

SYNTHETIC LUBRICANTS FOR AIRCRAFT

*HERBERT SCHWENKER
JOHN A. KING, CAPT, USAF
JAMES C. MOSTELLER*

MATERIALS LABORATORY

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FOREWORD

This report was prepared by the Organic Materials Branch, Materials Laboratory, Directorate of Research, Wright Air Development Center, and initiated under Task Number 73310 (formerly RDO Number 613-11, "Aircraft Lubricating Greases") with H. Schwenker, Lt J. A. King and J. C. Mosteller acting as project engineers.

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ABSTRACT

As military aircraft fly at higher altitudes and speeds, the requirements for lubricants to afford greater resistance to thermal effects, wider liquid or usable temperature ranges, and longer life has and will continue to be an interesting research and development program. Petroleum oils as a source for greases, aircraft hydraulic fluids, engine oils and special purpose lubricants are rapidly being replaced by synthetic materials and will soon be only of historical interest.

Development of a -65° to 450° F grease for use in anti-friction bearings, electronic devices and other types of aircraft equipment is in progress. Emphasis is being placed on improving the wear characteristics of this grease and extending the low temperature limits. In addition, research and development in greases is being devoted to improvement of grease availability and the investigation of oils and thickening agents.

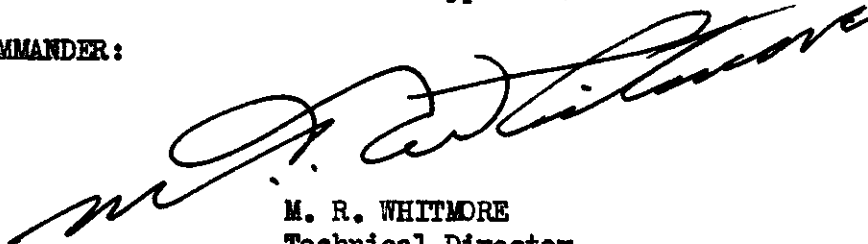
Synthetic lubricants for turbo-prop and turbo-jet applications have been formulated for use at temperature of -65° to greater than 400° F. High gear loadings encountered in some engine and accessory applications have required extensive research and development of suitable anti-wear additives.

The presently available hydraulic fluids offer a maximum usable temperature range of approximately 250° F. Speed, miniaturization of equipment, together with the necessity of operating hydraulic systems near heat producing bodies has increased this temperature range to greater than 400° F. Experimental fluids of the diester and the organosilicon classes have been developed and are being evaluated.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



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

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INTRODUCTION

The problem of providing an adequate lubricant for Air Force use is not new. As far back as World War I, as a necessity, castor oil was used as an engine lubricant. Later, the lubrication of exposed engine rocker arms presented a problem. This was solved by a "dab-on" grease technique using a latex containing grease. These two examples are but a few of the aircraft lubrication problems which have faced engineers.

During the piston engine era, the temperatures encountered in aircraft were dictated by the natural environment. The minimum temperature encountered on the earth's surface is about -65°F . At high altitudes, temperatures as low as -100°F have been measured. The maximum temperature on the earth's surface, due to solar heating, has been reported to be 160°F . In later model aircraft, temperatures of 200°F were obtained in areas of close proximity to accessory electronic equipment and mechanical components. Lubricants and hydraulic fluids for this equipment could generally be obtained from petroleum products. Such materials offered the required liquid range, stability, and were excellent lubricants.

The jet age in aircraft marks a demarcation from the maximum 200°F temperature limit. This has been brought about by increased speed, miniaturization of equipment, high heat producing power plants and arrangement of equipment which prevents dissipation of heat. These factors will increase the maximum operating temperatures for hydraulic fluids and lubricants to above 450°F . In addition to the high temperature requirement, there is the always present low temperature requirement imposed by the necessity to operate aircraft globally. Generally then, lubricants and hydraulic fluids will be required to operate from -65° to greater than 450°F .

A study of various applications in which higher operating temperatures will be imposed on hydraulic fluids and lubricants pointed out several necessary properties. Of primary importance is the stability of the fluid or lubricant to such temperatures, combined with good oxidation resistance and low volatility. In addition to the primary requirement, the fluid or lubricant should be non-corrosive to such metals as steel, aluminum, titanium, magnesium, copper alloy, silver, and in some cases, even lead and finally, it must lubricate steel on steel, and steel on non-ferrous alloy (sliding and rolling) surfaces.

In view of the known deficiencies of petroleum base materials, a comprehensive survey was made for materials which would possess the properties desirable for lubricants and hydraulic fluids for extended temperature applications. Studies included organo-phosphorous compounds, esters of mono and dicarboxylic acids, carbonates, borates, polyglycols, polysiloxane, silicates, and halogen-containing compounds. Briefly, some of the advantages and disadvantages of these materials are as follows:⁶

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INTRODUCTION (Continued)

Organo-Phosphorous Compounds — The organo-phosphorous compounds, particularly the phosphates and phosphonates, have reduced flammability and in general, good lubricating properties. They have viscosity temperature characteristics equal to high refined petroleum stocks and are superior in volatility properties. The viscosity properties of these materials rival those of the silicates but are inferior to the polysiloxanes. Compounds of this class require additives to thicken them to the desired viscosity level at high temperatures as well as to impart oxidation and hydrolytic stability. The phosphates, phosphonates, phosphonites, and phosphites studied as base materials showed that these materials have questionable thermal stability above 250°F for extended period of use in the presence of metals. The exception to this are the arylphosphates. The organo-phosphorous compounds are being studied as lubricity additives.

Dicarboxylic Acid Esters — The dicarboxylic acid esters exhibit lubricating properties slightly less or equal to light mineral oils but far exceed these materials in viscosity-temperature and viscosity-volatility relationships. Monoesters were found to be inferior to diesters in viscosity-volatility properties and flash points. The diesters possess high flash points combined with low pour points, good thermal stability and when properly inhibited, are non-corrosive to metals. The viscosities of the more conventional diesters at -65°F, range from less than 1000 cs for bis(3-methylbutyl) adipate to 7600 cs for bis(2-ethylhexyl)sebacate. These materials require the use of a viscosity index improver to meet the viscosity requirements for high temperature fluids and lubricants.

Polyglycols — These materials have questionable thermal stability at 400°F over prolonged usage in the presence of metals catalyst. The polyglycols exhibit excellent lubricating qualities, good viscosity-temperature and viscosity-volatility properties. Additives are required to thicken glycols for most fluid and lubricant applications.

Silicates — The name silicate applies here to esters of orthosilicic acid and represents a class of materials which show viscosity-volatility relationships superior to all other synthetics studied, except the silicones. The alkoxysilanes, represented by such compounds as tetrakis(2-ethylhexoxy) silane and tetrakis(2-ethylhexoxy)silane dimers and higher polymers, have excellent viscosity-temperature and viscosity-volatility properties. Compounds of this type are stable to shear and when properly inhibited are stable to oxidation and hydrolysis. The silicates offer an excellent source of base material for hydraulic fluids and specialty lubricants. Their use as engine lubricants has been limited by the tendency to form silicious material on pyrolysis. Studies have shown that either polysiloxanes or the acryloids are suitable for thickening the silicates to the desired viscosity requirements for high temperature hydraulic fluids.

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INTRODUCTION (Continued)

Polysiloxanes - The polysiloxanes or silicones have excellent viscosity-temperature and viscosity-volatility properties entirely suitable for high temperature hydraulic fluid and lubricant applications. Some polymers of this class are the most thermally stable base materials found to date. However, as liquid lubricants and hydraulic fluids, the silicones exhibit poor lubrication on steel on steel (sliding and rolling) surfaces. No additives are known which have adequate liquid range and impart lubricity to the silicones for this high temperature application. Blending of polysiloxanes with other superior lubricants may result in a satisfactory material. Research and development by industry will undoubtedly result in polysiloxanes with improved lubricity.

The other base materials investigated, namely the borates and carbonates, were found to be hydrolytically unstable while the halogenated compounds lacked either stability, good viscosity-temperature characteristics, or liquid range. A list of properties for representative members of these synthetics is given in Table I.

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SECTION I

HIGH TEMPERATURE HYDRAULIC FLUIDS

Hydraulic systems have been extensively used in modern aircraft because they offer a source of precise control with the maximum reliability as well as a source of power for operating various aircraft components. A major part of any hydraulic system is the hydraulic fluid. Experience has taught us that for optimum operation, a hydraulic fluid should have certain fundamental properties; the fluid should have an adequate liquid range, be non-corrosive to the metals used in the hydraulic system, be stable to oxidation, and hydrolytically stable. The fluid must also have adequate lubricity to lubricate high speed hydraulic pumps and last, but not least, the fluid should have a low coefficient of viscosity.

For the conventional aircraft of today, the red mineral oil, Specification MIL-O-5606, Fluid; Hydraulic, Petroleum Base, has performed admirably. This fluid is operable from -65°F to 200°F . By pressurizing the hydraulic reservoir, the upper limits could be raised to 275°F . With the advent of high speed aircraft, the upper temperature requirements for hydraulic fluids have risen considerably above 400°F . For a global Air Force, a -65°F is still a necessary part of this requirement. The high temperature hydraulic fluid is required to operate with the same stability, have the same low temperature properties as the current petroleum base hydraulic oil, but at 400°F , this fluid must have viscosity properties equal to the petroleum oil at 200°F . The requirements for a high temperature hydraulic fluid are given in Table II.

Previous studies have shown that petroleum oil does not have the necessary liquid range for this application. Synthetics, such as diesters and organosilicon compounds, offered the most promise as high temperature hydraulic fluids.

The California Research Corporation was contracted for the development of a fluid to meet these requirements. After examination of base materials, it was found that silicates offered the most promising approach. Diesters were precluded from this development since they could not meet the viscosity requirement. Silicones were not considered because of the poor lubricity of these materials for steel on steel applications.⁴

Extensive formulation studies were conducted utilizing stabilized alkyl silicates and polymeric thickening agents for the desired viscosity. The polysiloxanes and the polymethacrylates both functioned satisfactorily as viscosity index improvers. Table III gives the properties of the more desirable silicates and their thickened blends. In the evaluation of wear properties, the silicates were found to be equal to a light petroleum oil when measured on the Shell four-ball wear tester.

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The original requirements for a high temperature hydraulic fluid were achieved using a mixed 2-ethylhexyl-2-ethylbutyl silicate thickened with a polysiloxane. This hydraulic fluid had a flash of 350°F. The fluid was distributed to industry as MLO 5277. A deficiency was noted in MLO 5277 which can be described as a vapor phase oxidation which occurred at about 500°F. Two possible means were available to circumvent this undesirable condition. The first solution was through the use of additives, such as dialcyl selenide, which suppress vapor phase oxidation. This was successful; however, the inhibitor had a deleterious effect on the hydrolytic stability. Therefore, it was decided to increase the flash point of the fluid to above 400°F and by so doing decrease the volatility of the hydraulic fluid. This step entailed reformulating fluids and the synthesis of less volatile base stocks. Alkoxydisiloxanes were synthesized and found to have the desirable low volatility for an improved hydraulic fluid. A hydraulic fluid, MLO 8200, was formulated from a polysiloxane and hexa-2-ethylbutoxy-disiloxane which had a flash point above 415°F.

Oxidation-corrosion tests were run on this new material (MLO 8200) at 400°F for 72 hours. Phenyl-2-naphthylamine was found to be the most effective oxidation inhibitor, but this material deposited sludge. Although the sludge formation was prevented by the addition of calcium alkylphenate, the resulting blends were unstable to storage at room temperature. Other diamyl amines were also found to be effective and sludging was prevented by introducing alkyl groups into the aromatic ring. When inhibited with these materials, the polysiloxane-hexa-2-ethylbutoxy-disiloxane fluid showed good stability after a 72 hour oxidation-corrosion test at 400°F. The composition and properties of the silicone-disiloxane hydraulic fluid are given in Table IV.

Hydraulic Fluid Evaluation - The actual operating conditions for high temperature hydraulic systems, where the pressure is maintained at 3000 psi and temperatures may reach 400°F or above, are severe tests for hydraulic fluids. Not only is a fluid required to operate in this environment, but it must refrain from lacquering of precision parts or corroding the metals in the system. Besides this, the fluid is required to lubricate high pressure and high speed hydraulic pumps. To insure that a high temperature hydraulic fluid meets these requirements, extensive chemical and physical evaluations are being conducted by the U. S. Air Force.

The chemical evaluation consists of such tests as high temperature oxidation-corrosion tests at 400°F or hydrolytic stability tests to insure a stable fluid in storage as well as in use.

Physical evaluation of a fluid begins with laboratory wear tests, pump wear and shear stability. A fluid that is satisfactory under these conditions is then tested in cycling rigs and mock-up tests to evaluate the overall effect of the hydraulic fluid in a simulated hydraulic system.

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Lubricity - The Shell four-ball wear tester has been employed in this program to measure the lubricity of various base materials and finished fluids. Table V gives a comparison of the wear values of silicates and disiloxanes compared to other lubricants. In these tests, slightly higher wear values have been noted for the silicates than for diester-tricresyl phosphate compositions and petroleum lubricants. However, at elevated temperatures, the superior viscosity of the silicates will be a definite advantage when one compares these various fluids employing the hydrodynamic component of lubrication.

The silicate base hydraulic fluids have exhibited satisfactory lubricity when tested in both gear and piston pumps at 160°F. At temperatures of 250°F, 300°F, 350°F and 400°F, failure occurred before 500 hours of operation in standard aircraft hydraulic pumps and these failures were more likely due to mechanical failure than to a lack of lubricity. These standard aircraft hydraulic pumps are designed for -65°F to 160°F use. This fact was brought out by an examination of the pumps and by conducting additional pump tests with high temperature petroleum base engine oils. These tests exhibited the same pattern of failure.

Mock-up Tests - Silicate base, high temperature hydraulic fluid MLO 5277 was tested in a hydraulic system mock-up test which utilized the important parts of an aircraft hydraulic system at temperatures of -65°F to 400°F. Difficulty was noted in this test at -65°F when the fluid was cold soaked for 96 hours. This was traced to an inferior viscosity index improver and has been corrected. At room temperature and at 160°F, normal operation was experienced. At higher temperatures, a pattern of pump failure similar to the preliminary pump tests was noted. Hereto, no high temperature pumps were used or available. The 300°F run was continued for 89 hours. No lacquering or corrosion of the metal parts was noted and the lap fit values used in the assembly operated with ease.

