Commander
Wright Air Development Center
Air Research and Development Command
Wright-Patterson Air Force Base
Ohio

Attention: WCLDE-1

Subject: Contract No. AF 33(616)-6090
Report No. 1, Project A-414

Dear Sir:

I. Laboratory

Adequate chemical laboratory space has been set aside and organized for the exclusive use of this project. Equipment available includes a laboratory furnace with thermostat control for high temperature examination of raw materials and formulations, and solvent-dry ice systems for low temperature experiments. Also included is mixing apparatus for compounding the sealant in gram, multi-gram and pound quantities.

II. Materials

A survey of the literature is being carried forward as rapidly and thoroughly as possible. Several oils which nearly meet the temperature requirements have been identified as industrial materials, and one such compound has been procured. Requests for suggestions concerning oils and solids which may be applicable have been sent to several manufacturers who have research and development experience in the field of high temperature materials. Several polar solid materials from the industrial samples on hand have been selected for preliminary screening and compounding experiments.

Preliminary compounding experiments with the applicable oils and solids already on hand has commenced.
III. Test Devices

Local metal suppliers are being contacted for fittings suitable for construction of the test assemblies and oxygen bomb test equipment. Although these devices are primarily intended for a more advanced phase of the project, it is anticipated that limited use may be made of them in earlier phases of the work.

Respectfully submitted,

W. H. Burrows
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division
December 5, 1958

Commander
Wright Air Development Center
Air Research and Development Command
Wright-Patterson Air Force Base
Ohio

Attention: WCLDE-1

Subject: Contract No. AF 33(616)-6090
Report No. 2, Project A-414

Dear Sir:

I. Materials

A thorough survey of recent technical literature is under way. This survey includes journal literature, manufacturers' releases, and ASTIA listings. Reference has been found to several oils and several solids which might be combined into an anti-seize compound with properties approaching the specifications of this contract. As rapidly as sources of these materials could be identified, they were placed on order for immediate shipment. Several samples of solids and three samples of oil were received on November 26, permitting initiation of more compounding and testing experiments.

II. Experimental

Dow Corning fluid QF6-7009 has been compounded with various solids, such as talc, graphite, aluminum powder, copper powder, and finely divided calcium silicate. The oil blended well with all of these solids, and no great difficulty was encountered in making a homogeneous paste, either by ball milling or by grinding with mortar and pestle.

Low temperature performance was satisfactory. As the oil was cooled by a mixture of acetone and dry ice, a waxy solid was observed to form prior to hardening and crystal formation. The temperature achieved by this refrigerant is approximately -100°F.
High temperature performance was unsatisfactory. Each of the greases was heated in an open crucible at 600°F, and in every case the oil in the sample decomposed, leaving a charred mass. The exposure was repeated with fresh samples at 500°F for 24 hours, and most of the oil evaporated, with only slight charring.

These greases have not as yet been exposed to these elevated temperatures in the confines of a threaded joint, under which conditions their performance may be quite different. However, their performance in an atmosphere of air at elevated temperature does not indicate the possibility of their passing the oxygen bomb test prescribed in the specifications.

It has been noted that less decomposition of the oil occurs in those samples compounded with talc or other light colored filler than in those compounded with graphite or carbon black.

III. Present and Future Work

The investigators believe that the success of a formulation will depend upon the availability of an oil stable over the specified range, or upon chemical or physical inhibition of oil phase decomposition. Several silicone oils with slightly more heat tolerance than the GF6-7009 fluid will be used in future compounding. Solid materials will be chosen for the antioxidizing as well as their lubricating properties.
Heat testing of threaded joints lubricated with these greases will be conducted in addition to the open crucible tests. A comparison can then be made as to the stability of these materials under confinement and in open vessels.

Respectfully submitted

W. H. Burrows,
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division
Commander
Wright Air Development Center
Air Research and Development Command
Wright-Patterson Air Force Base
Ohio

Attention: WCLDE-1

Subject: Contract No. AF 33(616)-6090
Report No. 3, Project A-414
Period December 1 through December 31, 1958

Dear Sir:

I. Materials

Of the materials thus far referenced in our literature surveys, four silicone fluids and a number of lubricating solids have been secured and subjected to preliminary tests. The literature surveys are being continued along previous lines to bring into the experimental program additional materials which appear to be applicable.

Preliminary tests have been conducted at high temperature and atmospheric pressure. Fittings for high pressure test assemblies have been placed on order.

II. Experimental

It is expected that the most difficult specification to meet will be that of high temperature performance of the fluid phase. For this reason, attention has been directed primarily towards development of a formulation which would withstand a temperature of 600°F for 24 hours or more in an open vessel.

When subjected to these conditions, all of the base fluids showed some fuming, thickening, or charring. The same effects were observable upon extended heating at 500°F, also, indicating that chemical decomposition was taking place rather than physical separation of a more volatile component.
Thickening, accompanied by fuming or smoking of the fluid, leads one to suspect a form of polymerization, in which a volatile by-product is split out and subsequently decomposed. Various additives have been included in the formulations to inhibit such polymerization, but results have thus far been negative.

Formulations of these oils with certain solids showed a marked improvement in temperature stability over that of the oils alone. There was still some fuming or smoking during initial phases of the heating at 600°F, but the mix retained the physical properties required for thread sealing and antiseize. If the tendency to fume or smoke during initial heating is not overcome by further modifications in formulation, it is expected that it might be overcome by subjecting the finished formulation to a cooking cycle.

Fluids subjected to preliminary examination to date are:

1. Dow Corning Fluid 710. This material has shown the most satisfactory performance thus far.

2. Dow Corning Fluids QF-258 and 710-R. These have been discarded because of excessive drying or decomposition of the anti-sieze compounds after several hours of heating.

3. General Electric Silicone Fluid 81705. Anti-sieze compound prepared from this material thickened during a 24 hour period at 600°F and continued to produce fumes during the entire experiment.

4. Two additional fluids, initially considered for experimentation, were not examined, as the literature indicated that they would be less stable than the materials cited above.

The high lubricity solids tested include molybdenum disulfide, graphite, carbon black, pyrogenic silica, plumbous oxide, finely divided alumina, aluminum powder, copper powder, and finely divided talc. Of these materials, Vulcan XC-72R\textsuperscript{1} furnace black, Cab-o-Sil\textsuperscript{1} high surface
area silica, and No. 1 Lo-Micron talc produced formulations which showed little or no deterioration and exhibited very slight thickening and fuming under the test conditions. Generally, formulations compounded with Cab-o-Sil and No. 1 Lo-Micron talc exhibited slightly less thickening in 24 hours at 600°F than did the anti-seize materials compounded with carbon black. These two formulations were selected for release testing.

Release Tests

Preliminary tests of the anti-seize properties of the above formulations were conducted using standard (not military specification) brass and steel tube fittings. The material was applied to the threads, and the fittings were tightened using torques representative of those in Mil-T-5542B (ASG), 12 July, 1954, for the thread diameters employed. The assemblies were heated at 600°F for 24 hours, cooled and disassembled.

In no case did the release torque exceed 60% of the initial torque. There was no discoloration or distortion of the threads in areas protected by the anti-seize compounds. A film of the compound was still observable on the threads.

Present and Future Work

As stated, specification fittings have been placed on order. Upon receipt of these fittings, high pressure (2000 lb. oxygen) and bomb testing of the most promising formulations will be initiated.

Immediate investigations are directed towards eliminating the tendency to fume during initial phases of the heating period. It is believed that

2. Whittaker, Clark and Daniels
this defect can be removed or reduced to a very low level by increasing the amount of high surface area silica to absorb excess oil. Inclusion of a final heating step in the compounding procedure may still be necessary in order to remove this defect entirely.

Respectfully submitted

W. H. Burrows
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division
Dear Sir:

I. Introductory Summary

Literature searches are being continued along the lines previously outlined in an effort to bring in additional leads as to materials applicable to this problem. No additional primary lubricating fluids have been referenced since Report No. 3; consequently, attention has been given largely to exploring supplementary materials which might add a few degrees to the upper limits of temperature stability of those lubricants previously examined.

There has been some difficulty in getting delivery on some of the AN Specification parts needed for testing these materials at high temperature under 2000 psig oxygen; consequently, this phase of the program has been somewhat delayed.

II. Experimental

Previous experimentation had established that the tendency of some of the silicic oils to fume and thicken upon prolonged exposure at 600°F was reduced by the addition of high surface area silica. This silica has the further advantage of serving as a filler and thickener. Further formulations have been prepared and examined to determine whether this protective quality of silica might be further exploited without exceeding the proportions permissible for a filler. A point
has been reached beyond which further addition of silica fails to increase stability of the oil, although there is still room for the addition of more silica if required for thickening purposes.

