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BOUNDARY LUBRICATION OF FORMULATED C-ETHERS IN AIR TO 300° C

II - Organic Acid Additives

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Cleveland, Ohio 44135

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16. Abstract

Friction and wear measurements were made on CVM M-50 steel lubricated with three C-ether (modified polyphenyl ether) formulations in dry and moist air. Results were compared to those obtained with a formulated Type II ester and the C-ether base fluid. A ball-on-disk sliding friction apparatus was used. Experimental conditions were a 1-kilogram load, a 17-meter-per-minute surface speed, and a 25° to 300° C (77° to 572° F) disk temperature range. The three C-ether formulations yielded better boundary lubricating characteristics than the Type II ester under most test conditions. All C-ether formulations exhibited higher friction coefficients than the ester from 150° to 300° C (302° to 572° F) and similar or lower values from 25° to 150° C (77° to 302° F).

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II - ORGANIC ACID ADDITIVES

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SUMMARY

Friction and wear measurements were made on consumable electrode vacuum melted (CVM) M-50 steel lubricated with three C-ether (modified polyphenyl ether) formulations (organic acid additives) in dry (<100 ppm H_2O) and moist air (relative humidity (RH) 50 percent at 25° C (77° F)). Results were compared to those previously obtained with a fully formulated Type II ester and the C-ether base fluid. A ball-on-disk sliding friction apparatus was used. Experimental conditions were 1-kilogram load (initial Hertz stress, 1×10^9 N/m^2), a 17-meter-per-minute disk surface speed, a 25° to 300° C (77° to 572° F) disk temperature range, and a 25-minute test duration.

The three C-ether formulations yielded better boundary lubricating characteristics than the Type II ester under most test conditions. Formulation V (perfluoroglutaric acid boundary additive) (0.1 weight percent) exhibited lower or similar wear compared to the C-ether base fluid for most of the test conditions. In both dry (<100 ppm H_2O) and moist air (RH 50 percent at 25° C (77° F)), formulations IV (polyacid boundary additive) and VI (phenylphosphinic acid boundary additive) (0.08 weight percent) yielded higher wear than the C-ether base fluid over the entire temperature range.

In general, all C-ether formulations exhibited higher friction coefficients than the Type II ester from approximately 150° to 300° C (302° to 572° F). Lower or similar friction coefficients were observed for the formulations from 25° to 150° C (77° to 302° F).

Formulation V (perfluoroglutaric acid additive) (0.1 weight percent) exhibited lower wear at low temperatures and higher wear at high temperatures when tested in moist air as compared to a dry air atmosphere. No moisture effects were observed with the other two formulations.

INTRODUCTION

Advanced aircraft and re-entry vehicles will place increased thermal stresses on hydraulic fluids and lubricants. Maximum fluid temperatures in excess of 316° C (600° F) have been estimated for future applications (refs. 1 to 5). At these elevated temperatures, fluids must operate without appreciable degradation and must also provide effective lubrication for bearings and hydraulic system components.

Presently available fluids such as the super-refined mineral oils (refs. 6 and 7), hindered esters (refs. 3, 8, and 9), fluorinated polyethers (refs. 2, 3, and 10), and polyphenyl ethers (refs. 3, 7, 11, and 12) have one or more deficiencies which limit their use at high temperatures. These deficiencies include poor oxidation stability, poor boundary lubricating characteristics, and corrosivity. In addition, because of the optimization of the high temperature properties, these fluids exhibit poor low temperature fluidity (high pour point).

The C-ethers, which are structurally related to the polyphenyl ethers, are a promising class of fluids for possible high temperature applications (refs. 13 and 14). They have excellent thermal stability (thermal decomposition temperature of 390° C (734° F) measured by isoteniscope), good oxidation stability to 260° C (500° F), and adequate pour points (-29° C (-20° F)). They also exhibit low vapor pressure, high surface tension, and excellent shear stability. The main deficiencies of the C-ethers have been their poor boundary lubricating ability and poor wetting characteristics (refs. 7 and 15). Heat transfer (cooling) problems have also been encountered with this fluid class (ref. 2) and are probably a result of its poor wetting properties. Additives can, however, improve the boundary lubricating ability of C-ethers (ref. 16).