From the experience gained in this development, a specification will be prepared for a high temperature hydraulic fluid for use in aircraft from -65°F to slightly above 400°F.

JET ENGINE LUBRICANTS

In 1949, a synthetic lubricant was developed by Pennsylvania State University under Air Force Contract for use in aircraft alternator drive assemblies. This development was necessary because the petroleum base hydraulic fluids previously used foamed considerably and were too volatile for this application. The use of a synthetic lubricant composed of bis(secamyl)sebacate, acryloid and 1% tricresyl phosphate operated remarkably well in the high speed gear and hydraulic arrangement present in the equipment. When petroleum base materials were used, the lubricant required changing every twenty-four hours while the synthetic lubricant gave excellent service for 500 hours. This lubricant, Specification MIL-L-6387 Lubricant; Synthetic, General Purpose, marked the entry of diester based synthetics into gear and accessory applications. The composition and properties of this material are listed in Table VI. This development showed that diesters are suitable base materials for use in gear and hydraulic oil formulations.

Since this early development, the requirements for a lubricant usable in turbo prop engines became a reality.² The lubricant for this application was required to have : a usable temperature range of from -65°F to 350°F with intermittent operation at higher temperatures; the ability to lubricate highly loaded gears; stability to oxidation; and no corrosive attack to the engine metals. A di-2-ethylhexyl sebacate-acryloid blend was formulated using a conventional sulfur-chlorine E.P. additive and phenothiazine as an anti-oxidant. The acryloid polymer was used as the VI improver and the viscosity was adjusted to 3400 cs at 40°F and 5.5 cs at 210°F. This material, known as PRL 3059, exhibited excellent load carrying capacity and adequate stability for use in this application. Engine tests showed an undesirable amount for dirtiness and engine corrosion; however, no corrosion was noted in the gear box assembly.

A second lubricant composition, known as PRL 3161, was developed to give adequate high temperature oxidation-corrosion and good wear characteristics without using active sulfur chlorine additives. This lubricant was tested in the T-38 turbo prop engine and lubricity was found to be adequate for normal running, but under "ram" loading, the gear lubrication was inadequate. PRL 3161 was formulated from di-2-ethylhexyl sebacate, acryloid, 5% tricresyl phosphate and phenothiazine as an oxidation inhibitor.³

In order to overcome the borderline lubricity of the PRL 3161 composition in highly loaded gears without jeopardizing high temperature oxidation and corrosion stability, a lubricant was developed using an alkyl acid phosphate-tricresyl phosphate-diester composition. In actual engine test and laboratory gear tests, there is evidence which indicates that the improved lubricity of this formulation, PRL 3313, over the PRL 3161 type of lubricant is due not only to the active acid phosphate but also to the increased viscosity level of this lubricant. The composition and properties of these lubricants are given in Table VII. A comparison of gear wear values obtained with the Pratt and Whitney Ryder Gear Tester is given in Table VIII.

The technological advancements in gas turbine engines resulted in aircraft engines that operated with bearing temperatures approaching 450°F. Thus a requirement resulted for a lubricant that would provide a usable liquid range

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from -65°F to 450°F . Taking advantage of the earlier development work in MIL-L-6387 type lubricants, the turbo prop lubricant studies and the excellent development studies of industry, a synthetic gas turbine lubricant was quickly developed and is known today under Specification MIL-L-7808. The specification requirements establish that diesters are most suited for base oil materials. Studies indicate that esters of sebacic, azelaic and adipic acid are suitable.

However, like hydraulic systems, the operating temperatures of future gas turbine engines are on an upward trend. Combined with this, it is the desire of the military services to maintain a minimum number of lubricants in stock. Hence, a synthetic gas turbine lubricant is desired that will operate over a liquid range of -65°F to temperatures considerably above 450°F and at the same time, possess lubricity characteristics sufficient to satisfy the extreme high loading encountered in turbo prop engine gear boxes.

Development work is in progress on lubricant base materials, additives to improve lubricity, prevent oxidation and retard foaming. These studies are in progress within industry and under contract to the United States Air Force and within USAF research laboratories. Considerable effort is being placed on lubricity improving additives stable at high temperatures and to the metals of construction proposed for future aircraft engines.

Briefly then, studies on lubrication additives are centered around organo-phosphorous compounds.

Lubrication Additives - The E.P. oils developed in the past proved to be too corrosive to be used in the power section and the non-E.P. lubricant was unsatisfactory in the gear assembly due to the excessive gear loads. Therefore, it became necessary to investigate additives which would impart high load carrying capacity to an oil and be stable at temperatures above 400°F . Lubricity had to be achieved in a base stock medium which had a usable liquid range of greater than 450°F . Materials previously used for E.P. additives, such as sulfur chlorine and chlorine compounds, were found to be too corrosive for the application.

In order to obtain anti-wear and E.P. additives for use in high load carrying engine oils and gear oils, organo-phosphorous compounds were investigated. The following discussion presents the information obtained to date on phosphorous compounds:

The organo-phosphorous compound studies include the aryl and alkyl phosphates, the phosphonate, substituted acid phosphates, phosphites, and alkyl acid phosphites. The aryl phosphites, most notably tricresyl phosphate, have been used as anti-wear additives in mineral oil. The extension of this material to synthetics has shown that varying degrees of wear improvement have been obtained with various diesters. That is, one diester or class of esters had wear improve-

ment have been obtained with various diesters. That is, one diester or class of esters had greater improvement from tricresyl phosphate than another class. The actual lubricant formulation using tricresyl phosphate will be discussed later; however, the concept of tricresyl phosphate susceptibility for esters has been examined.

Tricresyl Phosphate Susceptibility - Tricresyl phosphate does not give the same degree of wear improvement for a given additive concentration in the various esters studied. The esters are typical of those considered useful as base stock materials for synthetic lubricants. The effect in esters is more pronounced than in mineral oils and extends over a wider concentration range of tricresyl phosphate than was previously noted in hydrocarbons. The susceptibility of various esters to tricresyl phosphate, as a means of obtaining a reduction in wear, as measured in the Shell Four-Ball Wear Tester, is illustrated in Table IX. The evaluation of higher concentrations of tricresyl phosphate in several esters on the Shell Four-Ball E.P. Lubricant Tester is given in Table X. These data indicate that the relative effectiveness of tricresyl phosphate as an anti-wear additive extends into the mild E.P. range. Compounds with good tricresyl phosphate susceptibility exhibit incipient seizure loads. In general, these data indicate that tricresyl phosphate is more effective in esters of poly-basic acids and mono-hydroxy alcohols than in esters of polyhydroxy alcohols and mono-basic acids. (2) (1)

In the study of tricresyl phosphate susceptibility in esters, two effects were noted. In some cases more tricresyl phosphate is required to lower the wear but with additional tricresyl phosphate, a normal wear curve is observed. That is, the lowest wear value is noted at the lowest load and, for the most part, the greatest wear improvement over the ester without an additive, occurs at the lowest load. In many cases, concentrations of tricresyl phosphate have been evaluated which lower the wear value at the 1 kilogram load in the Shell Four-Ball Wear Tester without effecting the wear of the ester at the 10 and 40 kilogram loads. An increased tricresyl phosphate concentration in this case will normally cause wear improvement at a higher load. This case is illustrated in Table XI.

TABLE XI

WEAR CHARACTERISTICS OF BIS SEC-AMYL SEBACATE WITH TRICRESYL PHOSPHATE ANTI-WEAR ADDITIVE

Tricresyl Phosphate Concentrations Weight %	Average Wear Scar Diameter, mm, Steel on Steel Bearing Surfaces		
	1 Kg.	10 Kg.	40 Kg.
0.0	0.32	0.63	0.84
0.3	0.22	0.63	0.88
0.5	0.19	0.60	0.84
1.0	0.16	0.29	0.91

The second effect noted with tricresyl phosphate is illustrated in Table XII below:

TABLE XII

WEAR CHARACTERISTICS OF A SPECIFICATION MIL-L-6387 TYPE FLUID
WITH TRICRESYL PHOSPHATE ANTI-WEAR ADDITIVE

Tricresyl Phosphate Concentration Weight Percent	Average Wear Scar Diameter, mm, Steel on Steel Bearing Surfaces		
	1 Kg.	10 Kg.	40 Kg.
0.0	0.44	0.60	0.88
1.0	0.43	0.54	—
1.5	0.35	0.26	—
2.0	0.32	0.28	0.42

The wear remains high at the 1 kilogram load even though wear reduction over that of the ester without anti-wear additive is noted for the 10 kilogram load.

It has been observed that tricresyl phosphate becomes less effective in the presence of polar compounds which have an affinity for the metal surfaces. Petroleum sulfonates are a good example of this type of compound. Tricresyl phosphate becomes less effective as an anti-wear additive in the presence of compounds which are more chemically reactive and readily attack bearing surfaces under lubrication conditions.

The effects of the individual ortho, meta and para isomers of tricresyl phosphate and several other tricresyl phosphates have been studied in four-ball wear testers. All of the tricresyl phosphates exhibit essentially the same degree of effectiveness in 1.0 weight percent as an anti-wear additive and in 5.0 weight percent as an anti-wear and mild E.P. additive.

Alkyl Phosphates and Phosphonates and Acid Phosphates - The alkyl phosphate and phosphonates show wear improvement somewhat comparable to tricresyl phosphate. The data indicates that the lower molecular weight phosphates and the thio-phosphates offer an improvement over tricresyl phosphate in the Shell Four-Ball E. P. Tester. The most thermally stable phosphate, to date, for lubrication purposes is tricresyl phosphate. This material has little effect on the oxidation-corrosion characteristics of a finished lubricant. Alkyl phosphates and phosphonates have displayed poorer thermal stability than tricresyl phosphate.

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Preliminary studies have indicated that the effectiveness of an acid phosphate is directly proportional to the amount of replaceable hydrogen present in the acid phosphate. It was also found that small concentrations of phosphoric acid itself was quite effective as an anti-wear agent. Table XIII presents a comparison of tricresyl phosphate, an alkyl acid phosphate and phosphoric acid. It is evident that a concentration of 0.01 weight percent phosphoric acid is as effective as 1 to 5 weight percent tricresyl phosphate. The effectiveness of phosphoric acid is particularly marked in the cases of certain esters which show tricresyl phosphate susceptibility. A test was conducted with 1.0 weight percent alkyl acid phosphate to illustrate the chemical erosion obtained with larger amounts of acid phosphates. When 0.01 weight percent of phosphoric acid was used, the neutralization number increased about 0.1. For a given concentration, phosphoric acid exhibits more activity than long chain alkyl acid phosphates.

It is known that acid phosphates have a greater tendency to be absorbed on the metal surfaces than tricresyl phosphate or other alkyl or aryl phosphates. This suggests that the increased effectiveness of the acid phosphate is due to its presence on, or very near, the bearing area under lubrication conditions to take part in the chemical polishing reaction. Certain compounds known to be absorbed at the bearing surface and to react chemically with the bearing surface tend to nullify the effect of tricresyl phosphate but do not reduce the anti-wear properties of an acid phosphate. The comparison of the wear properties of tricresyl phosphates and active phosphates is given in Table XIV.

Organo-Phosphites - The study of wear improvement has been extended to the general class of compounds called phosphites. In this investigation, the acid phosphites, the tri-substituted phosphites and phosphorous acid have been investigated. In general, the acid phosphites show increased load carrying capacity with increased concentration. Compared to the acid phosphates, the acid phosphites show a larger response to increase in concentration. This response is shown in the point of incipient seizure and the weld point as measured on the Shell Four-Ball E.P. Tester. Table XV presents wear data obtained on phosphites. A representative group of acid phosphates is included for comparison purposes. Phosphorous acid has shown wear improvement comparable to phosphoric acid, but is less acidic in nature than phosphoric acid. Acid phosphites of lower molecular weight show wear improvement greater than the higher homologues.

In several cases, a slight increase in wear values is noted with increasing additive concentrations of phosphites. This may be considered a normal trend for active extreme pressure additives. The wear values for additives of this general class decreases with increasing concentration through a minimum wear value for the optimum concentration of the additive as an anti-wear agent. Further increases above this optimum anti-wear value tend to increase the wear values slightly but improve the E. P. properties. This increase in wear is interpreted as an increase in chemical erosion of the bearing surfaces. The data presented here indicates that organo-phosphorous compounds will improve

Continued

the lubricating properties of diester base materials as measured on the Shell Four-Ball Lubrication Tester and E.P. Tester. It should be noted that these data obtained on these laboratory testers do not measure the hydrodynamic property of the lubricant.

Additive Persistence - Thus far we have considered the lubrication improvements of phosphorous compounds as measured by the Shell Four-Ball Wear Testers. In all cases the temperature of 75°C was maintained. Other properties of a lubricant, such as oxidation, stability and non-corrosiveness are equally important. An attempt has been made to predict the persistence of various additives in the laboratory by subjecting the fluid to an oxidation-corrosion test and then re-evaluating the wear properties of the lubricant. Phosphates and phosphites have been subjected to a 72 hour oxidation test at 347°F in accordance with a Specification MIL-L-7308 procedure. One inch square of copper, aluminum, steel and magnesium are present. In the case of phosphorous acid and secondary acid phosphites, the fluids have been evaluated after a 168 hour oxidation test conducted in the presence of water and a copper strip at a temperature of 200°F. Each of the additives have been incorporated in a blend of acryloid polymer, phenothiazine, and di-2-ethylhexyl sebacate. The point of incipient seizure of the dialkyl phosphates reverts back to that shown by the base fluid but the point of permanent welding does not change. The ethyl diacid phosphate and the amyl diacid phosphate show good incipient seizure and weld points following the 347° oxidation test. Little difference was noted for the di-butyl acid phosphate. In all cases where the acid phosphites were subjected to the oxidation test containing water, the resultant wear values for the fluid were reduced to those shown by the base fluid without additives. This may have been caused by either extraction by water of the additive or hydrolysis followed by extraction during the course of the test. The data obtained in this study are included in Table XVI.