Industrial literature has indicated the possibility that certain dyestuffs of unusually high temperature stability might provide protection against oxidation and increase the stability of silicone oils. Accordingly, a series of greases is now being prepared containing Indanthrene Blue RS and/or silica in the previously examined silicone oils. These greases will be subjected to exposure at 600°F in the manner employed in original screening of oils. Should this material prove to add to the stability of the oil at high temperature, it would also have the further advantages of thickener and coloring additive. A colored material has the advantage of showing clearly the extent of coverage obtained and indicating the presence of leaks where such leaks are associated with loss of sealing compound from the joint.

The dye employed for this purpose is very difficult to dissolve in the oil when used in the form of a dry powder. Instead, it is provided in the form of a presscake containing, apparently, approximately 50 percent moisture. The presscake is ball milled into the oil to obtain a gritless suspension. The moisture is removed by gradually reducing the pressure until a high vacuum is obtained, then heating while still in vacuo. The solution of dye in oil obtained by this "flushing" technique is then compounded by the addition of silica, talc and/or additional oil.

III. Test Assemblies

Two test assemblies are described in MIL-T-5542B(ASG), 12 July 1954; "Suggested assembly for antiseize and sealing properties tests," (Figure 1, p. 5) and "Bomb assembly," (Figure 2, p. 8). Parts for the latter assembly have been secured, as have the necessary parts for attaching this assembly to a suitable pressure gauge, shut-off valve and oxygen cylinder, and for inserting the test assembly, under pressure, into the furnace. These parts have been assembled and subjected to initial tests under pressure. However, some leakage has been observed
at silver-soldered joints and at valve fittings. The complete assembly
is being overhauled and retested to eliminate all such leaks.

Parts for the "Suggested assembly for antiseize and sealing prop-
erties tests" have not all been delivered as yet. All possible
measures have been taken to insure their delivery at the earliest
possible date.

IV. Literature Searches

At the initiation of this project request was made for access to
ASTIA material pertinent to the problem. Past ASTIA listings were
surveyed, a close check was kept on all new ASTIA listings, and copies
of certain releases were requested. Approval of our request has now
been received. As yet there have been no significant contributions to
the technical information of this project from these releases, but the
search is being continued for information on additional heat-stable lub-
ricating materials and supplementary additives.

In addition, literature work is continuing in such fields of general
scientific knowledge as might uncover leads to new materials applicable
to this problem.

V. Present and Future Work

The program of work for the present and immediate future is implied
in the preceding review of our present status. This program may be
summarized as follows:

1. Completion of bomb assembly, preliminary testing of equipment
   under 2000 psig pressure and at 600°F.

2. Bomb testing of those formulations which have proved most
   satisfactory in preliminary examination of heat stability and lubricity.

3. Completion of test assembly for antiseize and sealing properties.
   Application of this assembly in testing formulations which have success-
   fully passed other tests.
4. Examination of formulations incorporating additional antioxidants and polymerization inhibitors.

5. Further exploration of ASTIA and other information sources.

Respectfully submitted,

W. H. Burrows
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division
Commander
Wright Air Development Center
Air Research and Development Command
Wright-Patterson Air Force Base
Ohio

Attention: WCLDE-1

Subject: Contract No. AF 33(616)-6090
       Report No. 5, Project A-414
       Period February 1 through February 28, 1959

Dear Sir:

I. Introductory Summary

    Literature searches are being continued.

    Results obtained in preliminary testing under 2000 psig. oxygen have indicated the need for certain modifications, which are now being incorporated in the formulation.

    A sample of a preliminary formulation has been submitted to the sponsor, although performance of this material is not adequate for a final formulation.

II. Experimental

    Report No. 4 described a method of incorporating into the formulation a dyestuff, such as Indanthrene Blue RS. This material is reported to have an inhibiting effect upon the tendency for silicone oils to fume at high temperatures. Compounds have been prepared using various levels of this material in formulations made up of silicone oils, silica, and talc. While it has not been possible to eliminate smoking altogether, it has been found that a 5 per cent level of this dyestuff has a definite inhibiting effect on the tendency of the sealant to fume and thicken in open vessel tests at 600°F.
Samples of sealant, compounded of silicone oil, talc, silica and dyestuff were prepared and subjected to bomb and release testing. Under 2000 psig. oxygen, there was some separation of the oil from the talc filler, particularly at brass-to-brass and brass-to-aluminum connections. This separation was followed by slow leakage around the threads. Stereomicroscopic examination indicated a deposit of talc on the threads and minute oil droplets on the sample in the bomb assembly. It is anticipated that an increased loading of the material with talc, or the incorporation of additional filler material, such as carbon black or graphite, may reduce the tendency to separate. Samples involving both possibilities are being prepared and will be tested under pressure as soon as they are ready.

III. Test Assemblies

The bomb assembly was put out of service by failure of an AN816-2D nipple, which seized during a high temperature test and cracked during removal from the bomb assembly. Seizure of this part occurred when the furnace went out of control during an extended test, and the part was held at 1000°F for several hours. Procurement of a replacement part proved more difficult than expected; however, a replacement steel part has been fabricated locally and has been found to function satisfactorily.

Sufficient parts have been delivered for construction of the "Suggested assembly for antiseize and sealing properties tests." This assembly is being placed in service for the further evaluation of formulations which exhibit promise in bomb testing.

IV. Literature Searches

Surveys of current technical literature are being continued. One ASTIA document (AD 131001) has been received and studied. No materials which had not previously been screened on this project were referenced in the literature reviewed during this period.
V. Present and Future Work

The program of present and future work is implied in the preceding review of our present status. This program may be summarized as follows:

1. Formulation of sealant compounds involving molybdenum disulfide, carbon black, and larger quantities of talc as filler materials to impart more "body" to the sealant at elevated pressures.

2. Bomb testing of these formulations under 2000 psig. oxygen at 600°F and at 744°F.

3. Application of test assembly for antiseize and sealing properties to formulations which have passed other tests.

4. Further exploration of ASTIA and other information sources.

Respectfully submitted,

[Signature]

W. H. Burrows,  
Project Director

Approved:  

[Signature]

Wyatt C. Whitley, Chief  
Chemical Sciences Division
Commander
Wright Air Development Center
Wright-Patterson Air Force Base
Ohio

Attention: WCLDE-1

Subject: Contract No. AF 33(616)-6090
Report No. 6, Project A-414
Period March 1 through March 30, 1959

I. Introductory Summary

New literature is continually being surveyed for new materials applicable to this problem.

Difficulty in obtaining certain fittings has limited the experimental program involving exposure to oxygen at 2000 psig.

Modifications have been made in the formulation to overcome some of the difficulties encountered with the formulation submitted last month.

Unexpected delays in the experimental program have necessitated a request for a sixty day extension of time without an increase in appropriation.

II. Experimental

Report No. 5 mentioned certain difficulties encountered with the formulation involving silicone oil, a dyestuff, silica and talc. The principal handicap has been the tendency of the oil to separate from the filler material upon prolonged heating, and under 2000 psig pressure. This effect is apparently brought about by the comparatively low absorption capacity of the filler materials for oil. Recent formulations have substituted Fuller's earth for part or
all of the talc with the result that the tendency to separate has been greatly reduced.

The Fuller's earth, however, does not have the lubricity of talc; consequently, this substitution was made at the expense of antiseize properties. This deficiency has been offset by the introduction of additional quantities of molybdenum disulfide and graphite.

A difficulty which was not anticipated in applying MIL-T-5542B(ASG), 12 July 1954, tests at 600°F is the pronounced effect of the differences in coefficients of thermal expansion of the metals comprising the fittings from which the test assemblies are made. At the temperatures for which this specification was written, these effects are minor. At 600°F, thermal expansion causes sufficient separation between certain fittings that oxygen at 2000 psig can leak through. When the temperature is reduced, the joints again become sufficiently tight to hold the pressure without loss.

Recent formulations have attempted to overcome this difficulty through the introduction of finely divided aluminum powder. Thus far, this material has shown some promise of mitigating thermal expansion effects, particularly at connections involving brass male to aluminum female and steel male to brass female threads.

III. Test Assemblies

The bomb and release testing assemblies have been put out of service by failure of AN 818-2D and AN 819-2D fittings on the filler assembly. Delivery of replacement parts is anticipated within the next few days.

One difficulty encountered in applying these test assemblies is the inability of AN 816-2D nipples to hold up under the torques specified in MIL-T-5542B(ASG), 12 July 1954. This part shows pronounced deformation and cracking at lesser torques, even before heating. For this reason, it has been necessary to confine torque values for these fittings to about seventy-five per cent of that specified.
The extreme test conditions specified for these fittings has led to frequent failures requiring replacement. Stocks of suppliers who bid on these items seem to remain sufficiently low that back-orders are often required. These factors have so delayed this phase of the experimental program, that request for an extension of time recently became necessary. This request was made formally by Mr. Harry L. Baker, Jr. of Georgia Tech Research Institute, in a letter dated 18 March 1959, to Commander, Wright Air Development Center, Attention: Contracting Officer, Mr. A. L. Sidnell, WCKSC. While the primary need for this extension arises from the delay in procuring replacement parts for the test assemblies, it is believed that recent developments in formulation will enable us to provide a more satisfactory product with the additional time than could have been produced during the original time period of the contract.