The objectives of this investigation were (1) to determine the friction and wear of consumable electrode vacuum melted (CVM) M-50 steel lubricated with three C-ether formulations (organic acid additives) in dry (<100 ppm H_2O) and moist air (relative humidity (RH) 50 percent at 25° C (77° F)) at temperatures from 25° to 300° C (77° to 572° F); and (2) to compare these results with those previously obtained (ref. 16) with a fully formulated Type II ester (MIL-L-23699) and the C-ether base fluid. Other experimental conditions included a 1-kilogram load (initial Hertz stress, 1×10^9 N/m^2), a 17-meter-per-minute surface speed, and a test duration of 25 minutes.

APPARATUS

The ball-on-disk sliding friction apparatus is shown in figure 1. The test specimens were contained inside a stainless-steel chamber. The atmosphere was controlled with respect to moisture content. A stationary 0.476-centimeter-radius ball was placed in

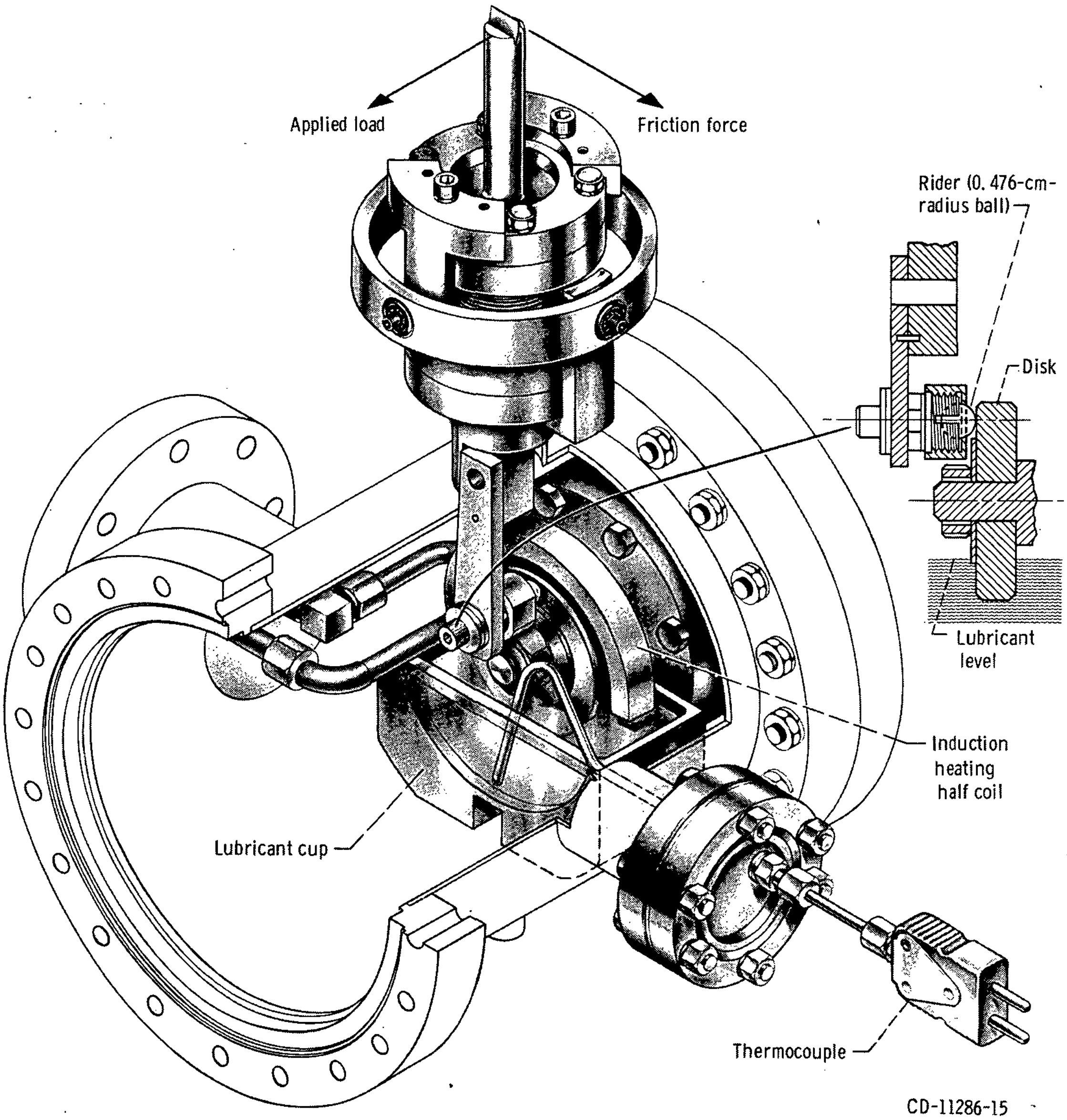


Figure 1. - Friction and wear apparatus.

