours of engine test duration.

(c) Same nominal formulation as O-60-27.

The effect of copper, as exhibited by the neutralization number data of Table 7, was significant only for O-60-23 and O-60-27. The use of copper gave a considerable decrease in neutralization number for O-60-23, but a large increase for O-60-27. On the whole, the correlation with engine test was slightly worsened with copper present.

F. 18-Hr 425 to 500°F Oxidation-Corrosion Test Results

Using the basic 18-hr oxidation-corrosion procedure, tests were made on eleven experimental lubricants selected by ASD. The objective of this work was to determine the oils' breakpoints with respect to oxidation stability by increasing the bath temperature from an initial value of 425° F, in 25° F increments, until a sample viscosity increase of 100 percent or more at 100°F was obtained. The test apparatus and procedures were identical for all tests with the exception of bath temperature. It was shown earlier that the lubricant temperature in the sample tube was, as a rule, slightly lower than the bath temperature. At a bath temperature of 425° F and the specified air flow of 197 liters/hr, the measured sample temperature was 422 to 423° F. The corresponding sample temperature at 500°F bath temperature (the maximum operating capability of the apparatus) was 496 to 497°F.

Tables 8 through 19 present the individual test results for the eleven lubricants included in this investigation. While duplicate tests were not run on all samples, it will be noted that very good repeatability was in evidence throughout the temperature range (425 to 500° F) for the samples which were tested more than once The following tabulation is a summary of average values for viscosity increase for the eleven fluids:

	I	Percent 100°F \	liscosity Increa	se
		for 1	fest at	
	425° F	450° F	<u>475°F</u>	500°F
0-60-23	48.7	1220		
0-60-27	29.4	980		
0-61-17	11-6	860		
MLO-62-1000	11.0	640		
MLO-62-1003	80.5	1835		
MLO-62-1004	38 6	690		
MLO-62-1008	32.5	158	890	
MLO-62-1011	3.4	86	1100	
MLO-62-1012	22.4	45 1	210	
ELO-62-50	91.4	72 1	26.8	820
MLO-62-1005	7.5	11.0	23.0	34.4

TABLE 8. RESULTS OF 18-HOUR OXIDATION-CORROSIONTESTS ON O-60-23

Bath temperature, °F		425(a)	450
Viscosity at 100°F, cs:	Initial .	16.0	16.0
·	Final	23.8	212.6
	% Increase	48.7	1220
Neut. no., mg KOH/g:	Initial	0.05	. 0. 05
	Final	4. 7	10.62
Overhead product neut. no., mg KOH/g		20. 9	73.5
Overhead product collect	ted, g	84. 0	196. 5
Oil loss, g		86. 2	208.0
Metal weight change, mg/cm ² : Al		0.0	+0.02
	Ti	-0.01	0.0
	Ag	-0.06	-0.32
	Steel	+0.01	+0.02
	SS	0.0	-0.02

(a) Average values for three tests.

TABLE 9. RESULTS OF 18-HOUR OXIDATION-CORROSIONTESTS ON 0-60-27

Bath temperature, °F		<u>425(a)</u>	450
Viscosity at 100°F, cs:	Initial	15.0	15.0
	Final	19.4	171.3
	% Increase	29.4	980
Neut. no., mg KOH/g:	Initial	0.07	0.07
• •	Final	0.8	13. 31
Overhead product neut. no., mg KOH/g		2.0	34. 9
Overhead product collected, g		143.0	230. 0
Oil loss, g		143. 7	236. 0
Metal weight change, mg/cm ² : Al		0.0	+0. 01
	Ti	+0.01	+0. 02
	Ag	-0.02	-0.34
	Steel	+0.04	+0.09
	SS	+0.02	+0. 91

34

(a) Average values for three tests.

TABLE 10.RESULTS OF 18-HOUR OXIDATION-CORROSIONTESTS ON 0-61-17

	<u>425(a)</u>	450
itial	15. 92	15.92
nal	17.72	152. 1
Increase	11. 3	860
itial	0.06	0.06
nal	0. 48	12.26
, mg KOH/g	4. 42	47. 5
, g	79.5	192. 4
	82. 2	201. 5
m ² : Al	0.0	+0.02
Ti	+0.02	0.0
Ag	-0.02	-0.17
Steel	+0.04	+0.02
SS	+0.02	-0.01
	itial nal Increase itial nal , mg KOH/g , g m ² : Al Ti Ag Steel SS	$ \frac{425(a)}{425(a)} $ itial 15.92 nal 17.72 Increase 11.3 itial 0.06 nal 0.48 , mg KOH/g 4.42 , g 79.5 82.2 m ² : Al 0.0 Ti +0.02 Ag -0.02 Steel +0.04 SS +0.02

(a)Average values for two tests.

TABLE 11RESULTS OF 18-HOUR OXIDATION-CORROSIONTEST ON ELO-62-50

Bath temperature, °F		425	450	475	500
Viscosity at 100°F, cs: Initia	al	20.62	20.62	20.62	20. 62
Fina	II.	39.46	35.48	26.15	189. 7
% Inc	crease	91.4	72. 1	26.8	820
Neut no , mg KOH/g. Initia	al	0.08	0.08	0.08	0 08
Fina	Ţ	1.96	2.54	1.30	8 43
Overhead product neut: no., 1	mg KOH/g	0.39	1.28	5. 30	32. 3
Overhead product collected, g	b 0	>270	279.9	202.4	.228. 7
Oil loss, g		303.0	285.0	207.0	247.0
Metal weight change, mg/cm ²	AI	0.0	(a)	+0.13	+0.04
	Τ.	0.0	(a)	+0.10	+0.12
	Ag	-0.02	-0.04	+0, 13	+0.12
	Steel	+0.05	+0.04	+0.16	+0.22
	SS	0.0	+0.04	+0.12	+0.24

(a) Error in metal weight suspected

 TABLE 12.
 RESULTS OF 18-HOUR OXIDATION-CORROSION

 TESTS ON MLO-62-1000

Bath temperature, °F		4	25		450
Viscosity at 100°F, cs. Initial	19. 21	19. 21	19.21	19. 21	19. 21
Final	21.35	21.40	21.24	21.33	142.8
% Increase	11.1	11.4	10. 6	11.0	640
Neut. no., mg KOH/g. Initial	0.07	0.07	0.07	0.07	0.07
Final	0.43	0.53	0. 43	0.50	12.32
Overhead product neut. no. , mg KOH/g	6.28	6. 34	6.10	5.23	72.9
Overhead product collected, g	39. 6	42.5	39. 5	41.0	134.2
Oil loss, g	41. C	43, 0	43.0	41.0	149.5
Metal weight change, mg/cm ² : Al	0.0	0.0	0.0	0.0	0.0
Ţ	<u>0</u> .0	0.0	0.0	0.0	+0.01
Ag	-0.06	-0,05	0.0	+0.02	-0.26
Steel	+0.04	+0.03	+0.02	+0.01	+0.05
SS	0.0	+0.01	0.0	+0.01	+0.08

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TABLE 13RESULTS OF 18-HOUR OXIDATION-CORROSIONTESTS ON MLO-62-1003

Bath temperature, "F			4	25		4	50
Viacosity at 100°F', cs:	Initial Final % Increase	19.40 35.81 85	19.40 30.80 59	19.40 37.45 93	19.40 35.97 85	19.40 355.5 1730	19.40 309.5 1500
Neut. no mg KOH/g:	Initial Final	0.06 7.83	0.06 6.87	0.06 10.51	0.06 8.81	0. 06 12. 23	0. 06 12. 33
Overhead product neut. Overhead product collec	no. , mg KOH/g ited, g	48. 5 52. 6	41.4 44.7	48.9 54.7	46. 2 53. 5	74. 3 149. 2	73. 6 147. 0
Dulless, g		55. 0	46. 0	57.0	60.0	166.5	162. 5
Metal weight change, m	g/cm ² . Al Ti	0.0 0.0	-0.01	+0.02	+0.02 +0.04	+0.01	0.0
	Ag	-0.06	-0.08	-0.04	-0.05	-0.17	-0. 09
	SS	+0.02	+0.03	0.0 +0.01	-0.05	+0. 05 +0. 19	+0.11 +0.12

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TABLE 14.RESULTS OF 18-HOUR OXIDATION-CORROSIONTESTS ON MLO-62-1004

Bath temperature, "F		425	450
Viscosity at 100°F, cs:	Initial	15.66	15.66
	Final	21.71	123.1
	% Increase	38.6	690
Neut. no., mg KOH/g:	Initial	0.05	0.05
•	Final	2.88	12.76
Overhead product neut. no., mg KOH/g		7.40	34. 3
Overhead product collect	eted, g	114.5	204. 7
Oil loss, g		114.0	220. 0
Metal weight change, m	.g/cm ² : Al	~0.02	(a)
	Ti	-0.02	(a)
	Ag	-0.05	· (a)
	Steel	+0.07	+0,05
	SS	+0.02	+0.01

(a) Error in metal weight suspected

a

TABLE 15. RESULTS OF 18-HOUR OXIDATION-CORROSION TESTS ON MLO-62-1005

56.58 35.2 0.33 -0.11 -0.34 -0.01 0. 11 41.84 +0.01 71.8 61.8 0.0 S 102. 500 0.28 -0.29 55.84 -0.11 0.11 0.0 -0.01 41.84 71.4 59.7 99.0 0.0 ഹ 33. 51.39 0.11 0.13 -0.13 -0.25 41.84 -0.01 22.8 78. 5 24. 2 0.0 59.0 0.0 475 0.13 51.53 -0.26 0.11 41.84 -0.13 23.2 0.0 57.5 0.0 0.0 23.1 79.1 0.09 46. 29 10. 6 0.11 -0.02 41.84 -0.14 -0.03 -0.02 +0.05 81.4 8.9 27.0 450 0, 11 Ø. 10 46. 66 +0.01 -0.28 +0.08 41. 84 +0.01 +0.02 82. 7 8. 2 11.5 25.0 0.56 -0.13 41.84 45.14 0.11 +0.02 89.4 0.0 7.9 0.0 **1**.6 0.0 Ś 12. 425 0. 11 0. 31 41.84+0.02 44.81 +0.01 2.0 0.0 11.5 Overhead product neut. no., mg KOH/g 91.6 (a) 0.0 7.1 Steel Ag SS , हन हिन्न A % Increase Metal weight change. mg/cm²: Overhead product collected, g Initial Initial Final Final Neut. no , mg KOH/g: Viscosity at 100 °F, cs. **ئے** م Bath temperature. Oil loss, g

(a) Specimen damaged in handing

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TABLE 16. RESULTS OF 18-HOUR OXIDATION-CORROSION TEST3 ON MLO-62-1006

0.05 11.87 -0.02 44.80 +0.22 +0.33 -0.01 +0.01444. l 890 40.8 127.6 147.0 475 0.05 11.92 +0.09 +0.04 44.80 -0.03 -0.06 -0.02 1:8.3 31. 1 80. 5 9i. 0 164 450 0.05 +0.04 +0.02 10.55 44.80 +0.01 -0.03 -0.06 113.2 33. 1 76. 2 88.5 153 *4*4. 80 59. 32 32. 4 0. 05 3. 30 12.47 34.5 -0.11 +0.03 +0.09 -0.02 €. 03 39.0 425 59.34 32.5 0.053.25 12.09 32.1 -0.07 +0.01 +0.06 44.80 -0.01 0.0 38.0 Overhead product neut. no., mg KOH/g Ag Steel SS % Increase יהי [--] Ā Initial Initial Final Final Metal weight change, mg/cm²: Overhead product collected, g Viscosity at 100° F, cs' Neut. no., mg KOH/g: Call temperature, °F Oil loss, g

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TABLE 17.RESULTS OF 18-HOUR OXIDATION-CORROSIONTEST ON MLO-62-1011

Bath temperature, °F			425	450	475
Viscosity at 100°F, cs:	Initial		14.80	14.80	14.80
	Final		15.31	16.08	177.2
. · · · ·	% Incre	ase	3.4	8.6	1100
Neut. no., mg KOH/g:	Initial		0.01	0.01	0.01
	Final		0.06	0.20	10.66
Overhead product neut. no., mg KOH/g		1.60	3.31	3.43	
Overhead product collected	ed, g	-	115.8	194. 4	252.2
Oil loss, g			117.0	199.0	293.0
Metal weight change, mg	/cm ² :	A1	+0.02	(a)	0.0
		Ti	0.0	-0.01	-0.01
		Ag	-0.07	-0.16	-0.20
		Steel	+0.05	-0.03	+0.02
		SS	-0.02	-0.02	0.0

(a) Error in metal weight suspected

TABLE 18. RESULTS OF 18-HOUR OXIDATION-CORROSION TESTS ON MLO-62-1012

Datificemperature, r		450	475
Viscosity at 100°F, cs: Initial	26.84	26.84	26.84
Final	32.85	38.94	53.37
% Increas	e 22.4	45.1	210
Neut. no., mg KOH/g: Initial	0.15	0.15	0.15
Final	1.61	2.13	5.92
Overhead product neut. no , mg K	OH/g 17.32	22.64	27.08
Overhead product collected, g	35.1	65.7	125.6
Oil loss, g	40.6	76.0	138.0
Metal weight change, mg/cm ² : Al	0.0	(a)	+0.01
Ti	-0.02	-0.02	0.0
Aş	-0.07	-0.07	-0.12
St	el +0.04	0.0	+0.07
SS	-0.01	+0.02	+0.02

(a) Error in metal weight suspected.

5.0

Using a maximum of 100 percent viscosity increase at $100^{\circ}F$ as the criterion for satisfactory performance, it will be observed that seven oils were unsatisfactory at 450°F, two failed at 475°F, and one was unsatisfactory at 500°F. One lubricant, MLO-62-1005, showed very good oxidation stability up to 500°F.

ELO-62-50, one of the two lubricants of the group to merit testing at 500° F, demonstrated an unusual performance at the lower test temperatures. The results obtained at the initial temperature of 425° F indicated a passing test; however, the performance appeared marginal with a viscosity increase at 100° F of 91.4 percent. In addition, excessive oil loss was noted at 425° F for ELO-62-50. Subsequent tests at 450 and 475°F on this oil showed a lessening in deterioration with increasing temperature, reflected by both viscosity increase and oil loss. At 500°F, severe degradation of the oil was evident with a viscosity increase of 820 percent.

MLO-62-1005 exhibited excellent oxidation properties within the temperature range of 425 to 500°F. Final sample viscosity for this oil at the various test temperatures increased generally in proportion to the operating temperature, i. e., oil deterioration showed no tendency toward acceleration within the range of temperatures investigated. Moreover, final sample neutralization number measured less than 0.6 mg KOH/g for all tests on MLO-62-1005 (Table 15).

An interesting aspect of MLO-62-1005 is shown by the metal weight changes given in Table 15. At 475 and 500°F, a measurable weight loss was recorded for the mild steel specimen. Significant corrosion of this metal has never previously been encountered with other lubricants in the 18-hr oxidation corrosion test.

Of the remaining oils evaluated, the majority indicated some silver attack at 450° F or higher. This was particularly noticeable with oils O-60-23, O-60-27, and MLO-62-1000 (Tables 8, 9, and 12).

G. Conclusions

An 18-hr 425°F oxidation test was developed which provided good correlation with engine tests results obtained by ASD. Twenty selected lubricants were evaluated by this test. The test exhibited very good repeatability. A cooperative test program was undertaken by five laboratories to evaluate the reproducibility of the test. In the initial tests, close agreement was shown for three participants with the two remaining laboratories reporting lower values for oil deterioration. Repeat tests by these two laboratories served to bring one into close alignment with the majority. The lack of reproducibility previously obtained by this laboratory was attributed to low oil sample temperatures. No clarification of results was shown by repeat tests for the other participant. The oil of most interest, O-60-13, indicated signs of contamination, thus the rerun data must be considered suspect for this laboratory.

The 18-hr 425°F oxidation-corrosion test was found to be quite sensitive to changes of ± 5 °F from the standard bath temperature of 425°F, but not sensitive to ± 10 percent changes in air flow from the standard rate of 197 liters/hr. Introduction of copper test specimen affected adversely the correlation of the test with the engine test.

Eleven lubricants were evaluated in an 18-hr 425 to $500^{\circ}F$ oxidationcorrosion test program. Of these eleven lubricants, seven failed at $450^{\circ}F$, two were unsatisfactory at $475^{\circ}F$, and one failed at $500^{\circ}F$. One lubricant, MLO-62-1005, exhibited very good oxidation stability throughout the test temperature range of 425 to $500^{\circ}F$; however, this lubricant showed significant corrosion of the mild steel specimen at the higher test temperatures.

III. LUBRICANT OXIDATION-CORROSION (500°F AND ABOVE)

A. General Remarks

This portion of the lubricant oxidation-corrosion phase was concerned with the design, construction, and development of apparatus and procedures for evaluating lubricant oxidation and corrosion characteristics at temperatures of 500°F and above and the evaluation of the oxidation and corrosion characteristics of candidate lubricants.

It was noted in the preceding chapter that the available oxidationcorrosiontest apparatus using a liquid bath had a maximum temperature capability of 500°F. For the development of a lubricant screening test for the supersonic transport engine, it was deemed indispensable that a test apparatus of higher temperature capability be developed. Consequently, a decision was made to design and construct a high-temperature oxidation-corrosion test apparatus. The design target for the high-temperature apparatus was 800°F, hence the use of an aluminum heating block in place of a liquid bath was necessary. Further, since it was shown in the preceding chapter that the lubricant degradation was very sensitive to small changes in test temperature and that the lubricant temperature in the sample tube was usually lower than the temperature of the heating medium, it was decided that the new design must provide for direct measurement of the lubricant sample temperature. Further, variations of this temperature throughout the test apparatus should be held to an absolute minimum.

Under the current program, a high-temperature oxidation-corrosion test apparatus using a forged aluminum heating block with provision for eight sample tubes was designed, constructed, and put into operation. The apparatus was tested at temperatures up to 700°F, although higher temperatures were possible. In the work associated with this development, close liaison was maintained with the Bench Tests Panel of the CRC Aviation Group on Gas Turbine Lubrication, which is currently attempting to establish a 500°F oxidation-corrosion test(8).

In an exploratory study of the test apparatus, the lubricant deterioration characteristics of two selected lubricants were evaluated at 500°F sample temperature. In addition, the deterioration characteristics of a 5P4E polyphenyl ether were investigated at 500, 550, and 600°F sample temperature, thereby initiating the establishment of the high-temperature baseline performance of this fluid for comparison with the performance of future hightemperature lubricant candidates.



B. Test Apparatus

1. Heating Block

A cylindrical aluminum heating block was designed and constructed of forged 6061-S aluminum alloy. It was decided to forge the block rather than cast it, in order to insure freedom from voids. Other workers in this field have reported a lack of uniform temperature distribution using a cast aluminum block, presumably due to the presence of voids within the metal.

The unit was designed to accommodate eight sample tubes and for a temperature capability of 800°F. Thermal insulation of the block is illustrated in Figure 9. Base insulation is provided by 4 in. of Johns-Manville Marinite 23A. The block rests directly on this material, which possesses good strength characteristics. By this means, metal supports for the block were eliminated along with the accompanying high heat losses. The top insulation consists of 3 in. of Marinite 23A, and the side of the block is encased by a 3-in. thickness of Johns-Manville Thermobestos pipe insulation. Thermal conductivities for these materials at 500°F are 0.61 and 0.50 Btu/ hr/ft²/in./°F, respectively.

The block is equipped with sixteen 300-watt, 230-volt, cartridge heaters. These units are 1 in. in diameter and 12 in. in length with a stainless steel sheath material. Eight of the heaters are equally spaced in a ring just outside the circle of the sample tube wells. These elements are operated in a continuously-on circuit with control accomplished by a variable transformer. The remaining heaters are equally spaced in a ring just inside the circle of the tube wells. These units are controlled by means of a variable transformer and an on-off controller as illustrated in Figure 10. An independent safety switch is provided to shut off the heating circuits in the event of a temperature rise of more than 10 to 15°F above the preset control temperature.

As will be discussed in a later section, it was found necessary to employ a heat-transfer medium between the block wells and the glass sample tubes in order to obtain maximum heat input to the sample. For this purpose, a 60/40 tin/lead solder has been used which permits operation at a minimum temperature of approximately 400 °F.

2. Test Glassware

The glassware employed in the high-temperature oxidationcorrosion test is of the same general configuration as that used in the 425°F





test except as noted and described in the following paragraphs. The sample tubes are constructed of standard wall 51-mm Pyrex tubing with a round bottom. A standard taper 71/60 outer joint was provided at the top of the tube. Overall tube length is 450 ± 10 mm, and the tube immersion depth within the aluminum block is 250 ± 10 mm.

In tests conducted with nonreflux of oil condensate, the configuration of the tube head was as illustrated in Figure 11. The overhead sidearm of each sample tube was attached to a water-cooled Graham condenser with a 250-ml round-bottom two-neck flask used as a condensate receiver. Vapors from the receiver were exhausted through a ring manifold which was vented to the building exterior. A standard taper 12/30 glass joint, positioned in the tube head 90° from the sidearm, permitted oil sampling and oil temperature measurements without disturbing the assembly. An air delivery tube of standard 6-mm Pyrex tubing, approximately 600 mm in length, was fixed in the upper end of the head by means of a one-hole cork. The tip of the air tube was cut at a 45° angle and rested directly on the bottom of the sample tube. A small collar of sufficient size to hold the metal specimens was located 15 mm from the tip of the air tube. The bottom metal specimen rested directly on this collar, and succeeding specimens were separated by 1/4-in. glass spacers cut from standard 9-mm Pyrex tubing.

In tests conducted with condensate reflux, the reflux glassware assembly used was a water-cooled Allihn condenser attached immediately above the sample tube by a ground-glass reducing adapter (71/60 to 24/40 joint). An oil sampling port was added to each adapter. An air tube of approximately 1100-mm length extended through the condenser into the sample tube.

3. Air Supply System

A precision air regulator was used to provide a constant air pressure to individual fine-thread needle valves from the laboratory air line. The air was passed through a 4-in. glass-pipe drying column, containing a calcium sulfate drier, whence to a manifold before reaching the individual test tube control valves and flowmeters. Each of the eight air flowmeters was calibrated by means of a wet test meter in order to provide accurate measurement of the air flow rate.

4. Metal Test Specimens

The metal specimens were of the round washer type with dimensions 3/4-in. O. D. and 1/4-in. I. D. by 0.032-in. thickness. The following material designations apply to the metals which were used:



FIGURE 11. HIGH-TEMPERATURE OXIDATION-CORROSION TEST APPARATUS

Aluminum alloy	QQ-A-355
Titanium alloy	MIL-T-009046B (ASG), Class 1 (8% Mn)
Silver	Electrolytic grade
Mild steel	QQ-S-636
Stainless steel	MIL-S-5059, Grade 301 Half Hard
Copper	QQ-C-576
Magnesium	QQ-M-44a (1)

C. Test Procedures

It has been the objective of this effort to explore the oxidationcorrosion characteristics of high-temperature lubricant candidates over a wide range of test conditions. Therefore, experimentation was not restricted to the use of a specific set of test conditions. Tests were conducted at 500, 550, and 600*F, with the major effort at the latter temperature. The normal test duration was 48 hr; however, in some runs at 500°F, the test duration was extended to 72 hr. Air flow rate to the sample tube was varied over the range of 0.5 to 130 liters/hr. An intermediate sampling procedure was used (20-ml samples drawn at 16, 24, and 40 hr). No makeup oil was added for the samples drawn or the oil losses. The test series at 500 and 550°F were performed with an initial sample charge of 200 ml; however, at 600°F, the sample size was increased to 250 ml in order to partially overcome the severe oil losses experienced at that temperature. Metal specimens usually consisted of a five-metal group: aluminum, titanium, silver, steer, and stainless steel. In some tests, copper and/or magnesium were added to the specimen group.