Another difficulty encountered with test assemblies arises in applying at 600°F test methods originally designed for use at 250°F. This is the large increase in gas pressure encountered at the higher temperature. The bomb or other test assembly is charged, prior to heating, with 2000 psig of oxygen. An increase in temperature to 250°F may increase this pressure to about 2500 psig., but at 600°F the pressure may easily be 4000 psig. This effect has been offset to some degree in bomb tests by the inclusion of an expansion chamber in the line between bomb and shut-off valve. A similar modification is anticipated in testing with the "Suggested assembly for antiseize and sealing properties tests," as it appears to be the intent of the specification that the sealant withstand a pressure of 2000 psig at 600°F, rather than 4000 psig at that temperature.

IV. Literature Search

A number of documents selected from ASTIA listings have been requested. Of those thus far received, none have suggested any materials which have not already been screened on this project.
Literature work is continuing in such fields of general scientific knowledge as might uncover leads to new materials applicable to this problem.

V. Present and Future Work

The program of work for the present and immediate future is implied in the preceding review of our present status. This program may be summarized as follows:

1. Repair of the filler assembly and preliminary testing of equipment under 2000 psig pressure of oxygen at 600°F.
2. Bomb testing of those formulations which have proven most satisfactory in earlier examinations of heat stability, lubricity, and sealing properties under elevated temperatures and pressures.
3. Application of test assembly for antiseize and sealing properties in testing formulations which have successfully passed other tests.
4. Preparation and submission of a modified preliminary sample at a very early date.
5. Examination of formulations incorporating additional fillers, antioxidants, and polymerization inhibitors.
6. Further exploration of ASTIA and other information sources.

This program is based upon the assumption that the requested extension of time for this contract will be granted. In the event that this request is not granted, the program for the immediate future will consist in preparation of a final report and of twenty 4-oz. jars of that compound which at present appears to be most acceptable.

Respectfully submitted,

W. H. Burrows,
Project Director

Wyatt C. Whitley, Chief
Chemical Sciences Division
Commander
Wright Air Development Center
Wright-Patterson Air Force Base
Ohio
Attention: WCLDE-1

Subject: Contract No. AF 33(616)-6090
Report No. 7, Project A-414
Period April 1 through April 30, 1959

I. Introductory Summary

New literature is continually being surveyed for new materials applicable to this problem.

Repeated failure of aluminum fittings in the bomb test assembly has necessitated their replacement after nearly every high temperature exposure to 2000 psig oxygen.

Modifications in the formulation described in Report No. 6 have been found to have an adverse effect upon antiseize properties. Further modifications are at present being made.

Notice has been received that the requested sixty-day, no cost extension of time has been granted.

II. Experimental

Report No. 6 mentioned substitution of fuller's earth for talc in the filler material, addition of aluminum powder to counteract thermal expansion effects, and increased level of molybdenum disulfide or graphite to improve lubricity of the formulation. These modifications increased the time at 600°F under 2000 psig oxygen before significant pressure drops were observed, but the lubricity of these compounds disappeared completely upon heating. Bomb samples were recovered as a coked powder and the aluminum fittings in the bomb assembly were broken or severely deformed after each trial.
Talc has been the most satisfactory filler employed in these formulations to date, but talc-filled compounds have been observed to thin out on long standing. At present, larger quantities of talc are being milled into the base oil stock by adding small portions of talc as rapidly as each previous addition is absorbed. Formulations of this type are being prepared in Dow Corning 710 silicone fluid and in a solution of indanthrene dyestuff in the same fluid.

The following items of information gleaned from ASTIA documents are considered significant in the choice of methyl phenyl silicone (Dow Corning 710) as a base fluid, and offer some explanation of the behavior of sealants in which it is used.

1. The relative oxidation stability of DC 710 fluid is greater than that of polyphenyl ethers, certain substituted polyphenyl ethers and diester base jet turbine oils.

2. DC 710 fluid gives better wear in a Shell 4-ball tester than polyphenyl ethers or diester base oils.

3. The decomposition temperature of DC 710 fluid only approaches 600°F, but base fluids having higher temperature resistance are less satisfactory because of high pour point, or low oxidative stability.

4. Certain diesters with selenide inhibitors appear to exhibit superior oxidative and temperature resistance, but the toxicity of selenium compounds prohibits their use in an aircraft oxygen system.

5. Both copper and iron appear to catalyze oxidative attack on intermolecular linkages of several possible base fluids at elevated temperatures.

III. Test Assemblies

A supply of AN 816-2D, AN 818-2D and AN 819-2D fittings for the bomb and release testing assemblies has been secured. As the principal cause for delay in this program has been in securing replacement of these parts, more nearly continuous testing should now be possible.

It should be noted that copper is frequently mentioned in the literature as a catalyst for cleaving carbon-to-carbon or carbon-to-silicon bonds. While it has not been specifically established for this program, it is suspected that some form of copper catalyzed decomposition may be significant in the failure of the oil stock at the brass fittings.
IV. Literature Search

A number of documents selected from ASTIA listings have been received, and additional documents are at present on request. Of those thus far received, none has suggested any materials which have not already been screened on this project. As noted in Section II, information gleaned from several ASTIA documents has served to corroborate the findings of our laboratory.

Literature work is being continued in such fields of general scientific knowledge as might indicate additional leads to materials applicable to this project.

V. Present and Future Work

The program of work for the present and immediate future is as follows:

1. Testing of formulations containing higher concentrations of talc as a filler at 600°F and 2000 psig oxygen.
2. Testing the most promising of these formulations at 550°F and 2000 psig oxygen.
3. Submission of a preliminary sample of the most promising formulation.
4. Examination of formulations incorporating additional fillers, antioxidants, and polymerization inhibitors.
5. Preparation of twenty 4-oz. jars of the formulation, if approved by the sponsor.
6. Further exploration of ASTIA documents and other literature sources.

Respectfully submitted,

W. H. Burrows,
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division
I. Introductory Summary

Literature searches, formulations of sealants and testing at 600°F under 2000 psig oxygen have been continued. A second preliminary sample of the most successful thread sealant thus far developed in this program has been submitted for preliminary examination. It is felt that the performance of this sample excels that of the first preliminary sample. Components for the twenty four-ounce jars of sealant are being readied, and a preliminary draft of the final technical report is being prepared.

II. Experimental

Previous experimental work had indicated methyl phenyl silicone (Dow Corning 710) as the most promising base fluid available and an increased quantity of talc as a practicable method of preventing separation of liquid and solid components on standing. An indanthrene dye (Helio Fast Blue RS) had appeared to inhibit slightly the decomposition of the oil stock at elevated temperatures under 2000 psig oxygen. Talc alone did not afford sufficient lubricity to permit the aluminum fittings to be separated without severe damage after heating, indicating a need for addition of other solid lubricants. Of the solid lubricants available, only graphite and molybdenum disulfide appeared to have significant resistance to decomposition under the specified test conditions.
Several sealants representing varying levels of the materials cited above along with a pyrogenic silica (Cab-O-Sil\textsuperscript{2}) thickener were prepared and tested at 600°F under 2000 psig oxygen (for twenty-four hours) or until significant leakage appeared in the test assembly.

No formulation met completely the specifications of MIL-T-5542B(ASG), as amended. In all cases, after twenty-four hours under 2000 psig oxygen at either 550°F or 600°F the oil phase had deteriorated. Test assemblies using sealants containing both molybdenum disulfide and indanthrene dye in the sealant were most satisfactory, in that they required relatively low torques for disassembly and were more resistant to leakage. Under similar test conditions, sealants containing graphite failed to release readily and left a harder, drier, caked residue in the bomb assembly than did the molybdenum disulfide formulations. Sealants containing indanthrene dye as an oxidation inhibitor were more resistant to leakage than those in which the dye was omitted. They were also recovered as a cake containing some tarry material rather than the completely dry cake left by sealants formulated without the dye.

A four ounce sample of the most successful of these formulations has been forwarded to Wright Air Development Command\textsuperscript{3} for preliminary testing. This formulation includes methyl phenyl silicone, pyrogenic silica, molybdenum disulfide and an indanthrene dye at their optimum levels and has performed more satisfactorily than any other sealant tested.

III. Test assemblies

Testing has been restricted to a bomb assembly rather than a more complex release assembly. This procedure seemed justifiable in view of the several sealants to be examined and the large differences in their properties.

IV. Literature Search

A survey of ASTIA documents, industrial literature, and technical periodicals has suggested no material or compounding technique not previously screened on this project.
V. Present and Future Work

Sufficient materials are on hand or on order to permit rapid preparation of twenty four-ounce jars of the sealant if the preliminary sample is approved by WADC. Work on a preliminary draft of the final technical report has been initiated.