sliding contact with a rotating 6.3-centimeter-diameter disk. A sliding speed of 17 meters per minute was maintained. A normal load of 1 kilogram (initial Hertz stress, 1×10^{9} N/m^2) was applied with a deadweight. Balls and disks were made of CVM M-50 tool steel. Disk and ball hardness was Rockwell C 62 to 64.

The disk was partially submerged in a polyimide cup containing the test lubricant and was heated by induction. Bulk lubricant temperature was measured with a thermocouple. Disk temperature was monitored with an infrared pyrometer. Frictional force was measured with a strain gage and was recorded on a strip chart recorder.

MOISTURE MONITORING AND CONTROL

The two atmospheres used in this study were (1) moist air at a relative humidity (RH) of 50±5 percent at 25° C (77° F) and (2) dry air (<100 ppm H_2O). The relative humidity was monitored by a direct reading hygrometer accurate to ±1.5 percent. The low water concentrations were monitored by a moisture analyzer with an accuracy of ±10 ppm.

Dry air was obtained by drying and filtering service air. Moist air was obtained by bubbling the dry gas through a water reservoir. The relative humidity of the moist air was controlled manually to 50±5 percent at 25° C (77° F).

PROCEDURE

Disks were ground and lapped to a surface finish of 10×10^{-8} to 20×10^{-8} meter (4 to 8 μin.) and balls to 2.5×10^{-8} meter (1 μin.) rms. Specimens were scrubbed with a paste of levigated alumina and water, rinsed with tap water and distilled water, then placed in a desiccator.

All lubricants tested in dry air were degassed at approximately 150° C (302° F) at 2 torr for 1 hour. Measurements using the Karl Fischer technique indicate that this degassing procedure reduces dissolved water content in C-ethers to less than 20 ppm.

The specimens were assembled and 70 cubic centimeters of lubricant were placed in the lubricant cup. The test chamber (3.7×10^{3} cm^3 volume) was purged with the test atmosphere for 10 minutes at a flow rate in excess of 50×10^{3} cubic centimeters per hour. The disk was heated by induction to the test temperature while rotating (100 rpm). The ball was then loaded against the disk. Test atmosphere flow rate was reduced to 35×10^{3} cubic centimeters per hour, and a 6.9×10^{-3}-newton-per-square-meter (1-psig) pressure was maintained in the chamber. The lubricant was heated only by heat transfer from the rotating disk. The bulk lubricant temperature was essentially the same as the disk temperature at disk temperatures to 100° C (212° F). At disk temperatures of 200° and 300° C (392° and 572° F), the bulk oil temperatures stabilized at approximately 150° and 200° C (302° and 392° F), respectively.

Frictional force and bulk lubricant temperature were continuously recorded. Disk temperature was continuously monitored. Experiments were terminated after 25 minutes and the rider (ball) wear scar diameter was recorded (from which wear volume was calculated).

EXPERIMENTAL LUBRICANTS

The experimental and reference fluids used in these experiments were a formulated Type II ester, a C-ether base fluid, and three C-ether formulated fluids. Some typical properties of the Type II ester and the C-ether base fluid appear in table I. Table II contains the additive contents of the test fluids.

Formulated Type II Ester

A fully formulated Type II ester was chosen as a reference fluid for these experiments. This lubricant is commercially available and meets General Electric D50TF1,

TABLE I. - TYPICAL PROPERTIES OF THE EXPERIMENTAL FLUIDS

Properties[a]	C-ether base fluid	Type II ester
Kinematic viscosity, m^2/sec (cS)		
At 38° C (100° F)	2.5×10^{-5} (25)	2.8×10^{-5} (28)
At 99° C (210° F)	4.1×10^{-6} (4.1)	5.3×10^{-6} (5.3)
At 300° C (572° F)	6.9×10^{-7} (0.69)	[b]6.8×10^{-7} (0.68)
Pour point, °C (°F)	-29 (-20)	-60 (-75)
Flash point, °C (°F)	239 (445)	280 (535)
Fire point, °C (°F)	285 (540)	---------------
Density at 38° C (100° F), g/cm^3	1.19	[c]0.990
Thermal decomposition (isoteniscope), °C (°F)[d]	390 (734)	316 (600)
Vapor pressure at 371° C (600° F), torr	140	---------------
Surface tension at 23° C (73° F), N/cm (dynes/cm)[d]	44.8×10^{-4} (44.8)	---------------
Erdco bearing rig deposit rating (Type II conditions)[a,e]	---------------	26