D. Preliminary Test Results

1. Performance of Heating System

Initial operation and temperature checkout of the aluminum heating block itself indicated very satisfactory performance at temperatures up to 700°F. The adequacy of the block insulation was exemplified by the fact that at 500°F only 500 watts, of the total 4800 watts available, were required for operation (one-half of this power being used for on-off control) Further, temperature measurements by means of four thermocouple holes, equally spaced in a semicircle around one tube well, indicated absolutely

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uniform temperature from a 9-in, depth up to approximately 1 inch from the top surface of the block. In addition, excellent temperature distribution throughout the block was shown in that no difference in temperature was recorded between the eight sample tube wells at 500 and 600 °F.

After satisfactory checkout of the heating block, the eight sample tubes were placed in the block, and oil LRO-11 (a 4P3E polyphenyl ether) was introduced into the sample tubes. Temperature checks were first made with dry tube wells, i.e., without using a heat-transfer medium between the block and the sample tubes. At a block temperature of 510°F and with 130 liters/hr air flow bubbling through the sample tubes, the sample temperatures ranged from 492 to 503°F. After some investigation, these variances were attributed to small variations (within manufacturing tolerances) in the O.D. of the glass sample tubes, which affected the clearances between tubes and block. In view of the requirement for very close sample temperature control discussed earlier, the use of a 60/40 tin/lead solder mixture was decided upon in order to obtain maximum heat transfer to the sample tubes. With the solder mixture in the tube wells, a sample temperature of 500°F was obtained for all eight test tube positions using a block temperature of 502°F. In addition, the sample temperatures remained unchanged throughout the air flow range of 25 to 130 liters/hr (measurements were made at 25, 50, 90, and 130 liters/hr). Similar performance was obtained with LRO-13 (a 5P4E polyphenyl ether) at a sample temperature of 600°F with a block temperature of 602°F. This implies that the heat loss due to air flow represents only a minor portion of the total loss contributed by other factors.

As stated before, the use of the tin/lead heating medium limits the operation of the apparatus to a minimum temperature of about 400°F. However, this is not considered serious for the current work. If lower test temperatures are ever required, an oil may be used as the heating medium.

2. 500°F Test Results on MLO-62-1005

Three samples of an experimental oil MLO-62-1005 obtained from different sources were run in the high-temperature apparatus at 500°F to determine any differences in performance among the samples and to compare the results thus obtained with those obtained previously using the modified 18-hr test at 500°F. MLO-62-1005 (F-1036) previously exhibited excellent oxidation properties in the 18-hr test within the temperature range of 425 to 500°F. The oil was thus tested in the high-temperature apparatus under the following conditions: 500°F sample temperature, 130 liters/hr clean, dry air flow, 200-ml sample volume, and a five-metal specimen set. The results of these determinations are given in Table 19. It should be noted
							andrineo
)ii Code	Tests	0	Test 1	Fime, hr	QV	Acidity,	Vis,
	01037			14	40	mg KUH/g	cs/100°F
2001-29-0 ⁰	Vis, cs at 100°F	41.84	55.07	70.79	(a)	68.9	25,18
F-1036)	% Vis Increase	ı	31.6	69.2	>100) ;)
	NN, mg KOH/g	0.18	0.08	0.15	1.04		
	Overhead Wt, g	1	27.9	42.2	60.5		
0-62-1005	Vis. cs at 100°F	41.84	55.69	77.35	(a)	70.9	25,90
F-1036)	% Vis Increase	ł	33.1	84.9	>100	•	
	NN, rng KOH/g	0.18	0.16	0.31	1		
	Overhead Wt, g	ł	30.8	44.5	60.6		
0-62-1005	Vis, cs at 100°F	41.89	53.91	69.07	(a)	65.2	25.90
F-1046)	% Vis Increase	ł	28.7	64.9	>100	•	
	NN, mg KOH/g	0.19	0.67	0.13	0.99		
	Overhead Wt, g	١	28, 2	43.3	62.1		
0-62-1005	Vis, cs at 100°F	42,04	54.00	70.44	(a)	61.9	25.31
F - 1048)	% Vis Increase	ł	28.4	67.6	2100		
	NN, mg KOH/g	0.18	0.11	0.15	0.80		
	Overhead Wt, g	ł	24.9	39.2	56.3		

Sample temperature 500°F. Air rate 130 liters/hr. Sample volume 200 ml. Without condensate return. Metal specimens Al, Ti, Ag, steel, stainless steel. •

(a) Too thick for determination.

TABLE 19. RESULTS OF 500°F OXIDATION-CORROSION TESTS ON MLO-62-1005

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that the air rate of 130 liters/hr in the high-temperature apparatus corresponds proportionally to the 197 liters/hr flow used in the 18-hr oxidationcorrosion test based on the linear velocity of air flow through the sample tube, neglecting temperature effects.

The data in Table 19 show that the performance of MLO-62-1005 was quite satisfactory up to 24 hr. At the end of 40 hr, however, a severe increase in viscosity was noted, thus the tests were terminated. Good agreement was indicated by results for the three different samples, and by the data for the duplicate tests.

Although the high-temperature test and the modified 18-hr test cannot be directly compared due to differences in test durations, the following tabulation shows very close agreement for F-1036 between the 16-hr data from the high-temperature test and the final results from the modified 18-hr test:

		Modified 18-hr Test	High-Temp Test, 16-hr Sample
Viscosity, cs at 100°F:	Initial	41.84	41.84
	Final % Increase	55.84, 50.58 33.5, 35.2	31.6, 33.1
Neut. No., mg KOH/g:	Initial Final	0.11 0.28, 0.33	0.11 0.08, 0.16
Weight Change, mg/cm ² : (end of test values)	Al Ti Ag Steel SS	0.0, +0.01 -0.01, 0.0 -0.11, -0.11 -0.29, -0.34 0.0, -0.01	$\begin{array}{cccccccccccccccccccccccccccccccccccc$

3. 500°F Test Results on 4P3E Polyphenyl Ether

A brief investigation was conducted at 500°F on the oxidationcorrosion properties of a 4P3E polyphenyl ether, LRO-11. A limited study was also made of the effect of air flow rate for this oil. The results obtained are presented in Table 20.

LRO-11 showed relatively high oil losses at 500°F due to volatilization and/or oil mist entrainment. Using an air flow of 130 liters/br, the entire sample was removed from the test tube within a 48-hr test period. Succeeding tests at lower air rates served to reduce oil loss, generally in

dir Rate				Ĥ	est Time,	hr			Overhead Acidity.
iters/hr	Tests	0	15	24	36	48	60	72	mg KOH/g
130	Vis, cs at 100°F	71.00	72.66	ı	72, 64	-(a)			0,0
	% Vis Increase	1	2.3	ł	2.3	t			> >
	NN, mg KOH/g	0,0	0,02	Ŧ	0.13	•			
	Overhead Wt, g	è	62.3	ì	138.4	177.2			
130	Vis, cs at 100°F	71.00	71.72	ł	73.00	-(a)			0.0
	% Vis Increase	ł	1.0	ł	2.8	ı			•
	NN, mg KOH/g	0.0	0.03	ł	0.13	ı			
	Cverhead Wt, g	ł	65, 1	ł	154.2	174.3			
100	Vis, cs at 100°F	71.00	72.20	72.42	71.69	76.92			0.0
	% Vis Increase	\$	1.7	2,0	1.0	8,3			•
	NN, mg KOH/g	0.0	0.0	0.0	0.0	0.0			
	Overhead Wt, 6	1	45.8	72.5	106. 2	140.5			
4	Vis. cs at 100°F	71.00	72.47	72.05	72.63	73, 52			0.0
	% Vis Increase	ŧ	2.1	1.5	2.3	3.7			
	NN, mg KOH/g	0.0	0. G	0.0	0.0	0.0			
•	Overhead Wt, g	ł	27.2	44. 2	55.5	88. 3			
20	Vis, cs at 100°F	71°00	70, 32	72.45	73, 22	73, 54	74.69		0.07
	% Vis Increase	ł	-1.0	2,0	3.1	3, 6	5, 2		
	NN, mg KOH/g	0.0	0.0	0.0	0°0	0,0	0,04		
	Overhead Wt, g	ŧ	18.0	29, 2	43.3	58, 1	73.9		
50	Vis, cs at 100°F	71.00	72.01	72, 68	73.81	72, 59	74.48	74, 18	0.22
	% Vis Increase	ŧ	1, 4	2,4	4.0	2, 2	4.9	4, 5	
	NN, mg KOH/g	0.0	0,0	0.0	0.0	0,0	0.0	0, 03	
	Overhead Wt, g	t	5.7	9.0	13.0	18.0	24, 2	27.2	

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TEST RESULTS OF 500°F OXIDATION-CORPOSION TESTS ON LBO-11 TABLE 20.

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Air Rate				Ţ,	est Time,	hr			Overhead Acidity.
liters/hr	Tests	0	15	24	36	48	60	72	mg KOH/g
130	Vis, cs at 100°F	71.00	72.00	71.64	73.14				0.0
Copper	% Vis Increase	ł	1.4	0.9	3.0				
added	NN, mg KOH/g	0.0	0.0	0.0	0,0				
	Overhead Wt, g	<b>t</b>	56.7	90.3	131.3				
130	Vis, cs at 100°5	71.00	71.64	72.10	75.34				0.0
N. M	% Vis Increase	3	0.9	1. 6	6.1				i 9
added	NN, mg KOH/g	0.0	0.0	0.0	0.0				
·	Overhead Wt, g	ł	64.0	101.5	147.5				

Sample temperature 500°F. Metal specimens Al, Ti, Ag, steel, stainless steel, with Cu or Mg Without condensate return. in place of stainless steel as indicated. Sample volume 200 ml.

(a) Test tube dry at 48 hours.

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proportion to the air flow used. It was also observed that oil loss varied linearly with respect to time at any given air rate.

In all cases, oil deterioration as indicated by viscosity increase and neutralization number was very moderate for LRO-11. One sample (at the lowest air rate used, 20 liters/hr) was run to a total of 72 hr with a resultant viscosity increase of less than 5 percent. In addition, the extent of oil oxidation was generally unaffected by air flow rate within the range investigated. Similarly, no change was noted in oil performance for the LRO-11 samples containing a magnesium or a copper test specimen.

No evidence of significant metal corrosion was observed for LRO-11 using the metal specimens listed in Table 20.

## E. Test Results on 5P4E Polyphenyl Ether

Two batches of 5P4E polyphenyl ether were used in these investigations. The first batch, LRO-13, was already on hand at the initiation of this program and was thus used for the preliminary studies of test variables. In the meantime, a sufficient supply of the second batch, F-1041, was procured for use as a reference fluid for the entire program. After receipt of F-1041, tests were made on this batch which, as will be shown later, gave the same performance as LRO-13.

As a result of the use of an intermediate sampling schedule, a considerable volume of data has been generated in this work. In most cases, therefore, data presentations have been summarized by tabulating only results for the final (48-hr) sample.

Relatively mild oxidative deterioration was obtained with LRO-13 at sample temperatures of 500 and 550°F, too mild to permit a study of test variables. As shown by the results given in Table 21, the maximum viscosity increase at 100°F was approximately 22 percent at 550°F test temperature, and final neutralization number was negligible in all cases.

Overhead condensate weights in the 550°F test series showed a direct dependence on air flow rate. The amounts collected in the tests at 130 liters/ hr represented an oil loss in 48 hr of approximately 45 percent of the initial sample charge.

For the test series at 600°F, an initial sample volume of 250 ml was used in order to compensate for the severe oil losses. Even with the increased volume, however, test operation at the higher air flows could not be extended beyond 40 hr because of the insufficiency of sample remaining.

# TABLE 21. SUMMARY OF OXIDATION-CORROSION TEST RESULTS ONLRO-13 AT 500 AND 550°F

	48-hr Sa	mple	Ov	erhead Sample	e
Air Rate, liters/hr	% Vis Increase at 100°F	NN, mg KOH/g	Vis, cs/100°F	NN, mg KOH/g	Weight,
	<u>500°F</u>	, 5-Metal Spe	ecimen Set		
130	7.5	.0.0	-	0.04	33
130	7.3	0.0	-	0.03	30
	<u>550°F</u>	, 7-Metal Spe	ecimen Set		
130	21.6	0.0	346.3	0.31	103
130(a)	.17.1	0.0	338.5	0.06	112
100	16.5	0.0	325.4	0.03	81
75	14.5	0.0	331.8	0.05	60
20	15.5	0.03	326.1	0.28	14
5	22.6	0.09	(b)	0.94	3

5-metal set: Al, Ti, Ag, steel, stainless steel. 7-metal set with Cu and Mg added. Sample volume 200 ml. Without condensate return.

(a) 250 ml initial sample volume.

(b) Insufficient sample volume.

Table 22 provides a summary of the high-temperature oxidation data on LRO-13 at 600°F. The major effort was directed toward investigation of the effect of air flow rate using a 5-metal specimen set consisting of Al, Ti, Ag, steel, and stainless steel. The results of these tests, with few exceptions, permitted several generalized observations concerning the effect of air rate on the performance of LRO-13 at 600°F.

Decreasing air flow rate generally resulted in an increase of both test oil and overhead oil neutralization number. Test oil sample acidity remained low in all cases, however. Viscosity measurements on the overhead oil samples indicated that this value remained relatively constant at air flows of 5 liters/hr and below. At higher air rates, overhead oil viscosity for the individual tests followed a general increase. It should be noted that the major isomer component (mmm-5P4E) of LRO-13 has a viscosity of about 332 cs at 100°F. This isomer is also the least viscous of the three which constitute the 5P4E mixture. Thus, it appears that the materials which have been collected as overhead oil were probably some form of various oxidation products since overhead viscosities as low as 32 cs at 100°F were recorded.

The most notable effect produced by varying air flow rate in the 600°F test series was observed for sample viscosity increase. This property is generally considered the primary criterion of lubricant oxidation stability and was therefore a characteristic of major importance in this investigation. As evidenced by the data presented in Table 22, viscosity increase was significant in the 600°F tests on LRO-13. The most interesting feature of these results, however, is presented in Figure 12 which illustrates the effect of air rate on the viscosity of LRO-13 for all 5-metal tests which completed 48 hr. An anomalous trend was indicated in that the oil deterioration curves described both a maximum and a minimum within the range of air flow studied. The trend was most pronounced for the 40 and 48-hr sample data. Results obtained at 0.5, 3 and 5 liters/hr demonstrated a consistent increase in lubricant viscosity as air flow rate was increased. At approximately 15 liters/hr, viscosity increase reached a maximum. Further increase in air rate resulted in a general decrease in oil deterioration until a minimum in viscosity increase was encountered at the 50 liters/hr air flow. At higher air rates, the lubricant exhibited a mild, progressive decline in oxidation stability, particularly for the later sample periods.

A definitive explanation of the unusual effect on LRO-13 produced by varying air flow rate is not feasible with present knowledge. Certainly, the effect is contrary to that normally expected in oxidation studies and, therefore, is assumed to be a distinguishing characteristic of either the fluid under investigation, the test apparatus geometry, or both. A plausible interpretation of the minimum and maximum shown in Figure 12 may be related

	48-hr Sa	mple	Ov	erhead Sample	е
Air Rate,	% Vis Increase	NN,	Vis,	NN,	Weight,
liters/hr	at 100°F	mg KOH/g	<u>cs/100°F</u>	mg KOH/g	g
130 (40 hr)	156	0.0	316.4	0.03	214
100	(a)	0.0	318.8	0.04	194
100 (40 hr)	153	0.0	332.0	0.06	186
90	(a)	(a)	321.6	0.03	182
90	(a)	(a)	326.7	0.04	195
75	89	0.13	293.8	0.24	141
75	110	0.04	303.5	0.09	148
75	123	0.07	308.6	0.10	154
60	93	0.31	313.6	1.91	140
60	97	0.08	302.7	0.28	135
60	119	0.04	315.9	0.07	147
50	74	0.06	265.4	0.18	90
50	86	0.09	283.1	0.09	95
50	91	0.18	263.5	0.18	90
50	92	0.08	284.9 🔩	0.15	98
50 <b>(b)</b>	92	0.08	256.4	0.24	87
50(c)	89	0.07	275.2	0.26	93
50(C)	90	0.08	285,6	0.28	99
42	93	0.71	261.8	0.88	79
42	110	0.07	253.8	0.33	89
42	108	0.07	262.7	0.31	93
35	105	0.16	213.9	0.35	61
35	138	0.11	203.9	0.31	65
35	105	0.11	223.5	0.25	66

# TABLE 22.SUMMARY OF OXIDATION-CORROSION TEST RESULTS ON<br/>LRO-13 AT 600°F

5-metal set Al, Ti, Ag, steel, stainless steel. Sample volume 250 ml. Without condensate return.

(a) Insufficient sample available.

(b) Bottled air used instead of laboratory compressor supply.

(c) No intermediate sampling.

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	48-hr Sa	mple	Ov	erhead Sampl	e
Air Rate,	% Vis Increase	NN,	Vis,	NN,	Weight,
liters/hr	at 100°F	mg KOH/g	<u>cs/100°F</u>	mg KOH/g	g
27	128	0.39	149.5	0.88	44
27	138	0.16	158.0	0.52	45
27	149	0.16	155.6	0.54	46
27	162	0.30	158.3	0.64	49
20	118	0.19	132.6	0. 79	31
20	139	0.20	131.1	0.20	35
20	132	0.19	127.5	0.62	34
20	190	0. 29	106.5	0.73	33
20	172	0.24	120.5	0.64	31
20	198	0.18	129.5	0.72	39
20(Ъ)	139	0. 20	142.9	0.59	42
20(b)	179	0.18	102.8	0.92	31
20(c)	204	0.24	104.5	0.91	39
20(c)	179	0.21	103.3	0.90	36
16	145	0. 22	107.9	0.88	28
16	173	0. 21	96.5	0.96	26
16	220	0.19	82.1	1.16	26
16	191	0.19	93.1	0. 98	26
16	208	0.19	102.0	1.04	30
16	197	0. 27	69.8	1.28	21
12	169	0. 42	93.5	0.88	25
12	149	0.25	87.9	0.96	23
12	197	0.17	75. 2	1.13	23
12	232	0.33	78.9	1.61	26
5	150	0.20	36.9	1.66	9
5	209	0.31	46.0	2.08	14
5	203	0.33	44. 2	2.47	13
5	168	0.47	(a)	(a)	11

## TABLE 22.SUMMARY OF OXIDATION-CORROSION TEST RESULTS ON<br/>LRO-13 AT 600°F (Cont'd)

5-metal set Al, Ti, Ag, steel, stainless steel. Sample volume 250 ml. Without condensate return.

(a) Insufficient sample available.

(b) Bottled air used instead of laboratory compressor supply.

(c) No intermediate sampling.

	<u>48-hr Sa</u>	mple	Ove	erhead Sample	е
Air Rate, liters/hr	% Vis Increase at 100°F	NN, mg KOH/g	Vis, cs/100°F	NN, mg KOH/g	Weight,
3	136	0.24	38.7	3.94	7
3	166	0.35	43.1	2,11	12
3	165	0.40	31.5	2.58	10
0.5	70	0.29	41.5	4.30	7
0.5	81	0.15	(a)	1.35	7
0.5	71	0.27	(a)	(a)	7

# TABLE 22. SUMMARY OF OXIDATION-CORROSION TEST RESULTS ON LRO-13 AT 600°F (Cont'd)

5-metal set Al, Ti, Ag, steel, stainless steel. Sample volume 250 ml. Without condensate return.

(a) Insufficient sample available.



EFFECT OF AIR FLOW RATE ON LUBRICANT DET RIORATION FIGURE 12

with a vapor-phase oxidation phenomenon. At the low air rates, it is conceivable that such a mechanism could be the predominant factor influencing oil deterioration in that, due to the low air flow velocities within the test tube, a large portion of the products of vapor-phase oxidation was subject to condensation and ran back down the sample tube, and thereby had a significant effect on the bulk oil condition. As the air flow was increased a reasonable assumption is that the residence time of the oil vapors was progressively decreased so that vapor-phase oxidation, and its effect on the bulk sample, was also decreased. It is further expected that at still higher air rates, the effect of vapor-phase oxidation became insignificant, thus the prevailing mechanism was deterioration of the bulk oil sample.

A detailed description of 5P4E glassware deposits and cil sludge is not presented for the individual tests. These phenomena were quite consistent throughout the test program and will be described only in summary form. No sludge was noted for any test, using the nonreflux configuration, either by 100-mesh screen filtration or 1-hr centrifuging at a relative centrifugal force of 840 g's. At low air rates, a black carbon deposit of a thin, grainy consistency was observed on the bottom of some tubes, but usually in small streaks running axially up the tube side, normally above the sample oil level Glassware deposits in tests at air rates of 50 liters/hr and higher were nil The most consistent phenomena noted with respect to sample tube appearance was in the coloration of condensed oil droplets within the tube head. At air flows above 50 liters/hr. these droplets were a light, clear yellow and seemed to coalesce into an oil film with increasing air rates. The tube heads in tests at lower air flows showed the droplets with a brownish coloration, increasing in darkness as air flow was decreased. This effect was quit a pronounced, and by close observation it was possible to distinguish the level of air flow by visually rating the color depth of the droplets.

A comparison of the results obtained in repeat tests on LRO-13 using a five-metal specimen set showed a significant range of scatter, particularly with respect to final viscosity increase. As a consequence of this scatter, two factors were briefly investigated which could be expected to affect test repeatability, viz, the purity of the air supply and the intermediate sampling procedure. Accordingly, tests were conducted at air rates of 20 and 50 liters/ hr using high-purity bottled air rather than the normal laboratory compressor supply in addition, two tests were run at each air rate in which the intermediate sampling procedure was constrated any effect on the level of lubricant deterioration or the degree of test repeatability. A close examination of test data likewise eliminated any deviation in repeatability attributable to sample tube position within the block or to a particular glassware set. All of these items, including the air flowmeter, are identified by number, and no relationship has been determined between the extent of lubricant degradation and the test assemblies.

It is felt that several factors, many uncontrollable, contribute to the apparent lack of good repeatability for final viscosity data at 600°F. Two prominent factors are the relatively long test duration and the sensitivity of LRO-13 to air flow rate variation. Table 23 presents data on the repeatability of the test expressed as a fraction of the average viscosity increase. It will be seen that the fractional repeatability was quite acceptable and without significant variation for the 16- and 24-hr results. Test repeatability after 40 hr was, however, considerably poorer.

The influence on 5P4E deterioration at 600°F produced by the presence or absence of certain metal types was examined for three distinct cases: without metals, a five-metal group (Al, Ti, Ag, steel, stainless steel), and a seven-metal group (Cu and Mg added). Extensive test data were obtained with the five-metal set (Table 22). Results for the two remaining cases are given in Table 24. Although only single determinations were obtained in most instances using seven metals, or none, the general effect of these specimen changes was quite evident. Data obtained in tests with seven metals (Cu and Mg added) indicated considerably milder oil deterioration than the five-metal series at almost all air flow rates studied. The sole exception was the test at 0.5 liters/hr which gave essentially the same viscosity increase using five or seven metals. Figure 13 illustrates these differences as a function of air rate. The range of values obtained for the five-metal series is also shown by this illustration. The improved performance of LRO-13 using seven metals was presumably due to the presence of the copper specimen which has been reported to have an inhibitory action on the oxidation of 5P4E.