Respectfully submitted,

W. H. Burrows,
Project Director

Wyatt C. Whitley, Chief
Chemical Sciences Division

1. General Dyestuff Company
2. Godfrey L. Cabot, Inc.
3. May 25, 1959 to Transportation Officer, AFB 2300, Building 258, M/F Department 49, Attention: WCLDE-1 AF 33(616)-6090, Wright-Patterson Air Force Base, Ohio; from W. H. Burrows
   May 27, 1959 to Commander, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio; from Milton W. Bennett
I. Introductory Summary

Literature searches have been continued for information on base liquid and solid components of higher thermal stability and superior lubricity. Formulation of new compounds and testing at 600°F under 2000 psig oxygen have also been continued.

Preparation of twenty four-ounce jars of the sealant has been initiated, although there has been some delay in receiving from the sponsor approval of the preliminary sample previously submitted.

The final report is being prepared.

II. Experimental

Among new products screened for use in the sealant composition is zirconium chloride. Preliminary examination indicated that inclusion of this material in the sealant would be deleterious. Thus far, no composition offering properties superior to those demonstrated by the second preliminary sample (see Report No. 8) has been discovered.

Preparation of sufficient sealant for the twenty four-ounce jars has presented some difficulties, as a rather severe form of ball milling is required in order to disperse the solids uniformly in the small amount of base fluid used. These difficulties have been largely overcome, and it is expected that the samples will be ready for shipment upon receipt of approval of the preliminary sample.
III. Present and Future Work

A preliminary draft of the final technical report for this project has been prepared and reviewed. The text is now being revised and rearranged in conformation of the format specified in ARDC Manual No. 5-1. Final editing and preparation of copies for the sponsor will be handled by the Reports Section of Technical Information Service.

Respectfully submitted,

W. H. Burrows,
Project Director

Approved:

Wyatt C. Whitley, Chief
Chemical Sciences Division
ANTISEIZE AND THREAD SEALING COMPOUND
FOR AIRCRAFT OXYGEN SYSTEMS

W. H. Burrows
L. W. Elston

Engineering Experiment Station
Georgia Institute of Technology

Contract No. AF 33(616)-6090

AEROSPACE MEDICAL LABORATORY
WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO
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ANTISEIZE AND THREAD SEALING COMPOUND
FOR AIRCRAFT OXYGEN SYSTEMS

W. H. Burrows
L. W. Elston

Engineering Experiment Station
Georgia Institute of Technology

Contract No. AF 33(616)-6090

AEROSPACE MEDICAL LABORATORY
WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO
FOREWORD

This report covers research on antiseize and thread sealing compounds conducted by the Engineering Experiment Station of Georgia Institute of Technology, with Mr. W. H. Burrows as principal investigator. The objective of this research was the development of a compound, incorporating solid and liquid lubricants, capable of sealing threaded pipe joints against pressures of oxygen up to 2000 psig and operative at temperatures from -65° to +600° F.

This report was prepared for the Aircraft Equipment Section, Engineering and Development Branch, Aerospace Medical Laboratory, Wright Air Development Center, under Contract No. AF 33(616)-6090, Project No. A-414. Work began October 1, 1958 and was completed May 31, 1959.

The authors are indebted to the staff of the Georgia Tech Research Institute for their help in handling the many administrative details of this project, to the staff of the Engineering Experiment Station for assistance in the preparation of reports, and to the staff of the Industrial Products Branch for constant assistance in the conduct of the research. Contracting agency for this project was the Georgia Tech Research Institute, Research Building, Georgia Institute of Technology, Atlanta 13, Georgia.
ABSTRACT

A study has been conducted to formulate and develop an antiseize and sealing compound from commercially available materials for use in aircraft oxygen systems in the -65° to 600°F temperature range. No formulation was stable under 2000 psig oxygen at 600°F for extended periods, but one compound exhibited satisfactory antiseize and sealing properties despite decomposition of its liquid phase. Compounds have been formulated by blending finely divided solids as antioxidants, thickeners, fillers, and inhibitors in the several commercially available silicone and siloxane fluids stable at temperatures near 500°F. Suitable thickeners, fillers, and solid lubricants are available, but further work is necessary to improve the stability of base fluids in the environment specified.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:
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I. INTRODUCTION

The purpose of this first study is the development of an antiseize and sealing thread compound for aircraft oxygen systems. The requirements for this compound as defined in Specification MIL-T-5543B(ASG) are that it be a stable, homogeneous material of greaselike consistency, that it be suitable for use from \(-65^\circ\) to \(600^\circ\) F under 2000 psig oxygen, and that it be physiologically harmless in the environment. The scope of the study was formulation from existing materials rather than synthesis of new temperature-resistant liquids and solids.

A literature survey of materials suitable over the specified temperature range revealed no liquid stable under oxygen at \(600^\circ\) F and only a very limited number of fluids serviceable in the \(500^\circ\) F area. Inquiries directed to manufacturers of high temperature lubricants brought forth even less encouraging suggestions. Experimental effort was directed toward blending various solid lubricants, antioxidants, and inhibitors with several of the most thermally stable liquids available to form a thread compound which would perform for as long a time as possible at temperatures in excess of the decomposition temperature of the fluid alone.

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II. EXPERIMENTAL WORK

A. Screening of Base Fluids

A survey of technical literature, industrial bulletins, and documents from the Armed Services Technical Information Agency indicated several possible base fluids.\textsuperscript{2,3,4,5} As lubricity in the formulation could readily be imparted through the solid phase, shear strength was considered less important than thermal stability and low solidification points. We could not find any reference to a fluid that was designed for operation in open systems or in systems containing oxygen under high pressures at elevated temperatures.

Initial screening of the base fluids was carried out using 5-gram samples of each liquid in open porcelain crucibles. The samples were placed in a muffle furnace and maintained at 600° F for 24 hours. Periodic observations of physical characteristics are summarized in Table I.

Performance in these trials indicated that Dow Corning Silicone fluid 710 (methyl phenyl silicone) shows the least deterioration in the presence of air at 600° F. This observation is in agreement with observations made by other investigators of extreme high temperature lubricants.\textsuperscript{6,7} Polyphenyl ethers were considered and rejected because of their high melting points and low oxidative stability.\textsuperscript{6,7}

<table>
<thead>
<tr>
<th>Material</th>
<th>10 Minutes</th>
<th>1 Hour</th>
<th>6 Hours</th>
<th>24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dow Corning QF-6-7009</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor, some thickening</td>
<td>Very little, slightly charred, residue</td>
</tr>
<tr>
<td>GE 81644</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor, some thickening</td>
<td>Gel, slight fuming</td>
</tr>
<tr>
<td>GE Versilube F-50</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Very little, slightly charred, residue</td>
</tr>
<tr>
<td>GE 81705</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Gel, very slight fuming</td>
</tr>
<tr>
<td>Dow Corning QF</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor, some drying</td>
<td>Very slight residue</td>
</tr>
<tr>
<td>Dow Corning 200</td>
<td>Fuming, charred odor</td>
<td>Fuming, charred odor</td>
<td>Thickening, fuming, charred odor</td>
<td>Slight charred residue</td>
</tr>
<tr>
<td>Dow Corning 710</td>
<td>Slight fuming, odorless</td>
<td>Slight fuming, odorless</td>
<td>Slight fuming, odorless</td>
<td>Slightly thickened yellow liquid, slight fuming</td>
</tr>
<tr>
<td>Dow Corning 710R</td>
<td>Fuming, sharp, acrid fumes</td>
<td>Acrid fumes, test discontinued</td>
<td>---</td>
<td>---</td>
</tr>
</tbody>
</table>

TABLE I

OPEN CRUCIBLE SCREENING OF BASE FLUIDS AT 600° F
B. Screening of Solid Materials

1. Thickeners and Fillers

As the silicone base fluids possess only limited shear strength and oxidative stability, it is necessary that a large portion of the compound's lubricity and oxidative stability be derived from the solid materials incorporated as thickeners and fillers. These solids must possess sufficiently high surface area to adsorb and hold the base fluid to form an homogeneous, grease-like substance which neither thins nor separates on standing. They must also impart sufficient body to the sealant to resist displacement under extreme pressure and temperature. As no single material possesses all of these properties, it was necessary to include several solids in the formulation in such proportions as to produce maximum benefit without exceeding for each material some limit beyond which the over-all effectiveness of the sealant is decreased. No literature was found describing lubricants to function in an environment of 2000 psig oxygen at 600 °F and with low tolerance for toxicity of either the lubricant or its decomposition products. The choice of the solid materials and their optimum levels was made by trial and error after a study of the literature with respect to each component. The sealants formulated with each of these solids and diphenyldidodecyl-silane (Dow Corning fluid QF-6-7009) are listed in Table II. These materials exhibited such decomposition in open-crucible tests that bomb testing under 2000 psig oxygen was omitted.