[a]Manufacturer's data.
[b]Extrapolated.
[c]Specific gravity (15.6° C/15.6° C (60° F/60° F)).
[d]Measured by author.
[e]Bulk oil 227° C (440° F), oil in 204° C (400° F), bearing 260° C (500° F).

TABLE II. - ADDITIVE CONTENTS OF TEST FLUIDS

Formulated Type II ester	C-ether base fluid	C-ether formulation IV	C-ether formulation V	C-ether formulation VI
Antiform, anticorrosion, aromatic amine antioxidant, combined antioxidant and load carrying agent	Antifoam	Antifoam, polyacid	Antifoam, perfluoroglutaric acid (0.10 weight percent)	Antifoam, phenylphosphinic acid (0.08 weight percent)

Pratt and Whitney PWA 521B, and MIL-L-23699 lubricant specifications.

C-ether Base Fluid

The C-ether base fluid used in this study was originally reported in reference 13. This fluid is a blend of three-ring and four-ring components which are structurally similar to the polyphenyl ethers. This base fluid contains an antifoam additive.

C-ether Formulations (Organic Acid Additives)

Formulation IV. - Formulation IV was the base fluid plus a proprietary organic polyacid boundary additive.

Formulation V. - Formulation V was the base fluid plus 0.1 weight percent of a boundary additive (perfluoroglutaric acid).

Formulation VI. - Formulation VI was the base fluid plus 0.08 weight percent of a boundary additive (phenylphosphinic acid). None of these formulations has been studied previously.

RESULTS AND DISCUSSION

Wear

Formulated Type II ester. - This fluid was chosen as a reference fluid because it appeared to be a typical example of the polyol ester group of MIL-L-23699 lubricants. Wear results for this fluid appear in figure 2 (from ref. 16). No significant differences in wear were observed between the dry and wet air results. Therefore, a single wear temperature curve appears in figure 2. The wear rate was essentially constant at 1.4×10^{-13} cubic meter per minute over the entire temperature range.

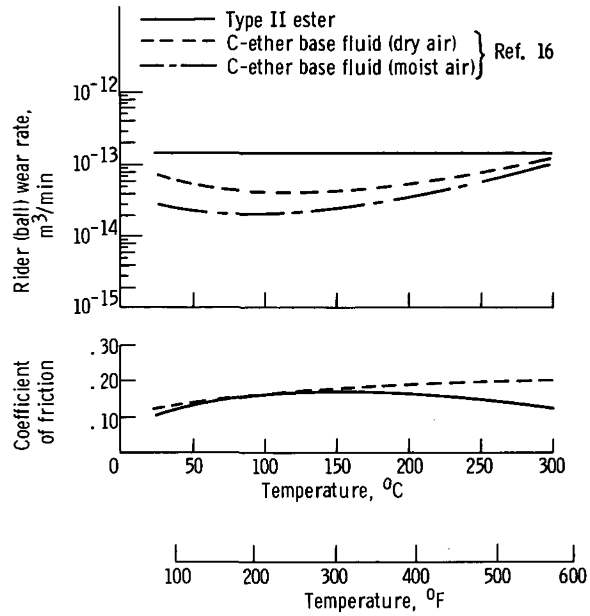


Figure 2. - Coefficient of friction and rider (ball) wear as a function of temperature for a fully formulated Type II ester and a C-ether base fluid. Test conditions: M-50 steel specimens; 1-kilogram load; 17-meter-per-minute (100-rpm) sliding speed; dry (<100 ppm H_2O) and moist (RH 50 percent at 25° C (77° F) air; 25-minute test duration.

C-ether base fluid. - Wear results for this base fluid also appear in figure 2 (from ref. 16). The C-ether base fluid yielded lower wear than the ester in both atmospheres over almost the entire temperature range. Lower wear rates occurred when the C-ether was tested in moist air (RH 50 percent at 25° C (77° F)) as compared to a dry air (<100 ppm H_2O) atmosphere.