In general, the test series conducted without metals (Table 24) demonstrated no significant change from viscosity data with the five-metal group. In addition to these tests, four tests were also performed without metals in a brief examination of the influence of air dispersion on sample viscosity increase. These runs were made using a small fritted glass disperser attached to the end of the air tube instead of the normal open-end tube. Use of the gas disperser served to accelerate lubricant deterioration at air rates of 5, 20, and 50 liters/hr. The run at 35 liters/hr, however, indicated no significant effect due to the change in air tube configuration.

In the later stages of the program, the supply of oil LRO-13 was depleted and investigations were carried out using the new supply of 5P4E

Air R	ate,	Fractional I	Repeatability ^(b) of	Percent Viscosit	y Increase
liters/1	$hr^{(a)}$	<u>16 hr</u>	<u>24 hr</u>	<u>40 hr</u>	<u>48 hr</u>
0.5	5 (3)	0.08	0.08	0.08	0.08
3	(3)	0.09	0.07	0.18	0.11
5	(4)	0.08	0.10	0.14	0.15
12	(4)	0.09	0.12 .	0.16	0.19
16	(6)	0.07	0.10	0.11	0.14
20	(6)	0.09	0.10	0.14	0.21
27	(4)	0.06	0.07	0.10	0.10
35	(3)	0.10	0.12	0.15	0.16
42	(3)	0.06	0.04	0.08	0.09
50	(4)	0.08	0.05	0.10	0.10
60	(3)	0.07	0.05	0.08	0.14
75	(3)	0 - 08	0.07	0.11	0.16
Doo	~ 0	0.06 - 0.10	0.04 - 0.12	0.08 - 0:18	0 08 - 0 21
Kan	ge	0.00 - 0.10	$0, 0 \neq = 0. 22$	0,08 - 0,18	0.00 - 0.21
Ave	rage	0.08	0 08	0.12	0.14

# TABLE 23. REPEATABILITY OF OXIDATION-CORROSIONTEST RESULTS ON LRO-13 AT 600°F

Test temperature 600°F. 5-metal specimen set. Without condensate return.

- (a) Figures in phrentheses indicate number of test determinations.
- (b) Standard de liation of percent viscosity increase divided by average percent increase at 100°F.

	48-hr Sa	mple	Ove	erhead Sample	e
Air Rate,	% Vis Increase	NN,	Vis,	NN,	Weight,
liters/hr	at 100°F	mg KOH/g	<u>cs/100°F</u>	mg KOH/g	g
	<u>7-N</u>	letal Specime	n Set		
130 (40 hr)	(a)	0.0	29.0	0.03	223
100	(a)	0.0	334.2	0.03	200
75	71	0.0	307.9	0.06	139
50	64	0.02	283.6	0.11	96
35	77	0.08	229.8	0.22	67
20	92	0.13	146.2	0.56	32
5	113	0.15	(a)	1.44	11
3	106	0.24	54.9	3.46	10
0.5	76	0.29	48.7	3.42	7
	]	No Metals Pre	esent		
130 (40 hr)	181	0.0	332.2	0.03	214
75	99	0.13	300.0	0.21	132
50	78	0.06	261.2	0.18	85
50(b)	127	0.19	262.0	0.18	106
35	187	0.33	188.7	0.47	69
35	109	0.35	229.3	0.40	64
35	133	0.54	204.7	0.43	62
35(b)	89	0.72	248.7	0.36	66
20	156	0.19	114.4	0.81	31
20	180	2.36	125.8	1.08	38
20(b)	246	0.96	130.0	0.56	43
5	146	0.22	41.4	2.00	10
5(b)	>500	2.56	(a)	(a)	19
			• •	• •	

# TABLE 24.EFFECT OF ME TALS ON OXIDATION-CORROSIONTEST RESULTS ON LRO-13 AT 600°F

7-metal set: Al, Ti, Ag, steel, stainless steel, Cu, Mg. Sample volume 250 ml. Without condensate return.

(a) Insufficient sample available.

(b) Using a fritted glass air tube.



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fluid, F-1041. Several determinations were made at 600°F with F-1041 in order to compare its performance with that for LRO-13 under similar test conditions. The results on F-1041 are presented in Table 25. The series without condensate return showed good agreement between data for the two 5P4E fluids. In particular, the deterioration trend produced by varying air rate was comparable for both oils.

Additional results on F-1041 shown in Table 25 include a study of the effect of condensate reflux. Once again, the data indicated a maximum in viscosity increase within the range of air rates used. However, a much higher level of oil degradation was evident using condensate return, except for the test at 75 liters/hr. This run was slightly lower in final viscosity than similar tests using nonreflux. Some caution must be exercised however, in assessing the effect of reflux versus nonreflux at the higher air rates. As illustrated by the oil loss results in the reflux tests, condenser efficiency was severely impaired by increasing test dir flow. For example, net oil loss at 75 liters/hr was approximately 50 percent of that which could be expected without a condenser. Thus, for most reflux tests, only a partial return of effluent products was obtained, and the degree of return was dependent on the rate of air flow.

Condensate return was likewise deleterious to 5P4E performance with respect to other test evaluations in addition to viscosity increase. A slight, general increase in sample neutralization number was obtained (Table 25). Glassware appearance showed increased deposits just above the oil level. A 3-inch band of hard, streaked carbon was noted on all test tubes. The density of these deposits was lessened at higher air flows. In addition, very small amounts of sludge were recovered by 100-mesh screen filtration of the oil samples The amounts were too small to allow a quantitative measurement by weighing. Conclusive evidence of oil sludge by centrifuging could not be obtained due to the opaqueness of the samples. Less than 0.1 ml of material was collected by centrifuging and then pouring off the sample; however, it was difficult to differentiate between sludge and oil in evaluating this residue. It was noted that the material collected by centrifuging was soluble in benzene, whereas the sludge collected by filtration was essentially insoluble

Evidence of significant metal specimen corrosion by 5P4E occurred only at the 600°F test temperature, and was confined to three metals--silver, copper, and magnesium. Typical data on weight change for these metals are given in Table 26. These weights are representative of the extent of corrosion obtained throughout the entire program, and are typical for both batches of 5P4E used. Further, although the metal weight changes given were all obtained in tests without condensate return, corrosion results from the reflux tests did not differ substantially.

	48-hr Sa	mple	Ove	er <mark>head</mark> Sample	e
Air Rate,	% Vis Increase	NN,	Vis,	NN,	Weight,
liters/hr	at 100°F	mg KOH/g	<u>cs/100°F</u>	mg KOH/g	<u>g</u>
	w	ithout Conden	sate Return		
75	76	0.11	310.8	0.0	156
	72	0.09	303.3	0.0	150
50	67	0.02	274.6	0.29	99
	81	0.12	256.9	0.12	101
35	90	0.11	218.8	0.29	69
27	147	0.22	154.4	0.47	51
	132	0.18	145.3	0.47	47
20	165	0.17	128.7	0.65	42
	149	0.30	118.9	0.12	36
16	190	0.19	83.9	2.06	30
	169	0.34	102.7	0.82	34
5	170	0.35	39.96	1.99	13
	190	0.26	(a)	1.95	12
	v	Vith Condensa	te Return		
				Oil Loss, g	
75	77	0.10		86	
50	190	0.24		56	
35	440	0.49		38	
27	1850	1.03		23	
20	1500	1,12		16	
	1140	0.94		18	
16	1140	0.90		12	
5	350	0.85		9	

# TABLE 25. SUMMARY OF OXIDATION-CORROSION TEST RESULTS ON F-1041 AT 600 $^{\circ}{\rm F}$

Metal specimens Al, Ti, Ag, steel, stainless steel. Sample volume 250 ml. (a) Insufficient sample available.

	48-hr Sample		Metal Weight Change,		
Air Rate,	% Vis Increase	NN,	$mg/cm^{2(a)}$		
liters/hr	at 100°F	mg KOH/g	Ag	Cu	Mg
130 (40 hr)	156	0.0	-0.12	-	-
130 (40 hr)	(b)	0.0	+0.11	-0.04	+i.31
100 (40 hr)	153	0.0	-0.04	-	-
100	(b)	0.0	+0.12	-0.12	+1.08
75	89	0.13	-0.54	-	-
75	71	0.0	0.0	+0.04	+0.04
50	74	0.06	-0.02	-	-
50	64	0.02	-0.23	-0.67	+0.09
35	105	0.16	-0.05	-	-
35	77	0.08	-0.36	-0.81	-0.05
20	118	0.19	-0.07	-	-
20	92	0.13	-0.30	-1.08	-0.05
5	150	0.20	-0.21	-	-
5	113	0.15	+0.55	-1.42	+0.09
3	136	0.24	-0.23	-	-
3	106	0.24	+0.29	-1.86	-0.11
0.5	70	0.29	-0.23	-	-
0.5	76	0.29	-0.54	-1.44	+0.49

# TABLE 26. TYPICAL DATA FOR SPECIMEN WEIGHT CHANGE IN<br/>OXIDATION-CORROSION TESTS ON 5P4E AT 600°F

Sample volume 250 ml. Without condensate return.

(a) Metal weight changes for Al, Ti, steel, and stainless steel were negligible.
(b) Insufficient sample available.

As shown in Table 26, corrosion of the silver specimen was random with no apparent relationship existing between metal weight loss and oil performance. The maximum silver loss was  $0.54 \text{ mg/cm}^2$ , which was obtained in one run at 75 liters/hr air flow and in one test at 0.5 liters/hr. An equally significant weight gain was shown by silver in a 600°F test at 5 liters/hr air flow. This weight increase was caused by a dark gray, ingrained deposit over the entire specimen and was not removable by the usual cleaning procedures.

Severe copper corrosion was initially observed in the 600°F test conducted at 50 liters/hr air flow. In tests conducted at lower air flows, corrosion of the copper specimens increased in severity as air flow was decreased The largest weight change shown for copper was -1.86 mg/cm², which corresponds to a total specimen weight change of approximately -10 mg.

The effect of 5P4E on magnesium was not in corrosive attack, but in the formation, in certain tests, of measurable quantities of deposits similar in appearance to those mentioned previously for some silver specimens. In tests at 100 and 130 liters/hr air flow, a weight gain in excess of  $1 \text{ mg/cm}^2$  was obtained for magnesium.

#### F. Viscosity-Temperature Relationship of 5P4E Polyphenyl Ether

In the following chapter on lubricant deposits and degradation employing the roller bearing test, oil deterioration for the 5P4E fluid (F-1041) was evaluated on the basis of viscosity increases measured at 210°F. However, in order to permit a comparison of the bearing test results with the oxidationcorrosion test data, sample viscosity in the two tests was determined at both 100 and 210°F upon initiation of the bearing test program.

The viscosity data obtained at the two reference temperatures revealed that, due to the nature of the 5P4E viscosity-temperature relationship, viscosity increase rose at a slower rate at 210°F than at 100°F as shown in Figure 14. For example, an increase of 100 percent at 210°F corresponded to an increase of approximately 350 percent at 100°F, whereas a 50 percent increase at 210°F gave approximately 150 percent at 100°F

The viscosity-temperature function of organic fluids is normally exponential. Neglecting any change in the viscosity-temperature properties of 5P4E upon deterioration, a plot of kinematic viscosity at 210°F versus that at 100°F should yield un exponential curve. It was noted that the results showed some deviation from the theoretical curve at the higher viscosities. Data analysis using ASTM viscosity-temperature charts revealed that as deterioration progressed, a very slight change occurred in the viscositytemperature relationship of the cil, i.e., the rate of viscosity change from 210 to 100°F was accelerated, and the slope of the viscosity-temperature line increased.

The visco...ty data shown in Figure 14 for the oxidation-corrosion test include results for runs with and without condensate return. The marked differences in deterioration between these two conditions were not reflected by any change in the viscosity characteristics of 5P4E. However, the results obtained from bearing tests indicate a consistent deviation from the exidationcorrosion test data curve, chiefly at the higher viscosity levels. While the oxidative reaction was undoubtedly the major factor influencing oil properties in both test types, an additional element was evident in the case of bearing test results. This difference was probably due to a mechanical shear of the oil by the dynamic action of the roller bearing and auxiliary pumps--a phenomenon not present in the static oxidation-corrosion test.

### G. Conclusions

Operation and performance of the high-temperature oxidation-corrosion test apparatus have been very satisfactory. Excellent temperature uniformity was attained, and it is expected that the temperature capability of the unit will be much higher than the design target of 800°F.

Tests with the high-temperature test apparatus on MLO-62-1005 at 500°F sample temperature indicated close agreement between the 16-hr data from this test and results from the earlier 18-hr oxidation-corrosion test at 500°F bath temperature. The similarity for the two procedures was reflected in both oil analysis and metal specimen corrosion.

A brief study conducted with 4P3E polyphenyl ether (LRO-11) showed excellent oxidation stability at 500°F sample temperature and air flows up to 130 liters/hr. Oil loss, however, was severe at air rates above 50 liters/hr. No effect on 4P3E performance was evident when copper or magnesium specimens were present.

Results obtained with 5P4E lubricant also indicated only moderate deterioration at 500 and 550°F. At 600°F sample temperature, substantial oxidation was demonstrated as evidenced by lubricant viscosity. The effect of test air flow rate on 5P4E performance at 600°F was significant, with a maximum and a minimum in oil deterioration encountered within the range of air flow studied

An examination of the influence of certain metal specimen groups on the oxidation of 5P4E showed no difference between tests without metals and those with a five-metal set (Al. Ti, Ag. steel, stainless steel). Use of a



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FIGURE 14 CORRESPONDENCE OF PERCENT VISCOSITY INCREASE FOR 5P4E AT TWO REFERENCE TEMPERATURES seven-metal specimen group (Cu and Mg added), however, indicated a distinct suppression of lubricant oxidation, presumably due to the presence of copper.

A 600°F test series with condensate return revealed a marked acceleration in 5P4E viscosity increase at air rates below 75 liters/hr.

Significant metal corrosion was noted for 5P4E only at 600 F, with and without condensate return, and was confined to silver and copper. Silver attack was random, i. e., no relationship was evident between metal loss and cil performance. Corrosion of the copper occurred at test air rates of 50 liters/hr and below. Magnesium showed weight gains due to formation of deposits, particularly at high air flow rates.

#### IV. LUBRICANT DEPOSITS AND DEGRADATION

#### A. General Remarks

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The objectives of the lubricant deposits and degradation phase of the program were to develop apparatus and techniques for determining the deposits and degradation characteristics of lubricants and to evaluate candidate lubricants under environmental conditions representative of Mach 3 class gas turbine engine designs.

For the performance of this work, the Erdco 100-mm roller bearing machine, with its extensive background in MIL-L-7808 and MIL-L-9236 applications^(7,8), was selected as the basic equipment. However, it was decided to employ a different test oil system design from the one commonly used, in order to minimize heat losses and other mechanical problems. The test procedure adopted in this study was patterned after that used in previous work of this nature^(3,7) and later standardized by the Deposits and Oil Degradation Characteristics Panel of the CRC Aviation Group on Gas Turbine Lubrication⁽⁸⁾, but with appropriate modifications where required.

During the period covered by this report, two Erdco 100-mm roller bearing machines available under Contract AF 33(616)-7223 were installed on available drive stands. A special test oil system was designed, and two such systems were constructed and installed. Operational checks were made on both installations, using a used sample of a 5P4E polyphenyl ether, over the anticipated range of operating conditions including test oil temperatures up to 700°F and test bearing outer-ring temperatures up to 750°F. As an aid to test method development and establishment of repeatability between the two machines, exploratory tests were conducted on a MIL-L-9236 lubricant at 425°F test oil temperature and 500°F test bearing temperature.

Following the preliminary tests, a program to establish the baseline performance characteristics of 5P4E polyphenyl ether was initiated. Six tests were completed on this fluid at high operating temperatures, with and without air flow being introduced through the test oil sump. It was shown that 5P4E gave excellent performance at 5C0°F test oil temperature and 550°F test bearing temperature, marginal performance at 550°F test oil temperature and 700°F test bearing temperature, and unacceptable performance at 700°F test oil temperature and 750°F test bearing temperature.

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#### B. Test Apparatus

#### 1. Erdco 100-mm Roller Bearing Machine

Two Erdco 100-mm roller bearing machines were used in the experimental work reported herein. A cross-section drawing of the Erdco 100-mm roller bearing machine is shown in Figure 15. The test bearing, A, is a 100-mm, unshielded, cylindrical roller bearing. A 4700-watt tubular heater, B, wrapped around the test bearing outer race mount, supplies the required heat to the outer race of the bearing. Aluminum oxide is packed around the heater to provide more even heat distribution to the bearing. Test oil is supplied to the bearing through a 0.040-in. jet, F, which is directed toward a point on the bearing midway between the C.D. of the roller cage and the I.D. of the outer race. The test oil is scavenged from the machine through two scavenge ports, G, one located in front of the bearing and one behind the bearing. A screw-thread nonrubbing seal, H, separates the test oil and support oil sections of the machine. Load is applied to the shaft, C, and hence to the test bearing by means of a hydraulically controlled load piston. E, acting through a ball bearing, D, which is free to float in a plane perpendicular to the axis of the shaft. The bail bearing also acts as a thrust bearing to limit the axial movement of the shaft. A small straight roller bearing, P, supports the splined end of the shaft.

## 2. Drive Stand

The drive stands used for the Erdco 100-mm bearing machines are of SwRI design, each consisting of a 50-hp motor coupled to a jack shaft through a fixed-ratio pulley and belt arrangement. The jack shaft drives a 9.25:1 step-up gearbox providing an output speed of 10,000 rpm. Coupling of the bearing machine to the drive stand is effected by an Erdco type adapter block and splined coupling. The support oil system, located on the drive stand, supplies the required lubrication to the drive unit, the support ball bearing, and the support roller bearing in the bearing machine; in addition, it provides the hydraulic load pressure used to load the bearing machine. A photograph of the drive stand with the bearing machine installed is presented in Figure 16.

## 3 Test Oil System

In the standard Erdro bearing rig installation⁽⁸⁾, the test oil system comprises a 2-gallen test oil sump connected to a test oil pump which meters and supplies the test oil to the jet located on the front cover of the bearing machine. The oil is then drawn from the bottom of the bearing machine by means of a scavenge pump, whence it is returned to the test oil sump. The test oil sump, test oil pump, and the oil lines connecting the sump to the pump



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FIGURE 15. CROSS SECTION OF THE ERDCO 100-MM ROLLER BEARING MACHINE



FIGURE 16 PHOTOCRAPH OF 100-MM ERDCO ROLLER BEARING MACHINE INSTALLATION AT SWRI and the pump to the jet are insulated; but experience indicates that the heat loss from these lines (including the pump) is such that a temperature drop of as much as 40°F may be obtained at a test oil inlet temperature (or jet temperature) of 400°F. This temperature drop is apt to be even greater at higher test oil temperatures. It was felt by ASD that this temperature drop should be minimized so that the performance capabilities of the lubricants could be accurately defined. A maximum temperature drop target of 10 to 15°F was established by ASD.

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Experience also indicates that the test oil pump and the scavenge pump seals are prone to leak, particularly at high temperatures. Further, differential expansion of the pump components due to the high fluid temperature inside and the lower surrounding temperature has caused operational difficulties. The leakage and differential expansion problems were therefore minimized as described in the succeeding paragraphs.

In the test oil system designed and constructed for this program, the basic features of the Erdco test oil system have been retained. The most significant departure from the Erdco system has been in the use of a specially designed test oil sump in which both the test oil pump and the scavenge pump are placed. By placing these pumps within the test oil sump, possible loss of test oil through leakage at the pump seals is eliminated. In addition, the pressure pump is mounted such that it is below the level of the test oil. This insures against any possible test oil temperature loss as the oil passes through the pump. The location of the pumps also eliminates the differential expansion of pump components since both the interior and exterior of the pumps are subjected to nearly the same temperatures. Figure 17 is a photograph showing how the components are fastened to the lid of the test oil sump for ease of assembly, disassembly, and cleaning.

The test oil pump and scavenge pump are operated through directdrive couplings. The test oil pump is of 0.5 gpm capacity at 1750 rpm and is driven by a variable-speed motor of 1/3-hp rating. The scavenge pump capacity is 9.0 gpm at 1750 rpm, and it is driven at this speed by a constantspeed 1/3-hp motor. In combination, the two provide excellent control of the test oil flow.

The 2-gallon test oil sump is of stainless steel construction and cylindrical in configuration (12 in. diameter  $\times$  10 in. high). It is provided with a thermocouple for controlling test oil temperature and a drain plug to facilitate removal of test oil. The test oil is heated by three band heaters mounted about the outer periphery of the sump wall and two concentric ring heaters located on the bottom of the sump. Together, the heaters supply 4700 watts to heat the sump with a uniform watt density at all heated surfaces. Power to the band heater farthest from the sump bottom is connected such

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that it is not used when the test oil is cold. Through this arrangement, the surface level of the lubricant is normally a minimum of 1 in. above any area of the sump being subjected to direct heat. This precaution was taken to minimize the occurrence of coking within the sump. Figure 18 illustrates the test oil levels relative to the heater configuration.

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A test oil filter recommended for use by the CRC Deposit and Oil Degradation Characteristics Panel⁽⁹⁾ was installed in the pressure line between the test oil sump and the test oil jet. In doing so, a test oil temperature drop between the sump and the jet of 25°F was realized at a sump temperature of 700°F. No amount of insulation appeared to reduce this temperature drop significantly. Therefore, a smaller filter housing was designed and fabricated to accommodate the 100-mesh screen element of the standard filter. When fully insulated, this modified filter housing reduced the temperature drop from 25°F to a maximum of 15°F at a 700°F sump temperature--a figure considered satisfactory by ASD.

At the request of ASD, a metal specimen holder and an air sparger tube were incorporated into the test oil sump (Fig. 17). The metal specimens were included as a means of obtaining additional information concerning the corrosive effects of test fluids on different types of metals under the various operating conditions. The holder consists of a 3/8-in. rod which is mounted on the sump lid and extends 8 in. into the sump (or 2 in. below the cold oil level). The metal samples are secured to the rod by a screw and separated from each other by 1/8-in. spacers. The rod, retaining screw, and spacers are made of stainless steel. The metal specimens are 3/4-in. diameter disks with 3/16-in. center holes by which they are mounted. A five-specimen set consisting of aluminum, titanium, silver, steel, and stainless steel was used. The air sparger tube is constructed of 1/4 in. stainless steel tubing and reaches 9 in. into the sump (or 3 in. below the cold oil level). Air is introduced, as dictated by the test condition, through eight 0.062-in. diameter holes spaced 1 in. apart and located on the bottom of the 11. in, horizontal run,

The flow path of the test oil, as well as the overall integration of the test oil sump, test machine, and accessories is shown in Figure 19. The inlet to the pressure pump is located in the general vicinity of the sump thermocouple, approximately 1-1/2 in. from the sump bottom. The pump delivers test oil to the test machine through fully insulated 1/4-in. lines and the 100mesh filter. The test oil is scavenged from the machine by way of two ports to the accumulator and then through a 3-way valve which allows flow measurement and sampling. From the 3-way valve, the test oil passes through a 40mesh screen filter (recommended by the CRC) into the scavenge pump and is then returned to the sump for recirculation.



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FIGURE 18. SCHEMATIC DIAGRAM OF TEST OIL SUMP HEATER ARRANGEMENT



### 4. Multipoint Recording Potentiometer

In order to assure maximum performance data during the testing of a lubricant, a multipoint recording potentiometer was integrated into the test instrumentation. Thus, a continuous and permanent record of all pertinent temperatures was generated for each test.