It has been observed that the acid phosphites exert a synergistic effect on a hindered phenolic oxidation inhibitor used in some ester compositions. This effect has also been noted with alkyl acid phosphites. Neither the acid phosphites or the hindered phenolic inhibited ester blends by themselves meet the requirements of the 347°F oxidation-corrosion test. Also both oxidation inhibitor and the lower molecular weight phosphites have a relatively high vapor pressure at these temperatures. However, in combination, this volatility deficiency is noted. Table XVII summarizes the estimated stable life of gear lubricants using the phosphites, phenolic inhibitor separately and in combination.

The investigation of organo-phosphorous compounds for lubricity improvement is continuing. The results to date have shown that these materials are effective additives. Further evaluation and development is necessary to insure a corrosion free and stable lubricant. The effect of acid phosphates and phosphites on the oxidation stability and corrosion resistance and storage stability warrants further study before these materials can be considered as suitable lubrication additives for diester base lubricants. In addition, considerable research is necessary to provide base oils that are stable to temperature above 450°F and to develop oxidation inhibitors for these temperatures. Additional research will undoubtedly be required to provide anti-foam additives for new base oil materials.

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SECTION III

GREASE DEVELOPMENT

High Temperature Greases

Temperature requirements for military aircraft greases are essentially the same as those required for liquid lubricants. Items of grease lubricated aircraft equipment currently in use are encountering ambient temperatures as high as 450°F. Others in the development stage may require lubrication at temperatures as high as 700°F. Since these items may be exposed to temperatures as low as -100°F, the low temperature properties of aircraft greases cannot be relaxed to any great extent.

To provide adequate lubrication over the -100° to 300°F temperature range, three Air Force Specification greases are now required. There are no Air Force Specification greases designed for temperatures in excess of 300°F. The ideal solution to the current lubrication problem would entail the development of a single grease capable of providing adequate lubrication over a -100° to 450°F range. It was realized that development of such a grease was not possible at the present time because oils possessing the necessary rheological properties at -100°F lack the necessary thermal stability at 450°F. Thermal stability of the base oil to be used in the development program was used as the factor in determining the temperature range of the grease to be developed. It was decided that an attempt should be made to develop a grease suitable for operation over a -65° to 450°F temperature range.

The Air Force contracted with the Standard Oil Company (Indiana) for the development of a grease which would operate satisfactorily over a temperature range of -65° to 450°F. Early development work by Standard Oil Company (Indiana) has been reported previously. ⁽⁸⁾ ⁽⁷⁾

All types of thickeners both organic and inorganic have been investigated with the best high temperature oil available, a silicone oil DC-550. These greases have been tested and evaluated for their performance at 450°F. The criterion for evaluation of high temperature greases is the ABEC-NLGI bearing tester operation at 450°F and 10,000 r.p.m. Results of these evaluations proved the organic type thickener to be the most successful in high temperature greases.¹² Organic materials that have been investigated as thickeners for high temperature greases are:

1. Aromatic hydrocarbons
2. Mono, di and polyaromatic amides
3. Mono and di substituted ureas

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The urea materials have proved to be the most promising as thickeners in high temperature oils. Experimental effort has therefore been mainly directed towards the development and improvement of greases made from the substituted ureas.

Aryl-substituted ureas have proved to be the best of the urea thickeners. Greases made from arylureas and silicone fluid have high temperature properties superior to any other types of grease in addition to most of the other desirable physical and chemical properties of aircraft lubricating greases. Arylurea-silicone oil greases have been developed which meet all but three of the requirements for a -65°F to 450°F grease. A list of these requirements are found in Table XVIII. Those requirements which have not been met are low temperature torque, apparent viscosity and gear wear. Arylurea-silicone oil greases do not meet these requirements because of the DC-550 silicone fluid used as the base oil for the greases. The DC-550 oil limits low temperature operation to -40°F for most applications, however, if a reasonable amount of torque is available, satisfactory operation may be obtained at -50°F . The poor wear properties of silicone oils is well known and this characteristic disadvantage is evident in the arylurea greases.

The performance of arylurea-silicone oil greases in the High Temperature Bearing Tester at 450°F is outstanding. Several greases gave consistent performance tests in excess of 600 hours at 450°F .

One of the most promising arylurea greases, MLG 9300, was produced in a small pilot batch of approximately 30 pounds. Samples of this grease were then distributed to members of the CRC Panel on High Temperature Greases for correlation tests on the High Temperature Bearing Tester. Several samples of MLG 9300 were distributed to manufacturers of aircraft and aircraft equipment for service evaluation tests. Only a few reports have been received on service evaluation tests. One manufacturer has reported obtaining good results in the motor bearings of two types of electromechanical actuators. Both of these actuators were subjected to ambient temperatures of 250°F . The lubrication problem was also complicated by a high input to the motors which are very small in size.

Tests were performed using MLG 9300 in comparison with MIL-C-3278 Grease. It was found that the use of MLG 9300 grease increased life of the bearings from 250 hours to over 400 hours. The manufacturer has therefore changed to the MLG 9300 type of grease as the accepted lubricant for these actuators.

The promising results obtained with the arylurea greases made it desirable to study the feasibility of commercial production of these greases. Methods of adaptation of urea thickened greases to large scale production are now being determined. One of the most important problems to be overcome is the availability of starting materials used in the preparation of the urea thickeners. Materials used in the preparation of the urea thickener used in MLG 9300 grease were of a highly purified nature and were not available in sufficient amounts. Cost of these purified materials was also very high and would make the cost of MLG 9300

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excessive for most applications. Subsequent efforts of the Standard Oil Company (Indiana) have resulted in the development of MLG 9301 grease. The silicone fluid and one of the amines used in preparing this grease are readily available, but the isocyanate and other amine are not. A chemical company promised to make available pilot quantities of the isocyanate. At the time the MLG 9301 grease was made the second amine was available but since then it has become increasingly difficult to obtain. Commercial production of the amine has been eliminated because of its identification as a material having possible carcinogenic properties. Efforts are underway to find suitable replacements for this material.

MLG 9301 was evaluated in the High Temperature Bearing Tester at 450°F and performance runs exceeding 500 hours were obtained. Samples of this grease have been distributed to various aircraft manufacturers for service evaluation tests. Factors such as the surface being lubricated, high speeds and heavy loads may cause the grease to fail in one application and perform satisfactorily in another at 450°F. It is hoped that these service tests will make it possible to determine for which purposes these greases are most suitable.

Efforts are continuing to improve the characteristics of these greases. Attempts are being made to improve the wear properties of arylurea-silicone greases by the use of additives. Test results have shown that the amount of wear varies with the test conditions and the metal combination being lubricated.

Samples of improved silicone oils were submitted to Materials Laboratory for evaluation. The low temperature properties and thermal stability of these fluids were then determined. One of the experimental fluids, XF-258, gave indications that it will give satisfactory performance, temperature-wise, over the -65°F to 450°F range. Further evaluation work conducted by the contractor substantiates this belief. This fluid is now being used in the development of arylurea-silicone greases.

Since future aircraft greases may be required to withstand 700°F temperatures, a portion of the work devoted to development of high temperature greases will be aimed at developing thickeners for 700°F use. Since there are no commercial or experimental oils capable of performing satisfactorily at 700°F, these new thickeners will have to be evaluated for their ability to function as grease thickeners in the synthetic type lubricants now available.

They will then be ready for use with 700°F fluids when they become available. A large amount of effort is being directed by the Materials Laboratory towards the development of such fluids.

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Grease Availability

Current Air Force Greases, such as MIL-G-3278, High and Low Temperature, Aircraft and Instrument Grease and MIL-G-7421, Extreme Low Temperature Grease are made from materials which are either imported or in limited supply. In time of War, these materials might be cut off or become insufficient for the needs of the Air Force. Effort is therefore being directed towards the development of aircraft greases from non-critical domestic materials, which are equal to or better than current Air Force Greases composed of critical or imported materials.

The investigation of both thickeners and base oils is included in this development work. Materials were chosen for their availability and on the basis of their physical and chemical properties.

Thickeners investigated were Silica Aerogel, Bentone 34 and Lithium 9/10 hydroxystearate. New base oils used in the development work were dipropylene glycol dipelargonate, tri-decyl azelate and various silicate esters. A list of all tests used in this work may be found in Table XXX.

Silica Gel Greases

The thickening agent used was a silica aerogel. (*) It has the following physical properties:

Agglomerate Particle Size	1-3 microns
Appearance	White powder
Oil Absorbtion	2.5 g/g of Silica Aerogel
Density	3.5 - 4 lb. per ft ³

Electronmicrographs were taken of the silica aerogel thickener. (Figure 1). The silica aerogel was used with the following oil types in the preparation of greases: silicone oil, petroleum oil, fluorocarbon oil, diester oil and silicate ester oils. Grease was prepared by mixing the silica aerogel together with the desired base oil in the cold, and then milling the resulting grease on a three roll-roller mill to produce the finished product. Figure 2, is an electronmicrograph of a silica-aerogel grease.

The gelling power of the silica aerogel was very good with all types of oils. It required approximately 8 to 12% of the thickener to produce greases of suitable consistency. Table XIX contains information on the thickening power of silica-aerogel in various types of oils. Mechanical stability of these greases is greatly dependent on the operating temperatures to which they are subjected while being worked. Excellent mechanical stability was exhibited by the silica aerogel when worked in the "Hain Microworker".⁹

* Santocel, Grade ARD, Monsanto Chemical Company

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Results of these tests are contained in Table XVIII. Mechanical stability of silica aerogel greases at temperatures above 250°F is very poor for greases using polar type oils. This fact was noted by Standard Oil(Indiana) in their development work on high temperature greases. Data concerning the mechanical stability of silica aerogel greases after heating are found in Table XX.

Resistance to oil separation and evaporation loss of the silica aerogel greases varied from fair to good depending upon the base oil used. The results of oil separation and evaporation tests are found in Table XXI.

Wear characteristics of the silica aerogel greases were determined by the Navy Gear Wear Tester. Wear properties of these greases were good with the exception of the silica-aerogel-silicone oil greases. The silica aerogel seemed to add to the already notoriously poor wear properties of the silicone oil. One of the surprising results noted during the wear tests was the excellent anti-wear properties exhibited by the silica-aerogel-silicate ester type greases. The wear characteristics of these greases were as good or better than those of best soap type greases. There was no appreciable increase in wear when the gear load was increased from 5# to 10#. Wear test results are found in Table XXII.

Water resistance of silica gel greases was determined by the Water Resistance Test and the Water Immersion Test. The degree of water resistance of the greases was dependent upon the base oil used with the silica aerogel. An improvement in water resistance was noted when the thickener and base oil were similar in structure. None of the silica aerogel greases could withstand immersion in boiling water for longer than 5 minutes however. Results of the Water Resistance and Water Immersion Tests are given in Table XXIII.

Bentone 34 Greases

Bentone 34 is a finely divided, light cream colored powder, the reaction product of an organic ammonium cation with montmorillonite. This material was used as a thickening agent with various synthetic oils in preparing greases! Greases were prepared on a beaker scale, no batch exceeding 4 lbs. in size. The Bentone 34 was mixed together with the desired base oil at room temperature. The resulting mixture was then heated at 200°F for a period of two hours to obtain maximum gelation. The material was then milled on a three roll-mill to produce the finished grease.

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If additives were added they were blended into the oil before mixing with the thickener. It was found that approximately 20% by weight of Bentone 34 was necessary to gel most oils to a point where a grease of good consistency is obtained. The greases exhibited good mechanical stability when tested in the Hain Microworker and the ASTM Grease Worker. The only greases tested which showed an appreciable effect due to mechanical working, were the greases having silicone base oils. The composition of the greases evaluated may be found in Table XXIV along with the results of their mechanical stability tests.

Water resistance of the greases was determined by the Water Resistance Test and the Water Immersion Test. Water resistance of the Bentone Greases is excellent. All of the greases tested performed well in these tests.

Oil separation of the greases was dependent on the type of oil used and the amount of thickener used. Resistance to bleeding was good.

The evaporation loss of Bentone Greases at 210°F was found to depend entirely on the base oil used. The Bentone 34 thickener did not accelerate or retard the original rate of evaporation of the base oils used. Low evaporation losses were recorded for those greases having base oils with low evaporation rates and vice versa.

Oxidation resistance of the greases was determined by Bomb Oxidation Method. An uninhibited Bentone 34 Silicone Oil grease showed good oxidation resistance. Those greases tested which contained oxidation inhibitors all exhibited good oxidation resistance.

Wear characteristics of the Bentone 34 greases were determined by the Navy Gear Wear Tester and the Shell Four-Ball Wear Machine. These tests show the greases to have good wear properties. Shell Four-Ball Wear Tests were run on both soap and non-soap type greases in order to obtain comparative wear data. Results of the Navy Gear Wear Tests and the Shell Four-Ball Wear Tests are given in Tables XXV and XXVI.

The rust preventive properties of the Bentone 34 greases were investigated under high relative humidity conditions in a humidity cabinet.¹⁰ The uninhibited Bentone 34 greases exhibited very poor rust preventive properties. Uninhibited soap-type thickeners when used in the same base-oils have shown a marked superiority over the Bentone 34 greases with respect to rust prevention. The best result obtained with rust-inhibited Bentone 34 greases was a No. 2 rating for the test bearings after 28 days in the humidity cabinet.

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Since the good rust resistance of certain soap-type greases has been attributed in part to their ability to absorb slight amounts of water, a 1% addition (by weight) of a water soluble soap was added to a Bentone 34 grease. The bearings protected by this grease showed a marked improvement, in the humidity cabinet test. The test bearings were given a #1 rating after 28 days in the humidity cabinet.

High Temperature Performance Tests were made on a Bentone 34 grease which was not inhibited against oxidation. Results of the High Temperature Performance Tests at 250°F are as follows:

Run #1	Run #2	Run #3	Run #4	Average
663 hrs.	320 hrs.	240 hrs.	344 hrs.	392 hrs.

Further tests of oxidation inhibited greases are under way.

The low temperature properties of Bentone 34 greases containing low temperature base oils are satisfactory at -65°F. The torque tester was used as a means of determining low temperature limits of the greases.