C-ether formulations (organic acid additives). - Wear results for C-ether formulations I, II, and III, containing phosphorus ester additives, were reported in reference 16. Wear results for C-ether formulations IV, V, and VI, containing organic acid additives, appear in figure 3. The wear rate for the Type II ester also appears in figure 3 for reference.

Moisture in the test atmosphere did not greatly affect the wear results for formulations IV and VI. However, formulation V yielded lower wear below and higher wear above 185° C (365° F) when tested in moist as compared to dry air.

As is evident from figure 3, formulations IV and V yielded lower wear than the ester over essentially the entire temperature range. Formulation VI yielded lower wear than the ester from 25° to 150° C (77° to 302° F) and about the same wear from 150° to 300° C (302° to 572° F).

Comparisons between the wear rates for the three formulations and the C-ether base fluid appear in figure 4. In both dry (fig. 4(a)) and moist air (fig. 4(b)) formulations IV

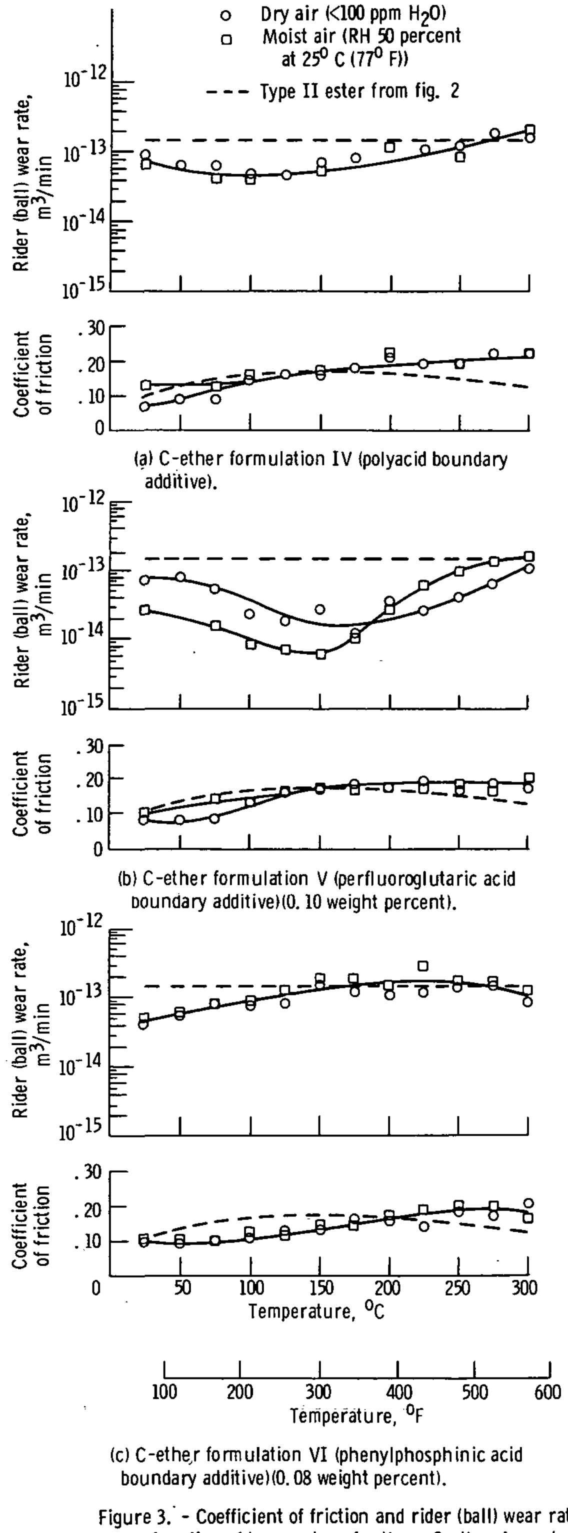
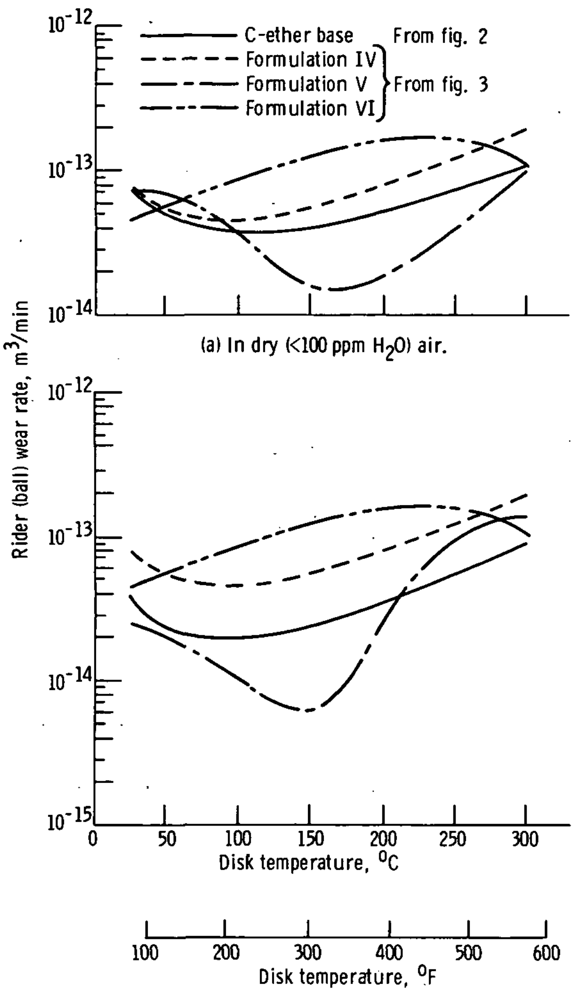