#### C. Test Procedures

#### 1. Operating Procedure

The operating procedure developed was patterned as closely to the CRC procedure(9) as the installation and program requirements would allow. The total test duration is normally 48 hr, divided into three 16-hr periods with 4-hr shutdowns between them. The procedure provides for initial stabilization of support oil and test oil temperatures at 180°F and 280°F, respectively. If the test conditions require air to the sump, lcfm of air is introduced into the sump through the air tube following the turning on of the test oil heaters. Upon reaching the conditions of stabilization, the air flow to the bearing machine (through the end cover) is set at 0.35 cfm, the test oil flow is set at 600 ml/min, the machine is started, and the load is applied. The bearing machine is allowed to operate for 1 hr without additional heat to the test bearing. If the test bearing temperature does not exceed 375°F during this period, the test bearing heater is turned on, the required test conditions are set, and the test proceeds. Each of the 16-hr periods begins when the test oil heaters are turned on and terminates when the test bearing and test oil heaters are turned off. After the heaters are turned off, the machine is allowed to run until the test bearing temperature has dropped to 400°F. At this time, the machine is stopped, t vend cover removed, and an interim evaluation made of visible deposits (the purpose of this evaluation is discussed later). The pressure and scavenge oil filters are replaced after each 16-hr period of operation. The used elements are allowed to drain for 1 hrat a temperature of 185°F. The sludge and deposits remaining are determined by weight difference. Since the time required for the test bearing to reach the 400°F shutdown temperature varies directly with the operating temperature of the test bearing, a need for a standardized downtime is necessary. A 4-hr period was selected for the duration of downtime as this allows the test bearing adequate time to cool to 400°F from the maximum operating temperature of 750°F.

The following temperatures were continuously monitored with the recording potentiometer. test bearing (3 positions around the outer ring, 120° apart), test oil sump, test oil in (jet), test oil out (2 positions at front and rear scavenge ports), support oil in, support oil out, and load bearing. In addition, instrumentation was such that temperatures of the test bearing, test oil sump, support oil in, and load bearing could be monitored at will independent of the potentiometer.

The test oil flow rate was checked each 3 hr or whenever faulty flow regulation was suspected. A 50-ml test oil sample was taken for laboratory analysis each 4 hr. Test oil was added each time, after samples were drawn, to compensate for losses resulting from consumption and the 50-ml sample.

### 2. Deposit Demerit Rating Procedure

The deposit demerit rating system provides a method for numerically describing the lubricant deposits accumulated in the test bearing section of the machine during a test. This method of rating was first adopted for use in an industry-Air Force cooperative program in 1958(3) and later standardized by the CRC Deposit and Oil Degradation Characteristics Panel(8) for CRC cooperative test work. This method is briefly reviewed in the following paragraphs.

Six specific items are visually inspected and rated in the test bearing section of the machine in order to obtain the overall rating. These items are the end cover, spacer and nut (considered to be one piece), heater mount-front, heater mount-rear, seal plate, and test bearing. The test bearing is divided into four sections: rollers, outer ring, inner ring, and cage. These sections are in turn broken down into eleven specific areas. The rollers are rated on the contact surfaces, plus the front and rear roller ends. For the outer ring, a rating is made of the contact path and the areas to the front and rear of that path. Deposits on the exposed surfaces of the inner ring are rated front and rear. Cages are rated on the front surface, the rear surface and between rollers. The test bearing rating is then obtained by dividing the summation of the demerits thus obtained by the number of areas rated, namely eleven.

A demerit rating number, ranging from 0 to 20 and defined in Table 27, is selected to identify the different types and thicknesses of deposits present on each item or area being inspected. This demerit sumber is then multiplied by a number from 0 to 10, corresponding to the percent area (0 to 100%) covered by that deposit, to obtain a rating value. Inspection should account for 100 percent of the area or item being inspected. In the event that more than one type of deposit is present on the area being inspected, the rating for that area or item is the total of the individual rating values. In any case, the deposit rated is that which is visible without the removal of another deposit (excluding test oil) Double ratings, such as sludge over varnish, are not used

Demerit Rating Number			
Light	Medium	Heavy	
1	3	5	
6	7	8	
9	10	11	
12	13	. 14	
<b>15</b> -	16	17	
18	19	20	
	Dem Light 1 6 9 12 15 15 18	Demerit Rating Nur           Light         Medium           1         3           6         7           9         10           12         13           15         16           18         19	

# TABLE 27. DEMERIT RATING NUMBERS USED FORNUMERICALLY DESCRIBING DEPOSITS

An arbitrary factor is used with each of the six items rated to account for what is considered to be the relative importance of that particular item. The overall deposit demerit rating is obtained by multiplying each of the individual item ratings by the assigned factor, totaling, and dividing this sum by the number of items which is six. The general appearance of a deposit rating sheet showing the weight factors for the individual items is as follows:

	Factor	Rating	Demerits
End cover	1	$\mathbf{x}_{\mathbf{l}}$	$\mathbf{x_1}$
Spacer and nut	2	x ₂	2X ₂
Heater mount front	3	<b>x</b> ₃	3X3
Heater mount rear	3	x ₄	3X4
Seal plate	· 1	<b>x</b> 5	<b>x</b> 5
Test bearing	. 5	<b>x</b> ₆	5% ₆
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 $\frac{\text{Total demerits}}{6} = \text{Overall demerit rating}$ 

## 3. Test Termination

Because of the expense involved in a research effort of this type, an appropriate means for maintaining costs at a minimum, while not sacrificing the ultimate objective of the test, was considered desirable. Consequently, an inspection scheme has been devised to permit the termination of a test prior to the completion of 48-hr test time, if it becomes evident that the test lubricant will not satisfactorily meet the minimum requirements imposed by the program

Interim inspections are performed following each 16-hr period of testing. At this time, the test bearing is not removed, but the end cover is removed to permit a rating of all visible deposits. In addition to these scheduled interim inspections, unscheduled interim inspections are made at any time, during the course of a test, that the test oil viscosity shows an increase of 100 percent at 210°F. If any of these interim ratings of the visible deposits indicates that the corresponding estimated overall deposit demerit rating is in excess of 60, the test is terminated. Finally, in the event that the test oil viscosity at 210°F shows a 100 percent increase after 40 hr of testing, the test is also terminated.

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#### 4. Test Oil Changes

During a test, the test oil is completely drained and replenished in accordance with the following procedure. A 50-ml sample of test oil is removed from the system at 4-hr intervals and its viscosity at 210°F immediately determined. Any sample indicating that the test oil viscosity has increased 100 percent or more requires that the bearing machine be shut down in accordance with the procedure previously outlined. The end cover is then removed and a partial deposits rating made. If this rating indicates that the test is to be continued, then the test oil sump is drained and recharged with two gallons of fresh test oil. Following this, the test is continued toward the completion of the 48-hr operating schedule. The test oil is not changed once the test has proceeded beyond 40 hr.

#### 5. Viscosity Determinations

As stated in the preceding paragraphs, test oil viscosity is determined at 4-hr intervals at a reference temperature of 210°F. Since severe degradation was anticipated in the high-temperature bearing tests, this departure from the usual viscosity reference temperature of 100°F was made in order to avoid the possibility of non-Newtonian flow at the latter temperature, particularly in view of the high pour point (+40°F) of 5P4E polyphenyl ether and the relatively poor viscosity-temperature characteristics of this fluid. A discussion of the viscosity-temperature relationship of 5P4E has been given in the preceding chapter.

#### D. Preliminary Test Results

#### 1. Operational Checks

Following the construction and assembly of the test oil system, several 8-hr checkout tests were carried out over the range of prescribed operating temperatures, using a used sample of 5P4E polyphenyl ether accumulated from a variety of different tests conducted under previous contract periods. The following maximum temperature losses between the test oil sump and the test oil in (jet) were recorded:

Bearing Temp. *F	Sump Temp, *F	Test Oil In, Temp, *F	<u>ΔT, *</u> F
500	425	417	8
550	500	490	10
650	600	588	12
750	700	685	15

The air flow and test oil flow were also varied over the probable test requirements, and equipment operation was found satisfactory for both rigs.

#### 2. Sump Skin Temperature Survey

A skin temperature survey of the test oil sumps was conducted. Three thermocouples, equally spaced, were attached to the inside walls of the test oil sumps so that the thermocouples were measuring temperatures produced at the center of a heating element. These measurements revealed that in attaining a lubricant temperature of 400°F, the sump skin temperature reached 440°F before stabilizing at 4.0°F. The period of overheating was less than 5 min. At 700°F lubricant temperature, the highest skin temperature measured was 725°F, again the duration of overheating was less than 5 min before the system stabilized at 700°F.

#### 3 Test Results on O-61-17

After experiencing some difficulty in achieving repeatability during the early stages of this study, both test machines were considered to be rating similarly when the tests to be discussed were conducted.

Five tests were completed using O-61-17 (a MIL-L-9236 fluid) as the test lubricant. A test oil flow of 600 ml/min was maintained while the test machines were operated at a bearing temperature of 500°F and a test oil sump temperature of  $425^{\circ}$ F. Also the tests were run under varied schedules with respect to shutdown times and air flow rates to the test oil sump.

Tests A and B were performed in accordance with the following time table The bearing machines were operated 16 hr and shut down 8 hr until a total of 48 hr was accumulated (3 cycles). For Test C, the schedule was unchanged. However, air was allowed to flow into the test oil sump at the rate of 1 cfm through the air sparger tube. In the case of Tests D and E, 1 cfm of air was introduced into the test oil sump, and the 48-hr test time was continuous except for very short interruptions to change filter elements. Two such filter changes were made at 16-hr intervals, and the time required to accomplish this was approximately 20 min each time. During these stops, the test oil was maintained at the operating temperature of 425°F.

The results of these tests are presented in Figures 20 and 21. Referring to Figure 20, it is seen that air bubbling through the test oil sump and continuous test operation had marked effects on deposit accumulation. Comparing the tests with the 8-hr shutdowns, the addition of 1 cfm of air to the sump increased the overall deposit demerit rating by about 35 percent.



FIGURE 20 DEPOSIT DEMERIT RATINGS OF 0-61-17 UNDER VARIOUS TEST CONDITIONS



FIGURE 21. VISCOSITY INCREASE OF 0-61-17 UNDER VARIOUS TEST CONDITIONS

If in addition to the 1 cfm of air to the sump, the tests were carried out without the 8-hr shutdowns, an increase in overall deposit demerit rating of approximately 65 percent was observed. As shown in Figure 21, oil degradation as indicated by viscosity increase was mild for all tests with the 8-hr shutdowns, and introduction of 1 cfm of air to the sump did not affect oil degradation. However, the oil degradation was markedly accelerated when the tests were conducted without the 8-hr shutdowns.

The viscosity data presented in Figure 21 showed excellent agreement between the two bearing rigs when the viscosity increase was mild. At high level of viscosity increase, the agreement, though poor, was within the range of normal experience for such degradation level (see also discussion in the preceding chapter). As to the deposit demerit ratings, Figure 20 shows that the results were in excellent agreement in both instances where comparable tests were run on the two bearing rigs.

Tables 28 through 32 present the general data obtained for the five tests. In addition to the deposit demerit ratings presented, additional evaluations of the overall demerit ratings were made independently by different operators. A summary of these separate ratings is as follows:

Operator	Test A	Test B	Test C	<u>Test D</u>	Test E
No 1 (Reported in Tables 28 through 32)	70.3	68.3	94.6	112.8	115.9
No. 2	-	-	93. 7	117. 2	110.1
Nc. 3	69.0	60. 3	90.3	118.8	-

Note that there was very good agreement between the results reported and those obtained in the additional evaluations.

E

Test Results on 5P4E Polyphenyl Ether

A total of six high-temperature tests were completed on the 5P4E polyphenyl ether F-1041 during the period covered by this report. Test conditions were 600, 650, and 700*F test oil sump temperature and.

### TABLE 28. SUMMARY DATAON 0-61-17, BEARING TEST A

### Deposit Demerit Rating

Item	Rating	Factor	Demerits
End cover	17.5	1	17.5
Spacer and nut	10	2	20
Heater mount, front	28 5	3	85 5
Heater mount, rear	58,0	3	174
Seal plate	13	1	13
Test bearing	22.3	5	111.5
			421 5

Overall Rating:  $\frac{421.5}{6} = 70.3$ 

Not included in official rating: Test oil sump wall, medium sludge Test oil sump bottom, heavy sludge

Oil consumption rate: 93 ml/hr Total accumulated filter weight: Pressure 2.9 g. Scavenge 1.1 g.

Test Time, hr	Viscosity, cs at 100°F	Neut. No., mg KOH/g
0	15 92	0.03
8	16 65	0.20
16	17.26	0.47
24	17.36	0.52
32	17.83	0.54
40	18.21	0.58
48	18.25	0.63
56	18.80	0.81
64	19.36	1.18
72	19.47	1.10

Test Oil Performance

Rig No. 1, without metal specimens	in test oil sump.
Test oil sump temperature. 'F	425
Test sil in temperature, *F	414
Test bearing temperature, 'F	500
Air flow to bearing machine, cfm	0.35
Air flow to test oil sump, cfm	None

Item Rating Factor Demerits 17.5 17.5 End cover 1 2 12.5 25 Spacer and rut 3 112.5 Heater mount, front 37 5 Heater mount, rear 54.5 3 163 5 8.5 1 8.5 Seal plate 16.5 5 Test bearing 82 5 409.5

#### TABLE 29. SUMMARY DATA ON 0-61-17, BEARING TEST B

Deposit Demorit Rating

Overall Rating:  $\frac{409.5}{6} = 68.3$ 

Not included in official rating: Test oil sump wall, medium sludge Test oil sump bottom, medium sludge

Oil consumption rate: 91 mil/hr Total accumulated filter weight: Pressure 3.8 g. Scavenge 1.5 g.

Test Time, hr	Viscosity, cs at 100°F	Neut. No , mg KOH/g
0	15.92	0 03
8	16.78	0, 20
16	17.34	0.43
24	17 41	0.52
33	17.69	0.49
40	18.16	0.65
48	18.29	0.79
56	19.11	1.03
64	20.33	1. 28
72	20. 56	1.55

#### Rig No. 2, without metal specimens in test oil sump Test oil sump temperature, °F 425 Test oil in temperature, °F 417 Test bearing temperature, °F 500 Air flow to bearing machine, cfm 0 35 Air flow to test oil sump, cfm None

Test Oil Performance

#### TABLE 30. SUMMARY DATA ON O-61-17, BEARING TEST C

#### Deposit Demerit Rating

Item	Rating	Factor	Demerits
End cover	28	1	28
Spacer and nut	22.5	2	45
Heater mount, front	67	3	201
Heater mount, rear	47	- 3	141
Seal plate	36	1	36
Test bearing	23.3	5	116.5
-			567.5

Overall Rating:  $\frac{567.5}{6} = 94.6$ 

Not included in official rating: Test oil sump wall, medium sludge Test oil sump bottom, medium sludge

Oil consumption rate: 104 ml/hr Total accumulated filter weight: Pressure 3.3 g. Scavenge 1.7 g.

#### Test Oil Performance

Test <u>Time, hr</u>	Viscosity, cs at 100°F	Neut. No., mg KOH/g
0	15. 92	0. 03
8	16.62	0, 11
16	17.27	0.34
24	17.32	0.38
32	17.54	0.47
40	17.81	0. 52
48	17.91	0. 52
56	18, 13	0, 53
64	18, 15	0, 56
72	18.50	0, 72

Rig No. 2, without metal specimens in test oil sumpTest oil sump temperature, °F425Test oil in temperature, °F418Test bearing temperature, °F500Air flow to bearing machine, cfm0.35Air flow to test oil sump, cfm1.0

TABLE 31. SUMMARY DATA ON 0-61-17, BEARING TEST D

Item	Rating	Factor	Demerits
End cover	31	1	31
Spacer and nut	37.5	2	75
Heater mount, front	59.5	3	178.5
Heater mount, rear	71.5	3	214.5
Seal plate	4	1	4
Test bearing	34.8	5	174
3			677

#### Deposit Demerit Rating

Overall Rating:  $\frac{677}{6} = 112.8$ 

Not included in official rating: Test oil sump wall, medium sludge Test oil sump bottom, heavy sludge

Oil consumption rate: 115 ml/hr Total accumulated filter weight: Pressure 12 g. Scavenge 1.3 g.

Test <u>Time, hr</u>	Viscosity, cs at 100°F	Neut. No., mg KOH/g
0	15. 92	0.03
8	16. 92	0.24
16	17.45	0.42
24	19.99	2.76
32	33, 34	7.02
40	49.05	7.85
48	79.43	11.44

#### Test Oil Performance

Rig No. 1, without metal specimens in test oil sump.Test oil sump temperature, °F425Test oil in temperature, °F415Test bearing temperature, °F500Air flow to bearing machine, cfm0.35Air flow to test oil sump, cfm1.08-hr shutdown periods not observed.

#### TABLE 32. SUMMARY DATA ON 0-61-17, BEARING TEST E

Item	Rating	Factor	Demerits
End cover	10	1	10
Spacer and nut	45.5	2	91
Heater mount. front	55	3	165
Heater mount. rear	73	3	219
Seal plate	33	1	33
Test bearing	35.5	5	177.5

#### Deposit Demerit Rating

Overall Rating:  $\frac{695.5}{6} = 115.9$ 

Not included in official rating: Test oil sump wall, medium sludge Test oil sump bottom, heavy sludge

Oil consumption rate: 112 ml/hr Total accumulated filter weight: Pressure 4.8 g. Scavenge 1.5 g.

Test <u>Time, hr</u>	Viscosity, cs at 100°F	Neut. No., mg KOH/g
0	15, 92	0.03
8	16. 78	0.18
16	17.30	0.38
24	17.56	0.49
32	19.64	2.09
40	26.19	6. 19
48	36.01	8. 29

Test Oil Performance

Rig No.without metal specimens in test oil sump.Test oil sump temperature, °F425Test oil in temperature, °F415Test bearing temperature, °F500Air flow to bearing machine, cfm0.35Air flow to test oil sump, cfm1.08-hr shutdown periods not observed.

correspondingly, 650, 700, and 750°F test bearing temperature. Each temperature condition was run with and without 1 cfm of air flow to the test oil sump. A summary of pertinent test results is given in Table 33. The individual test summaries are presented in Tables 34 through 39.

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These tests were conducted with 4-hr shutdowns between three 16-hr cycles toward a total test time of 48 hr. For these tests, the 16-hr interim deposit inspections proved to be of no value in predicting the final overall deposit rating. The items visible with the end cover removed remained unusually free of deposits through 32 hr of testing and, consequently, it was impossible to utilize these items as a basis for estimating the overall deposit rating. As can be seen in Tables 34 through 39, the major portions of deposits generally accumulated on the rear of the heater mount and test bearing, and these items could not be evaluated without disassembling the bearing machine.

Therefore, all tests were allowed to complete the 48-hr operating schedule where feasible. In doing so, a more complete indication of the performance characteristics of 5P4E under the various operating environments was obtained. Figure 22 presents a comparison of the viscosities at 210°F obtained during the tests. From this comparison, it is evident that the sump temperature of 600°F and bearing temperature of 650°F (Tests 10 and 11) resulted in rather mild oil deterioration with or without air to the sump. The actual viscosity increase amounted to approximately 10 percent. There was a total absence of deposit formation within the test oil sump, and only small amounts of sludge deposited on the filter elements. Although the filter elements showed a weight gain of more than 1 g, the weight gains did not reflect actual deposit accumulation since it was evident that test oil remained on the elements after the specified drain period. Indeed, this was true of all filter elements used in the study of 5P4E.

For Tests 14 and 15, with operating temperatures of 650°F sump and 700°F bearing, the test oil viscosity increase remained below 75 percent throughout 48 hr of testing There seemed to be no significant difference in these two tests which might be attributed to effects of air on the test oil. Figure 22 shows that both test conditions resulted in similar viscosity increase trends. The sumps remained free of deposits throughout these tests.

With respect to Tests 12 and 13, Figure 22 shows that at temperatures of 700°F sump and 750°F bearing, F-1041 experienced rather rapid breakdown. In the course of these tests, it was necessary to drain and recharge the test oil sump twice for Test 12 and once for Test 13 due to viscosity increases (see Tables 36 and 37) Neither of these tests completed the 48-hr operating schedule since they exceeded a 100 percent viscosity increase soon after 40 hr of test time. Also, it appeared that at this temperature air to the sump

SUMMARY OF BEARING TEST RESULTS ON F-1041 TABLE 33.

lest	Test Tem	perature, °F	<b>De</b> posit Demerit	% at	Vis Increat	se at 210°F Test Time	Oil Consumption	Ria
Š	히	Bearing	Rating	16 hr	32 hr	48 hr	ml/hr	No.
	Air to s	umpnone						
10	600	650	35. 7	4. 2	<b>6.</b> 3	10.0	108	1
15	, 650	200	82.7	12.7	32.7	71,5	337	1
13	700	750	98.4	52.8	37. 7 (a)	103.7(41 hr)	455	2
	Air to su	<u> 1 dm</u>						
11	600	650	59.3	5, 1	8.2	10.2	227	2
14	650	700	74.8	11.1	28.5	58.0	419	2
12	700	750	111.8	71.0	138. 0 (b)	162 4(44 hr)	290	T

Test oil changed at 24 hr Test oil changed at 20 and 32 hr (a) (d)

#### TABLE 34. SUMMARY DATA ON F-1041, BEARING TEST NO. 10

Item	Rating	Factor	Demerits
End cover	0	1	0
Spacer and nut	0	2	0
Heater mount, front	1. 2	3	3.6
Heater Mount, rear	4.6	3	13.8
Seal plate	0	1	0
Test bearing	39.4	5	197
J	•		214.4

#### Deposit Demerit Rating

Overall Rating: 214.4/6 = 35.7

Not included in official rating: Sump 100% clean Oil consumption rate: 108 ml/hr Total accumulated filter wt. Pressure 1.4 g. Scavenge 1.5 g.