One Bentone 34 Diester Oil formulation also passed the Instrument Performance Test at -65°F.

Lithium 9 or 10 Hydroxystearate Greases - The 9 or 10 hydroxystearic acids are prepared from readily available domestic materials. Samples of these acids were obtained from Emery Industries and the Department of Agriculture. Lithium soaps were prepared from the following acids:

Material	Melting Point	Source
(a) 9/10 Hydroxystearic Acid	66.5 - 73.5°C	Emery Industries
(b) 9/10 Hydroxystearic Acid	70.0 - 75.0°C	N.R.R.L. Department of Agriculture
(c) 12 Hydroxystearic Acid	75.0 - 76.0°C	Emery Industries

The 12 hydroxystearate acid was included as a basis of comparison with the 9/10 hydroxystearic acids. Some of the MIL-G-3278 and MIL-G-7421 greases use Lithium 12 Hydroxystearate as a thickening agent. It was

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hoped that the lithium soap of 9/10 hydroxystearic acid could be used as a substitute for the lithium soap of 12 hydroxystearic acid which is obtained from castor oil, an imported material. The following melting points were obtained for the lithium soaps:

Material	Melting Point	Source
(a) Lithium 12 Hydroxystearate	207-208.5°C	Emery Industries
(b) Lithium 9/10 Hydroxystearate	201-204.5°C	Emery Industries
(c) Lithium 9/10 Hydroxystearate	207-210°C	N.R.R.L. Department of Agriculture

These soaps were used with the following oils to make greases:

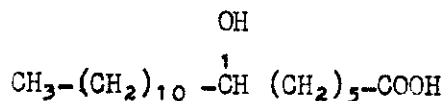
Oil	Source
(1) Di(2-ethylhexyl)sebacate	Rohm and Haas
(2) Tri-decyl azelate	Emery Industries
(3) Di-propylene glycol dipelargonate	Emery Industries

The gelling power of the lithium 9/10 hydroxystearates proved to be less than that of lithium 12 hydroxystearate. The lithium soaps of the 9/10 hydroxystearic produce a somewhat softer grease than the same percentage of lithium 12 hydroxystearate when used with the same base oil, but they appear to be good gelling agents.¹²The composition of some of the greases and the results of their mechanical stability tests are found in Table XXVII.

Mechanical stability of the greases was determined with the Hain Microworker. Lithium 12 hydroxystearate greases exhibited excellent mechanical stability whereas that of the lithium 9/10 hydroxystearate greases was only fair. Electronmicrographs were made of the two different types of soap greases to determine if there was a difference in their fiber structures. An examination of the electronmicrographs revealed that the Lithium 12 hydroxystearate grease (Figure 3) had a twisted symmetrical type of fiber structure (a characteristic previously noted of this soap) which has been credited with giving good mechanical stability. The lithium 9/10 hydroxystearate grease (Figure 4) had a rod like structure and the fibers were packed together in bundles. It was generally known that lithium stearate greases had straight rod like fibers.

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These greases have fair mechanical stability but they do not have the excellent mechanical stability of the lithium 12 hydroxystearate greases. This outstanding characteristic of the lithium 12 hydroxystearate greases has been attributed to their twisted symmetrical fiber structure. This fiber structure is thought to be the result of the -OH group attached to the stearic acid structure:



However, the electronmicrographs of the lithium 9/10 hydroxystearate greases make it appear that the location of the -OH group is important in order to obtain the twisted symmetrical type of fiber structure. The location of the -OH group on the 9 or 10 carbon atom of the stearic acid structure did not produce the fiber structure of the lithium 12 hydroxystearate greases.

On working both the lithium 12 hydroxystearate and the lithium 9/10 hydroxystearate fibers appear to be packed closer together. The following explanation is offered as a possible cause for the difference exhibited in their mechanical stability: Lithium 12 hydroxystearate fibers when packed together do not form a solid mass but retain a lattice type structure with many interstices which still presents a large surface area of the thickener for retention of the oil; thereby resulting in a very limited amount of breakdown of the grease due to working. (Figure 5) The fibers of lithium 9/10 hydroxystearate are packed together on working but in this case a solid mass is formed which results in a reduction of surface area of the thickener for retention of the oil component. This results in the greater amount of breakdown noted with this type of grease. (Figure 6)

Oil separation of the greases made with lithium 9/10 hydroxystearate was much higher than that of greases made with the same percentage of lithium 12 hydroxystearate and the same base oils. Increasing the amount of lithium 9/10 hydroxystearate to give greases of consistency comparable to that obtained with lithium 12 hydroxystearate would undoubtedly reduce the amount of oil separation in the lithium 9/10 hydroxystearate greases.

Lithium 9/10 hydroxystearate greases were tested for their water resistance by the following tests: Water Immersion Test and the Water Resistance Test. On the basis of these tests, the water resistance of the lithium 9/10 hydroxystearate greases is considerably less than that of lithium 12 hydroxystearate greases. A comparison of the water resistance of these greases is given in Table XXVIII.

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The lithium hydroxystearate greases were tested on the Navy Gear Wear Tester. Wear characteristics of both types of the hydroxystearate greases were good on the basis of this test. There was no appreciable difference in wear between the greases. Wear Test Data are shown in Table XXIX. Melting points of the hydroxystearate greases were determined. There was no appreciable difference in their melting points. Melting points ranged from 350°F to 370°F.

Summary on Grease Availability

Greases made using silica aerogel as a thickening agent lacked the necessary water resistance and mechanical stability required of aircraft synthetic base oil lubricating greases. Efforts by this Laboratory to improve the water resistance of these greases have been unsuccessful. Silica aerogel may be of use in formulating special purpose greases. The excellent wear characteristics of silica-aerogel-silicate ester greases lend credence to this belief.

Acceptance of Bentone 34 as a thickening agent appears to depend on: (1) Improvement of the High Temperature Bearing Performance of Bentone 34 greases. (2) Achievement of satisfactory rust preventive properties for Bentone Greases. If both of these requirements can be met then Bentone 34 can be added to the list of thickeners suitable for use in certain Air Force Greases.

Preliminary investigations of lithium 9/10 hydroxystearate greases reveal that they compare favorably with lithium 12 hydroxystearate greases except for mechanical stability and water resistance. The water resistance of the lithium 9/10 hydroxystearate greases while not as good as that of lithium 12 hydroxystearate greases is not a serious problem and is adequate for most aircraft greases. The greatest problem to be overcome is their mediocre mechanical stability. Attempts to improve this characteristic are being carried out. Correction of this factor would provide the Air Force with a soap type thickener which is obtained from domestic sources for use in aircraft lubricating greases such as MIL-G-3278 Grease.

TABLE I

PROPERTIES OF VARIOUS SYNTHETIC MATERIALS

	Requirements of High Temp. Fluid	Organo-Phosphorous Compounds	Diesters	Car-bonates	Borates	Poly-glycols	Silicones	Silicates
Viscosity at -65°F, cs	2,500	1,440	7,600	180(ex.)	17,350	40,000(ex.)	1,100(ex.)	2,500
Viscosity at -40°F, cs	—	264	1,420	59.1	1,781	3,500	600(ex.)	781
Viscosity at 0°F, cs	—	—	213.0	—	—	270	290.0	—
Viscosity at 100°F, cs	—	4.84	17.5	2.93	6.6	11.5	74.0	29.0
Viscosity at 210°F, cs	—	1.58	4.50	1.17	1.84	2.78	30.0	8.02
Viscosity at 400°F, cs	3.5	—	—	—	—	—	11.0(ex.)	—
Vapor Pressure at 400°F, mm Hg	3	—	—	—	—	—	—	3.0
Hydrolytic Stability	Satisfact.	Satisfact.	Satisfact.	Unsatis.	Unsatis.	Satis.	Satis.	Satis.
Thermal Stability at 400°F	Satisfact.	Unsatisfact.	Satisfact.	Unsatis.	Unsatis.	Satis.	Satis.	Satis.
Oxidation and Corrosion (inhibited)	Satisfact.	Unsatisfact.	Satisfact.	Unsatis.	Unsatis.	Satis.	Satis.	Satis.
Flash Point (min.), °F	400	300	460	275	345	275	575	350
Pour Point, °F	-65	-65	-65	-65	-70	-70	-65	-100
Wear - Steel on Steel	Satisfact.	Satisfact.	Satisfact.	Satis.	Satis.	Satis.	Unsatis.	Satis.

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TABLE II

REQUIREMENTS FOR HIGH TEMPERATURE HYDRAULIC FLUID

	Original Requirements	Present Requirements
Viscosity at -65°F	2500 C.P.	2500 cs
Viscosity at 400°F	3.5 C.P.	2.5 cs
Hydrolytic Stability Coke Bottle Test 200°F Copper Catalyst Specification MIL-L-6387 Method	Satisfactory	Satisfactory
Oxidation Stability Specification MIL-L-6387 Method With Temp. 400°F Metals Present, Al, Mg Steel, Cu	Satisfactory	Satisfactory ¹ (1) Mg not required
Volatility	At 400°F Equal to Spec. MIL-O-5606 Fluid at 200°F	Same
Flash Point Minimum	None	410°F
Fire Point Minimum	None	425°F
Pour Point	Less -75°F(Less than)	-75°F(Less than)
Low Temperature Stability at 72 hours at -65°F	Satisfactory	Satisfactory for 168 hrs at -65°F
Wear	Satisfactory Pump Lubrication at 400°F	Satisfactory Pump Lubrication at 400°F
Foaming	Non-foaming in Modern Equipment	Non-foaming in Modern Equipment

TABLE III

PROPERTIES OF ORGANO-SILICON COMPOUNDS AND BLENDS
OF VISCOSITY INDEX IMPROVERS

Base Fluid	Additive	Viscosity at			
		-65F,cs	-40F,cs	100F,cs	210F,cs
C ₆ Silicate	204	67	3.94	1.55
C ₆ Silicate	Polysiloxane A 6%	1466	. .	25.4	9.38
C ₆ Silicate	Polysiloxane A 8%	2351	. .	42.2	14.7
C ₆ Silicate	Polysiloxane B 14%	1682	. .	32.7	11.8
C ₈ Silicate	1320	316	6.95	2.26
C ₈ Silicate	Polysiloxane A 6%	6315	. .	34.5	10.9
Mixed Silicate	472	136	5.02	1.82
Mixed Silicate	Polymethacrylate	6845	. .	27.9	9.52
Mixed Silicate	Polysiloxane C 10%	2500	781	27.0	9.45

TABLE IV

COMPOSITION AND PROPERTIES OF DISILOXANE HYDRAULIC FLUIDS

Composition - wt. %	(a)		(b)		MLO 8205	MLO 8200	MLO 8202	MLO 8203	MLO 8204	MLO 8207
	Hexa(2-ethylbutoxy)disiloxane	90.73	93.18	89.98	97.98	—	—	—	—	—
Hexa(2-ethylhexoxy)disiloxane	—	—	—	—	—	—	—	—	85.98	—
Tetra(2-ethylhexyl)silicate	—	—	—	—	—	—	—	—	—	97.98
2-Ethylbutoxy polysiloxane	7.25	4.8	8.0	—	—	—	—	—	12.0	2.0
XF 371 100,000 cs.	2.0	2.0	2.0	2.02	—	—	—	—	2.0	—
Dioctylphenyl-o-naphthylamine	—	—	—	—	—	—	—	—	—	—
p,p'-Dioctyldiphenylamine	—	—	—	—	—	—	—	—	—	—
Quinizarin	0.02	0.02	0.02	0.02	—	—	—	—	0.02	0.02
<u>Properties</u>										
Flash Point, of	410	415	475	475	—	—	—	—	400	420
Viscosity at -65°F, cs	2453	2557	9737	3947	—	—	—	—	—	2868
Viscosity at -65°F, cp	2408	2543	9464	3852	—	—	—	—	—	—
Viscosity at -40°F, cs	—	797	2475	922	—	—	—	—	2128	—
Viscosity at -40°F, cp	—	783	2379	881	—	—	—	—	2018	—
Viscosity at 100°F, cs	37.9	33.77	54.8	14.94	—	—	—	—	54.77	21.64
Viscosity at 400°F, cs	4.64	3.82	5.50	1.47	—	—	—	—	5.95	6.64
Viscosity at 400°F, cp	3.60	3.02	4.31	1.15	—	—	—	—	4.52	—
Pour Point, of	Below -100	Below -100	Below -100	Below -100	Below -100	Below -100	Below -100	Below -100	Below -100	Below -100
Low Temperature Stability, 120 hrs. at -65°F	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Vapor Pressure at 400°F, mm. Hg	1.2	1.2	0.09	0.09	1.2	1.2	0.09	0.09	1.3	—
Evaporation, 6 1/2 hrs. at 400°F	21.0	22.3	5.52	6.51	—	—	—	—	—	—

(a) Distilled
(b) Stripped

TABLE V
FOUR-BALL WEAR VALUES OF SILICATES
 800 rpm - 30 -Minute Duration - Steel-on-Steel
 10 Kilograms

	Wear Scar Diameter, mm	
	200°F	400°F
Hexa(2-ethylhexoxy)disiloxane + 1% PAN	—	0.62
Tetra(undecyl) silicate + 1% PAN	—	0.63
Poly(2-ethylhexyl)polysiloxane	—	0.36
Ditridecyl azelate	0.50	0.64
Mixed(2-ethylbutyl,2-ethylhexyl)silicate	0.44	0.58
Tetra(2-ethylbutyl)silicate	0.55	
Tetra(2-ethylhexyl)silicate	0.33	
25:75% mixture of hexyl and butyl silicates	0.47	