Figure 3. - Coefficient of friction and rider (ball) wear rate as a function of temperature for three C-ether formulations. Test conditions: M-50 steel specimens; 1-kilogram load; 17-meter-per-minute (100-rpm) sliding speed; dry (100 ppm H_2O) and moist (RH 50 percent at 25° C (77° F) air; 25-minute test duration.



(a) In dry (<100 ppm H_2O) air.

(b) In moist (RH 50 percent at 25° C (77° F)) air.

Figure 4. - Rider (ball) wear rate as a function of temperature for a C-ether base fluid and three C-ether formulations. Test conditions: M-50 steel specimens; 1-kilogram load; 17-meter-per-minute (100-rpm) surface speed; 25-minute test duration.

and VI yielded higher wear than the base fluid over almost the entire temperature range. Formulation V exhibited a somewhat more complex behavior yielding lower wear than the base fluid under most conditions but higher wear under others.

Coefficient of Friction

Figure 2 contains a comparison between the friction coefficients for the Type II ester and the C-ether base fluid. Figure 3 shows the friction-temperature curves for each of the three formulations. All C-ether formulations exhibited higher friction coefficients than the ester from 150° to 300° C (302° to 572° F) and similar or lower values from 25° to 150° C (77° to 302° F). Testing in a moist as opposed to a dry air atmosphere had little or no effect of the friction coefficients for all fluids.