#### Test Oil Performance

Test Time, hr	Viscosity, cs at 210°F	Percent Viscosity Increase at 210°F	Viscosity, cs at 100°F
0	12. 88	••	354. 5
4	13, 14	2. 0	367, 3
8	13, 31	3. 3	308.8
12	13, 36	3. 7	391.9
16	13.43	4. 3	397.4
20	13.46	4. 5	399, 3
24	13, 58	5.4	405.1
28	13.60	5.6	409.7
32	13, 69	6, 3	414. 3
36	13.83	7,4	422, 4
40	13.84	7.5	424. 3
44	14. 15	9.9	421.4
48	14, 17	10.0	427.0

#### Neutralization number: 0.0 mg KOH/g for all samples

Rig No. 1, with 5 metal specimens	in test oil sump
Test oil sump temperature, "F	600
Test oil in temperature, *F	593
Test bearing temperature, "F	650
Air flow to bearing machine, cfm	0.35
Air flow to test oil sump, cfm	None

#### TABLE 35. SUMMARY DATA ON F-1041, BEARING TEST NO. 11

#### Deposit Demerit Rating

Item	Rating	Factor	Demerits
End cover	0	1	0
Spacer and nut	48	2	96
Heater mount, front	3	3	9
Heater mount, rear	14	3	42
Seal plate	0	1	0
Test bearing	41.7	5	208.5
			355.5

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**Overall Rating:** 355/6 = 59.3

Not included in official rating: Sump 100% clean Oil consumption rate: 227 ml/hr Total accumulated filter wt. Pressure 1.5 g Scavenge 1.3 g

Test	Viscosity,	Percent Viscosity	Viscosity,		
lime, hr	<u>cs at 210° F</u>	Increase at 210°F	<u>cs at 100°F</u>		
0	12.88		354 5		
4	13. 21	2.6	367.8		
8	13, 29	3. 2	378. 3		
12	13.47	4 6	383.1		
16	13.54	5, 1	385.5		
20	13.45	4.4	391.7		
24	13.77	6.9	398.4		
28	13.85	7, 5	400.8		
32	13.94	8, 2	405.4		
36	14.00	8. 7	404.5		
40	13,99	<b>8</b> . ú	420.7		
44	14.02	8.9	430, 3		
48	14.20	10.2	420 D		

#### **Test Oil Performance**

### Neutralization Number: 0.0 mg KOH/g for all samples

Rig No. 2, with 5 metal specimens	in test oil sump
Test sump temperature, 'F	600
Test oil in temperature, 'F	590
Test bearing temperature, *F	650
Air flow to bearing machines cfm	0.35
Air flow to test oil sump. cfm	1

#### TABLE 36. SUMMARY DATA ON F-1041, BEARING TEST NO. 12

Item	Rating	Factor	Demerits
End cover	3	1	3
Spacer and nut	49.5	2	99
Heater mount, front	36.5	3	109.5
Heater mount, rear	69	3	207
Seal plate	3	1	3
Test bearing	49.9	5	249.5
•			671

#### Deposit Demerit Rating

Overall Rating: 671/6 = 111.8

Not included in official rating: Sump - Moderate coking at oil surface Oil consumption rate: 790 ml/hr

Total accumulated filter wt. Pressure 1.5 g. Scavenge 1.4 g

Test	Viscosity,	Percent Viscosity	Viscosity,
Time, hr	$cs at 210 \cdot F$	Increase at 210°F	<u>cs at 100°F</u>
0	12. 88	••	354. 5
4	13.46	4.5	393.0
8	15, 22	18, 2	520.4
12	17.19	33.5	725. 5
16	22.02	71,0	1263.3
20	32. 45	151.9	3186.0
24	14. 79*	14.8	485. 3
28	21, 78	69.1	1187.0
32	30, 65	138.0	2730.0
36	15, 98*	24, 1	562. 2
40	21. 28	65.2	1133.0
44	33, 80	162.4	3677.0

#### Test Oil Performance

Neutralization number: 20-hr 0. 2 mg, 28-hr 0. 3 mg, 32-hr would not dissolve in titration solvent, 44-hr 0. 2 mg, all others 0. 0 mg KOH/g

Rig No. 1, with 5 metal specimens	in test oil sump
Test oil sump temperature, 'F	700
Test oil in temperature, *F	691
Test bearing temperature, *F	750
Air flow to bearing machine, cfm	0.35
Air flow to test oil sump, cfm	1

[©]Changed oil

### TABLE 37. SUMMARY DATA ON F-1041, BEARING TEST NO. 13

	Deposit Den		
Item	Rating	Factor	Demerits
End cover	0	1	0
Spacer and nut	35	2	70
Heater mount, front	21	3	63
Heater mount, rear	63	3	189
Seal plate	6	1	6
Test bearing	52.1	5	260.5
-			588.5

Overall Rating: 588.5/6 = 98.1

Not included in official rating: Sump - Light coking at oil surface Oil consumption rate: 455 ml/hr Total accumulated filter wt. Pressure 2 g Scavenge 1, 4 g

#### Test Oil Performance

Test Time, hr	Viscosity, cs at 210°F	Percent Viscosity Increase at 210°F	Viscosity, <u>cs at 100°F</u>
0	12.88		354 5
4	12.98	0.8	371 6
8	15.03	16.7	507.0
12	16.52	28.3	633.8
16	19.68	52.8	957.0
20	20.65	60.3	1056.0
24	26.96	109.3	1962.0
28	14.77*	14. 7	483.6
32	17.73	37 7	733 8
36	20 71	60 8	1055.0
40	23.72	84.2	1415.0
41	26.24	103 7	1725.0

Neutralization number 24-hr 0. 1 mg. 40-hr 0. 1 mg. 41-hr 0. 2 mg. all others 0. 0 mg KOH/g

Rig No. 2, with 5 metal specimens	in test oil sump
Test oil sump temperature, *F	700
Test oil in temperature, "F	689
Test bearing temperatures "F	750
Air flow to bearing machine, cfm	0.35
Air flow to test oil sump, cfm	None

*Changed oil

#### TABLE 38. SUMMARY DATA ON F-1041, BEARING TEST NO. 14

#### Deposit Demerit Rating

Item	Rating	Factor	Demerits
End cover	0	1	0
Spacer and nut	57	2	114
Heater mount, front	15	3	45
Heater mount, rear	19	3	57
Seal plate	0	1	0
Test bearing	46.5	5	232.5
•			448.5

Overall Rating: 448.5/6 = 74.8

Not included in official rating: Sump 100% clean Oil consumption rate: 419 ml/hr Total accumulated filter wt. Pressure 1.6 g Scavenge 1.4 g

### Test Oil Performance

Test Time, hr	Viscosity, cs at 210° F	Percent Viscosity Increase at 210°F	Viscosity cs at 100° F
0	12.88	• · · · · · · · · · · · · · · · · · · ·	354. 5
4	13.26	3.0	375. 7
8	13.54	5.1	403.2
12	13.99	8.6	429, 3
16	14.31	11.1	458.7
20	14.93	15.9	496.6
24	15.30	18.8	529. 5
28	15.81	7.55	572.7
32	16.55	28.5	622.6
36	16.39	27.3	631,6
40	17.59	36.6	727. 5
44	18,06	40. 2	784.1
48	20. 35	58.0	1006. 0

#### Neutralization number: 0.0 mg KOH/g for all samples

Rig No. 2, with 5 metal specimens in	test oil sump
Test oil sump temperature, *F	650
Test oil in temperature, 'F	641
Test bearing temperature, *F	700
Air flow to bearing machine, cfm	0.35
Air flow to test oil sump, cim	1

#### TABLE 39. SUMMARY DATA ON F-1041, BEARING TEST NO. 15

Item	Rating	Factor	Demerits
End cover	0	1	0
Spacer and nut	21	2	42
Heater mount, front	18	3	54
Heater mount, rear	57	3	171
Seal plate	0	1	0
Test bearing	45.8	5	229
0			496

#### Deposit Demerit Rating

#### Overall Rating: 496/6 = 82.7

Not included in official rating: Sump 100% clean Oil consumption rate: 337 ml/hr Total accumulated filter wt. Pressure 1.6 g. Scavenge 1.4 g

	168	t off Feriorinance	
Test Time, hr	Viscosity, cs at 210°F	Percent Viscosity Increase at 210°F	Viscosity, cs at 100°F
0	12.88		354, 5
4	13.26	3.0	374.9
8	13.62	5. 7	402.4
12	14.08	9.3	431.3
16	14.52	12.7	468.2
20	15.25	18.4	522.2
24	15.66	21.6	552.7
28	16. 49	28.0	627.8
32	17.09	32.7	681.2
36	17.71	37.5	740.3
40	20. 45	58.8	1016 0

#### Test Oil Performance

Neutralization number: 0.0 mg KOH/g for all samples

19.85

22.09

44

48

Rig No. 1, with 5 metal specimens	in test oil sum	p
Test oil sump temperature, °F	650	
Test oil in temperature, °F	639	
Test bearing temperature, °F	700	
Air flow to bearing machine, cfm	0.35	
Air flow to test oil sump, cfm	None	

109

54.1

71.5

951.0

1222.5



FIGURE 22 VISCOS.TY INCREASE OF F-1041 UNDER VARIOUS TEST CONDITIONS

further encouraged deterioration of the test oil. Some coking in the sump resulted in these tests, but it is felt that this was a consequence of the high test oil consumption rates (455 and 790 ml/hr) and the addition of makeup oil at intervals which allowed the test oil to drop below the desired level discussed in the section on the test oil system. To have prevented this occurrence, it would have been necessary to add makeup oil every hour- a departure from the operating procedure for this program. These tests also point to the probability, as seen in Figure 22, that the degradated test oil remaining in the system after draining induced premature deterioration of the fresh oil when the test oil system was recharged.

The relationship between temperature and deposit formation appeared quite consistent as illustrated by Figure 23. However, it cannot be concluded from these data what significant effect the introduction of 1 cfm of air to the test oil sump had upon deposit formation.

The summary data given in Tables 34 through 39 require special comments. In every case, the addition of air to the sump brought about a considerable increase in the test oil consumption rate since the air stream served to accelerate the escape of oil vapors through the sump vent. Oil vapors passing out the sump vent were trapped and collected, with as much as two-thirds of the consumed volume recovered. Laboratory analysis of these condensates showed a slight decrease in viscosity and no significant change in neutralization number.

Tables 34 through 39 show that, with 5P4E as the test lubricant, the greatest contributing item to the overall deposit demerit rating was the test bearing. A close examination of the detailed demerit evaluations, given in Tables 40 through 45, further discloses that from 40 to 60 percent of the test bearing rating was due to the cage alone. While the presence of carbon on the cage was unquestionable, it was actually quite light. Generally speaking, the test bearings from the 5P4E tests appeared to be cleaner than the bearings from the earlier O-61-17 tests. However, because of the different types of deposits and the weighted values assigned to these deposits, this observation was not reflected in the respective bearing ratings.

The metal specimens, which were placed in the test oil sump for all 5P4E tests, failed to comonstrate any significant weight changes at any of the operating temperatures or air flow conditions.

#### F. Conclusions

Operational checks on both test installations were satisfactory, and this was further substantiated by the completion of a number of 48-hr tests







## TABLE 40.DETAILED DEMERIT EVALUATION FOR<br/>BEARING TEST NO. 10

CALL AND A

It	em	Deposit Description	Evaluation
End cover		100% clean	0
Spacer and nu	t	100% clean	0
Heater mount,	front	2% L sludge 98% clean	1.2 0
Heater mount,	rear	2% H varnish 3% L sludge 2% L smooth carbon 93% clean	1 1.8 1.8 0
Seal plate		100% clean	0
Test bearing			
Outer race:	Contact surface	50% L varnish	5
	_	50% clean	0
	Front	80% M varnish	24
		20% L smooth carbon	18
	Rear	20% L varnish	2
		55% M varnish	16.5
		5% L sludge	3
		20% L smooth carbon	18
Inner race:	Front	80% L varnish	8
		20% L smooth carbon	18
	Rear	100% L varnish	10
Rollers:	Contact surface	100% L varnish	10
	Front	80% L varnish	. 8
		10% H varnish	.5
		10% clean	Ŭ
	Rear	90% L varnish	9
		10% L smooth carbon	9
Cage:	Between rollers	100% L smooth carbon	90
-	Front	100% L smooth carbon	90
	Rear	100% L smooth carbon	90

	Item	Deposit Description	Deposit [*] Evaluation
End cover		100% clean	0
Spacer and n	ut	80% L sludge	48
		20% clean	
			v
Heater mount	t, front	5% L sludge	3
		95% clean	0
Heater mount	t, rear	10% L varnish	1
		5% H varnish	2.5
		10% L sludge	6
		5% L smooth carbon	4.5
		70% clean	0
Seal plate		100% clean	0
Test bearing			
Outer race:	Contact surface	100% clean	0
	Front	60% L varnish	6
		40% M varnish	12
	Rear	25% L varnish	2,5
-		15% M varnish	4.5
		40% H. varnish	20
,	· ·	20% L sludge	12
Inner race:	Front	90% M varnish	27
•		10% L smooth carbon	9
	Rear	70% L varnish	7
	• •	30% L smooth carbon	27
Rollers:	Contact surface	100% clean	0
	Front	20% L varnish	2
		30% M varnish	9
	: :	10% H varnish	5
		30% L smooth carbon	27
		10% clean	0
•	Rear	35% L varnish	3.5
•		35% M varnish	10.5
		10% H varnish	5
		20% clean	0
Cage:	Between rollers	100% L smooth carbon	90
	Front	100% L smooth carbon	90
· · ·	Rear	100% L smooth carbon	90

# TABLE 41. DETAILED DEMERIT EVALUATION FORBEARING TEST NO. 11

	Item	Deposit Description	Deposit Evaluation
End cover		5% L sludge	3
Spacer and nut			U
-1 and 1		15% M varnish	4.5
		15% L sludge	45
		10% clean	0
Heater moun	t, front	30% L varnish	3
		30% M varnish	9
		35% L sludge	21
		5% M sludge	3.5
Heater moun	t, rear	20% L varnish	2
		15% L sludge	9
		5% M sludge	35
		55% L smooth carbon	49.5
		5% M smooth carbon	5
Seal plate		5 % L sludge	3
-		95% clean	0
Test bearing			•
Outer race:	Contact surface	100% clean	0
	Front	10% L varnish	1
		20% M varnish	6
		35% H varnish	17.5
		35% L smooth carbon	31.5
	Rear	10% H varnish	5
		10% L sludge	6
		70% L smooth carbon	63
		10% M smooth carbon	10
Inner race:	Front	50% L varnish	5
		50% M varnish	15
	Rear	40% L varnish	4
		30% H varnish	15
		30% L smooth carbon	27
Rollers	Contact surface	100% clean	0
	Front	45% H varnish	22. 5
		45% L smooth carbon	40.5
		10% clean	0
	Rear	20% H varnish	10
		80% clean	0
Cage	Between rollers	100% L smooth carbon	90
	ront	100% L smooth carbon	90
	Kesi	100% L smooth carbon	90

## TABLE 42.DETAILED DEMERIT EVALUATION FOR<br/>BEARING TEST NO. 12

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## TABLE 43. DETAILED DEMERIT EVALUATION FOR<br/>BEARING TEST NO. 13

(C) (

	Item	Denosit Description	Deposit Evaluation
		Deposit Description	
End cover		100% clean	0
Spacer and n	ut	10% H varnish	5
		50% L sludge	30
		40% clean	0
Heater moun	t, front	30% L varnish	3
		30% L sludge	18
		40% clean	0
Heater moun	t, rear	5% H varnish	2.5
	·	20% L sludge	12
		5% M sludge	3.5
		10% L smooth carbon	9
		5% M smooth carbon	5
		15% L crinkled carbon	18
		10% M crinkled carbon	13
		30% clean	0
Seal plate		10% L sludge	6
Test bearing		90% clean	0
Outer race:	Contact surface	100% clean	0
	Front	100% L smooth carbon	90
	Rear	15% L sludge	9
		85% L smooth carbon	76.5
Inner race:	Front	50% L varnish	5
		25% M varnish	7.5
		25% L smooth carbon	22.5
	Rear	40% L varnish	4
		60% L smooth carbon	54
Rollers:	Contact surface	100% clean	0
	Front	35% L varnish	3, 5
		35% M varnish	10.5
		10% L smooth carbon	9
• •		20% clean	0
	Rear	30% L varnish	3
		10% L smooth carbon	9
		60% clean	0
Cage:	Between rollers	100% L smooth carbon	90
	Front	100% L smooth carbon	90
	Rear	100% L smooth carbon	90

## TABLE 44. DETAILED DEMERIT EVALUATION FOR<br/>BEARING TEST NO. 14

I	tem	Deposit Description	Deposit Evaluation
End cover		100% clean	0
Spacer and nu	ıt	95% L sludge	57
		5% clean	0
Heater mount	, front	25% L sludge	15
		75% clean	0
Heater mount	, rear	10% M varnish	3
		5% H varnish	2.5
		15% L sludge	9
		5% L smooth carbon	4.5
		65% clean	Ű
Seal plate		100% clean	U
Test Bearing			•
Outer race:	Contact surface	100% clean	U NO
	Front	60% M varnish	10
		30% H varnish	12
	D	10% L smooth carbon	<b>y</b>
	Kear	10% M varnish	)7 E
		35% H Varnish	17.5
		20% L studge	12
		55% L smooth carbon	JI. J
Inner race:	Front	20% L varnish	2
		50% L smooth carbon	45
		30% clean	0
	Rear	20% L varnish	2
		50% L smooth carbon	45
		30% clean	0
Rollers:	Contact surface	100% clean	0
	Front	60% M varnish	18
		5% H varnish	2.5
		5% L smooth carbon	4.5
		30% clean	0
	Rear	80% L varnish	8
		10% L smooth carbon	9
		10% clean	Ŭ
Cage:	Between rollers	100% L smooth carbon	90
	Front	1004 L smooth carbon	90
	Rear	100% L smooth carbon	90

## TABLE 45. DETAILED DEMERIT EVALUATION FOR<br/>BEARING TEST NO. 15

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Item		Deposit Description	Deposit Evaluation
End cover		100% clean	0
Spacer and nut		35% L sludge	21
		65% clean	0
Heater mount, front		30% L sludge	18
		70% clean	0
Heater mount, rear		20% M varnish	6
		40% L sludge	24
		10% L smooth carbon	9
		10% L flaked carbon	18
		20% clean	0
Seal plate		100% clean	0
Test Bearing	5		
Outer race:	Contact surface	100% clean	0
	Front	65% M varnish	19.5
		35% H varnish	17.5
	Rear	20% M varnish	6
		45% H varnish	22.5
		15% L sludge	9
		20% L smooth carbon	18
Inner race:	Front	70% M varnish	21
		30% H varnish	15
	Rear	100% M varnish	30
Rollers:	Contact surface	50% L varnish	5
		50% clean	0
	Front	90% H varnish	45
		10% clean	0
	Rear	70% L varnish	7
		20% L smooth carbon	18
		10% clean	0
Cage:	Between rollers	100% L smooth carbon	90
	Front	100% L smooth carbon	90
	Rear	100% L smooth carbon	90

embracing all test conditions. Reasonable repeatability was achieved between both bearing machines, and lubricants were not subjected to significant overheating in attaining operating sump temperatures.

Referring to tests on O-61-17, a MIL-L-9236 lubricant, air flow to the test oil sump evidently had an undesirable effect upon the deposit formation behavior of the lubricant. In addition, continuous test operation was shown to encourage degradation of this fluid. However, it should be kept in mind that these observations could well be characteristic of O-61-17 and not necessarily typical for all fluids.

Test data obtained for the 5P4E polyphenyl ether indicated that satisfactory performance, both from the standpoint of deposit formation and oil degradation, could be expected of this lubricant at a sump temperature of 600°F and a bearing temperature of 650°F. The rated items remained exceptionally clean throughout the tests at these temperatures. The test bearing cage was responsible for a considerable portion of the overall deposit demerit rating though its general appearance was not indicative of extreme deposits. Also, the moderate viscosity increase, coupled with no change in neutralization number, for the duration of the tests lent additional support to the excellent performance of this lubricant at these test conditions.

At temperatures of 650°F sump and 700°F test bearing, the 5P4E polyphenyl ether began to show more significant deposit accumulation. Small amounts of the more severe deposits, chiefly smooth and flaked carbon, were formed during the tests; also, the viscosity increase became more pronounced suggesting that, under these operating conditions, this lubricant might be considered marginal in performance.

With respect to the most strenuous temperature environment, the 5P4E polyphenyl ether was found to produce considerable amounts of carbon deposits of a smooth, crinkled, and flaked nature. At 700°F sump and 750°F bearing temperatures, this lubricant deteriorated more readily than at the lower test temperatures and did not complete the 48-hr test duration even with intermediate oil changes. It must be concluded that the 5P4E material did not give reasonable satisfaction at a sump temperature of 700°F and a bearing temperature of 750°F.

In general, the addition of air to the sump did not give any particular influence pattern in the 5P4E tests. It did, however, bring about a higher test oil consumption rate through entrainment of oil vapors in the air stream passing out the sump vent. Because trapped oil vapors indicated slight viscosity changes, it is recommended that any future work on 5P4E be designed to investigate the possibility of collecting and returning the condensate to the test oil sump.

Results with the five-metal specimen set showed that 5P4E has no particular corrosive effects upon aluminum, titanium, silver, steel, and stainless steel at any of the aforementioned test conditions.

#### V. GEAR LOAD-CARRYING CAPACITY

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#### A. General Remarks

The objectives of the gear load-carrying capacity phase of the program were to develop apparatus and techniques for determining the loadcarrying capacity of lubricants at high temperatures and to evaluate the gear load-carrying capacity performance of candidate lubricants under environmental conditions representative of Mach 3 class gas turbine engine designs.

Two WADD high-temperature gear machines previously developed and available at SwRI under Contract AF 33(616)-7223(5) were employed in this work. These two machines had accumulated nearly 1000 hr of satisfactory service in the prior program, mainly at low to moderate operating temperatures. Their load system had also been calibrated and found satisfactory under these conditions.

In the current program, operating temperatures exceeding those previously used were anticipated. In order to insure reliable test results at high temperatures, dynamic calibration of the load system was extended to test gear temperatures up to 700 °F. These calibration studies revealed that the diametral clearance of the support roller bearings in the machine previously used at low to moderate temperatures (0.0005 in.) was inadequate for the current program. Accordingly, the diametral clearance of the support roller bearings was increased to 0.0025 in., and this proved to give excellent machine calibration at test gear temperatures up through 600 °F.

The dynamic calibration also revealed that the backlash of the test gears used in the previous program (0,005 in.) was inadequate for hightemperature work. Consequently, the backlash was increased to 0.011 in., an amount adequate for test gear temperatures up to 700°F.

Load-carrying capacity determinations were made using Nitralloy N steel test gears on nine lubricants, selected for their varied composition, at test gear temperatures up to 700°F on one, 600°F on another, 500°F on four others, and 425°F on the remaining three. Of the lubricants evaluated beyond 425°F, all but one showed an increase in load-carrying capacity as the temperature was increased beyond 425°F. The 5P4E and 4P3E polyphenyl ethers were shown to give rather modest load-carrying capacities throughout the temperature range investigated.



An investigation was conducted to determine the possibility of a correlation existing between the load-carrying capacity results obtained with standard Ryder test gears at 165°F test conditions and those obtained with Nitralloy N steel test gears at either 165 or 425°F test conditions. The results showed no evidence of any organized correlation between the load-carrying capacity results obtained using standard Ryder test gears and those obtained using Nitralloy N steel test gears. However, a correlation appeared to exist between the load-carrying capacities of all lubricants evaluated at the 165 and 425°F test conditions, when only one test gear material, Nitralloy N steel in this case, was used.

#### B. <u>Test Apparatus</u>

### 1. WADD High-Temperature Gear Machine

The experiments reported herein were conducted using two WADD high-temperature gear machines developed under Contract AF 33(616)-7223(5) Figure 24 shows a cross section of the WADD hightemperature gear machine. The operating principle of this machine is almost identical to that of the Ryder gear machine (10). However, improvements in materials and design have been made to extend its operating capability. Briefly, the WADD high-temperature gear machine differs from the Ryder gear machine in that each shaft is supported by two doublerow roller bearings instead of three journal bearings. It has one load chamber located on the end of the driven shaft, rather than two load chambers located in the middle portion of both shafts. Screw-thread type nonrubbing seals. rather than elastomer seals, are used to separate the test oil and support oil chambers. The case is made of tool steel to improve structural stability at elevated temperatures. Tests have shown that the machine is capable of operating at speeds up to 30,000 rpm and at test gear temperatures up to 800°F, though not simultaneously.

The WADD high-temperature gear machine with high-temperature test gears and an induction heating coil installed is shown in Figure 25. The induction heating coil is normally rigidly attached to the test end cover and is removed with the cover each time the cover is removed.

In the operation of the gear machine, the test gear tooth load is obtained by the application of controlled hydraulic load in the load chamber. X (Fi_x, 24). This hydraulic load causes a slight axial movement of one shaft relative to the other and is converted into a tangential load on the replaceable spuritest gears. T and U, through the action of the integral helical slave gears, R and S. The test gear tooth load is computed from the applied hydraulic load and the geometry of the load system.