A more extensive selection of solids was screened using methyl phenyl silicone (Dow Corning 710 fluid) as a base liquid. As the upper limit of thermal stability for this fluid approaches the 600 °F operating temperature, sought for the sealant formulation, smaller contributions are required from each of the solid materials included. Generally, each of the solids makes a single principal contribution as a thickener, filler, lubricant, or antioxidant, although a material included for one purpose might produce additional benefits in another area (e.g., some increase in lubricity is obtained by incorporating talc as a filler). These trials are summarized in Table III.

2. Solid Lubricants

As nearly all of the samples tested exhibited some drying on prolonged exposure to air at 600 °F, it seemed desirable to substitute a solid lubricant for some or all of the filler and thickener in the sealant. This procedure, if practicable, would produce the necessary antiseize properties even if the base fluid were partially destroyed. Formulations employing two grades of molybdenum disulfide and three base fluids were examined. High levels of molybdenum disulfide produced a sharp increase in the decomposition rate of the base fluid. Decreasing the level of molybdenum disulfide permitted its use without complete hardening of the sealant in 24 hours' exposure to air at 600 °F. These trials are summarized in Table IV.

3. Inhibitors and Antioxidants

Failure of the liquid phase was the most obvious common characteristic of all the unsatisfactory formulations. This failure could be attributed to vaporization, oxidative cleavage followed by vaporization, polymerization of the fluid, or polymerization of oxidation products of the fluid. It seemed probable
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible 600°F, 24 Hours</th>
<th>Open Crucible 500°F, 24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>27 g DC QF-6-7009 fluid, 25 g Dixon's MicroFine graphite</td>
<td>Oil added gradually over 24 hours on ball mill</td>
<td>Dry, slightly charred powder</td>
<td>Hardened, no charring</td>
</tr>
<tr>
<td>2</td>
<td>24.5 g DC QF-6-7009 fluid, 25 g No. 1 Lo Micron talc</td>
<td>Oil added gradually over 24 hours on ball mill</td>
<td>Dry, slightly charred powder</td>
<td>Soft, no charring (hardened in 72 hours)</td>
</tr>
<tr>
<td>3</td>
<td>35 g DC QF-6-7009 fluid, 25 g Micro-Coal</td>
<td>Oil added gradually over 24 hours on ball mill</td>
<td>Charred cake</td>
<td>Not attempted</td>
</tr>
<tr>
<td>4</td>
<td>28 g DC QF-6-7009 fluid, 6.25 g graphite, 18.75 g No. 1 talc</td>
<td>Oil added periodically over 24 hours on ball mill</td>
<td>Dry, slightly charred powder</td>
<td>Hardened, no charring</td>
</tr>
<tr>
<td>5</td>
<td>10 g DC QF-6-7009 fluid, 0.1 g aluminum powder MD3100, 10 g graphite</td>
<td>Mortar and pestle, 50 minutes</td>
<td>Not attempted</td>
<td>Charred cake</td>
</tr>
<tr>
<td>6</td>
<td>10 g DC QF-6-7009 fluid, 0.2 g aluminum powder MD3100</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Cake, no charring</td>
</tr>
<tr>
<td>7</td>
<td>10 g DC QF-6-7009 fluid, 0.5 g aluminum powder MD3100, 10 g graphite</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Charred cake</td>
</tr>
<tr>
<td>8</td>
<td>10 g DC QF-6-7009 fluid, 1 g aluminum powder MD3100, 10 g graphite</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Charred cake</td>
</tr>
<tr>
<td>9</td>
<td>10 g DC QF-6-7009 fluid, 0.2 g aluminum powder, 9 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Powder, no char</td>
</tr>
<tr>
<td>10</td>
<td>10 g DC QF-6-7009 fluid, 9 g No. 1 talc, 0.2 g copper powder No. 132</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Charred, slightly caked</td>
</tr>
<tr>
<td>11</td>
<td>6 g DC QF-6-7009 fluid, 4 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Greasy, slightly charred appearance</td>
</tr>
</tbody>
</table>
### TABLE III
OPEN CRUCIBLE SCREENING OF FILLERS AND FLUIDS

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible 600° F, 24 Hours</th>
<th>Open Crucible 500° F, 24 Hours</th>
<th>Non-specification Brass Fittings 600° F, 24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>30 g DC 710 fluid, 10 g No. 1 talc, 1 g Cab-O-Sil M-6</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>No visible change</td>
<td>Not attempted</td>
</tr>
<tr>
<td>13</td>
<td>30 g DC 710 fluid, 10 g No. 1 talc, 1 g Alon C</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight drying on surface</td>
<td>No visible change</td>
<td>Assembly torque 50 ft lb, release torque 30 ft lb, no discoloration or thread distortion, sealant dried slightly</td>
</tr>
<tr>
<td>14</td>
<td>30 g DC 710 fluid, 1 g carbon black, Vulcan XC-72-R</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>No visible change</td>
<td>Not attempted</td>
</tr>
<tr>
<td>15</td>
<td>30 g DC 710 fluid, 2.5 g Cab-O-Sil</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight drying on surface</td>
<td>Slight drying on surface</td>
<td>Not attempted</td>
</tr>
<tr>
<td>16</td>
<td>30 g DC 710 fluid, 5 g Alon C</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Slight drying on surface</td>
<td>Not attempted</td>
</tr>
<tr>
<td>17</td>
<td>30 g DC 710 fluid, 5.5 g carbon black, Vulcan XC-72-R</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight drying on surface</td>
<td>No visible change</td>
<td>Assembly torque 50 ft lb, release torque 30 ft lb, no discoloration or thread distortion, sealant dried slightly</td>
</tr>
<tr>
<td>18</td>
<td>30 g DC 710 fluid, 2 g Cab-O-Sil M-6, 5 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight drying on surface</td>
<td>No visible change</td>
<td>Assembly torque 50 ft lb, release torque 30 ft lb, no discoloration or thread distortion, sealant dried slightly</td>
</tr>
<tr>
<td>19</td>
<td>30 g DC QF-258 fluid, 2.5 g Cab-O-Sil, 1 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Soft, but some drying throughout</td>
<td>Not attempted</td>
</tr>
<tr>
<td>20</td>
<td>30 g DC QF-258 fluid, 2.5 g Alon C, 5 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Not attempted</td>
<td>Soft, but some drying throughout</td>
<td>Not attempted</td>
</tr>
<tr>
<td>21</td>
<td>35 g GE fluid 81705, 3 g Cab-O-Sil, 2 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Thickened, some fuming after 24 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>22</td>
<td>30 g GE fluid 81705, 4 g carbon black, Vulcan XC-72-R</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Thickened, some fuming after 24 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
</tbody>
</table>
### TABLE IV

**SOLID LUBRICANTS AS THICKENERS AND FILLERS**

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible 600°F, 24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>23</td>
<td>30 g DC 710 fluid 30 g moly sulfide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked in 15 minutes hardened in 3 hours</td>
</tr>
<tr>
<td>24</td>
<td>30 g DC 710 fluid 30 g moly sulfide technical fine</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked in 6 minutes, hardened in 3 hours</td>
</tr>
<tr>
<td>25</td>
<td>30 g GE fluid 81705 30 g moly sulfide pure</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked in 6 minutes, hardened in 3 hours</td>
</tr>
<tr>
<td>26</td>
<td>30 g GE fluid 81705 35 g moly sulfide technical fine</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked in 6 minutes, hardened in 3 hours</td>
</tr>
<tr>
<td>27</td>
<td>30 g DC QF-258 fluid 40 g moly sulfide pure</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked in 6 minutes, hardened in 3 hours</td>
</tr>
<tr>
<td>28</td>
<td>30 g DC QF-258 fluid 40 g moly sulfide technical fine</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked in 6 minutes, hardened in 3 hours</td>
</tr>
<tr>
<td>29</td>
<td>30 g DC 710 fluid 2.5 g Cab-O-Sil 1 g moly sulfide technical fine</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight smoking after 15 minutes, some increase in viscosity after 24 hours</td>
</tr>
<tr>
<td>30</td>
<td>30 g GE fluid 81705 2.5 g Cab-O-Sil 1 g moly sulfide technical fine</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Smoked after 15 minutes, hardened in 24 hours</td>
</tr>
</tbody>
</table>

that at least two (and quite possibly all) of these routes were involved in base fluid decomposition. Screening of several nontoxic oxidation and polymerization inhibitors appeared to offer a means of extending the high temperature life of the fluid. Open-crucible tests were made using a commercially available inhibited methyl phenyl silicone (Dow Corning 710R) and with normal fluids to which an inhibitor was added. These experiments are recorded in Table V.