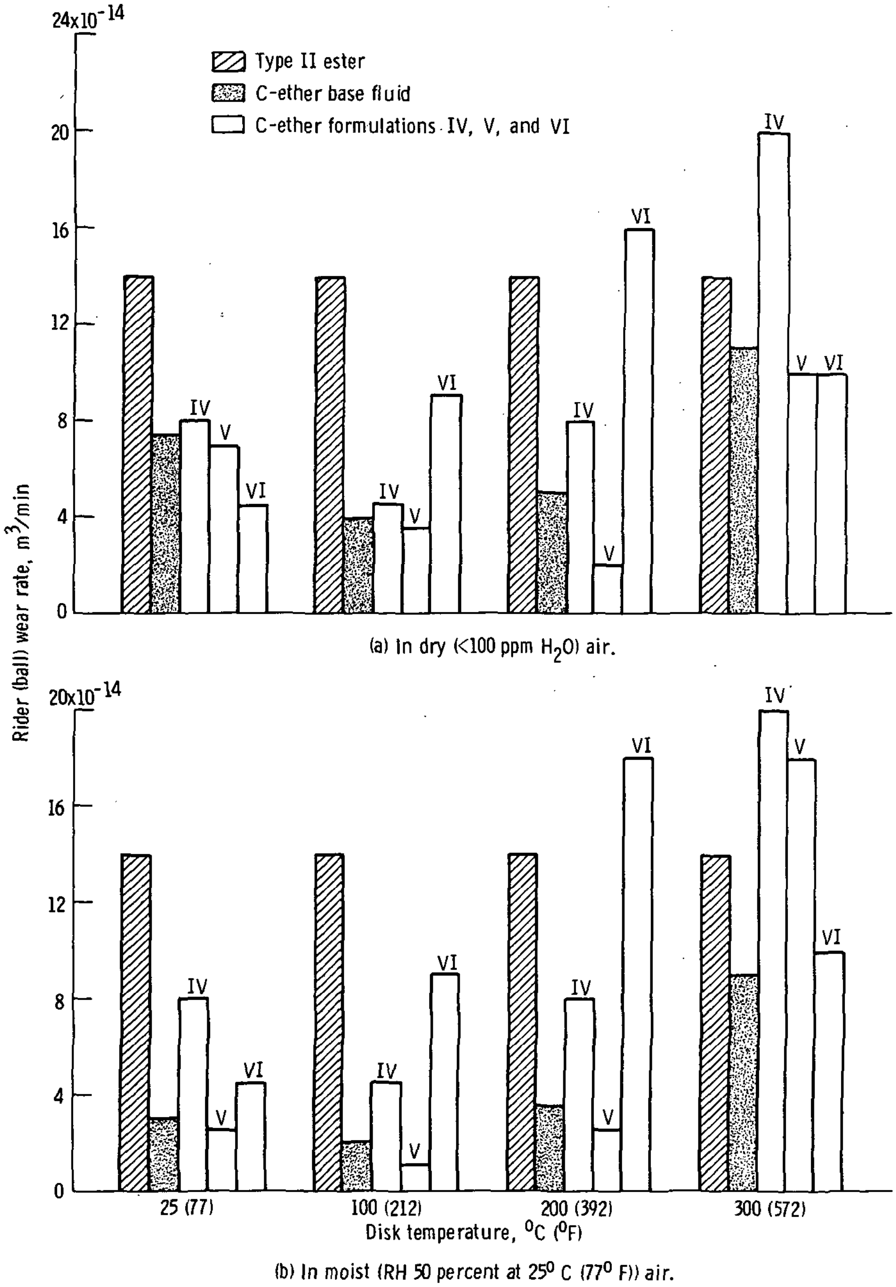
A summary of friction and wear results for all test fluids at four selected temperatures at 25°, 100°, 200°, and 300° C (77°, 212°, 392°, and 572° F) appears in table III. Figure 5 also summarizes the wear rates for all fluids at the aforementioned temperatures in both dry and moist air.

Care must be taken in interpreting these results. Obviously boundary additives are added to base stocks to improve their boundary lubricating characteristics (i.e., reduce friction or wear). However, whether a particular additive actually increases or decreases the wear associated with the unformulated base stock is dependent upon its chemical reactivity. The chemical reactivity, in turn, depends upon specimen metallurgy, type of atmosphere, and the severity of the test conditions.

TABLE III. - SUMMARY OF FRICTION AND WEAR RESULTS

Disk temperature		Test fluids				
°C	°F	Type II ester	C-ether base fluid[a]	C-ether formulation IV	C-ether formulation V	C-ether formulation VI
		Rider wear, m^3/min				
25	77	14×10^{-14}	7.5×10^{-14} [b]3.0	8.0×10^{-14}	7.0×10^{-14} [b]2.5	4.5×10^{-14}
100	212	14×10^{-14}	3.8×10^{-14} [b]2.0	4.5×10^{-14}	3.5×10^{-14} [b]1.0	9.0×10^{-14}
200	392	14×10^{-14}	5.0×10^{-14} [b]3.5	8.0×10^{-14}	2.0×10^{-14} [b]2.5	16×10^{-14}
300	572	14×10^{-14}	11×10^{-14} [b]9.0	20×10^{-14}	10×10^{-14} 16	10×10^{-14}
		Coefficient of friction				
25	77	0.10	0.12	0.07 [b].13	0.08 [b].10	0.10
100	212	0.16	0.16	0.14	0.12 [b].14	0.10
200	392	0.16	0.18	0.19	0.18	0.16
300	572	0.12	0.20	0.21	0.18	0.18

[a]Ref. 16.
[b]Moist air (RH 50 percent) results where they differ from dry air results.



(b) In moist (RH 50 percent at 25^o C (77^o F)) air.

Figure 5. - Rider (ball) wear rate at four disk temperatures for a Type II ester, a C-ether base fluid, and three C-ether formulations.