FIGURE 25 PHOTOGRAPH OF WADD HIGH-TEMPERATURE GEAR MACHINE WITH HIGH. TEMPERATURE TEST GEARS AND INDUCTION HEATING COIL INSTALLED The hydraulic load (or load oil pressure) is controlled automatically by means of a pneumatic controller-recorder. This system affords automatic setting and control of the load oil pressure to within  $\pm 0.25$  psi over a range of 0 to 120 psig. This corresponds to a sensitivity of approximately  $\pm 10$  lb/in. for tooth loads ranging from 0 to 5600 lb/in., which is the load range of the machine. The rate of load application is constant for any one load setting and is independent of the operator.

The WADD high-temperature gear machines are driven by standard Erdco 50-hp drive units which were modified earlier(4) to permit operation at speeds up to 30,000 rpm through a step-up gear ratio of 9.25:1.

#### 2. Test Oil System

For high-temperature lubricant tests, where some degree of test oil deterioration during test appears to be unavoidable and must therefore be minimized or controlled as much as possible, it was felt that direct contact of the test oil with a high-wattage immersion heater should be avoided. With this in mind, the test oil system. shown schematically in Figure 26 and in an "exploded view" in Figure 27, was previously designed and fabricated under Contract AF 33(616)-7223. This system is capable of being operated at bulk oil temperatures up to and including 400°F. The test oil is heated by means of a heat exchanger placed inside the sump. By this means, excessive localized heating of the test oil is avoided. The accessories, with exception of the drive motor for the pressure pump, are located inside the sump. The sump is made of stainless steel and is double-walled. Heat loss from the sump is minimized by means of a low-wattage band heater located in the space between the inner and outer walls. The lines between the sump and the gear machine are made very short, in a further effort to minimize heat loss. With these precautions, the temperature of the test oil in the sump need be only a few degrees higher than that entering the machine, and a minimum of deterioration of the test oil is obtained during a test. The location of the pump in the sump eliminates the problem of leakage through the pump seals. Further, by minimizing the temperature differential between the pump interior and its surroundings, the mechanical reliability of the pump is enhanced.

#### 3. Support Oil System

With induction heating to heat the test gears in the high-temperature load-carrying capacity studies, the standard Erdco support oil system⁽¹⁰⁾ is used to lubricate the support section of the high-temperature gear machine and to supply load oil pressure. Apart from the lubricating function, the 165⁴F support oil is also used as a control coolant in maintaining the test gear temperature by carrying away excess heat from the gear machine bearings and shafts.





#### 4. <u>High-Temperature Test Gears</u>

For the work described herein, special test gears made of Nitralloy N steel were used. These special gears have the same principal dimensions as the standard Ryder test gears (10) but conform to SwRI design with respect to the chordal thickness and the tooth width of the wide gear. A modification was made to the design of the wide test gear to increase the backlash of the gear set. This increased backlash (0.011 in.) was determined to be necessary for tests to be conducted at gear temperatures above  $400^{\circ}\text{F}$ . Details of this modification are described in a subsequent section of this report. The principal dimensions of the high-temperature test gears are shown in comparison with those of the standard Ryder test gears in Table 46. In Table 46, the case hardness is given in Rockwell 15 N units and the core hardness is given in Rockwell C units, the usual units of these measurements.

#### 5. Induction Heating of Test Gears

In the induction heating of the test gears, the induction coil is placed closely and accurately with respect to spacing around the hub of both the narrow and wide test gears. A photograph of one of the induction heating coils used is shown in Figure 25. With the gears turning in the induction field, the heat is evenly distributed from the hub outward to the gear teeth. This insures that the heat distribution and temperature are very nearly equal in both the narrow and wide gears which further insures that the lubricant in the gear mesh is subjected to nearly equal temperature conditions.

### 6. <u>Temperature Measurement of Test Gears</u>

The temperature of the test gear is measured by an industrial infrared radiometer. The radiometer measures the infrared radiation from the test gear. This measurement is related to temperature by means of calibration against thermocouple readings. These data are then plotted, and a radiometer output versus temperature curve is obtained. It is from this curve that the test gear temperature is determined. This method was developed and calibration curves obtained under Contract AF 33(616)-7223(5). The system of gear temperature measurement and control is shown in Figures 28 and 29.

The gear-blank temperature measurement is made at a point on the web of the narrow gear near the mesh of the test gears. Ideally, the temperature measurement and control should be at the mesh of the gear teeth, but, since the gear teeth undergo changes during a test from
# TABLE 46.COMPARISON OF PRINCIPAL DIMENSIONS OF STANDARDRYDER TEST GEARS WITH HIGH-TEMPERATURE TEST GEARS

	Standard Ryder Test Gears	High-Temperature Test Gears
Pitch Diameter, in.	3.500	3. 500
Face Width, Narrow Gear, in.	0.250	0.250
Face Width, Wide Gear, in.	0.937	0. 375
Number of Teeth	28	28
Diametral Pitch	8	8
Pressure Angle, degree	22. 5	22. 5
Tip Relief	None	None
Material	AMS-6260	Nitralloy N
Case Hardness, Rockwell 15N	90-92	90-92
Case Thickness, in.	0.025-0.040	0. 018-0. 024
Core Hardness, Rockwell C	30-40	30-40
Surface Finish, rms, in.	20-35 × 10-6	20-35 × 10-6
Backlash, in.	0.002-0.006	0.011-0.014







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the standpoint of infrared emissivity, it was decided that the gear web was the next most logical measuring point. Since the observation of the gear web is continuous while the gear is turning, it is expected that the emissivity factor will be very nearly constant, thereby insuring that little error in temperature measurement from the standpoint of emissivity will be obtained. To further insure a constant emissivity-temperature relationship during a test, it is necessary to obtain approximate black-body radiation from the test gear web. This is obtained by making the gear web black in color by electroplating the web of the gear with black chromium.

A calibration apparatus is located near the machine such that the output of the radiometer can be checked at any time it is felt necessary. This apparatus consists of two steel blocks so machined that when a gear is placed between them a form fit on the gear is obtained. The apparatus, shown in Figure 30, is heated by cartridge heaters placed in the steel blocks as shown The radiometer is focused on the plated portion of the gear web through a hole in the block. Thermocouples are fitted into the web of the gear from the side opposite the point at which the radiometer is focused, and to a depth such that they are within  $1/32 \mu$ in. of the face of the web. With this apparatus, thermocouple-radiometer output may be checked over the desired temperature range.

A more rapid but somewhat less accurate check has been made of the radiometer by painting the gear teeth of a used test gear with temperature-indicating paints. The gear is then placed on the machine and, with the induction heating coil in place, the gear is heated until the paints melt. The radiometer output is noted at the melting point of each of the paints. These paints are available in 50°F melting-point increments from 400 to 1000°F.

## 7. Closed-Circuit Television for Gear Inspection

To insure safety of the operating personnel from exposure to high-temperature parts and toxic fumes, it is now standard practice at SwRI to locate all high-temperature test rigs in individual test cells that are well ventilated, and to perform as many of the controlling and inspection operations as possible from outside the test cells. The closed-circuit television method for gear inspection, developed previously ⁽⁵⁾, has been adopted for general use in gear lubrication experiments at SwR! and approximately 400 scuff ratings have been obtained with this method. During a number of such tests, satisfactory checks of the television ratings with the standard visual-microscopic ratings were obtained



#### C. Test Procedures

## 1. High-Temperature Load-Carrying Capacity Tests

The procedure used in the high-temperature load-carrying capacity studies differs only slightly from Federal Test Method 6508 in that a different machine (WADD high-temperature gear machine), special test gears, and induction heating of the test gears are used. A comparison of the two test methods is shown in Table 47.

The specific WADD high-temperature gear machine operational procedure is as follows: A warm-up period is allowed with all systems functioning with the exception of the drive and the induction heating systems. After attaining the desired test oil and support oil temperature equilibrium, the drive system is activated and the machine is driven at 10,000 rpm. The induction heat control system is set at the desired temperature, and the test gear temperature is obtained and controlled automatically. The time required to heat the test gears to 500°F is approximately two minutes. The desired load is next set into the load system which automatically loads and controls the load on the gear teeth. After the load is obtained, the interval timer is set for the standard load duration time of ten minutes. Five minutes after start of the load duration, all temperatures and pressures are noted and recorded. At the end of the ten-minute period, the timer shuts down the drive. The operator then, in reverse order to that given above, turns off the load and induction heat to The machine is then stopped and the narrow gear teeth are the gears inspected. The procedure is then repeated for the next higher load. The test is terminated at least one load step after an average of 22.5 percent scuff is obtained on the narrow gear.

### 2. Gear Machine Calibration

The procedure used in the calibration of the WADD hightemperature gear machine is the same as that used in the high-temperature load-carrying capacity test procedure. The only exception is that the time at temperature equilibrium is extended so as to obtain three points at strain equilibrium. This is necessary because the sensitivity of the strain output to temperature variation is approximately  $5\mu$  in./in./*F. The strain data are recorded at each gear temperature and load level. These data are plotted versus load oil pressure or shaft torque at each gear temperature. The load deviation is then determined by a comparison of the strain-load curve with the static, dead-weight load curve.

TABLE 47. COMP	ARISON OF LOAD CI	ARRYING CAPACITY TEST	r methods
	Federal Test Method 6508	Methods Used in 165°F Test	Fresent Frogram <u>&gt; 400°F</u> Test
Test Machine	Erdco-Ryder gear machine	W ADD hígh-temperature gear machine	W ADD high-tempera gear machine
Test Gears	Ryder test gears	Special Nitralloy N test gears	Special Nitralloy N t gears
<b>Operating Conditions</b>	·		
Test gear speed, rpm Test oil four rate milmin	10,000 ± 10	10,000 ± 100	10,000 ± 200
	4 4 0 7 7		
			C ∓ 0/2
sear vir in temperature, r Support oil-in temperature	165 ± 5	103 # 3 165 # 5	400 ± 5 165 ± 10
			• • •
Test Gear Temperature	Not controlled	Not controlled	Controlled at requir- test temperature
Method of Loading			
Increment steps in tooth load (corresponding to 5-psi steps			
in load oil pressure), lb/in.	370	230	230
Duration of load-increment steps, mín	10	10	10
Criterion of Lubricant Rating	Tooth load at which 22.5% of	Tooth load at which 22.5% of working tooth	Tooth load at which 22. 5% of working toc
	working tooth area is scuffed	area is scuffed	area is scuffed

## D. Calibration of the WADD High-Temperature Gear Machine

## 1. Apparatus and Technique

In both the standard Ryder gear machine and the WADD hightemperature gear machine, load on the gear teeth is obtained by the application of an axial hydraulic pressure on the load shafts, which is converted into load on the gear teeth through the action of the helical slave gears made integral with the shafts. The tooth load is computed from the axial hydraulic pressure applied and the geometry of the load system (neglecting friction). In an effort to establish the validity of the computed relationship, a program was initiated under AF 33(616)-7223 to calibrate the load system of the WADD high-temperature gear machine⁽⁵⁾, and continued at higher temperatures in the present program.

A number of different approaches were considered as a means of obtaining a meaningful calibration of the machine.. Of the several approaches considered, it was decided that the calibration would be made by taking torque measurements of the gear machine drive shaft by means of strain gages located in the area of the screw-thread nonrubbing seal. This method presented several advantages in that a special gear would not be required, any test gear could be used and changed as necessary, the strain gage service would be less severe and could be air-cooled as necessary, and the changes to the drive shaft to accommodate the strain gages would de minor and would not affect the shaft with respect to its use in normal load-carrying capacity testing.

In developing the technique and method of applying the strain gages to the machine shaft, a considerable amount of time and effort went into trials and errors in the selection of an applin able strain gage and mounting technique from all those recommended. The main difficulty encountered in most instances was the bonding of the gages to the shaft such that the bond would withstand the extremely adverse conditions of temperature and the very high strain frequencies which exist during dynamic operation. The mounting technique now in use appears to be satisfactory from the standpoint of many hours of trouble free operation.

The load system calibration apparatus consists of four uniaxial etched-foil bakelite-backed FAB-12-12 strain gages, a four-channel mercury slip ring assembly and a strain-gage strain indicator. Two pairs of strain gages are mounted in a milled-out area 180° apart on the drive shaft of the gear machine in the area of the screw-thread nonrubbing air seal as shown in Figure 31. The strain gages are mounted in the receased area with EF 400 or xx connect, and the remaining void is filled with the same cement containing an asbestos binding fiber. The entire shaft is then



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F.GURE 31 STRAIN GAGE INSTALLATION TO ALLOW STANDARD NONRUBBING SEAL TO BE USED thermal-cycled in various increments of temperature to 500°F over a period of 24 hr. The protruding and irregular excess epoxy is then machined to the dimension of the air seal surface of the shaft. After stress relieving to 2000 in. -1b of torque in increments of 200 in. -1b, the system is ready for dynamic operation.

The static, dead-weight load system used in the calibration work is self-explanatory and is shown in Figure 32. A schematic diagram of the full bridge strain-gage system is shown in Figure 33.

#### Calibration Results

During the dynamic load system calibration carried out earlier(5), there were indications that the standard diametral clearance (0.0005 in.) of the support roller bearings in the WADD high-temperature gear machine was probably inadequate for the 400°F conventional loadcarrying capacity tests (10,000 rpm, 400°F test oil and support oil temperatures). This conclusion was reached because the calibration results had a greater scatter for the conventional 400°F test conditions than for the conventional 165°F conditions (10,000 rpm, 165°F test oil and support oil temperature). Although no difficulties were experienced with the 400°F induction heating load-carrying capacity tests (10,000 rpm, 165°F test oil and support oil temperatures, 400°F induction heated gear temperature). it was reasoned that the support roller bearing clearance was on the low side if the induction-heating tests were to be extended to gear temperatures substantially beyond 400°F. Accordingly, it was decided that studies should be made to establish the bearing clearance required to give satisfactory machine operation and repeatable load system performance over as wide a range of conditions as possible.

In the early studies, it was shown that with a diametral clearance of 0.005 in., a 0.0045-in. increase over the standard clearance of 0.0005 in., the dynamic calibration results for the 400°F conventional test conditions were comparable to those for the 165°F conventional test conditions. It was therefore decided that the bearing clearances for drastically different test conditions should be considered separately, and that an effort should be made to use a bearing clearance no more than needed for the particular set of test conditions. Further, since the current program is concerned primarily with operation at 10,000 rpm and gear temperatures above 400°F, the effort should primarily be directed toward this application.

With the above considerations in mind, dynamic load system calibration was made with a diametral support roller hearing clearance of



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0.0025 in., at 10,000 rpm and 165°F test oil and support oil temperatures. Bearings with 0.0025 in. diametral clearance were obtained by grinding the inner surface of the outer ring of the standard bearings. The results obtained are shown in Figure 34. Comparison of this figure with Figure 35 shows that the calibration results for 0.0025-in. diametral bearing clearance were comparable to those for 0.005-in. clearance.

Under the present program, calibration studies were made at 400, 500, 600 and 700°F test gear temperature conditions (10,000 rpm, 165°F support oil temperature, 400°F test-oil-in temperature). The test gears were heated by induction heating. The results are shown in Figures 36, 37, 38, and 39. As can be seen in Figure 36, the 400°F calibration results compared very favorably with those obtained at 165°F conditions (Fig. 34). A very slight inflection in the 500 and 600°F calibration curves was noted in Figures 37 and 38 at load oil pressures of 20 to 30 psig. At this time, there is no explanation for this deviation. It is felt that the deviation may be due to a temperature effect on the strain gage reading. Since the deviation is positive, the deviation cannot be due to any resistance to axial movement of the load shaft (due to a loss in clearance of the support roller bearings), loss of strain gage bond, strain gage cement creep, or change in the strain gage factor, for all these factors will always give a negative deviation. The only factors known which will give a positive deviation areachange in the strain gage environmental temperature or a resistance to turning of the driven or load shaft. A change in the strain gage temperature is not considered creditable, since great care has been exercised to obtain thermal equilibrium at each of the load increments. Also, the resistance to turning of the load shaft would have to be due to rubbing of the shaft on the air and/or load seal. It is felt that if any rubbing of the shaft occurred, there would be as much or more resistance to axial movement of the shaft, thereby cancelling or overriding the effect. The 700°F calibration results (Fig. 39) indicated a slight resistance to loading which is assumed to be due to inadequate diametral roller support bearing clearance. Further increases in the roller support bearing clearance will be deferred until such time that 700 °F load-carrying capacity determinations become the primary objective of the gear lubrication program. In the meantime, the results shown indicate that proper loading is being obtained by the load system and that the 0.0025-in. diametral roller support bearing clearance is sufficient at test gear temperatures up to and including 600 ° F .

## E. Preliminary Test Results

High-temperature gear lubrication investigations, with induction heating of the test gears, were carried out at 400°F under Contract



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FIGURE 35. COMPARISON OF STATIC AND DYNAMIC CALIBRATION CURVES AT 165°F WITH 0. 005-IN. DIAMETRAL SUPPORT BEARING CLEARANCE





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AT 500°F WITH 0.0025-IN. DIAMETRAL SUPPORT BEARING CLEARANCE







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AF 33(616)-7223(5). This work was done in order to compare the results obtained with the induction heating procedure with those obtained in the early high-temperature investigations, using the 400°F conventional heating procedure (400°F test oil and support oil temperature, test gears at temperature equilibrium of approximately 400°F). These data, shown in Table 48, were obtained using Nitralloy N steel test gears with standard backlash (0.005 in.) and standard diametral support bearing clearance (0.0005 in.). Since it was subsequently shown that the load-carrying capacity values obtained with the standard backlash and standard support bearing clearance were questionable, no emphasis is placed on the actual values shown.

It was observed in the course of the above study (Table 48) that the test gear temperature in the conventional 400°F tests, measured at the load at which 22.5 percent scuff was obtained, was always above 400°F. With the induction heating method, the gear temperature was controlled at 400°F. Therefore, the increased load-carrying capacity for the lubricants tested, using induction heating at a controlled gear temperature of 400°F, was attributed to this difference in gear temperature. From these data, it was concluded that the induction method of heating the test gears produced no adverse effects in load-carrying capacity determinations and could be used with confidence in high-temperature test method development.

In an effort to determine the effectiveness of induction heating and operation of the WADD high-temperature gear machine at test gear temperatures above 400°F, load-carrying capacity determinations were obtained on several lubricants using Nitralloy N steel test gears at controlled gear temperatures, varied in 50 or 100°F increments, from 400 to 700°F. The test lubricant supply temperature was maintained at 400°F. and the support oil supply temperature was maintained at 165°F. The test lubricants were selected on the basis of their 400°F load-carrying capacity ratings such that high, medium, and low load-carrying capacity lubricants were included in the study. No great amount of difficulty was encountered in the gear machine operation or temperature control of the inductionheated gears during the tests. The first difficulty encountered was excessive smoke in the gear case, at gear temperatures of 500°F and above, which interfered with the infrared radiation measurements This was overcome by installing a phenolic tube in the gear cover with the free end in close proximity to the area of the marrow test gear at which radiation measurements were made. With a small exhaust blower attached to the gear case, a slight draft of air was drawn through the tube thereby clearing the area of smoke and the small amount of oil mist normally present

## TABLE 48. COMPARISON OF 400°F LOAD-CARRYING CAPACITY RESULTS OBTAINED WITH THE INDUCTION HEATING METHOD AND THE CONVENTIONAL 400°F METHOD USING NITRALLOY N STEEL TEST GEARS

	400 °F Induction	Heating M	ethod	Conventional 400°F Method			
	Average	Load-C	arrying	Average	Load-Carrying		
	Gear Temp.	Capacit	y, 1b/in.	Gear Temp.	Capacity	r, 1b/in.	
Oil Code	at 22.5% Scuif, *F	<u> </u>	<u>B</u>	at 22. 5% Scuff, *F	<u>A</u>	_ <u>B</u>	
GTO-313	400	2060	2540	480	2340	4310	
	400	2620	2380	480	3080	2840	
				480	1560	1520	
				480	1270	1970	
		240	<u>o</u>		23	<u>60</u>	
GTO-770	400	5990(a)	6230 ^(a)	485	4580	4710	
				485	3810	4220	
				485	3940	4670	
		<u>611</u>	<u>0</u>		43	20	
GTO-855	400	2910	2490	430	1240	1080	
	400	3320	3220	430	790	1030	
				430	1330	1430	
				430	1360	1220	
		299	0		119	90	
GTO-915, O-60-23	400	1110	1620	420	1450	1150	
	400	2100	1450	420	950	1090	
				420	710	1670	
				420	740	1150	
		157	0		<u>11</u>	10	
GTO-939	400	1840	1570	490	1340	1670	
	400	1470	1600	400	1540	1220	
		162	0	· · · · ·	14	40	
LRO-11	400	1830	1710	420	1280	1310	
	400	1400	1640	420	1520	! 400	
				420	840	1 500	
	÷		÷.,	420	1250	1510	
	•	164	0		13	30	
LRO-13	400	1780	1990	•	1620	1720	
	400	2160	2460		1430	1640	
		·		•	1769	1880	
	•			•	1710	1410	
	•	210	<u>10</u>		16	50	
	•					•	

(a) Values obtained by extrapolation. Test was terminated at \$500 lb/in. tooth load.

During testing at gear temperatures of 500°F and higher, it was noted that the gear machine speed would drop somewhat while the gears were being heated to the desired test temperature. This drop in machine speed was believed to be due to thermal expansion of the test gears, thereby decreasing the test gear clearance and backlash, with interference loading of the test gears being the final result. A rough check on the gear machine starting drive torque was made by use of a torque wrench on the end of the driven shaft while the test gears were induction-heated over a temperature range of 400 to 600°F. A plot of these rough data is shown in Figure 40. A noticeable increase in torque was obtained at gear temperatures between 550 and  $600^{\circ}$ F. It was clear that means must be sought to eliminate the gear tooth interference, in order to permit gear lubrication studies to be made at high temperatures. The simplest way to do so, without otherwise changing the design characteristics of the test gears, appeared to be an increase in the gear backlash. Further, it was decided that the geometry of the narrow test gears (those used for rating purposes) should not be changed and that any additional backlash required should be obtained by modifying the wide test gears.

Measurements showed that the nominal backlash of the standard Nitralloy N steel test gears was 0.005 in.; and, with this backlash, it was found that tooth interference was likely to occur at a gear temperature of 450 to 500°F. From calculations based on the coefficient of expansion of Nitralloy N steel. it was found that the nominal backlash required to avoid interference was approximately 0.008 in. at 700°F and 0.010 in. at 800°F, In order to check these calculations, used wide gears with very low amounts of scuff on the gear teeth were reground to give backlashes of 0.008 and 0.010 in., respectively. These reground wide gears, as well as wide gears with standard backlash (0.005 in.), were then installed in a WADD high-temperature gear machine, along with the standard narrow gears. The gears were heated by induction heating and the gear temperature measured and controlled by an infrared radiometer in conjunction with an on-off controller-recorder. First, experiments were made by manually rotating the gears and noting the temperatures at which a slight resistance to smooth rotation was experienced. The data so obtained are shown in Figure 41. The gears were then run at 10,000 rpm with lubrication and at no load. at the temperatures so determined. There was no indication of tooth interference during these runs, and, upon inspection after the runs, no evidence of sculf due to tooth interference was found.

Based on the results of these experiments, a supply of Nitralloy N steel test gears with a backlash of 0 011 in. was ordered. As stated U.fore, standard geometry for the narrow test gear was retained, and the increased backlash was obtained by modifying the wide test gear.