TABLE VI

COMPOSITION AND PROPERTIES OF SPECIFICATION
MIL-L-6387 TYPE LUBRICANTS

	PRL 3039	PRL 3327
Composition wt. %		
Di-Sec-Amyl Sebacate	88.0	—
Di-3-Methylbutyl Adipate	—	44.0
Di-2-Ethylhexyl Sebacate	5.3	49.3
Tricresyl Phosphate	1.0	1.0
Pheonthiazine	0.5	0.5
Acryloid HF-25	5.2	5.2
Silicone Anti-Foam	0.001	—
Properties		
Centistoke Viscosity at 210°F	4.5	4.95
100°F	15.4	16.9
-40°F	1,150	1220
-65°F	6,300	6070
ASTM Slope (130 to -40°F)	0.634	0.60
Viscosity Index	212	204
ASTM Pour Point, °F	-75	-75
C.O.C. Flash Point, °F	415	360
C.O.C. Fire Point, °F	445	390
Percent Viscosity Decrease After 100 hrs. in Constant Speed Drive (Permanent Sheer)	12	—
Foaming Tendency	No Foam	No Foam
Oxidation-Corrosion Stable Life at 347°F in hours	220	—

TABLE VII
COMPOSITION AND PROPERTIES OF GEAR LUBRICANTS

PRL Designation	PRL 3161	PRL 3313	PRL 3059
Composition, Wt. %			
Di-2-Ethylhexyl Sebacate	90.6	85.0	87.1
Acryloid HF-25	3.9	9.0	3.9
Phenothiazine	0.5	0.5	0.5
Tricresyl Phosphate	5.0	5.0	1.0
Alkyl Acid Phosphate	—	0.5	—
Silicone Anti-foam Agent	0.001	0.001	—
Sulfur Chlorine Additive	—	—	7.5
Properties			
Centistoke Viscosity at 210°F	5.3	10.2	5.9
100°F	20.8	43.1	22.9
0°F	374	—	—
-40°F	2,700	—	3400
-65°F	16,000	41,400	—
ASTM Viscosity Index	181	164	174 (1)
ASTM Slope (210° to -65°F)	0.63	0.56	0.63
Neut. No. (Mg. KOH/gm. Oil)	0.2	2.0	0.1
C.O.C. Flash Point, °F	450	435	450
C.O.C. Fire Point, °F	500	500	475

TABLE VIII
WEAR STUDIES IN PRATT AND WHITNEY RYDIER GEAR TESTS

Test Fluid	Gear Scuffing Load	lbs/inch
PRL 3161	1850 2000	
PRL 3313	3900 4450	

TRICRESYL PHOSPHATE SUSCEPTIBILITY AS AN ANTI-WEAR AGENT
IN VARIOUS DIBASIC ACID ESTERS

Test Fluid Compositions in Weight Per Cent	Average Wear Scar Diameter, MM. Steel-On-Steel Bearing Surfaces		
	1 Kg	10 Kg	40 Kg
Di-2-Ethylhexyl Sebacate (Plasticizer Grade)	0.26	0.55	0.69
+ 1.0 Tricresyl Phosphate	0.14	0.26	0.37
Di-2-Ethylhexyl Adipate	0.32	0.64	0.84
+ 1.0 Tricresyl Phosphate	0.16	0.28	0.46
Di-2-Ethylhexyl Azelate	0.39	0.63	0.79
+ 1.0 Tricresyl Phosphate	0.17	0.27	0.86
Di-Isocetyl Adipate (Plexol 244)	0.23	0.65	0.80
+ 1.0 Tricresyl Phosphate	0.19	0.26	0.84
Di-3-Methylbutyl Adipate (Lot I)	0.37	0.78	1.04
+ 1.0 Tricresyl Phosphate	0.21	0.77	0.97
+ 2.0 Tricresyl Phosphate	0.16	0.56	1.04
+ 3.0 Tricresyl Phosphate	0.16	0.42	1.04
+ 0.2 Lauryl Acid Maleate + 1.0 Tricresyl Phosphate	0.18	0.58	0.95
+ 0.2 Oleic Acid + 1.0 Tricresyl Phosphate	0.19	0.63	0.95
Di-3-Methylbutyl Adipate (Lot II)	0.39	0.71	0.91
+ 1.0 Tricresyl Phosphate	0.38	0.71	0.97
+ 3.0 Tricresyl Phosphate	0.40	0.64	0.97
+ 5.0 Tricresyl Phosphate	0.23	0.25	0.78
Di-2-Ethylhexyl Phthalate	0.30	0.51	0.72
+ 1.0 Tricresyl Phosphate	0.22	0.45	0.78
+ 2.0 Tricresyl Phosphate	0.19	0.29	0.44
+ 0.2 Lauryl Acid Maleate	0.16	0.25	0.65
+ 0.2 Lauryl Acid Maleate + 1.0 Tricresyl Phosphate	0.16	0.25	0.53
+ 0.2 Oleic Acid + 1.0 Tricresyl Phosphate	0.28	0.42	0.58
Triethylene Glycol Di-2-Ethylhexanoate	0.43	0.63	0.82
+ 1.0 Tricresyl Phosphate	0.40	0.62	0.86
+ 2.0 Tricresyl Phosphate	0.33	0.50	0.84
+ 5.0 Tricresyl Phosphate	0.23	0.26	0.53
Polyethylene Glycol Di-2-Ethylhexanoate	0.41	0.66	0.97
+ 1.0 Tricresyl Phosphate	0.37	0.71	0.91
+ 2.0 Tricresyl Phosphate	0.37	0.61	0.91
+ 5.0 Tricresyl Phosphate	0.36	0.56	0.66

TABLE X

WEAR CHARACTERISTICS OF SOME EXPERIMENTAL GEAR LUBES CONTAINING VARIOUS SYNTHETIC BASE STOCKS

TEST CONDITIONS INCLUDE: FOUR-BALL WEAR TESTER: TEST TIME = 1 HOUR; TEST TEMPERATURE = 75°C; TEST SPEED = 850 R.P.M.
 EXTREME-PRESSURE LUBRICANT TESTER: TEST TIME = 1 MINUTE; TEST TEMPERATURE = ROOM TEMPERATURE (70-80°F);
 TEST SPEED = 1750 R.P.M.

STEEL BALLS = SKF INDUSTRIES GRADE #1 (0.5 INCH DIAMETER) STEEL BALLBEARINGS

	WEAR TESTER		EXTREME PRESSURE LUBRICANT TESTER		WELDED		
	AVERAGE WEAR SCAR DIAMETER, MM. STEEL ON STEEL BEARING SURFACES	STEEL ON STEEL BEARING SURFACES	APPROX. LOAD AVERAGE WEAR SCAR DIAMETER, MM. FOR INCIPIENT STEEL ON STEEL BEARING SURFACES	SEIZURE, KG.			
	1 KG.	10 KG.	40 KG.	60 KG.	70 KG. (1)	80 KG. (1)	120 KG.
3.9 Acryloid HF-25 + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in Di-2-Ethylhexyl	0.13	0.22	0.38	0.35	—	2.21	0.40
4.3 Acryloid HF-25 + 4.3 Di-2-Ethylhexyl Sebacate + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in Polyethylene Glycol Di-2-Ethylhexandate	0.28	0.39	0.71	0.43	2.16	2.21	0.97
4.8 Acryloid HF-25 + 4.8 Di-2-Ethylhexyl Sebacate + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in Flexol 244	0.16	0.22	0.36	0.36	0.39	2.12	0.78
10.0 Acryloid HF-25 + 10.0 Di-2-Ethylhexyl Sebacate + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in Di-3-Methylbutyl Adipate	0.16	0.22	0.40	0.35	0.42	2.27	2.20
4.2 Acryloid HF-25 + 4.2 Di-2-Ethylhexyl Sebacate + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in 60:40 (By weight) Herculox 600:Di-3-Methylbutyl Adipate	0.16	0.26	0.35	0.38	2.03	2.12	0.97
7.5 Acryloid HF-25 + 7.5 Di-2-Ethylhexyl Sebacate + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in Di-2-Ethylhexyl Adipate	0.16	0.30	0.36	0.35	2.53	2.38	2.20
4.4 Acryloid HF-25 + 4.4 Di-2-Ethylhexyl Sebacate + 5.0 Tricresyl Phosphate + 0.5 Phenothiazine in Di-2-Ethylhexyl Azelate	0.16	0.30	0.36	0.36	0.39	0.40	2.35

TABLE XIII

WEAR CHARACTERISTICS OF VARIOUS PHOSPHATES IN DI-2-ETHYLHEXYL SEBACATE

TEST CONDITIONS INCLUDE: FOUR BALL WEAR TESTER; TEST TIME = 1 HR, TEST TEMP. = 75°C, TEST SPEED = 850 R.P.M.
EXTREME PRESSURE LUBRICANT TESTER; TEST TIME = 1 MINUTE, TEST TEMP. = ROOM TEMP. (70-80°F), TEST SPEED = 1750 R.P.M.

Lubricant Fluid Composition	Average Wear Scar Diameter, mm.		Average Joint of Incipient Seizure		Average Wear Scar Diameter, mm.		Steel on Steel Bearing Surfaces		Welded	Welded	Welded	Welded
	1 Kg. 10 Kg.	40 Kg.	50	Seizure	40 Kg.	60 Kg.	80 Kg.	100 Kg.				
A 3-9 Acryloid HF-25 + 0.5 Phenobiazine in Di-2-Ethylhexyl Sebacate	0.34	0.63	0.78	50	0.12	2.12	2.66	3.12	WELDED			
B 5.0 Tricresyl Phosphate in A	0.13	0.22	0.38	80	0.32	0.36	0.42	2.92	WELDED			
C 5.0 Di-Nonyl-Iso-Octans Phosphonate in A	0.25	0.35	0.43	50	0.43	2.29	2.44	3.21	WELDED			
D Di-Hexyl Hexane Phosphonate in A	0.19	0.31	0.65	50	0.43	2.10	2.12	3.12	WELDED			
E 5.0 1,5 Pentane Diol Bis(Dibutyl Phosphate) in A	0.16	0.20	0.43	80	0.33	0.39	—	2.53	2.99	WELDED		
F 5.0 Triphenylthio Phosphate in A	0.16	0.24	0.40	80	0.33	0.35	0.42	—	1.82		2.34	WELDED
G 5.0% Tributyl Phosphate in A	0.17	0.22	0.52	80	0.35	0.36	0.41	—	2.94		WELDED	
H 5.0% Tri-2-Ethylhexyl Phosphate in A	0.19	0.26	0.37	60	0.39	0.39	2.79	—	WELDED		WELDED	
I 1.0% Alkyl Acid Phosphate in A	0.19	0.28	0.39	100	0.33	0.36	0.42	—	2.57		WELDED	
J 5.0% Alkyl Acid Phosphate in A	0.23	0.43	0.52	100	0.32	0.37	0.40	—	2.25		2.55	WELDED
K Di-2-Ethylhexyl Sebacate	0.34	0.56	0.69									
L K plus 1.0 Tricresyl Phosphate	0.15	0.26	0.40									
M K plus 0.1 Phosphoric Acid	0.35	0.58	0.67									
N K plus 0.001 Phosphoric Acid	0.16	0.27	0.65									
O K plus 0.01 Phosphoric Acid	0.16	0.22	0.40									

COMPARISON OF THE WEAR PROPERTIES OF TRICRESYL PHOSPHATES AND ACTIVE ORGANOPHOSPHOROUS COMPOUNDS

Test Fluid (Compositions in Wt. %)	Average Wear Scar Diameter, mm. Steel-on-Steel Bearing Surfaces		
	1 Kg.	10 Kg.	40 Kg.
Spec. MIL-L-7808 Type Gear Lubricant	0.16	0.22	0.37
9.0 Acryloid HF-25 + 0.5 Phenothiazine in Di-2-Ethylhexyl Sebacate	0.37	0.64	0.91
0.5 Ortholeum 162 in PRL 3379	0.27	0.37	0.41
0.5 Mono Dilauryl Acid Orthophosphate in PRL 3379	0.25	0.38	0.35
0.5 Mono-lauryl Acid Orthophosphate in PRL 3379 ¹	0.16	0.26	0.37
0.5 Ethyl Lauryl Acid Orthophosphate in PRL 3379	0.26	0.50	0.56
0.5 Mono-Di-2-Ethylhexyl Acid Orthophosphate in PRL 3379	0.25	0.32	0.39
0.5 Mono-2-Ethylhexyl Acid Orthophosphate in PRL 3379	0.16	0.45	0.61
0.5 Iso-Amyl-2-Ethylhexyl Acid Orthophosphate in PRL 3379	0.27	0.33	0.40
0.5 Diphenyl Phosphoric Acid in PRL 3379	0.18	0.26	0.51
0.5 Octyl Phenyl Phosphoric Acid in PRL 3379	0.19	0.28	0.41
0.5 Di-2-Ethylhexyl Acid Pyrophosphate in PRL 3379	0.30	0.37	0.54
0.5 Dimethyl Hydrogen Phosphite in PRL 3379	0.29	0.33	0.47
0.5 Diethyl Hydrogen Phosphite in PRL 3379	0.28	0.33	0.47
0.5 Dibutyl Hydrogen Phosphite in PRL 3379	0.19	0.28	0.48
0.5 Di-2-Ethylhexyl Hydrogen Phosphite in PRL 3379	0.18	0.24	0.43
0.5 Tri-2-Ethylhexyl Phosphite in PRL 3379	0.40	0.62	0.91

¹Green insoluble material present in fluid.