Rowe (ref. 17) has given an explanation of how lubricant (or additive) reactivity, wear, and test severity are related. A schematic plot (from ref. 17) illustrating these relations appears in figure 6. Curve B represents a set of test conditions more severe than those of curve A. Essentially, high wear can be obtained at low reactivities (adhesive wear) or at high reactivities (corrosive wear). Obviously, there is an optimum balance between the two types of wear which is dependent on the severity of the test conditions. It should also be obvious, that a change in test severity (such as an increase in load) could cause a re-ordering of additives of different reactivities from a wear standpoint.

The relative reactivities of the three additives included in this study have not been independently measured. However, it is apparent from figure 4 that both the polyacid (formulation IV) and phenylphosphinic acid (formulation VI) are operating in the corrosive wear regime. Both additives yield higher wear than the base fluid alone. A lowering of the additive concentration in these two formulations would ameliorate this effect.

Another interesting aspect shown in figure 4 is the effect of atmospheric moisture on wear rate. As previously mentioned, only formulation V (perfluoroglutaric acid) yielded different wear rates when tested in dry air as compared to moist air. It is also interesting to note that in the earlier study of C-ether formulations (ref. 16), only formulation III (containing a halogenated acid) yielded a similar result.

Apparently, water catalyzes boundary film formation with halogenated acids. This is consistent with some experiments with fatty acids which indicated that the rate of boundary film formation was accelerated by the presence of water (ref. 18).

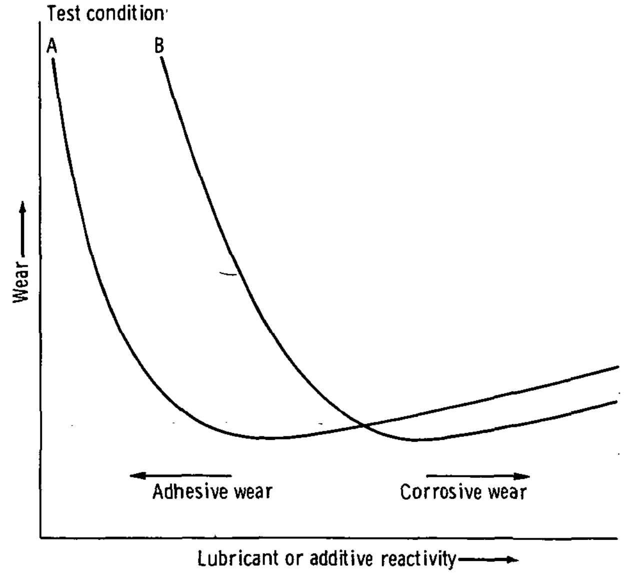


Figure 6. - Schematic illustrating relationship between wear, reactivity, and severity of test conditions (ref. 17). Severity of test condition B is greater than that of test condition A.

It must be stated that the test conditions of this study are only a part of the spectrum of conditions that a lubricant or hydraulic fluid would be subjected to in service. Quite different results may be obtained under higher speeds, higher loads, and different metallurgies.

SUMMARY OF RESULTS

The friction and wear of consumable electrode vacuum melted (CVM) M-50 steel lubricated with three C-ether formulations (organic acid additives) in dry (<100 ppm H_2O) and moist air (relative humidity (RH) 50 percent at 25° C (77° F)) were determined using a ball-on-disk sliding friction apparatus. Disk temperature range was 25° to 300° C (77° to 572° F). Other conditions were a 1-kilogram load (initial Hertz stress, 1×10^9 N/sq m), a 17-meter-per-minute (100 rpm) sliding speed, and a 25-minute test duration. Results were compared to those obtained with a formulated Type II ester and the C-ether base fluid. The major results were the following:

1. The three C-ether formulations yielded better boundary lubricating characteristics compared to the Type II ester under most test conditions.

2. Formulation V (0.1 weight percent perfluoroglutaric acid boundary additive) exhibited lower or similar wear while formulations IV (polyacid additive) and VI (0.08 weight percent phenylphosphinic acid boundary additive) exhibited higher wear than the C-ether base fluid for most test conditions.

3. In general, all C-ether formulations exhibited higher friction coefficients than the Type II ester from 150° to 300° C (302° to 572° F) and lower or similar values from 25° to 150° C (77° to 302° F).

4. In moist air, formulation V (perfluoroglutaric acid boundary additive) exhibited lower wear at low temperatures and higher wear at high temperatures then it did in dry air. No moisture effects were observed with the other two formulations.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 5, 1973,
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