FIGURE 41. GEAR BACKLASH VERSUS TEST GEAR TEMPERATURE AT START OF INTERFERENCE

### F. High-Temperature Test Results

The load-carrying capacities of nine selected lubricants were determined with the increased backlash Nitralloy N steel test gears, at test gear temperatures of 425, 500, 600, and 700 °F. WADD high-temperature gear machines with increased support roller bearing clearance were used. The nine lubricants included two polyphenyl ethers (0-61-20, LRO-8), two MII-L-9236 lubricants (MLO-61-1011 0-60-26), two polyglycols (GTO-770, E-1022), a silicone (GTO-615), and two mineral oils (Ref. Oil B, F-1055). The results obtained in this program are presented in Table 49, along with earlier results obtained at lower temperatures under Contract AF 33(616)-7223. Table 50 presents the same data in summary form.

The 425°F determinations were first made as a means of checking the results obtained here with the earlier results obtained at lower test gear temperatures with standard backlash test gears using machines with small clearance in the support roller bearings. Tables 49 and 50 show that the 425°F results were in line with previous experience, indicating that the earlier results were valid. It will be noted from Table 49, however, that the repeatability obtained on MJ O-61-1011, with a standard deviation/ mean of 46.3 percent, was poor. After the first large spread between the "A" and "B" determinations was obtained on MLO-61-1011, the test conditions and test procedure were carefully observed in the succeeding tests on the lubricant. However, no discrepancies were found, and since the scuff pattern appeared to be normal and dimensional and metallurgical examinations of the gears showed the test gears to be not causative in the deviation, the induction heating method was suspected. Load-carrying capacity dete :minations were then made on the lubricant with another WADD hightemperature gear machine at 400 °F conventional test conditions The repeatability, however, was roughly the same in that a deviation of 31.4 percent was obtained.

Satisfied that the repeatability difficulties were due to either the nature of the lubricant or some other unknown and indeterminable cause, the program was continued. The repeatability obtained on the remaining lubricants ranged from about 10 to 13 percent, considerably better than that obtained with the MLO-61-1011.

Load-carrying capacity determinations were next made at 500°F test goar temperature. Here, once again, the repeatability for MLO-61-1011 was poor, with the other lubricants remaining at about the same level (10 to 13 percent) as that obtained at 425°F.

## TABLE 49. SUMMARY OF INDIVIDUAL LOAD-CARRYING CAPACITY DETERMINATIONS OBTAINED USING NITRALLOY N STEEL TEST GEARS

						Loa	-Cerry	ing Car	acity, 1b/	in				
		5		00		10	at Gear	25	PARIZE, 'S	00	6	00	7	00
Oil Gode		B	Ă.	B	Ā	B	Ā	B	_ <u>A_</u>	B		8	A	B
Ref. Oil B	5280(a)						4740 4930	5030 3700	5550 5440(a)	(b) 5560(a)				
	528	0					46	00	556	0				
F-1055	4950 4530	5280 4760					3660 3760	3900						
	481	0					37	70						
GTO-615	2430 2500	2340 2380					1330 1080	1260 1130	700 590	720 870				
	24	9					12	10	72	0				
GTO-770	5190 5320	4430 4750			3200 4090	3880 3620	3690 4080	3730 4320	4390 3990	4090 4370	>5600(a) >5600(a)	>5600(a) 5490(a)		
	501	0			37	00	39	60	421	<u>0</u>	>5	600		
E-1022	3670	2620					2480 1850	0915						
	319	0					21	<u>40</u>						
LRO-è	2640 2440	2570 2470					1290 750	950 750		•				
0.41.30	223	2410	3050	2190	090	1110	1040	3060	1330	1630	1560	1846	3346	1120
0-01-40	2443	2560	6030	6110	1280	1350	1260	02.9	1360	1170	2200	2130	61.40	3340
	24	0	21	10	12	40	10	50	132	0		1840	29	30
MLO-61-1011	1640 2040	2170 1720			430 600	560 350	1000 1690 890 680 680	530 220 1090 1140 1590	470 570	1700 1930				
	181	0			4	20	2	40	117	0				
Q-60-26	1920 1540	(c) 2060			550 540	570 610	1090 1010	740	1530 1010	058 980				
	1800	2			3	10	2	<u>40</u>	101	0	,			

WADD high-temperature gear machines with 0.0023-in. dismatral support roller bearing clearance used for all determinations. Standard backlash (0.003 in.) test gears and conventional heating used for all determinations at 400°F and below. Increased backlash (0.012 in.) test gears and induction heating used for all tests above 400°F.

(a) Values obtained by extrapolation.
(b) Determinations not made due to tooth breakage during "A" en (c) Determinations lost due to failure in test oil system.

On the basis of the satisfactory repeatability obtained on all lubricants except MLO-61-1011, determinations were then made at 600 and 700°F test gear temperatures. However, most of the lubricants were not evaluated beyond 500°F, either because of their relatively low load-carrying capacity at this temperature or their poor high-temperature oxidative stability, or because they had other drawbacks as high-temperature gas turbine lubricants. Referring to Figure 42, which is a graphical presentation of the data in Table 50, it is seen that the different lubricant types gave widely different levels of load-carrying capacity. In particular, a 5P4E polyphenyl ether (O-61-20), a fluid of current interest primarily for its high-temperature oxidative stability, was rather modest in load-carrying capacity. The limited data for a 4P3E polyphenyl ether given in Table 50 show very similar performance.

Perhaps the most striking trend brought out by Figure 42 is that the load-carrying capacity of all but one of the lubricants tested reached a minimum value at about 425°F test gear temperature. An increase in load-carrying capacity was observed when the test gear temperature was either increased or decreased from this temperature. The reduction in load-carrying capacity with increasing temperature, in the low operating temperature range, was noted previously by Ku and Baber⁽¹¹⁾ who attributed it to the reduction of lubricant viscosity with temperature increase. The same authors also noted evidence of an increase in the load-carrying capacity of a lubricant (a MIL-L-7808 oil) in the 350 to 400°F range and attributed it to the formation of carbon deposits on the gears. In the present program, it was possible to attain temperatures considerably higher than in the early work; consequently, the reversal trend at high temperature could be shown in fuller perspective.

In the experiments at 500, 600, and 700°F, carbonaceous deposits were indeed observed on the gears at the completion of each determination. These deposits could readily be observed on the sides of the gear teeth, though not as evident on the working faces of the teeth because of the rubbing contact involved. Figure 43 shows a series of photographs of the sides of representative gear teeth, taken at the end of tests conducted on 5P4E(O-61-20) at progressively higher gear temperatures. In order to show more clearly the extent of the deposits, the area to the right of each tooth side was scraped slightly before the photograph was taken. Note that the amount of deposits increased progressively with increasing temperature.

The type of gear tooth surface damage obtained at 400 to 700°F test conditions differed considerably from that obtained at 165 to 400°F test conditions. This change in mode of damage appeared to occur gradually.



F.GURE 42. LOAD-CARRY.NG CAPACITY VERSUS TEST GEAR TEMPERATURE US.NG N.TRALLOY N STEEL TEST GEARS

## TABLE 50.SUMMARY OF AVERAGE LOAD-CARRYING CAPACITYRESULTS OBTAINED USING NITRALLOY N-STEEL TEST GEARS

	Load-Carrying Capacity, 1b/in							
			Test Gea:	r Temper	ature, °F			
Oil Code	165	300	400	425	500	600	700	
Ref. Oil B	5280(1)			4600(4)	5560(3)			
F-1055	4880(4)			3770(3)				
GTO-615	2410(4)			1210(4)	720(4)			
GTO-770	5050(4)		3700(4)	3960(4)	4210(4)	>5600(4)		
E-1022	3150(2)			2140(3)				
0-61-20	2460(4)	2110(2)	1240(4)	1050(4)	1320(4)	1860(4)	2930(2)	
LRO-8	2530(4)			940(4)				
MLO-61-1011	1890(4)		490(4)	940(10)	1170(4)			
0-60-26	1800(3)		560(4)	940(4)	1090(4)			

WADD high-temperature gear machines with 0.0025 in. diametral support roller bearing clearance used for all determinations. Standard backlash (0.005 in.) test gears used for all determinations at 400°F and below. Increased backlash (0.011 in.) test gears used for all tests above 400°F. Number in parentheses indicates number of determinations used to obtain average.







2a. 165°F





2Ъ. 400°F





2c. 425°F



2d. 500*F









21. 700°F

FIGURE 43. PHOTOGRAPHS OF GEAR TOOTH SURFACE DAMAGE AND LUBRICANT DECOMPOSITION PRODUCT DEPOSITS, 5P4E POLYPHENYL ETHER from a scoring type appearance to a burnishing of the surface, over the temperature range of from 400 to 700°F. It appeared as though metalto-metal contact was prevented by some medium separating the gear tooth surfaces, which became more pronounced as temperature was increased. In the 700°F determinations, the surface damage, though extensive over the working face, was smooth and reflected light similarly to a mirror finish. Figure 43 shows photographs of gear tooth surface damage over the experimental temperature range.

The consumption of the test lubricants was found to increase progressively as the test gear temperature was increased, with the consumption rates being approximately 40 ml/min at 600°F and 60 ml/min at 700°F test conditions, forO-61-20. Sludge accumulation in the test oil sump with frequent plugging of the filter caused some difficulty in the last load steps of the 700°F determinations.

#### G. Gear Type and Test Method Correlation

It has been suggested that the load-carrying capacity obtained for different lubricants using standard Ryder test gears at 165°F may be directly related to the load-carrying capacity of the same lubricants using Nitralloy N steel test gears at higher test temperatures. While this suggestion appeared to have little theoretical foundation, its practical implications could nevertheless be important. Obviously, if load-carrying capacity tests performed on lubricants using one test gear material at a lower temperature could predict the performance of the same lubricants using another test gear material at a higher temperature, then the former would constitute an adequate guide for lubricant evaluation, thereby saving the effort required for the development of the latter test. It was mainly for this reason that the decision was made to investigate the question of correlation between different test gear types and test methods.

Tests were made on the nine lubricants referred to in the preceding section, by means of WADD high-temperature gear machines with increased support roller bearing clearance, using standard Ryder test gears at  $165^{\circ}F$  test conditions (Federal Test Method 6508). The results so obtained are shown in Table 51, along with the data from Nitralloy N steel test gears at  $165 \text{ and } 425^{\circ}F$  test conditions. Attention is first drawn to Figure 44, which compares the data at  $165^{\circ}F$  test conditions for standard Ryder test gears obtained using machines with increased support roller bearing clearance with the earlier data⁽⁵⁾ obtained at same test conditions using machines with smaller bearing clearance. This figure shows that the two sets of results were directly comparable, thereby establishing that the earlier data were valid.

	Load-Car	Load-Carying Capacity, lb/in.					
	Standard Ryder	Nitrallcy N Steel					
	Test Gears	Test	Gears				
Oil Code	165°F	165°F(a)	425°F(b)				
Ref. Oil B	2540(4)	5280(1)	4600(4)				
F-1055	2350(4)	4880(4 <u>)</u>	3770(3)				
GTO-615	2410(4)	2410(4)	1210(4)				
GTO-770	4160(4)	5050(4)	3960(4)				
E-1022	1300(4)	3150(2)	2140(3)				
LRO-8	2240(4)	2530(4)	940(4)				
0-61-20	2260(4)	2460(4)	1050(4)				
MLO-61-1011	2080(4)	1890(4)	940(10)				
0-60-26	2150(4)	1800(3)	940(4)				

## TABLE 51. COMPARISON OF RESULTS OBTAINED USING STANDARDRYDER AND NITRALLOY N STEEL TEST GEARS

WADD high-temperature gear machines with 0.0025 in. diametral support roller bearing clearance used for all determinations. Number in parentheses indicates number of determinations used to obtain average.

(a) Test gears with standard backlash (0.005 in.) used.

(b) Test gears with increased backlash (0.011 in.) used.



FIGURE 44 COMPARISON OF STANDARD RYDER GEAR LOAD-CARRY.NG CAPACIT.ES OBTAINED AT 165°F TEST CONDITIONS USING DIFFERENT DIAMETRAL SUPPORT BEARING CLEARANCES Referring to Figures 45 and 46, which compare the results obtained using Nitralloy N steel test gears at 165 and 425°F test conditions, respectively, with those obtained using standard Ryder test gears at 165°F test conditions, it can be seen that no general correlation existed for all data obtained. Further, no organized correlation appeared evident even if the different lubricant types were considered separately. It is clear from Figures 45 and 46 that the load-carrying capacity of lubricants was strongly influenced by the test gear material. Hence, if the test gear material was varied, then no organized correlation of load-carrying capacity results was obtainable, regardless of the test temperature.

Figure 47 shows that if the same test gear material, Nitralloy N in this case, was employed, then the load-carrying capacity results obtained at two different temperatures, 165 and 425°F, exhibited a consistent though nonlinear relationship. It must be cautioned, however, that whether a similar correlation will exist at still higher test temperatures remains to be seen. Figure 42 indicates that the load-carrying capacity of nine lubricants decreased with an increase in test gear temperature up to about 400 or 425°F. However, with further increase in test gear temperature, most lubricants experienced an increase in load-carrying capacity due to the buildup of carbonaceous deposits on the rubbing surfaces. How this phenomenon would affect the correlation of the test results is not clear at present, and can only be established by further experimentation.

### H. Conclusions

Load-carrying capacity determinations were made on nine lubricants, at test gear temperatures up to 700°F on one, 600°F on another. 500°F on four others, and 425°F on the remaining three. Of the lubricants evaluated beyond 425°F, all but one showed an increase in load-carrying capacity as the temperature was increased beyond 425°F. An increase in lubricant deposits as well as a change in the mode of gear tooth surface damage were noted, as the temperature was increased. The 5P4E and 4P3E polyphenyl ethers gave rather modest load-carrying capacities throughout the temperature range investigated.

On the basis of the load-carrying capacity results obtained to date, no correlation was found to exist between the test results using standard Ryder test gears at 165°F test conditions and those using Nitralloy N steel test gears at either 165 or 425°F test conditions. On the other hand, a correlation appeared to exist between load-carrying capacities of lubricants at 165 and 425°F test conditions when only one test material. Nitralloy N steel in this case, was used. However, whether a similar correlation will exist at still higher temperatures remains to be seen.







FIGURE 46 RELATION BETWEEN LOAD-CARRYING CAPACITY OBTAINED WITH NITRALLOY N STEEL TEST GEARS AT 425'F WITH THAT OBTAINED WITH STANDARD RYDER TEST GEARS AT 165'F TEST CONDITIONS




The capability of the WADD high-temperature gear machine to operate at test gear temperatures through 700°F has been demonstrated. Calibration studies of the WADD high-temperature gear machine have shown the 0.0025-in. diametral clearance of the support roller bearings to be satisfactory for induction heated gear temperatures through 600°F.

### VI. THREE-BALL/CONE FATIGUE TESTER

### A. General Remarks

The overall objectives of the rolling-contact fatigue phase of the program were to develop apparatus and procedures for evaluating lubricants with respect to rolling-contact fatigue and to evaluate candidate lubricants under environmental conditions representative of Mach 3 class Las turbine engine designs.

This work was conducted along two parallel lines. The present chapter is concerned with a broad experimental investigation of the performance of lubricants with respect to rolling-contact fatigue under a wide range of temperature, load, and speed conditions, employing inexpensive bench-type test apparatus. The next chapter deals with the full bearing test, intended primarily to provide base-line data for the interpretation of the results from the bench-type test.

From a survey study conducted earlier under Contract AF 33(616)-7223 and recent conclusions of the Bearing Fatigue Panel of the CRC Aviation Group on Gas Turbine Lubrication, ASD decided that a 3-bail/cone fatigue tester should be developed. In the design of this tester, constant liaison was maintained with the CRC Panel. Construction of the tester is scheduled for completion in June 1963.

### B. Study of Bench-Type Fatigue Testers

### 1. SwRI Study

In 1960, SwRI was requested by ASD, under Contract AF 33(616)-7223, to make a brief survey of the state of the art of bench-type fatigue tests. It was recognized that the important advantages of bench-type fatigue tests were the simplicity and low cost of the test apparatus and the low cost of conducting the tests. However, the correlation between the bench-type tests with full bearing test needed to be resolved. In particular, a large number of bench-type testers were known to be available, and it was necessary to select a design that would best answer the requirements for evaluating lubricants for advanced gas turbine engines. The requirements considered important were geometric simplicity, provisions for varying stress level and stress frequency, provision for varying spin-to-roll ratio, high-temperature capability to 800°F or higher, recirculating lubrication systems with provision for operating in an inert gas atmosphere, provisions for measurement of speed, load, friction torque, and provision for reliable indication of fatigue failure.

A number of bench-type fatigue testers employing simple test specimens (usually balls) were examined:

Barnes one-ball tester⁽¹²⁾ GE one-ball tester⁽¹³⁾ NACA (two-ball) spin tester⁽¹⁴⁾ SKF three-ball/flat-washer tester⁽¹⁵⁾ Barwell four-ball tester⁽¹⁶⁾ Modified Barwell (3-ball/cone) tester⁽¹⁷⁾ NASA five-ball tester⁽¹⁸⁾

With some of these testers, considerable lubricant evaluation data were available; with others, practically no such information appeared to have been published. In some cases, correlation between the bench tests and full bearing test was claimed but not substantiated with organized data. However, in most instances, there were no valid reasons to suspect that reasonable correlation could not be obtained.

It appeared that many of the testers were available in several versions with presumably different performance capabilities. However, no single design was known to possess features that would satisfy all of the requirements set forth previously. Accordingly, it was concluded that all of the specified requirements could be met only by the design and construction of a special tester, based upon the principle of one of the basic designs listed above.

After careful consideration of all factors, it was felt the selection of a basic design would be among the following:

> SKF three-ball/flat-washer tester Barwell four-ball tester Modified Barwell (3-ball/cone) tester NASA five-ball tester

All of these basic designs oppeared to be capable of being developed for hightemperature operation and to be simple and low in initial cost and low in cost of operation. Among these, the modified Barwell (3-ball/cone) tester was considered to be the most versatile, due to its flexibility in varying the spinto-roll-ratio.

### 2. CRC Study

Almost parallel to the SwRI study has been a similar study conducted by the Bearing Fatigue Panel of the CRC Aviation Group on Gas Turbine Lubrication. This panel took into consideration a number of requirements similar to those given previously but examined a larger number of bench-type testers. These were then narrowed down to the same basic types examined in the SwRI study, from which three were selected as being most promising: The modified Barwell tester, the Barnes one-ball tester, and the NASA five-ball tester. However, no final choice has been made at this writing.

### C. Design of 3-Ball/Cone Fatigue Tester

### 1. Design Requirements

On the basis of the two aforementioned studies, ASD directed SwRI to design for their consideration a bench-type fatigue tester using the principle of the 3-ball/cone (modified Barwell) fatigue tester.

The ultimate purpose of the 3-ball/cone fatigue tester is to provide a reasonably simple device capable of evaluating the rolling-contact fatigue characteristics of lubricants over a wide range of conditions. It has been felt that, while it probably would not contribute to the evaluation of the fatigue characteristics of a lubricant, the measurement of friction torque would make the tester a more versatile research tool. Accordingly, this feature was made a part of the tester design. As a consequence, simplicity of design has been sacrificed in some respects.

The final design criteria for the fatigue tester are outlined below. Where the criteria were altered during the course of design, a note is added to this effect.

- (1) Test speciment
  - (a) Material High-temperature bearing-quality steel
  - (b) Simple geometry
- (2) Initial contact stress level:

÷.

(a) Variable up to 1,000,000 psi maximum (this was originally 700,000 psi)

- (3) Temperature:
  - (a) Specimen 1000°F maximum, 700 to 800°F acceptable; 150 to 250°F minimum
  - (b) Lubricant 750°F maximum, 100°F minimum
- (4) Kinematics of test elements:
  - (a) Variable stress frequency (maximum cone speed to be 10,000 rpm)
  - (b) Variable spin-to-roll ratio
- (5) Lubrication:
  - (a) Recirculating system
  - (b) Atmosphere control using inert gases at atmospheric pressure
- (6) Instrumentation and measurement capability:
  - (a) Failure detector to shut down the facility
  - (b) Timer or spindle revolution counter
  - (c) Spindle speed indicator
  - (d) Specimen and lubricant temperature control
  - (e) Load control
  - (f) Lubricant flow rate measurement
  - (g) Torque measurement (this was originally optional)

### 2. Design Features

<u>General Design</u>. The details of the test region, the lubrication system, and the spindle support of the 3-ball/cone fatigue tester are shown in Figure 48. Figure 49 is an overall pictorial view of the tester showing the frame, loading system, etc.



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FIGURE 48. CROSS SECTION OF THREE-BALL/CONE FATIGUE TESTER



In general, the conical specimen, A, (Fig. 48) rotates against three lower balls, equally spaced by a retainer. The lubricant is circulated by the submerged gear pump, B, located in the sump, and is directed against the ball-cone interface by the replaceable nozzle, C. Lubricant return is by gravity through drain holes. Loading is accomplished by dead weights and a lever arrangement through the spindle. The drive for the spindle consists of a 3600-rpm D. C. motor with pulleys and timing belt to give a continuously variable spindle speed up to 10,000 rpm. Heat is supplied to the test region by cartridge heaters, D, and to the sump by band heaters, E. The entire apparatus is insulated to reduce heat loss to the atmosphere.

Test Specimens and Test Region. The conical test specimen, A, driven by the spindle, F, through a self-actuating taper, is positioned in a cluster of three 1/2-in. diameter balls. The balls are equally spaced about the cone specimen by a retainer. The balls are confined in a chamber made up of a ring, G, and a washer, H. The ring and cone temperatures are measured by thermocouples. The signal from the latter thermocouple is removed from the rotating shaft through slip rings⁽¹⁹⁾. Heat is supplied to the test region by six cartridge heaters. D, inserted into snug-fitting holes in the block. Preliminary tests made using these heaters in a block similar to that proposed in the design indicate that a temperature of 1000°F can be reached within 1-1/2 hours.

The cone angle of the specimen may be varied from  $60^{\circ}$  to  $90^{\circ}$ , corresponding to contact angles of  $30^{\circ}$  and  $45^{\circ}$ , respectively. The spin-to-roll ratio of the cone against the balls and the number of stress cycles on the cone per revolution may be varied by varying this cone angle. With a  $30^{\circ}$  contact angle cone, each revolution of the spindle will result in 2. 2 stress cycles on the cone, and a spin-to-roll ratio of 0. 26. A 45° contact angle results in 2. 0 stress cycles per cone revolution, and a spin-to-roll ratio of 0. 53.

Lubricant Supply System. The test lubricant system is a semiclosed, positive-pressure, recirculating system capable of being blanketed with an mert gas. The sump, located below the test region, is separated from the test head through the static air seal, P. In this way, the sump may be made stationary; yet the test head is free to twist against flexural members (not shown) to indicate torque. The short lubricant flow lines between the pump and the nozzle, C, reduce temperature differential between the bulk oil and that entering the test area to a minimum.

A small electric motor drives the test oil pump, B, through a flexible shaft and a reduction gear box. The pump is a positive displacement

gear pump running submerged in the lubricant. This manner of pump operation reduces thermal distortion of the parts due to temperature variation throughout the pump.

The sump is heated by three band heaters, E, with provision for a plate heater underneath the sump if necessary. Previous experience with similar sumps and heaters indicates no difficulty in reaching 750°F test lubricant temperature.

The sump is heavily insulated, and, being physically separated from the test head, it is possible to regulate the sump and test region temperatures independently. Sump temperature is measured and controlled through thermocouples immersed in the lubricant. A thermocouple, J, extends to the tip of the nozzle to measure the temperature of the lubricant entering the test region.

