

## NOTICE

THIS DOCUMENT HAS BEEN REPRODUCED FROM  
MICROFICHE. ALTHOUGH IT IS RECOGNIZED THAT  
CERTAIN PORTIONS ARE ILLEGIBLE, IT IS BEING RELEASED  
IN THE INTEREST OF MAKING AVAILABLE AS MUCH  
INFORMATION AS POSSIBLE

# Improved Boundary Lubrication With Formulated C-Ethers

(NASA-TM-82808) IMPROVED BOUNDARY  
LUBRICATION WITH FORMULATED C-ETHERS (NASA)  
25 p HC A02/MF A01 CSCI 07C

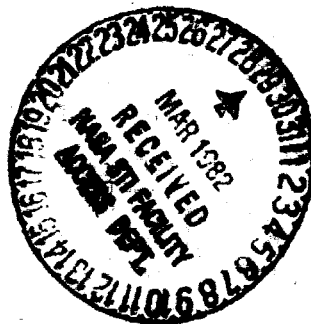
N82-20313

Unclas

G3/27 09349

**William R. Loomis**  
*Lewis Research Center*  
*Cleveland, Ohio*

Prepared for the  
Thirty-seventh Annual Meeting of the  
American Society of Lubrication Engineers  
Cincinnati, Ohio, May 10-13, 1982



**NASA**

# IMPROVED BOUNDARY LUBRICATION WITH FORMULATED C-ETHERS

by William R. Loomis\*

National Aeronautics and Space Administration

Lewis Research Center

Cleveland, Ohio 44135

## ABSTRACT

E-1149

A boundary lubrication study was made to compare five recently developed C-ether-formulated fluids with an advanced formulated MIL-L-27502 candidate ester. Steady-state wear and friction measurements were made with a sliding pin-on-disk friction apparatus. Conditions included disk temperatures up to 260° C, dry-air test atmosphere, 1-kilogram load, 50-rpm disk speed, and test times to 130 minutes. Based on wear rates and coefficients of friction, three of the C-ether formulations as well as the C-ether base fluid gave better boundary lubrication than the ester fluid under all test conditions. The susceptibility of C-ethers to selective additive treatment (phosphinic esters or acids and other antiwear additives) was demonstrated when two of the formulations gave somewhat improved lubrication over the base fluid. Also, the increased operating potential for this fluid was shown in relationship to bulk oil temperature limits for MIL-L-23699 and MIL-L-27502 type esters.

## INTRODUCTION

One anticipated trend in the development of advanced jet engines is toward higher operating temperatures to improve fuel efficiency and performance. Higher engine temperatures will place increased thermal demands on

---

\*Member, ASLE.

the lubricant. The lubricant will be required either to operate at higher oil temperatures or to be cooled by larger (heavier) heat exchanges.

The Air Force has funded programs with the goal of developing ester type lubricants with a bulk oil temperature (BOT) capability of 240° C (465° F) (1), (2), and has issued a target specification MIL-L-27502 which, slightly more conservatively, requires a BOT capability of 220° C (428° F). No oil has yet completely qualified under this specification, but a formulated hindered polyol ester of pentaerythritol is a promising candidate fluid which meets most of the requirements.

For future engines with Mach 4+ capability, it is estimated that lubricants with at least a 260° C (500° F) BOT capability will be needed (3). Polyphenyl ether fluids with the desired oxidative stability are available (4), but their pour points are too high, typically +5° C (40° F). However, polyphenyl ether analogs (C-ethers), which contain sulfur instead of oxygen in some or all of the phenyl to phenyl linkages, have much lower pour points, on the order of -29° C (-20° F), and still retain good oxidative stability at 260° C (500° F). They were first reported in Ref. (5) and were further studied under NASA contracts (6), (7).

In the present study, a pin-on-disk wear machine was used to determine the friction and wear characteristics of formulated C-ether blends. The scope of these studies included: (1) the determination of wear rates and friction coefficients of five C-ether formulations from 20° to 260° C, and (2) a comparison of these results with those of the MIL-L-27502 candidate ester of pentaerythritol and with those of the C-ether base stock.

#### APPARATUS

The sliding friction and wear apparatus is shown in Fig. 1. The test specimens, contained inside a stainless-steel chamber, consist of a station-

ary 0.476-cm-radius, hemispherically tipped rider in sliding contact with a rotating 6.35-cm-diameter disk. Rider holders of three different lengths were used to permit three concentric wear tracks to be run on a single disk, thereby eliminating the need to refinish disks after each run. Sliding velocities ranged from about 7.1 to 9.1 m/min as disk rotational speed was maintained at 50 rpm for all three rider holders.