4. **Indanthrene Dyestuff**

A successful extreme temperature grease consisting of methyl phenyl silicone thickened with an indanthrene dye suggested an additional means to extend the extreme temperature life of the sealant. The dyestuff, Helio Fast-Blue RSV presscake, was supplied as a cake containing 32 per cent by weight dye and 78 per cent water. Efforts to compound a sealant directly from this material were unsuccessful.
### Table V

#### FORMULATIONS INCLUDING OXIDATION AND POLYMERIZATION INHIBITORS

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible 600°F, 24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>31</td>
<td>15 g DC 710R fluid 1.5 g Cab-O-Sil 1 g No. 1 talc</td>
<td>Mortar and pestle, 50 minutes</td>
<td>Acrid smoke in 10 minutes viscosity increase in 24 hours</td>
</tr>
<tr>
<td>32</td>
<td>15 g DC 710R fluid 1.5 g Cab-O-Sil 1 g No. 1 talc 0.01 g hydroquinone</td>
<td>Mortar and pestle, 50 minutes</td>
<td>Acrid smoke in 10 minutes viscosity increase in 24 hours</td>
</tr>
<tr>
<td>33</td>
<td>15 g DC 710 fluid 1.5 g Cab-O-Sil 1 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Fuming, no odor in 10 minutes; no fuming viscosity increase in 24 hours</td>
</tr>
<tr>
<td>34</td>
<td>15 g DC 710 fluid 1.5 g Cab-O-Sil 1 g plumbous oxide 0.01 g hydroquinone</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Fuming, no odor in 10 minutes; no fuming viscosity increase in 24 hours</td>
</tr>
<tr>
<td>35</td>
<td>15 g DC 710 fluid 1.5 g Cab-O-Sil 1 g plumbous oxide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Fuming, no odor in 10 minutes; no fuming viscosity increase in 24 hours</td>
</tr>
<tr>
<td>36</td>
<td>30 g DC 710 fluid 2.5 g Cab-O-Sil 2 g No. 1 talc 1 g aluminum powder washed†</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight fuming, some drying in 24 hours</td>
</tr>
<tr>
<td>37</td>
<td>30 g DC 710 fluid 2.5 g Cab-O-Sil 2 g No. 1 talc 1 g copper powder</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight fuming, some drying in 24 hours</td>
</tr>
</tbody>
</table>

† Aluminum powder washed with acetone to remove stearic acid coating.

A stock solution of the dyestuff was prepared by ball milling 50 grams of the presscake with 200 grams of Dow Corning 710 fluid for 72 hours. The water was removed by application of partial vacuum followed by increasing infrared heating in vacuo until no further moisture was evolved. The solution thus contained approximately 5 per cent (by weight) dyestuff. This solution will be entered in the remainder of this report as "5 per cent dye solution." Preliminary evaluation of formulations containing this inhibitor indicated more satisfactory fluid life at high temperature than had been obtained in earlier trials. These data are presented in Table VI.

5. Levels of Included Solids

The tendency of these formulations to thin on long standing and to separate on heating under pressure suggested that larger quantities of thickener and solid lubricant would produce a significant improvement in both sealing and antiseize properties. A series of more viscous formulations was prepared to explore the maximum permissible level of each of several solid components.
### TABLE VI

**FORMULATIONS INCLUDING 5 PER CENT DYE SOLUTION**

(Formulations 40-47 Differ only in Amount of 5 Per cent Dye Solution and DC 710 Fluid. Each Contains 0.5 g Cab-O-Sil and 1.6 g No. 1 talc.)

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible 600° F, 24 Hours</th>
<th>Bomb Test 2000 PSIG Oxygen, 600° F</th>
<th>Release Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>1 g 5% dye solution, 9 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 6 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>41</td>
<td>0.5 g 5% dye solution, 9.5 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 12 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>42</td>
<td>0.25 g 5% dye solution, 9.75 g DC fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening after 12 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>43</td>
<td>2 g 5% dye solution, 8 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 3-1/2 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>44</td>
<td>3 g 5% dye solution, 7 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 3-1/2 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>45</td>
<td>4 g 5% dye solution, 6 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 8 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>46</td>
<td>5 g 5% dye solution, 5 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 8 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>47</td>
<td>2.5 g 5% dye solution, 7.5 g DC 710 fluid</td>
<td>Mortar and pestle, 30 minutes</td>
<td>No visible thickening or drying, no fuming after 5 hours</td>
<td>Not attempted</td>
<td>Not attempted</td>
</tr>
<tr>
<td>48†</td>
<td>32.5 g 5% dye solution, 97.5 g DC 710 fluid,  9 g Cab-O-Sil, 33 g No. 1 talc‡</td>
<td>Ball mill, 24 hours</td>
<td>No visible thickening or drying, no fuming after 3 hours</td>
<td>Leakage after 30 minutes, brass and steel threads distorted</td>
<td>Unsatisfactory</td>
</tr>
</tbody>
</table>

†A portion of this preparation was forwarded to Wright Air Development Center as an initial preliminary sample.
‡The final one-gram quantities of both Cab-O-Sil and No. 1 talc were added with an additional 24 hours ball milling when the sealant was observed to thin on standing.
Several filler materials not previously screened were employed to reduce separation of the liquid and solid phases in the sealant. These trials are reported in Table VII.

None of the solid materials incorporated as thickeners, fillers, lubricants, antioxidants, or polymerization inhibitors exhibited any useful property not predictable from literature describing the product.12

As a thickener, pyrogenic silica (Cab-O-Sil) produced a compound less susceptible to drying and caking than did finely divided alumina (Alon C) or a heat-resistant furnace black (Vulcan XC-72-R). Shear strength and lubricity of the base fluid remained unchanged.

Finely divided talc (No. 1 Lo Micron) proved to be the most satisfactory of the filler materials screened, although it did not completely overcome the separation tendency of the liquid and solid phases. Fuller's earth (Florigel SPL, DICALITE) showed reduced tendency to separate but lacked the solid lubricant properties necessary to prevent thread seizing, as did metal powders and indanthrene dyestuff. Solid lubricants (molybdenum disulfide and graphite) failed to prevent separation and decomposition of the liquid phase.

As an oxidation inhibitor, an indanthrene dye (Hello Fast Blue RSV presscake) proved more satisfactory than did aluminum powder. It was not satisfactory in preventing phase separation or as a solid lubricant when used as the sole thickener and filler under 2000 psig oxygen at 600° F. Aluminum powder became an abrasive under these conditions.

Thickening of some of the liquids screened indicated polymerization in the base fluid. In an attempt to overcome this effect, hydroquinone and copper powder, both well known in the literature of organic chemistry as free radical inhibitors, were included in several formulations. No beneficial effects were observed with hydroquinone. Samples containing copper, which is also widely used as a catalyst for cleavage of chemical bonds, showed complete decomposition.

Of the two materials screened as solid lubricants, molybdenum disulfide proved more satisfactory. Samples containing graphite showed lesser antiseize properties than did those formulated with molybdenum disulfide. This behavior agrees with the known absorption of oxygen between the layers of graphite and formation of "graphitic acids" under extreme conditions. The level of solid lubricant was restricted by the inability of these materials to prevent separation and decomposition of the liquid phase.

C. Test Assemblies

Testing under 2000 psig oxygen at 600° F was carried out in the bomb assembly shown in Figure 2 of MIL-T-5542B(ASG), Military Specification, Thread Compound, Antiseize and Sealing, Oxygen Systems. This assembly consists of mated steel, brass, and aluminum aircraft fittings in a variety of sizes. A muffle furnace was used for heating. Incorporation of a needle valve between the gauge and the oxygen tank and incorporation of a small surge chamber adjacent to the valve formed a closed system.
**TABLE VII**