To reduce lubricant oxidation and to simulate conditions in a gas-blanketed lubricant system, the lubrication system of the tester has been designed to operate under a small positive gage pressure using a nitrogen gas blanket. Escape of lubricant-entrained gas past the spindle is prevented by rotating screw seals, L. The static seal, P, between the sump and the test head, is also supplied with gas. Gas pressure buildup is prevented by venting through a reflux condenser attached at K. The condenser may be arranged to either reflux back into the sump, or to an external waste can. In the latter case, makeup lubricant must be added periodically.

Load System. The axial spindle load will vary from 850 lb to 1300 lb for contact angles of 30° and 45°, respectively. These loads give an initial maximum contact stress of 1,000,000 psi. The load is applied through the spindle by means of dead weights and a lever, M. The slight vertical motion of the spindle necessary for load application is obtained by mounting the spindle ball bearings in a sliding journal bearing, in the same manner as that used in a drill press.

<u>Spindle and Drive System.</u> The spindle is mounted on two preloaded size 208 precision angular-contact ball bearings. Lubrication is an oil-mist type. The upper end of the spindle couples through a spline shaft to a short, ball-bearing supported drive shaft, R The drive shaft is driven through pulleys and a timing belt by a variable speed, 1/2-hp, 3600-rpm D. C. motor. Maximum drive shaft and spindle speed is 10,000 rpm. By separating the drive shaft and spindle with the spline coupling, the drive shaft bearings are obliged to carry only radial loads and the spindle bearings only thrust loads. Such an arrangement tends to reduce spindle runout during operation. The test specimen is attached to the lower end of the spindle by a self-actuating taper. A thermocouple, S, extends the length of the spindle for measuring specimen temperature. The thermocouple output is removed through slip rings,  $T^{(19)}$ . Convection cooling rings, U, on the spindle help to prevent the flow of large amounts of heat from the specimen to the lower spindle bearing.

In order to gain access to the test region for purposes of replacing test specimens, the spindle assembly will be removable, much in the manner that a lathe collet may be removed. Tapered surfaces will assure alignment.

Torque Measurement. Friction between the rotating cone and the balls produces a torque on the test head assembly. This assembly is restrained from turning by flexural members (not shown) to which strain gages are attached. The deflection of the arm is a measure of the friction torque. The head is supported on an externally pressurized air thrust bearing, N, through a spider, V. The air bearing practically eliminates any restraint on the test head due to friction in the thrust bearing. It further allows the test head to move laterally in order to seek a position of equilibrium with respect to the rotating cone specimen.

Frame. The frame is of welded box construction, braced with gussets and pads at points of attachment or high stress. It consists of two uprights connected across the top by a horizontal member containing the spindle assembly. The uprights are attached to a supporting table.

Test Termination Control. When a specimen fatigues, with a resulting spalling of the surface, the irregularity will produce a vibration in the spindle assembly. This vibration will be picked up by a microphone transducer mounted on the frame. The transducer signal resulting from the vibration at failure will be used to shut off all electrical inputs, the inert gas supply, and stop all recording instrumentation. The final design of this portion of the rig will be made after the construction and initial operation of the tester.

### D Conclusions

A 3-ball/cone fatigue tester has been designed and is currently being fabricated. The estimated completion date is in June 1963.

### VII. BEARING FATIGUE

### A. General Remarks

The objectives of the bearing fatigue part of the rolling-contact fatigue phase were to develop apparatus and techniques for determining the bearing fatigue characteristics of lubricants in full bearings and to evaluate candidate lubricants with respect to bearing fatigue under environmental conditions representative of Mach 3 class gas turbine engine designs.

Two WADD 85-mm thrust bearing machines developed under Contract AF  $33(616)-7223^{(5)}$  were selected for this work. These machines were employed previously at speeds up to 15,000 rpm (DN =  $1.275 \times 10^6$  mm-rpm), thrust loads up to 18,000 lb (maximum Hertz stress = 400,000 psi), and bearing temperatures in excess of 750°F. They were considered adequate for the current programs, and no modifications were deemed necessary.

Except for the selection and ordering of the special test bearings and preparation for the tests upon receipt of the bearings, this part of the program has been dormant.

### B. Test Apparatus

#### . WADD 85-mm Thrust Bearing Machine

The WADD 85-mm thrust bearing machine was designed and developed under Contract AF 33(616)-7223⁽⁵⁾. Two such machines were made available for use on the current program.

Figure 50 presents a cross section of the 85-mm bearing machine. The inner rings of the two test bearings, A and B, are clamped to a cylindrical adaptor that turns with the cantilevered shaft-end. The cylindrical case of the bearing head is supported by the outer rings of these bearings and is restrained from turning with the shaft only by a torque arm (not shown) which is attached to a torque-measuring device⁽²⁰⁾ as shown in Figure 51.

Load is applied to the test bearings by means of hydraulic pressure acting on a thin steel diaphragm, C (Fig. 50), which in turn presses against the loading sleeve, D. The diaphragm has an annular convolution near its edge to insure flexibility. In order for bearing A to take the load, its outer ring must slide freely. Since bearing B must balance the thrust applied to bearing A, the two bearings become equally and oppositely loaded.

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FIGURE 51 SCHEMAT.C D.AGRAM SHOWING PRINCIPLE OF M.I.T. HYDRAULIC SCALE FOR TORQUE MEASUREMENT Test lubricant is introduced to the loaded side of the inner rings of the test bearings by means of a double jet, E, and is removed from the head through port  $\mathbb{F}$ , which leads to a scavenge pump. Leakage around the shaft is prohibited by the slinger and nonrubbing seal assembly, G. In normal operation, a vent hole located on the top of ring H is open to the atmosphere, and movement of air acts to sump the test oil back to the bearing head, and also to pump any support oil that passes the support bearing back to the support section. If it is desired to operate the test bearings in an inert gas atmosphere, all that is necessary is to apply a slight pressure of the inert gas to the vent. Positive seal against air leakage is accomplished. In that event, by the use of an elastomer "O" ring, which is held between rings H and I. A lower hole in ring H facilitates draining; it may be closed or left open as required.

Thermocouples are placed 120 degrees apart on the outer ring of each test bearing with one located at the bottom of each bearing. The outer rings may be heated, if necessary, by means of cartridge heaters, J, disposed around the bearings.

The machine is normally operated at 10,000 rpm (DN value =  $0.85 \times 10^6 \text{ mm-rpm}$ ) and a thrust load of 9,785 lb. However, the machines are capable of operating at speeds up to 15,000 rpm and thrust loads up to 18,000 lb which corresponds to a maximum Hertz stress of 400,000 psi. The maximum controlled test bearing temperature capability of the machine is in excess of 750°F.

### 2. 85-mm Test Bearings

The test bearings are angular-contact ball bearings made to ABEC-7 tolerances from consumable electrode M-50 steel. The major dimensions of the bearings are

Inside diameter, mm	85
Outside diameter, mm	150
Bearing width, mm	28
Ball complement	15
Ball diameter, in.	13/16
Contact angle, degree	25
Race curvalure, 🛸	5.2

The bearings are of split inner race design; and the retainer, of S-monel metal, is piloted on the inner ring. The balls and retainer assembly may be replaced by a new assembly after a test. This is possible since only one side of the cuter race and one side of the inner race are affected in a given test; thus the unused sides of each race may be utilized for another test. Quotations were requested and received from five bearing manufacturers to supply the special 85-mm thrust bearings. On the basis of these quotations, it was decided to purchase 150 complete bearings and 150 extra ball-retainer assemblies from Fafnir Bearing Company. An order was subsequently placed, and delivery of the bearings is scheduled for May 1963.

It was stated in the request for quotation that it was desired, for statistical reasons, to obtain all inner rings, outer rings, and balls from a single heat of consumable electrode M-50 steel. However, upon receiving the quotations, it was evident that it was not economically feasible to obtain such a large number of bearings from a single heat of material. As a result, all of the inner and outer rings of the 150 bearings on order will be from one heat of material, and all the balls will be from a different single heat of material.

### C. Conclusions

The special test bearings required for the program were selected and ordered. Except for preparation for the tests upon receipt of the bearings, no other work was done.

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### APPENDIXES

# APPENDIX I. DESCRIPTION OF TEST LUBRICANTS

Viscosity, cs			
Oil Code	100°F	210°F	Description
Ref Oil B	237 9	20 3	Mineral all MILL 6083P Curte 1100
F-1022	11 4	20.5	Bolughucol
E = 1022	354 5	12 88	Polyphonul other 5D4F mined is an and
F-1055	103 0	11 5	Minoral oil MIL I 6022 Crude 1065
GTO-313	17 1	4 61	MILE TRONC
GTO-615	61 3	20.7	Silicone (chlorinated)
GTO-770	64 6	10 43	Polyalycol
GTC-855	29.0	5.30	MIL-L-9236A type
GTO-915	16.0	3, 61	MIL-L-9236B
GTO-939	15.1	3.47	MIL-L-9236B
LRO-8	70.9	6.3	Polyphenyl ether, 4P3E mixed isomers
LRO-11	70.87	6.32	Polyphenyl ether, 4P3E mixed isomers
LRO-13	362.4	13.21	Polyphenyl ether. 5P4E mixed isomers
O-58-24	34.7		MIL-L-9236A type
O-59-15	18.7		MIL-L-9236B type
0-59-26	18.7		MIL-L-9236B type
O-60-3	17.2		MIL-L-9236B type
O-60-11	21.2		MIL-L-9236B type
O-60-12	16.1		MIL-L-9236B
O - 60 - 13	25.7		MIL-L-9236A type (CRC Oil RAO-1-60)
O - 60 - 19	20,8		ML-L-9236B type
O-60-23	16.0		Different batch of GTO-915
0-60-26	15.0	3.5	Different batch of GTO-939
0-60-27	15.0		Different batch of GTO-939
0-60-28	16.1		MIL-L-9236B type
0-61-17	15.9		Different batch of 0-60-12
0-61-19	15.7		Different batch of GTO-939
C-61-20	357. 3	13.2	Polyphonyl ether, 5P4E mixed isomers
MLO-61-1011	16.2	3.6	Different batch of GTO-915
MLO-61-1012	15.0		MIL-L-9236B type
MLO 61 - 1013	15.3		MIL-L-9236B type
MLO-61-1014	24.4		Advanced turbine oil candidate
ELO-62-50	20.62		Advanced turbine sil candidate
MLO-62-1000	10.51		Advanced turbine oil candidate
MLO • 62 • 100 3	19.40		Advanced turbine oil candidate
MLO-62-1004	15.66		MIL-L-9236B type
MLO - 62 - 1005	41.8		Advanced turbine oil candidate
NILU-62-1006	14,7 / 1 / M		M.1 L-9236B type
MLC 02-1008	64.69		Advanced turbine oil candidate
MLO-02-1011	14.50		MIL-L 9236B type
MLO-52-1012	26.84		Advanced turbine oil candidate

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## APPENDIX II. PROPERTIES OF 5P4E POLYPHENYL ETHER (F-1041)*

Material: Mixture of isomeric bis(phenoxyphenoxy)benzenes

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Isomer Distribution:	65% meta-meta- 30% meta-meta- 5% para-m <b>eta-</b>	meta para para
Refractive Index at 25°C:	1.6289	
Specific Gravity (20/20):	1.203	
Viscosily, cs:	At 100°F At 210°F	355 12.9
Halogen (Chlorine), %:	0.02	
Pour Point, °F:	+40	
Thernial Stability (Isoteniscope),	°F: 867	
Vapor Pressure, mm Hg at 500°	F: 0.96	
Evaporation Loss, 6.5 hr at 500 and 140 mm Hg, %:	°F 5. 2	
Oxidation-Corrosion, FS-791 a,	Method 5308.4,	48 hr at 600°F:
Weight Change, mg/cm ² :	Steel Silver Copper Aluminum Taanium Magnesium	+0.06 -0.17 -0.07 +0.02 +0.02 +0.21
Viscosity Increase, %:	At 190*F At 210*F	45.7 17.5
Autogenous Ignition Temperature	•. •F: >1100	

[©]Data furnished by the supplier, Monsanto Chemical Company.

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### APPENDIX III. 18-HR 425°F OXIDATION-CORROSION TEST*

### 1. SCOPE

1.5

1.1 This method describes a test procedure for determining the susceptibility to oxidation and the metal-corroding tendency of aircraft turbine lubricants and similar high-temperature lubricants.

### 2. OUTLINE OF METHOD

2.1 This test consists of exposing a lubricant sample to the combined action of high temperature, a high rate of aeration, and the presence of metal specimens. After a controlled exposure period, the extent of lubricant deterioration and loss are measured, and the metal specimens are evaluated for overall or local attack.

### 3. APPARATUS AND MATERIALS

3.1 Bath - Any bath may be employed that has provisions for maintaining a uniform temperature of  $425 \pm 2^{\circ}F$ . The depth of the heating fluid and the configuration of the sample tube supports must be such that the oil sample level (within the tubes) is  $2^{11} \pm 1/2^{11}$  below the heating fluid level. This measurement is to be made without aeration of the test oil, with both the test oil and the heating fluid at operating temperature.

### NOTE

Oil conforming to Specification MIL-L-6082, Grade 1100, is suitable as a heating fluid, provided low-intensity heaters are used for the bath. It is desirable to add an anti-oxidant (e.g., 1 percent phenyl a-naphthylamine) to this oil to extend its service life. For adequate temperature control, it is necessary that the bath be well insulated, well stirred, and controlled by a sensitive thermoregulator. The bath is equipped with a cover to accommodate the sample tubes, stirrer, and other

"Draft prepared on September 22, 1961.

necessary hardware, including a rack to support the bottoms of the sample tubes. The bath temperature may be read by any laboratory thermometer of the required range and accuracy; it is recommended that the accuracy of thermometer be checked periodically. It is further recommended that an auxiliary temperature record be provided by means of a recording instrument, and that an independent safety switch be provided to shut off the heating circuits in the event of a temperature rise of more than  $10-15^{\circ}F$  above the normal control point. It is also recommended that the bath should be located in a vented hood or provided with its own ventilating system.

3.2 Oil sample tube and auxiliary glassware - A sample tube and head in accordance with Figure 52 shall be used. An air delivery tube of standard 5-mm Pyrex tubing, approximately 610 mm in length, is fixed in the upper end of the head by means of a suitable one-hole cork. The remainder of the assembly includes a water-cooled Graham-type glass condenser and a 250-ml round-bottom two-neck flask used as a condensate receiver; the condenser is attached to the 19/38 joint on the head, and the flask is attached to the bottom of the condenser.

### NOTE

Aithough the details of the condenser and condensate flask are optional, it is suggested that the condenser be approximately 200 mm in length with 19/38 joints on both ends, and that the flask be a standard 250-ml two-neck distilling flask with two 19/38 joints. The sample tube and head are available from Scientific Glass Apparatus Co. : the condenser and condensate flask are standard shelf items.

3.3 Air supply system  $\sim$  The air supply system must be capable of metering 236 g/hr of clean dry air to each sample tube, maintaining this rate within  $\pm$  3 percent throughout the test period. This air flow rate is equivalent to 197 liters/hr measured at 76°F and 760 mm. The air shall be dried by passing through a column of Drierite or equivalent desiccant.

### NOTE

The Drierite column should be made of glass, and the desiccant should include some indicating Drierite to



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show when the desiccant needs changing. For a sixplace test unit, a 4-inch diameter column of Drierite is necessary; the length of column required will depend on the moisture content of the entering air and the frequency of Drierite replacement that can be tolerated. The use of predried air (e.g., from a calcium chloride drying unit) is desirable in order to avoid too frequent changes of the Drierite.

3.4 <u>Metal test specimens</u> - Specimens shall have dimensions as shown in Figure 53 and shall conform to the following metal specifications:

Aluminum alloy	QQ-A-355, T-3 or T-4
Titanium alloy	MIL-T-009046B (ASG), Class 1 (8% Mn)
Silver	Electrolytic grade (Handy and Harman 999 or equivalent)
Mild steel	QQ-S-636
Stainless steel	MIL-S-5059, Grade 301 Half Hard

3.5 Additional equipment

Analytical balance, sensitive to 0.1 mg

Laboratory scale, sensitive to 1.0 g, capacity 5 kg minimum

Microscope, 20× magnification

Sample tube racks, design optional (for handling and weighing)

Wire screen strainers, 200 mesh - Fisher Scientific #13-583 or equivalent

Corks, for air delivery tubes, size No. 1 or No. 2, depending on size tolerance of top entry tube (on head)

Wire, 0.016 in., Type 304 stainless steel - Wilbur B. Driver Co., Nilstain Type 304 or equivalent



.032"

DRILL 2 HOLES

TEST SPECIMEN



# TEST SPECIMEN CONFIGURATION

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FIGURE 53. METAL SPECIMEN DIMENSIONS AND ASSEMBLY CONFIGURATION Jigs for holding metal specimens for polishing and for assembly*

Lorg nose pliers

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Stamless steel forceps

Graduated cylinders, 500-ml

3.6 Cleaning and polishing supplies

Cotton gloves - H. Texture Glove Co., Plainfield, New Jersey, or equivalent

Cotton balls - Seamless Rubber Co., New Haven, Connecticut SR-9175 or equivalent

Cheesecloth, blached,  $28 \times 32$  count

Abrasive paper, 240 and 400 grit, 3M TRI-M-ITE Finishing Paper, Open Coat, Silicon Carbide

ASTM precipitation naphtha or ASTM iso-octane

Acetone, reagent grade, sulfur-free

Chleroform, reagent grade

Chromic acid cleaning solution Concentrated sulfuric acid 1 liter Saturated sodium dichromate solution

35 ml

#### 4. PROCEDURE

4.1 Test Conditions . The lubricant is exposed for 18 hours with a bath temperature of 425°F and an air flow rate of 239 g/hr. The lubricant sample volume is 350 ml, and one specimen of each of the five metals is present during the exposure period.

^oThe polishing jog should be made of plastic, to avoid scratching the metal spectmen surfaces: a suitable design is shown in Figure 54. The assembly jig may be made of metal, plastic, or a nonacidic hardwood such as maple, in any convenient design.



FIGURE 54. SPECIMEN POLISHING JIG

Preparation of metal specimens - After preliminary degreasing 4.2 and removal of any major pits or scratches by grinding, all surfaces of the specimen are polished with 240-grit silicon carbide paper to remove all previous marks, using fresh paper for each different metal. The specimens should not be touched with the hands, and should be handled only with clean cotton gloves or with forceps. A suitable jig should be used for holding the specimen during polishing. After the 240-grit polishing the specimens may be stored in a desiccator over Drierite or calcium chloride. The final polishing with 400-grit silicon carbide paper should be performed immediately before the test is to be started, using fresh paper for each different metal. Only longitudinal strokes are used, and the polishing is continued until a clean blemish-free metal surface is obtained. During the final polishing and in subsequent operations, the specimen is handled only with forceps. After polishing, the specimen is swabbed vigorously with clean naphtha (or iso-octane) and with acetone to remove all metal particles and abrasive dusts, weighed to the nearest ). I mg, and stored in a desiccator until the entire set of specimens is seary for assembly. The specimens are assembled in the configuration shown in Figure 53 and wired together. The wire (stainless steel as specified in Paragraph 3.5) is cleaned with acetone prior to use. All handling of specimens and wires during assembly is accomplished without hand contact. A metal or maple jig may be used to hold the specimens during assembly. The wire is looped through the holes, twisted loosely, and the excess is trimmed off. The assembled specimens are given a final rinse in naphtha or iso-octane, allowed to dry, and stored in a desiccator until ready for actual installation in the test cell.

4.3 Test startup - A 350-ml sample of the test oil is measured into a clean, dry test tube, avoiding wetting the ground glass joint. (Additional cil sample, 30 ml minimum, should be set aside for determination of initial oil properties). The metal specimen assembly is added to the sample tube, and the air delivery tube is inserted so that its lower end rests within one of the triangles formed by the specimens. The head is put in place, using a very small quantity of silicone grease, and the air delivery tube is positioned by means of a tightly fitting cork so that its lower end is 1/8 in. off the bottom of the sample tube. This assembly (sample, sample tube, head, air tube, cork, and specimen assembly) is weighed to the nearest gram. It is then placed in the heating bath at 425°F and allowed to warm up for 15 minutes. During this time the "overhead" assembly is completed; this includes connecting up the head sidearm to a condenser and tared  $(\pm 1 \text{ g})$  condensate flask and starting water flow through the condensers. The air delivery tube is connected to the metered air line, without any air flowing. At the end of the warmup period, timing of the test period is started, and air is introduced at a rate of 23% g/hr. A photograph of the assembled test apparatus and bath is presented in Figure 55.



FIGURE 55. PHOTOGRAPH OF OXIDATION-CORROSION TEST APPARATUS

4.4 Test period - The bath temperature is maintained at  $425 \pm 2^{\circ}F$ and the air flow rate at 236 g/hr  $\pm$  3 percent for a period of 18 hcurs  $\pm$  5 minutes.

4.5 <u>Test shutdown</u> - At the end of the test period, the air flow is stopped the air tube is disconnected, the head sidearm is disconnected from the condenser and the tube-head assembly is removed from the bath, wiped free of oil, and allowed to cool. This assembly is then weighed to the nearest gram to determine oil loss. The oil sample is filtered through a 200-mesh screen into a sample bottle and retained for determination of properties.

### NOTE

Since the neutralization number of some oxidized oils will change markedly on storage at room temperature, it is essential that the oxidized oil sample be tested on the same day as drawn, or that it be stored at 20°F or colder to prevent change.

The condensate flask is weighed to the nearest gram to determine the amount of overhead roduct collected, and this product is retained for the determination of neutralization number, observing the same precautions on sample storage as described above. Observitions are recorded on the appearance of the glassware with respect to sludge, varnish, or other deposits on all parts of the assembly, and sludge trapped by the filter screens. The metal specimen assembly is rinsed with acc one or naphtha as required to remove the oil and carefully disassembled. The individual specimens are placed on a mat of clean cheesecloth and swabbed vigorously with warm acetone and chloroform, using a series of fresh cotton swabs until clean swabs are noted. The specimens are not touched with the bare hands during disassembly and cleaning. After a final rinse in warm acetone and flash drying, the specimens are cooled in a desiccator and then weighed to the mearest 0.1 mg. The specimens are examined under 20X magnification for evidence of pitting, etching, or other attack.

4.6 <u>Glassware cleanup</u> - The glassware is rinsed with suitable organic solvents to remove all oil. The sample tubes, heads, and air delivery tubes are washed with soap or detergent and water. The air delivery tubes may be discarded if they are not readily cleaned. All of this glassware, whether new or precleaned by the methods described, is then soaked in chromic acid solution until all deposits are removed, then rinsed thoroughly with tap water and then with distilled water, and oven dried. The clean glassware may be protected with aluminum foil until ready for use. The condensers and condensate flasks are rinsed with suitable solvents as required to remove all oil and any deposits that are present. The condensers may be left in place during this rinsing. The joints on the condensers and condensate flasks are capped with aluminum foil after cleaning. 4.7 Determination of oil properties - The total acid number is determined on samples of the original oil, oxidized oil, and overhead product, using the electrometric titration method (Fed. Test Method Std. No. 791, Method No. 5106.4) but titrating to 11 pH. The viscosity at 100°F is determined on samples of the original oil and oxidized oil, using a kinematic viscosimeter (Fed. Test Method Std. No. 791, Method No. 305.3).

### 5. REPORT

The total acid number is reported in mg KOH per gram oil for the original oil, oxidized oil, and overhead product. The viscosity is reported in centistokes at 100°F for the original oil and oxidized oil. The weight loss of the oil sample during test and the weight of overhead product collected are reported in grams. Visual observations of sludge, lacquer, or other deposits are reported. The metal specimen weight changes are reported in milligrams per square centimeter of metal surface. The condition of the metal surfaces is reported in terms of visual observations of pitting, etching, or other evidences of corrosion.

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