TABLE XV
WEAR CHARACTERISTICS OF SOME EXPERIMENTAL ORGANOPHOSPHOROUS DIESTER BLENDS

TEST CONDITIONS INCLUDE: FOUR BALL WEAR TESTER; TEST TIME = 1 HR.; TEST TEMP. = 75°C. TEST SPEED = 850 R.P.M.
EXTREME PRESSURE LUBRICANT TESTER; TEST TIME = 1 MINUTE, TEST TEMP. = ROOM TEMP. (70-80°F), TEST SPEED = 1750 R.P.M.
STEEL BALLS = SKF INDUSTRIES GRADE #1 (0.5 INCH DIAMETER) STEEL BALL BEARINGS

Lubricant	Test Fluids, Composition in Weight %	Wear Tester			Approx. Load for Incipient Seizure, Kg.	Average Wear Scar Diameter, mm., Steel on Steel Bearing Surfaces							
		Average Wear Scar Diameter, mm., Steel on Steel Bearing Surfaces				40 Kg.	60 Kg.	80 Kg.	100 Kg.	120 Kg.	140 Kg.	160 Kg.	180 Kg.
		1 Kg.	10 Kg.	40 Kg.									
A	9.0 Acryloid HP-25 + 0.5 Phenothiazine in Di-2-Ethylhexyl Sebacate	0.37	0.64	0.91	60	0.12	0.65	3.07	3.98	WELDED	0.51	2.60	2.68
--	0.1 Phosphoric Acid In A	--	--	--	150	--	--	--	0.16	0.49	0.51	2.60	2.68
--	0.5 Phosphorous Acid In A	0.21	0.35	0.18	(1)	0.34	0.39	0.14	0.54	0.91	1.04	1.04	1.06
--	0.5 Dibutyl Acid Phosphate In A (2)	0.19	0.28	0.18	90	0.34	0.39	0.13	2.40	WELDED	WELDED	WELDED	WELDED
--	0.5 Dibutyl Acid Phosphate In A (3)	0.14	0.25	0.10	90	0.33	0.37	0.12	2.56	2.62	2.84	WELDED	WELDED
--	0.5 Diisopropyl Acid Phosphate In A	0.16	0.26	0.17	110	0.33	0.39	0.16	0.13	2.62	2.84	WELDED	WELDED
--	0.5 Di-2-Ethylhexyl Acid Phosphate In A	0.18	0.24	0.13	90	0.37	0.42	0.12	2.90	WELDED	2.27	3.05	WELDED
--	0.5 Bis(2-Octyl) Acid Phosphate In A	0.14	0.22	0.50	130	0.30	0.36	0.42	0.14	0.47	2.27	3.05	WELDED
--	0.5 Diphenyl Acid Phosphate In A	0.27	0.23	0.39	70	0.32	0.39	2.40	2.30	2.86	2.90	WELDED	WELDED

(1) Wear value at 340 Kilograms load is 1.11 Millimeters. No clear cut Point of Seizure is shown over this load range.

(2) Sample obtained from the Victor Chemical Company

(3) Sample obtained from the Monsanto Chemical Company

TABLE XVI

WEAR CHARACTERISTICS OF SOME EXPERIMENTAL GEAR BOX LUBRICANTS BEFORE AND AFTER OXIDATION

All values determined in the Shell Four Ball Extreme Pressure Lubricant Tester

TEST CONDITIONS INCLUDE: TEST TIME = 1 MINUTE, TEST TEMP. = ROOM TEMP. (70-80°F), TEST SPEED = 1750 R.P.M., STEEL BALLS = SKF INDUSTRIES GRADE #1 (0.5 INCH DIAMETER) STEEL BALL BEARINGS, PRL BATCH #11

PRL NO.	TEST FLUIDS COMPOSITIONS IN WEIGHT %	APPROX. LOAD INCIPENT SEIZURE	APPROX. WEAR SCAR DIAMETER, MM. STEEL ON STEEL BEARING SURFACES									
			60 Kg.	80 Kg.	100 Kg.	120 Kg.	140 Kg.	160 Kg.	180 Kg.	200 Kg.	220 Kg.	
3379	9.0 Acryloid HF-25 + 0.5 Phenothiazine in Di-2-Ethylhexyl Sebacate	60	0.65	3.07	2.98	WELDED	0.51	2.60	2.68	WELDED	2.70	WELDED
3405	9.0 Acryloid HF-25 + 1.0 Paranox 441 in Di-2-Ethylhexyl Sebacate	60	0.57	3.77	3.08	WELDED	0.54	WELDED	2.68	WELDED	2.70	WELDED
--	0.1 Phosphoric Acid in PRL 3379	150	--	--	0.46	0.49	0.53	2.79	WELDED	WELDED	2.70	WELDED
--	0.5 Diethyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	150	--	--	0.47	0.50	0.55	0.58	0.67	0.67	2.53	WELDED
--	0.5 Diethyl Acid Phosphate in PRL 3405 After Oxidation Test at 347°F for 72 hrs.	60	1.82	--	2.73	--	2.34	2.30	2.53	2.53	2.53	WELDED
--	0.5 Diethyl Acid Phosphate in PRL 3405 After Oxidation Test at 347°F for 72 hrs.	150	--	2.36	0.48	2.23	--	2.38	--	--	--	WELDED
--	0.5 Monoethyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	60	1.84	--	2.21	0.52	0.52	0.53	0.53	0.53	0.53	WELDED
--	0.5 Monoethyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	190	--	--	--	0.48	0.52	0.53	0.53	0.53	0.53	WELDED
--	0.5 Monoethyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	130	--	--	--	0.48	0.52	0.53	0.53	0.53	0.53	WELDED
--	Diphenyl acid Phosphate (anhydrous) in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	170	0.39	2.40	2.30	2.86	2.90	2.49	1.04	1.06	1.17	1.17
--	0.5 Phosphorous Acid in PRL 3379	150	0.42	2.27	2.41	--	2.86	2.49	1.04	1.06	1.17	1.17
--	0.5 Dibutyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	90	0.39	0.44	0.54	0.91	1.04	1.04	1.04	1.06	1.17	1.17
--	0.5 Dibutyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	80	0.41	0.65	2.40	2.47	2.60	2.60	2.60	2.60	2.60	2.60
--	0.5 Dibutyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	90	0.37	0.42	2.56	2.51	2.51	2.51	2.51	2.51	2.51	2.51
--	0.5 Diisopropyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	70	0.39	0.46	0.43	2.62	2.62	2.62	2.62	2.62	2.62	2.62
--	0.5 Di-2-Ethylhexyl Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	60	2.21	--	2.92	WELDED	2.36	2.75	WELDED	WELDED	WELDED	WELDED
--	0.5 Bis (2-Octyl) Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	90	0.42	0.42	2.90	2.90	2.66	2.66	2.66	2.66	2.66	2.66
--	0.5 Bis (2-Octyl) Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	100	--	0.17	0.65	2.47	2.47	2.47	2.47	2.47	2.47	2.47
--	0.5 Bis (2-Octyl) Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 72 hrs.	60	1.86	2.21	2.12	2.64	2.64	2.64	2.64	2.64	2.64	2.64
--	0.5 Bis (2-Octyl) Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 168 hrs.	130	0.36	0.42	0.44	0.44	0.44	0.44	0.44	0.44	0.44	0.44
--	0.5 Bis (2-Octyl) Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 168 hrs.	90	--	0.15	2.27	2.27	2.27	2.27	2.27	2.27	2.27	2.27
--	0.5 Bis (2-Octyl) Acid Phosphate in PRL 3379 After Oxidation Test at 347°F for 168 hrs.	60	1.88	--	--	2.64	2.64	2.64	2.64	2.64	2.64	2.64

Controls

(1) Wear value at 340 kilograms load is 1.41 millimeters. No clear cut point of seizure is shown over this load range.

(2) Oxidation test, water, copper strip, test temperature 200°F time 168 hours.

TABLE XVII

STABLE LIFE TESTS OF SOME SYNTHETIC COMPOSITION GEAR LUBRICANTS

TEST PROCEDURE AND TECHNIQUE IN ACCORDANCE WITH SPECIFICATION MIL-L-7808
EXCEPT TEST TIME

Test Fluid -- 9.0% Acryloid HF-25 in Di-2-Ethylhexyl Sebacate

Oxidation Inhibitor Weight %	Lubrication Additive Weight %	Approximate Stable Life at 347°F, Hrs.
1.0 Hindered Phenol ¹	None	27
2.0 Hindered Phenol ¹	None	32
5.0 Hindered Phenol ¹		52
None	0.5 Dibutyl Acid Phosphite	15
1.0 Hindered Phenol	0.5 Dimethyl Acid Phosphite	290+
1.0 Hindered Phenol	0.5 Di Ethyl Acid Phosphite	182
1.0 Hindered Phenol	0.5 Dibutyl Acid Phosphite	113
1.0 Hindered Phenol	0.5 Di-2-Ethylhexyl Acid Phosphite	65
1.0 Hindered Phenol	0.5 Monolauryl Acid Ortho Phosphite	65
1.0 Hindered Phenol	0.5 Tri-2-Ethylhexyl Phosphite	50

¹Test fluid -- Di-2-Ethylhexyl Sebacate

Contracts

TABLE XVIII
REQUIREMENTS FOR -65°F TO 450°F GREASE

Test	Requirements	Test Method
1. Penetration	Worked Penetration of 260-310.	Specification VV-L-791, Method 31.1.4
2. Dropping Point	As Determined.	Specification VV-L-791d, Method 142.1
3. Dirt Count	No abrasive foreign matter. Non-abrasive particles limits; 7500 max. per cm. ³ of 25 microns diam. or above. 1600 max./cm ³ of 75 microns diam. or above. None of 125 microns diam. or above.	Specification VV-L-791d, Method 300.5.1
4. Low Temperature Torque	15 seconds max. at -65°F under 2000 gm.-cm. load	See Specification AN-G-25
5. Apparent Viscosity	15000 poises at -65°F at a shear rate of 20 reciprocal seconds	See Specification AN-G-25
6. Bomb Oxidation	5 PSI pressure drop at 250°F after 100 hours	Specification VV-L-791d, Method 345.3
7. Copper Corrosion	Slight stain allowable. No brown stain or pitting.	Specification VV-L-791d, Method 530.9
8. Gear Wear	Not more than 2.5 mg. per 1000 cycles under 5 lb. load nor more than 3.5 mg. per 1000 cycles under 10 lb. load	See Specification MIL-G-3278
9. Evaporation Loss	4% maximum after 22 hours at 400°F	ASTM Method D-972-48T
10. High Temperature Endurance	Shall satisfactorily lubricate a 204-K bearing at 400°F for 500 hours. Tests at 450°F to be performed at the option of the project engineer.	Specification VV-L-791, Method 33.1
11. Stability After Mechanical Working	Shall not exceed a penetration of 375 after 100,000 work strokes	Specification VV-L-791d, Method 31.3

Contracts

TABLE XIX
COMPOSITION OF SILICA GEL GREASES AND THEIR MECHANICAL STABILITY

Type of Oil	% of Silica Aerogel	Micropenetrations After Working in "Hain Microworker"		
		50 Strokes	100 Strokes	500 Strokes
Di(2-ethylhexyl)sebacate	8%	129	145	137
Si(2-ethylhexyl)sebacate	12%	63	67	63
DC-550 Silicone	8%	75	78	79
Flurolube	4%	108	117	123
1010 Grade Petroleum Oil	8%	128	137	132
Orsil BF-1 Silicate Ester	11%	88	80	78

TABLE XX
MECHANICAL STABILITY OF SILICA GEL GREASES AFTER HEATING*
(Greases were heated to 300°F and milled each day.)

Oil	% Silica Aerogel	MICROPENETRATION		
		Before Heating	After 1 day at 300°F	After 2 days at 300°F
Solvent Extracts No. 20	10	50	57	59
Kel-F No. 10	5	40	43	—
DC-510 Silicone Fluid	10	54	Slurry	—
Flexol 201	10	85	325	Slurry
Tri-o-cresyl phosphate	10	62	Slurry	—
Tributyryn	10	95	Slurry	—

* Work performed under Contract AF 33(038)23687 by Standard Oil Company(Indiana).

TABLE XXI

OIL SEPARATION AND EVAPORATION LOSS OF SILICA GEL GREASES

Oil	% of Silica Aerogel	%Wt. Loss Oil Separation	% Weight Loss Due to Evaporation
Di(2-ethylhexyl)sebacate	12	3.72	0.31
DC-550 Silicone	8	5.19	0.21
Orsil BF-1 Silicate Ester	11	0.02	1.91
Dipropylene glycol dipelargonate	16	0.80	0.80

TABLE XXII

GEAR WEAR TESTS OF SILICA GEL GREASES

Oil	% of Silica Aerogel	Average Loss in mg./1000 cycles	
		for 5# load	for 10# load
Di(2-ethylhexyl)sebacate	12	0.30	1.06
DC-550 Silicone	8	4.16	9.61
Orsil BF-1 Silicate Ester	11	0.20	0.55
Dipropylene glycol dipelargonate	16	0.54	1.65
Orsil BF-2 Silicate Ester	10	0.08	0.11

TABLE XXIII

WATER RESISTANCE AND WATER IMMERSION TESTS OF SILICA GEL GREASES

Oil	% of Silica Aerogel	Water Resistance Test Weight Loss	Water Immersion Test
Di(2-ethylhexyl)sebacate	8	87.3	Failed
Orsil BF-1 Silicate Ester	11	2.92	Failed
DC-550 Silicone	8	3.04	Passed
Dipropylene glycol dipelargenate	16	17.85	Failed
Fluorolube F-S	4	98.0	Failed

TABLE XXIV
COMPOSITION AND MECHANICAL STABILITY OF BENTONE 34 GREASES

Oil	% Bentone 34	% Additive	Initial Micropenetration	Micropenetration after 500 strokes	ASTM Unworked Pene.	Worked Pene.	Worked Stability
Orsil BF-1-S Inhibited Silicate Ester	20	?	102	70	—	—	—
DC-550 Silicone	20	—	65	108	—	—	—
DI(2-ethylhexyl)sebacate	20	—	67	62	—	—	—
LB-135-X Inhibited Polyethylene glycol	20	?	135	156	—	—	—
Dipropylene glycol dipelargonate	22	2% Bryton HY Sulfonate	66	83	287	299	315
DC-170 Silicone Oil	12	—	88	181	—	—	—
Dipropylene glycol dipelargonate	20	1% Ortholeum 300	—	—	279	290	—
15% Tridecyl azelate, 85% Dipropylene glycol dipelargonate	24	1% Dilauryl selenide Calcium 2% Sulfonate	—	—	224	250	280
85% Dipropylene glycol dipelargonate	20	1% Dilauryl selenide	—	—	290	305	327
15% Orsil BF-1-S	20	2% Sulfonate	—	—	—	—	—

Contrails

TABLE XXV
NAVY GEAR WEAR TESTS OF BENTONE-34 GREASES

Oil	% Bentone-34	% Additive	Weight Loss of Gears mg/1000 cycles 5# Load	Wt. Loss of Gears mg/1000 cycles 10# Load
Orsil BF-1-S Inhibited Silicate Ester	20	?	1.5	3.5
DC-550 Silicone	20	—	1.7	4.8
Di(2-ethylhexyl)sebacate	20	—	1.2	3.7
LB-135-X Inhibited Polyethylene glycol	20	?	0.9	2.0
1010 Base Petroleum Oil	20	—	0.8	1.3
Dipropylene glycol dipelargonate	20	1% Ortholeum 300	0.6	1.5
Dipropylene glycol dipelargonate	22	2% Bryton HY Sulfonate	0.7	1.9
15% Tridecyl azelate 85% Dipropylene glycol dipelargonate	24	1% Dilauryl Selenide Calcium 2% Sulfonate	0.9	3.5