### SCREENING OF MORE VISCOUS FORMULATIONS

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible 600° F, 24 Hours</th>
<th>Bomb Testing 2000 PSIG Oxygen 600° F, 24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>49</td>
<td>20 g DC 710 fluid, 1.8 g Cab-O-Sil, 0.01 g moly sulfide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Noticeable drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>50</td>
<td>20 g DC 710 fluid, 2.8 g Cab-O-Sil, 2.5 g No. 1 talc, 0.01 g moly sulfide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Noticeable drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>51</td>
<td>20 g DC 710 fluid, 7 g Cab-O-Sil, 5 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Noticeable drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>52</td>
<td>20 g DC 710 fluid, 2 g Cab-O-Sil, 1.5 g No. 1 talc</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Noticeable drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>53</td>
<td>20 g DC 710 fluid, 2 g carbon black, 1.2 g moly sulfide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Charred, hard cake</td>
<td>Not attempted</td>
</tr>
<tr>
<td>54</td>
<td>20 g DC 710 fluid, 15 g No. 1 talc, 1 g Cab-O-Sil, 1.8 g moly sulfide</td>
<td>Ball mill, 72 hours</td>
<td>Dry, crumbled</td>
<td>Failed after 5 minutes</td>
</tr>
<tr>
<td>55</td>
<td>20 g DC 710 fluid, 20 g No. 1 talc, 29 g Cab-O-Sil, 15 g No. 1 talc</td>
<td>Ball mill, 72 hours</td>
<td>Partial separation of liquid followed by excessive drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>56</td>
<td>20 g DC 710 fluid, 2 g Cab-O-Sil, 1 g No. 1 talc, 2.5 g moly sulfide</td>
<td>Ball mill, 72 hours</td>
<td>Partial separation of liquid followed by excessive drying</td>
<td>Failed in 10 minutes, little, if any, thread distortion, satisfactory release</td>
</tr>
<tr>
<td>57</td>
<td>20 g DC 710 fluid, 1 g Cab-O-Sil, 3 g Dicalite White filler, 0.02 g moly sulfide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>58</td>
<td>20 g DC 710 fluid, 1 g Cab-O-Sil, 20 g DC 710 fluid, 49 g Florigel SPL, 0.029 g moly sulfide</td>
<td>Mortar and pestle, 30 minutes</td>
<td>Slight drying</td>
<td>Failed in 7 minutes, thread distorted, unsatisfactory release</td>
</tr>
<tr>
<td>59</td>
<td>20 g DC 710 fluid, 2 g Cab-O-Sil, 1 g No. 1 talc, 3 g Florigel SPL, 5 g No. 1 talc, 0.029 g moly sulfide</td>
<td>Ball mill, 24 hours, 30 minutes</td>
<td>Surface drying only</td>
<td>Not attempted</td>
</tr>
<tr>
<td>60</td>
<td>20 g DC 710 fluid, 30 g Florigel SPL</td>
<td>Ball mill, 48 hours</td>
<td>Surface drying only</td>
<td>Held 2000 psi over-night at 75° F, standard bomb failed in 5 minutes at 600° F, all-aluminum bomb lost 500 psi oxygen pressure in 18 hours at 600° F. Aluminum fittings in both assemblies broke when bombs were dismantled. Samples recovered as white, dry powder after 24 hours at 600° F under oxygen. Unsatisfactory release.</td>
</tr>
</tbody>
</table>

*All of the molybdenum disulfide used in sample No. 49 and succeeding samples is "technical fine" grade.*
<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Composition</th>
<th>Compounding Method</th>
<th>Open Crucible</th>
<th>Bomb Testing 2000 PSIG Oxygen 600° F, 24 Hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>61</td>
<td>10 g 5% dye solution 30 g DC 710 fluid 2 g Cab-O-Sil 30 g Florigel SPL .04 g moly sulfide</td>
<td>Ball mill, steel bar, 24 hours</td>
<td>Dry, crumbled</td>
<td>Pressure dropped in 10 minutes at 600° F, fittings released easily, no thread distortion, sample partly decomposed, satisfactory release</td>
</tr>
<tr>
<td>62</td>
<td>40 g DC 710 fluid 3 g Cab-O-Sil 20 g Florigel SPL 0.04 g moly sulfide</td>
<td>Ball mill, 24 hours</td>
<td>Very slight drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>63</td>
<td>40 g DC 710 fluid 4 g Cab-O-Sil 25 g Florigel SPL 5.04 g moly sulfide</td>
<td>Ball mill, 72 hours, adding Cab-O-Sil gradually</td>
<td>Caked residue</td>
<td>Not attempted</td>
</tr>
<tr>
<td>64</td>
<td>40 g DC 710 fluid 4 g Cab-O-Sil 30 g Florigel SPL 0.04 g graphite</td>
<td>Ball mill, 72 hours, adding Cab-O-Sil gradually</td>
<td>Slight drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>65</td>
<td>40 g DC 710 fluid 4 g Cab-O-Sil 50 g Florigel SPL 5.04 g graphite</td>
<td>Ball mill, 72 hours, adding Cab-O-Sil gradually</td>
<td>Caked residue</td>
<td>Extensive pressure drop at room temperature, Not heated</td>
</tr>
<tr>
<td>66</td>
<td>40 g DC 710 fluid 4 g Cab-O-Sil 50 g Florigel SPL 0.02 g graphite</td>
<td>Ball mill, 48 hours, adding Cab-O-Sil gradually</td>
<td>Slight drying</td>
<td>Pressure dropped after 5 minutes, unsatisfactory release</td>
</tr>
<tr>
<td>67</td>
<td>40 g DC 710 fluid 2 g Cab-O-Sil 50 g Diluex 0.02 g graphite 0.02 g moly sulfide</td>
<td>Ball mill, 48 hours</td>
<td>Slight drying</td>
<td>Not attempted</td>
</tr>
<tr>
<td>68</td>
<td>40 g DC 710 fluid 2 g Cab-O-Sil 0.02 g graphite 0.02 g moly sulfide</td>
<td>Ball mill, 24 hours</td>
<td>Slight drying</td>
<td>Pressure dropped after 2 hours, residue powdery, fittings distorted, unsatisfactory release</td>
</tr>
<tr>
<td>69</td>
<td>40 g DC 710 fluid 2 g Cab-O-Sil 20 g Diluex 10 g Aluminum (washed) MD5100 0.02 g graphite 0.02 g moly sulfide</td>
<td>Ball mill, 24 hours</td>
<td>Not attempted</td>
<td>Pressure dropped after 6 hours, Caked residue, fittings seized and fractured on release, Unsatisfactory release</td>
</tr>
<tr>
<td>70</td>
<td>40 g DC 710 fluid 1.5 Cab-O-Sil 37.5 g No. 1 talc 0.25 g moly sulfide</td>
<td>Ball mill, 5 days, gradually adding talc as absorbed</td>
<td>Very slight drying</td>
<td>Pressure dropped after 6 hours, white caked residue, Satisfactory release</td>
</tr>
<tr>
<td>71f</td>
<td>2.0 g 5% dye solution 90 g DC 710 fluid 37.5 g No. 1 talc 0.25 g moly sulfide</td>
<td>Ball mill, 8 days, adding talc as needed to stiffen sealant</td>
<td>No visible change</td>
<td>Pressure dropped after 1-1/2 hours, Caked residue, satisfactory</td>
</tr>
</tbody>
</table>

*Sample 71 was exposed to 2000 psig oxygen at 500° F. No pressure drop was observed for 12 hours. Bomb assembly was dismantled after 24 hours at 550° F. Fittings released readily and showed no thread distortion. Sample was recovered as a tan, caked material. A larger lot of this composition was submitted to WADC as a second preliminary sample.*
To facilitate rapid screening of sealants, the thermocouple connection was omitted. This approximation technique appeared to be justifiable, as any vigorous combustion would be indicated even more readily by a sharp rise in the pressure of the small, closed system.

The unlike coefficients of thermal expansion of the aluminum, brass, and steel materials in the bomb assembly restrict its usefulness at 600° F. This difficulty was indicated by gross leakage from the assembly at elevated temperatures and disappearance of this leakage when the apparatus was again cooled to room temperature. This leakage was not encountered in a bomb assembly made entirely of aluminum, although the sealant used in the trial failed, and the fittings fractured on release. The aluminum fittings in all cases lost their anodized coatings on heating and tended to fracture under the specified assembly torques. Most of the high temperature leakages observed occurred about the sleeve AN819-2D, the nut AN818-2D, and the nipple AN816-2D. In view of the inability of any of the formulations tested to overcome this difficulty in the small bomb assembly, no pressure tests at 600° F were made in the larger release assembly.

As there was a delay in obtaining delivery of specification parts, early release testing was accomplished with commercial high pressure tube fittings. Neither brass nor aluminum fittings withstood the specified assembly torques without fracture. Release torques after heating in air for 24 hours at 600° F were generally about 60 per cent of the torque applied for assembly of the threaded fittings. Section 4.2.21.4 of the specification MIL-T-5548B(ASB) permits release torques 25 per cent in excess of those required for assembly. A formulation was deemed to have exhibited satisfactory antiseize properties on meeting two qualifications:

1. Release torque after heating less than that applied in assembling the fittings.

2. No visible galling or thread distortion on the disassembled fittings as examined under a 40-power binocular microscope. These trials are summarized in Table VIII.

D. Formulation of Thread Compound

A stock suspension of indanthrene dye (Helio Fast Blue RSV presscake, 22 per cent) was prepared by placing 30 grams of the presscake (22 per cent dye-stuff and water) in a jar mill with 120 grams Dow Corning 710 fluid and 1/2-inch metal balls. The mixture was milled continuously for 72 hours. The water was removed from this mixture by evaporation under increasing vacuum followed by gradually increased infrared heating in vacuo until no further moisture was evolved. A 120-gram portion of this suspension was transferred to the jar mill in which the final formulation was to be prepared.

To the dye suspension were added 2280 grams Dow Corning 710 silicone fluid and 90 grams Cab-O-Sil M-6. This mixture was milled for 24 hours with 2 pounds of 3/4-inch steel balls. Fifteen grams of molybdenum disulfide and 300 grams of No. 1 Lo Micron talc were added to the mixture, and milling was continued. Additional No. 1 Lo Micron talc was added in portions of not more than 500 grams at intervals of not less than 24 hours until a total of 2280 grams of talc was reached. Ball milling was continued for 72 hours after the final addition of talc.
TABLE VIII
RELEASE TESTING WITH NONSPECIFICATION, HIGH PRESSURE,
THREADED FITTINGS AFTER EXPOSURE TO AIR AT 600° F FOR 24 HOURS

<table>
<thead>
<tr>
<th>Formulation No.</th>
<th>Thread DIA (Inch)</th>
<th>Material M</th>
<th>F</th>
<th>Applied Torque (Ft Lb)</th>
<th>Release Torque (Ft Lb)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>17</td>
<td>1/2</td>
<td>Brass</td>
<td>Brass</td>
<td>50</td>
<td>---</td>
<td>Fitting broke during assembly</td>
</tr>
<tr>
<td>17</td>
<td>5/8</td>
<td>Brass</td>
<td>Brass</td>
<td>65</td>
<td>---</td>
<td>Fitting broke during assembly</td>
</tr>
<tr>
<td>17</td>
<td>3/4</td>
<td>Brass</td>
<td>Steel</td>
<td>83</td>
<td>19</td>
<td>Oily film on threads after heating, no discoloration or marring of threads</td>
</tr>
<tr>
<td></td>
<td>7/8</td>
<td>Steel</td>
<td>Steel</td>
<td>103</td>
<td>70</td>
<td>Oily film on threads after heating, no discoloration or marring of threads</td>
</tr>
<tr>
<td>17a</td>
<td>5/8</td>
<td>Brass</td>
<td>Brass</td>
<td>65</td>
<td>20</td>
<td>Oily film on thread after heating, no discoloration or marring of threads</td>
</tr>
<tr>
<td>17a</td>
<td>3/4</td>
<td>Brass</td>
<td>Brass</td>
<td>83</td>
<td>40</td>
<td>Oily film on threads after heating, no discoloration or marring of threads</td>
</tr>
<tr>
<td>18</td>
<td>1/2</td>
<td>Brass</td>
<td>Brass</td>
<td>50</td>
<td>---</td>
<td>Fitting broke during assembly</td>
</tr>
<tr>
<td>18</td>
<td>5/8</td>
<td>Brass</td>
<td>Brass</td>
<td>65</td>
<td>20</td>
<td>Oily film on threads after heating, no discoloration or marring of threads</td>
</tr>
<tr>
<td>22</td>
<td>3/4</td>
<td>Brass</td>
<td>Brass</td>
<td>83</td>
<td>10</td>
<td>Oily film on threads after heating, no discoloration or marring of threads</td>
</tr>
<tr>
<td>22</td>
<td>7/8</td>
<td>Steel</td>
<td>Steel</td>
<td>103</td>
<td>50</td>
<td>Oily film on threads after heating, no discoloration or marring of threads</td>
</tr>
</tbody>
</table>

Note: As release trials were without exception characterized by complete seizure and extensive thread damage or by a torque noticeably less than that required for assembly and no thread damage, antiseize properties of the remainder of the formulations were scored simply as satisfactory or unsatisfactory.

† A second portion of sample 17 was exposed in an open crucible at 600° F for 24 hours before application to the fittings. (Sample 17a.)

‡‡ Sample No. 18 was exposed to air at 600° F for 24 hours before application to threads.
The resulting compound was homogeneous in appearance and had the consistency of a heavy grease.
III. DISCUSSION OF EXPERIMENTAL RESULTS

The antiseize and sealing compounds formulated in this study were designed to meet as nearly as possible the specifications of MIL-T-4452B(ASG) as amended to require prolonged exposure to 2000 psig oxygen at 600°F rather than at 160°F and under surge conditions at 744°F rather than at 302°F. Development of a material capable of complying fully with these specifications proved impossible due to the shortcomings of base materials now available on the chemical market. Experimental effort was therefore directed toward exploration of a larger number of formulations, utilizing the best materials available rather than exhaustive testing of compounds known to be inadequate. It is believed that the compound ultimately submitted for approval represents the nearest approach to complete compliance possible with available materials, and that development of a completely satisfactory compound must await further advances in the chemistry of base fluids.

Several of the sealants showed satisfactory release characteristics after exposure to 2000 psig oxygen at 600°F for 24 hours, but in no case was the compound recovered from the bomb in an unchanged condition. Easy release of the fittings in the bomb assembly could be attributed to the solid lubricant remaining on the thread after decomposition of the base fluid. The sample inside the bomb was recovered as a readily crumbled caked material at the end of 24 hours. Bomb samples held under 2000 psig oxygen at 600°F for shorter periods showed deterioration of the liquid phase increasing with time of exposure. There is no reason to suspect rapid combustion of the compound during any interval of the heating period. Inability of the sealant to prevent pressure drops at 600°F may be attributed to the varying thermal coefficients of expansion of the several metals used in the bomb fittings.

Formulation No. 71 described in Table VI of this report represents the most nearly suitable compound developed in this study. The levels of thickener, filler, oxidation inhibitor, and solid lubricant dispersed in the base fluid are those experimentally determined to be the maximum permissible without sacrifice of fluid life or of the properties contributed by one of the other components. This compound, though not serviceable at 600°F, has shown promising antiseize and sealing properties at 550°F and should be useful at temperatures significantly in excess of the 302°F maximum temperature cited in the unamended Specification MIL-T-5542B(ASG).
IV. CONCLUSIONS

This study was initiated to develop a thermally stable antiseize and thread-sealing compound. Substantial progress was made in this direction, although a compound meeting all requirements was not prepared.

The following components appear to offer the greatest resistance to oxidative, thermal, and hydrolytic decomposition among the commercially available materials in their respective classes:

1. Base fluids. Dow Corning 710 fluid, a methyl phenyl silicone, showed greater resistance to decomposition than any other fluid tested.

2. Solid lubricants. Molybdenum disulfide appears to be a stable lubricant under the environment specified in this contract.

3. Thickeners. Cab-O-Sil M-6 is a satisfactory thickener.

4. Fillers. Finely divided talc imparts resistance to hardening as well as some lubricity.

5. Oxidation inhibitors. An indanthrene dyestuff retards decomposition of the base fluid and serves as part of the thickener necessary in the compound.

Decomposition of the base fluid is the principal obstacle to the preparation of a suitable compound.
V. BIBLIOGRAPHY


VI. APPENDIX

Commercial Products Used in the Study

<table>
<thead>
<tr>
<th>Product</th>
<th>Type</th>
<th>Supplier</th>
</tr>
</thead>
<tbody>
<tr>
<td>DC QF-6-7009</td>
<td>Silicone fluid</td>
<td>Dow Corning Corporation</td>
</tr>
<tr>
<td>GE 81644</td>
<td>Silicone fluid</td>
<td>General Electric Company</td>
</tr>
<tr>
<td>Versilube, F-50</td>
<td>Silicone fluid</td>
<td>General Electric Company</td>
</tr>
<tr>
<td>GE 81705</td>
<td>Silicone fluid</td>
<td>General Electric Company</td>
</tr>
<tr>
<td>DC QF-258</td>
<td>Silicone fluid</td>
<td>Dow Corning Corporation</td>
</tr>
<tr>
<td>DC 200</td>
<td>Silicone fluid</td>
<td>Dow Corning Corporation</td>
</tr>
<tr>
<td>DC 710</td>
<td>Silicone fluid</td>
<td>Dow Corning Corporation</td>
</tr>
<tr>
<td>DC 710R</td>
<td>Silicone fluid</td>
<td>Dow Corning Corporation</td>
</tr>
<tr>
<td>Microfyne Graphite</td>
<td>Graphite</td>
<td>John H. Dixon Crucible Co.</td>
</tr>
<tr>
<td>No. 1 Lo Micron</td>
<td>Talc</td>
<td>Whittaker, Clark, and Daniels, Inc.</td>
</tr>
<tr>
<td>Micro-Cel E</td>
<td>Synthetic calcium silicate</td>
<td>Johns-Manville Corporation</td>
</tr>
<tr>
<td>MD 3100</td>
<td>Aluminum powder</td>
<td>Metals Disintegrating Co.</td>
</tr>
<tr>
<td>Copper lining 132</td>
<td>Copper powder</td>
<td>Crescent Bronze Company</td>
</tr>
<tr>
<td>Cab-O-Sil M-6</td>
<td>Silicon dioxide</td>
<td>Godfrey L. Cabot, Inc.</td>
</tr>
<tr>
<td>Vulcan XC-72-R</td>
<td>Furnace black</td>
<td>Godfrey L. Cabot, Inc.</td>
</tr>
<tr>
<td>Alon C</td>
<td>Alumina</td>
<td>Godfrey L. Cabot, Inc.</td>
</tr>
</tbody>
</table>
Helio Fast Blue
RSV Presscake

Moly Sulfide (Pure)

Moly Sulfide (Technical fine)

Dicalite White Filler

Florigel SPL

Diluex

Indanthrene dyestuff

Molybdenum disulfide
Climax Molybdenum Company

Molybdenum disulfide
Climax Molybdenum Company

Diatomaceous silica
Great Lakes Carbon Company

Fuller's earth
The Floridin Company

Fuller's earth and alumina
The Floridin Company