TABLE XXVI

SHELL 4-BALL WEAR TESTS ON SOAP AND NON-SOAP GREASES

Test Conditions: 2 hours, 1800 RPM, 10 Kg. Load

Composition of Greases	Wear Spot Millimeters
20% Bentone 34, 79% Dipropylene glycol dipelargonate 1% Ortholeum 300	0.61
20% Bentone 34, 80% LB-135-X Inhibited Polyalkylene glycol	0.64
12% Lithium 12 hydroxystearate, 88% Orsil BF-1-S Inhibited Silicate Ester	0.52
16% Silica Gel, 84% Dipropylene glycol dipelargonate	0.87
MIL-G-3278 Grease	0.74
MIL-L-7711 Grease	0.56

TABLE XXVII
COMPOSITION AND MECHANICAL STABILITY OF LITHIUM HYDROXYSTEARATE GREASES

Oil	% Soap	Initial Micro- penetration	Micropenetration After Working 50 Strokes	100 Strokes	500 Strokes
Di (2-ethylhexyl)sebacate	12 Soap A	62	74	71	94
Di (2-ethylhexyl)sebacate	12 Soap B	164	257	248	260
Di (2-ethylhexyl)sebacate	12 Soap C	81	129	143	207
Dipropylene glycol dipelargonate	12 Soap A	45	56	59	72
Dipropylene glycol dipelargonate	12 Soap B	72	100	122	140
Dipropylene glycol dipelargonate	12 Soap C	65	89	108	130
Tridecyl azelate	12 Soap A	42	52	57	69
Tridecyl azelate	12 Soap B	72	100	122	140
Tridecyl azelate	12 Soap C	66	101	115	142

TABLE XXVII

WATER RESISTANCE OF LITHIUM HYDROXYSTEARATE GREASES

<u>88% Oil</u> Type Oil	<u>12% Thickener</u> Type Soap	Water Resistance Test		Water Immersion Test
		% Wt. Loss Grease	Test	
Di (2-ethylhexyl) sebacate	Lithium 12 hydroxystearate	5.97		Passed
Di (2-ethylhexyl) sebacate	Lithium 9/10 hydroxystearate	24.32		Passed
Di (2-ethylhexyl) sebacate	Lithium 9/10 hydroxystearate	15.73		Passed
Dipropylene glycol dipelargonate	Lithium 9/10 hydroxystearate	2.57		Passed
Dipropylene glycol dipelargonate	Lithium 9/10 hydroxystearate	27.98		Passed
Dipropylene glycol dipelargonate	Lithium 9/10 hydroxystearate	13.75		Passed
Dipropylene glycol dipelargonate	Lithium 12 hydroxystearate	1.98		Passed

TABLE XXIX

NAVY GEAR WEAR TESTS HYDROXYSTEARATE GREASES

<u>88% Oil</u>	<u>12% Thickener</u>	Loss weight (mg/1000 cycles) 5# Load	Loss weight (mg/1000 cycles) 10# Load
Type Oil	Type Soap		
Di (2-ethylhexyl)sebacate	Lithium 12 hydroxystearate	0.63	1.27
Di (2-ethylhexyl)sebacate	Lithium 9/10 hydroxystearate	0.54	1.15
Dipropylene glycol dipelargonate	Lithium 12 hydroxystearate	0.55	1.22
Dipropylene glycol dipelargonate	Lithium 9/10 hydroxystearate	0.46	1.40

Contrails

TABLE XXX

Type of Test	Procedure or Method Used
1. Mechanical Stability	"Hain Microworker"
2. Worked Penetration	Specification VV-L-791, Method 31.1
3. Worked Stability	Specification VV-L-791, Method 31.3
4. Water Immersion	Specification MIL-G-3278
5. Water Washing	Specification VV-L-791, Method 325.2
6. Oil Separation	Specification VV-L-791, Method 32.1
7. Evaporation	Specification VV-L-791, Method 35.1
8. Gear Wear	Specification MIL-G-3278
9. Bomb Oxidation	Specification VV-L-791, Method 345.3
10. Humidity Cabinet	Specification MIL-G-4343
11. Dropping Point	Specification VV-L-791, Method 142.1
12. High Temperature Performance	Specification VV-L-791, Method 33.1
13. Instrument Performance	Specification MIL-G-3278
14. Low Temperature Torque	Specification MIL-G-3278
BS-1010 Petroleum Oil	Specification MIL-O-6081

Contrails

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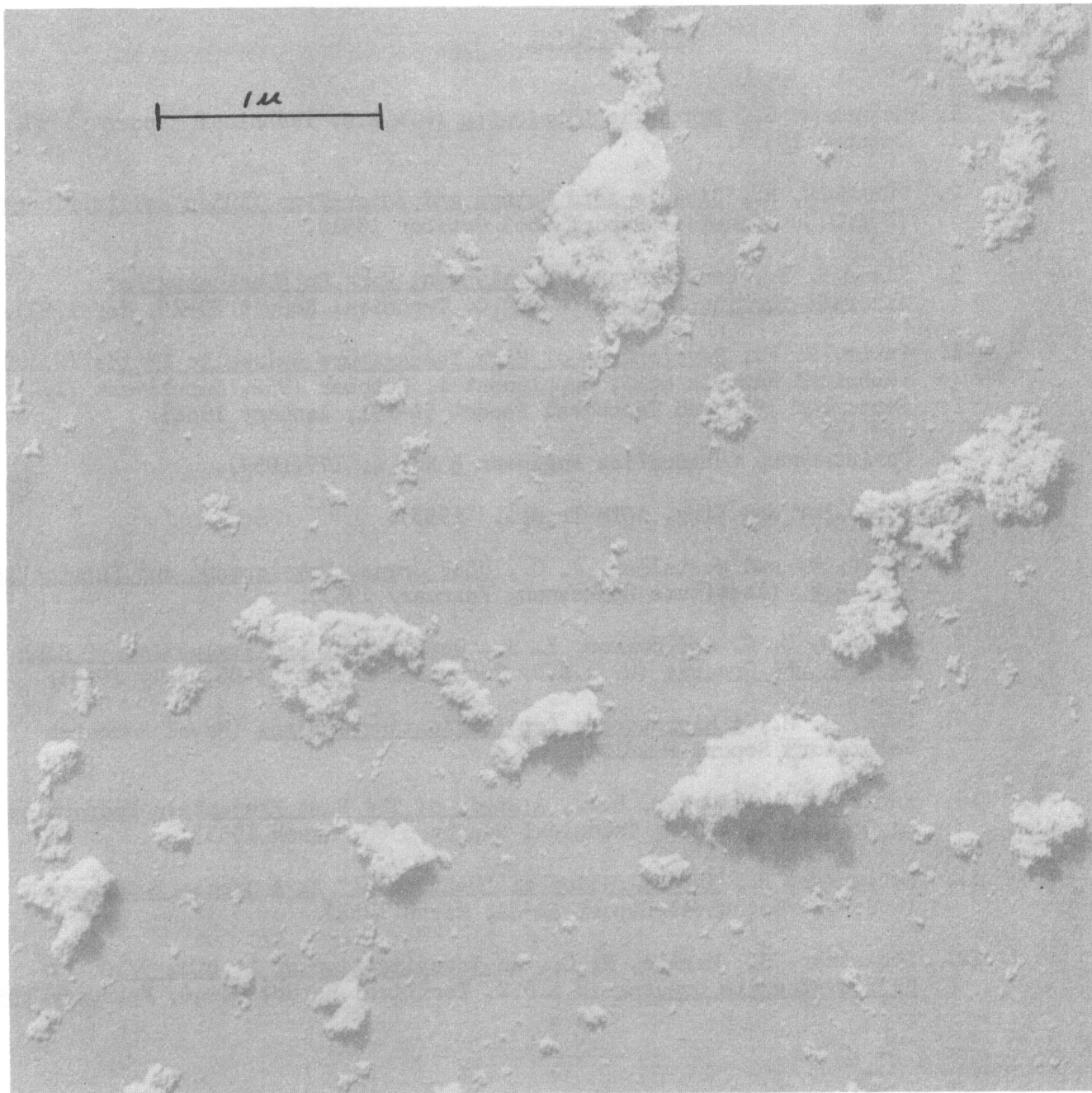


Fig. 1 Santocel ARD Silica Mag. 29, 000X

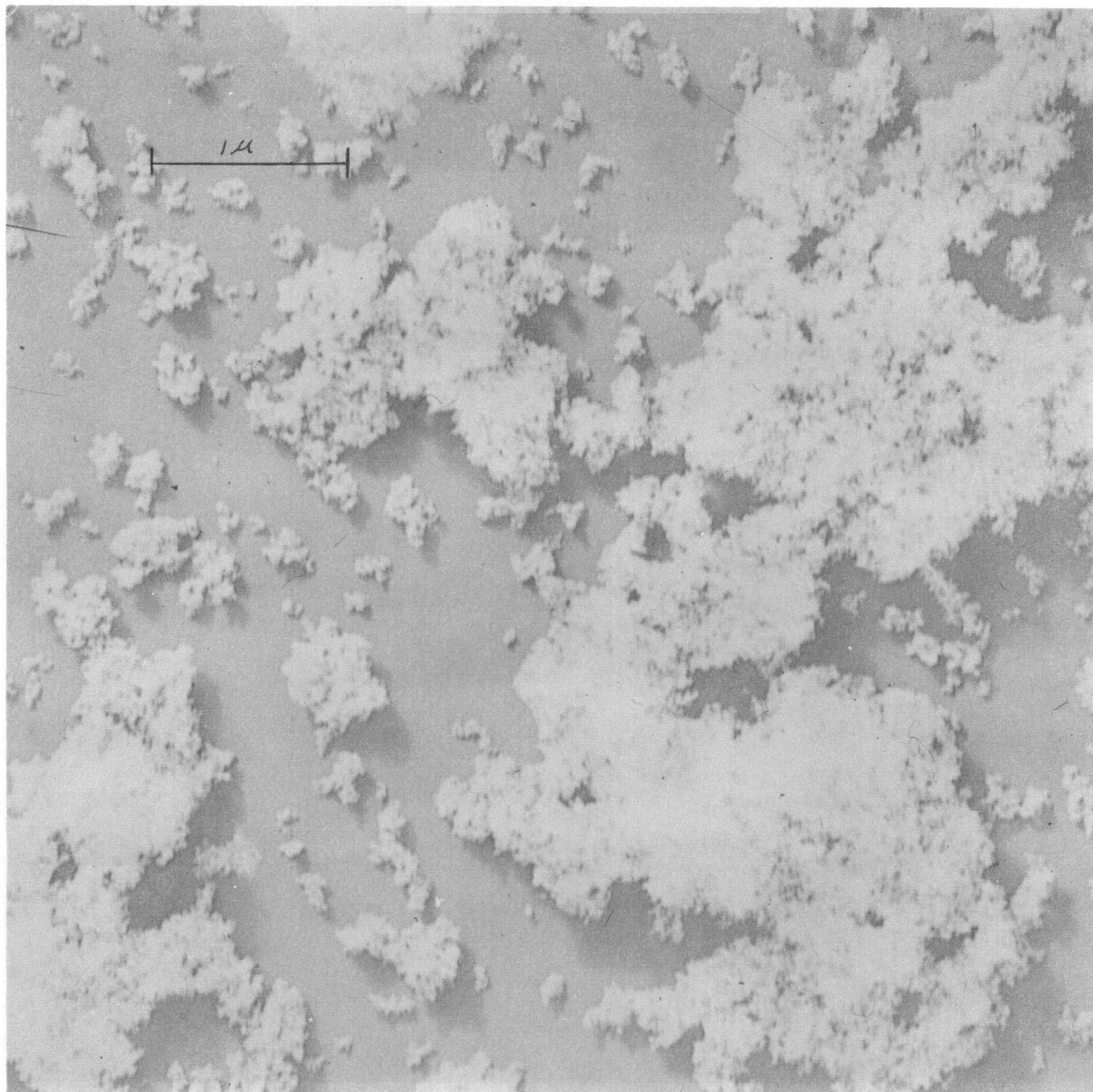
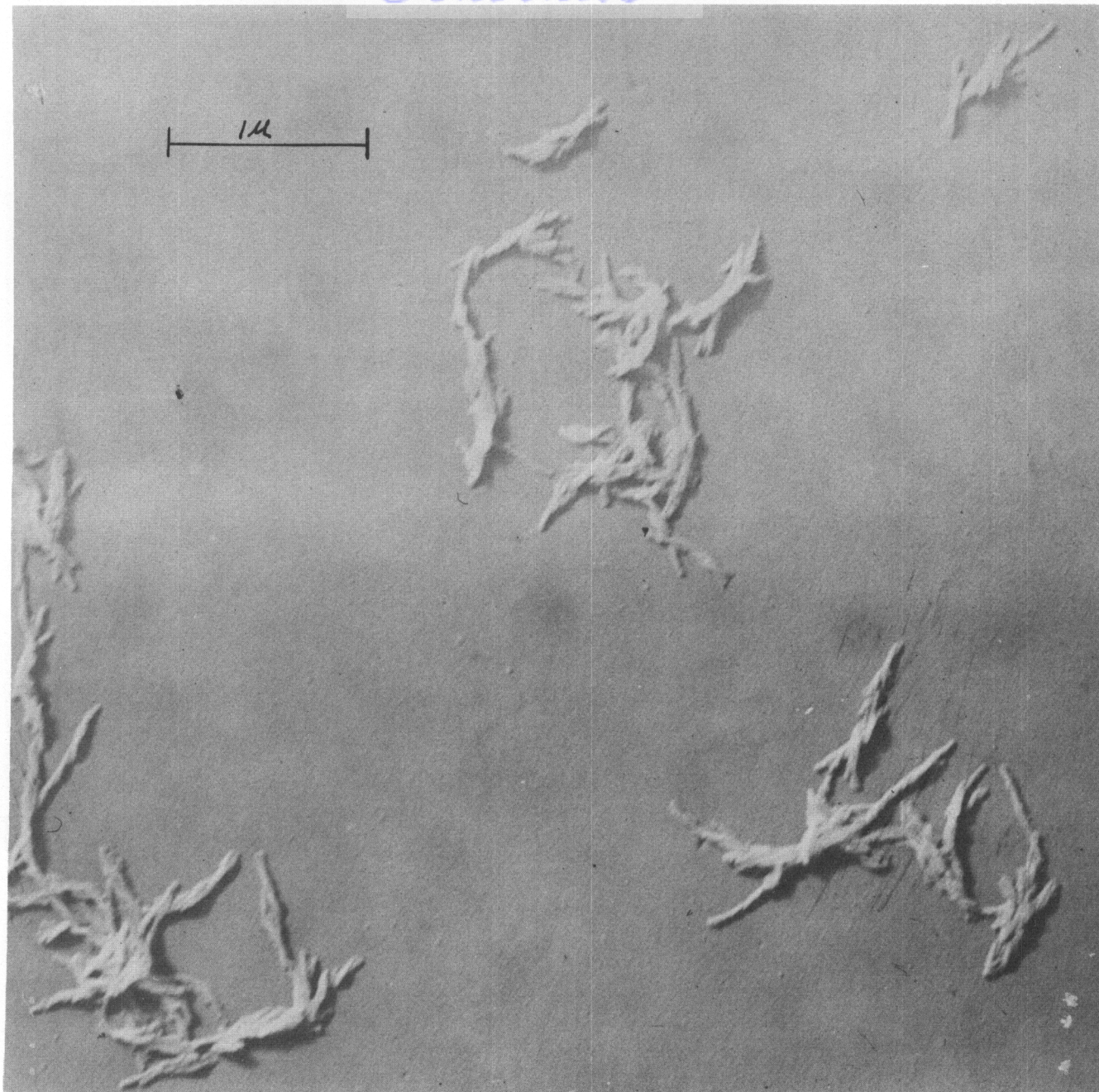
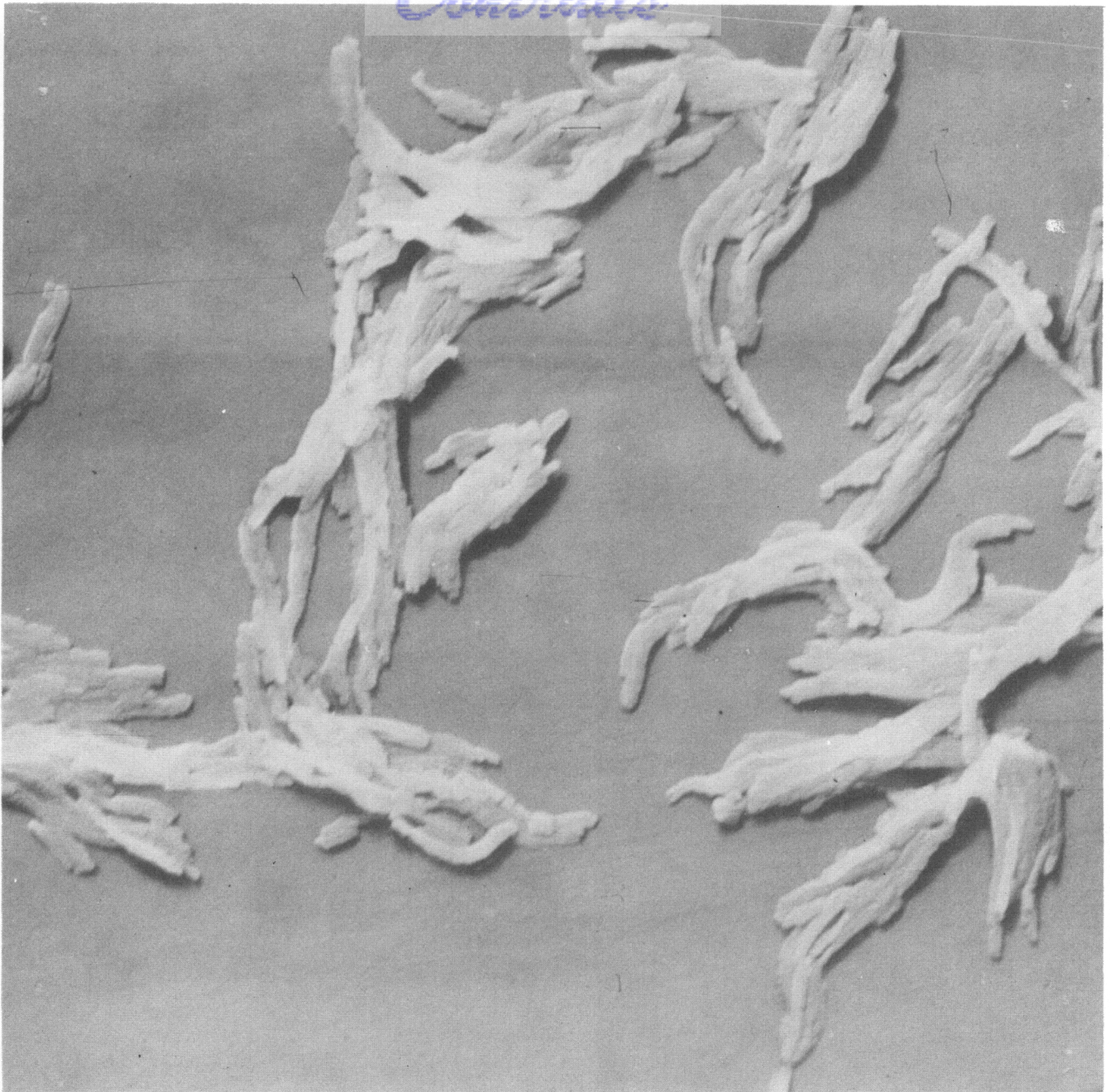


Fig. 2 A Silica Gel Grease Santocel ARD Silica Dipropylene glycol dipelargonate Mag. 32, 100X



**Fig. 3 Unworked Lithium 12 hydroxystearate grease
Mag. 32, 100X**



**Fig. 4 Unworked Lithium 9/10 hydroxystearate grease
Mag. 32, 100X**

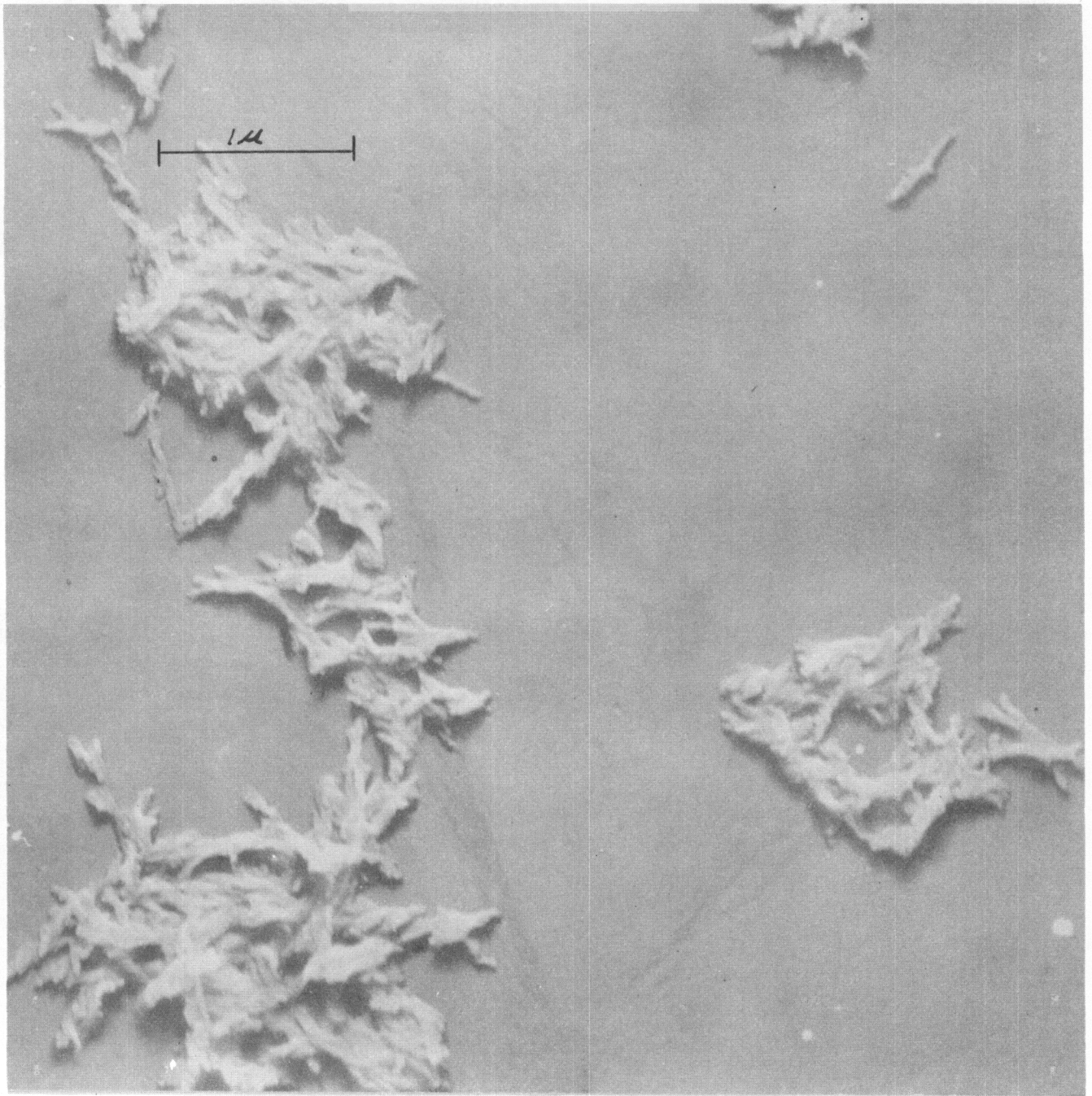


Fig. 5 Worked Lithium 12 hydroxystearate grease
Mag. 32, 100X

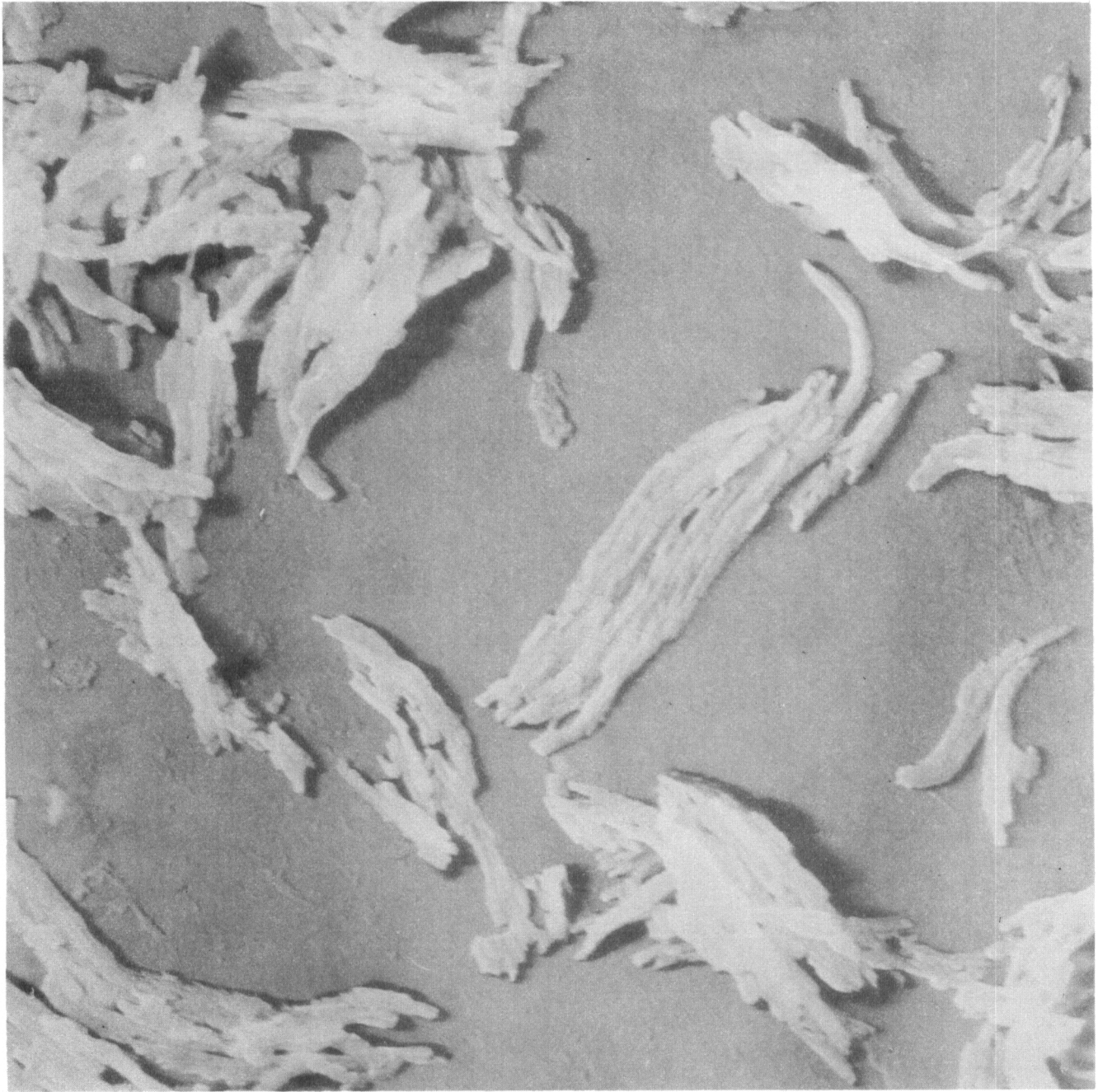


Fig. 6 Worked Lithium 9/10 hydroxystearate grease
Mag. 32, 100X