A normal load of 1 kg (initial Hertz stress,  $1 \times 10^9 \text{ N/m}^2$ ) was applied with a dead weight. Disks were made of consumable-vacuum-melted (CVM) M-50 tool steel and heat treated to a hardness of Rockwell C62 to C64. Riders were made of pure iron (99.99 percent iron) and were annealed to a hardness of 70 to 92  $\text{kg/mm}^2$  diamond pyramid hardness. Softer iron riders were used instead of M-50 tool steel to achieve larger and more easily measured wear scars than found in previous studies (8), (9). Also, the load and surface sliding velocities were selected to avoid elastohydrodynamic and "mixed" lubrication regions (10).

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 while disk temperature was monitored with an infrared pyrometer. Frictional force was measured with a strain gage and recorded on an X-Y recorder.

The test atmosphere was filtered air dried to less than 100 ppm water. The moisture content was monitored by a moisture analyzer with an accuracy of 110 parts per million. The moisture analyzer worked on the principle of weighing and indicating micro quantities of water vapor on a hygroscopically coated quartz crystal. A dry-air atmosphere was selected because it had been determined that, in general, lower wear rates were obtained when moist air was used (8).

## PROCEDURE

Disks were ground and lapped to a surface finish of  $1 \times 10^{-7}$  to  $2 \times 10^{-7}$  m (4 to 8  $\mu$ in.) rms. Rider tips were machined and polished to a surface finish of  $5 \times 10^{-8}$  to  $10 \times 10^{-8}$  m (2 to 4  $\mu$ in.) rms. Specimens were scrubbed with a paste of levigated alumina and water, rinsed with tap water and distilled water, and then placed in a desiccator.

Test lubricants were degassed at approximately  $150^\circ$  C ( $302^\circ$  F) at  $2.7 \times 10^2$  N/m<sup>2</sup> pressure for 1 hr. Measurements made using the Karl Fisher technique indicate that this degassing procedure reduces dissolved water content in the test fluids to less than 20 ppm. Approximately  $3 \times 10^{-5}$  m<sup>3</sup> (30 milliliters) of lubricant was employed. The test chamber ( $3.7 \times 10^{-3}$  m<sup>3</sup> volume) was purged with the dry air test atmosphere for 10 min at a flow rate greater than  $5 \times 10^{-2}$  m<sup>3</sup>/hr. During the high-temperature runs the disk was heated by induction to test temperature while rotating before the rider was loaded against the disk. The flow rate of the dry-air atmosphere was reduced to  $3.5 \times 10^{-2}$  m<sup>3</sup>/hr, and a pressure of  $6.9 \times 10^{-3}$  N/m<sup>2</sup> (1 psig) was maintained in the test chamber. The lubricant was heated only by heat transfer from the disk. At disk temperatures of  $150^\circ$  and  $260^\circ$  C ( $302^\circ$  and  $500^\circ$  F) the bulk oil temperatures stabilized at approximately  $140^\circ$  and  $230^\circ$  C ( $284^\circ$  and  $446^\circ$  F), respectively.

Frictional force, bulk lubricant and disk temperatures were continuously monitored. Rider wear scar diameters were measured after time intervals of 10, 40, 70, 100, and 130 minutes. Disk wear with pure-iron riders was found to be so small that it was not measurable.

## EXPERIMENTAL LUBRICANTS

The experimental fluids used in this study were a referenced fully formulated ester, a C-ether base fluid (modified polyphenyl ether), and five

formulated C-ether fluids. Some typical properties of the ester and the C-ether base fluids are given in Table 1. The formulated fluids, which contain no more than 0.10 percent by weight of any one additive, have essentially the same properties as the C-ether base fluid. Table 2 gives the additive contents, structures, and functions for all the test fluids.

#### Formulated Ester

The fully formulated ester is a special synthesized fluid whose base stock is a mixture of hindered polyol esters, polyester, and dipentaerythritol esters. It was developed (1), (2) as a MIL-L-27502 specification candidate lubricant with a potential -40° to 240° C operating range. Generic names for the additives are given in Table 2 for this referenced fluid where exact information is considered proprietary by the fluid manufacturer.

#### C-Ether Base Fluid

The C-ether base fluid is a mixture of a three-ring polyphenyl thioether and three four-ring polyphenyl ether-thioether components; the structures are presented in Fig. 2 (5), (6), (7). A dimethyl silicone antifoaming additive is also present.

#### C-Ether Formulations

The five formulated C-ether fluids were subject previously to extensive screening tests (7). Description and concentrations of the additives along with their general function or purpose are listed in Table 2. Comments on the rationale for selecting the additives shown in Table 2 are presented for each C-ether formulation, as follows:

Formulation I. - This formulation contained two antiwear additives consisting of 0.07-weight-percent perfluoroglutaric acid (PFGA) and 0.05-weight-percent di[2-ethylhexyl] perfluoroglutarate (ester of PFGA).

Previous bearing test studies with C-ether-plus-PFGA showed a quick loss of additive (6). Therefore, this formulation was devised to use the fast-adsorbing/reacting PFGA in combination with the more slowly reacting compound PFGA ester.

Formulation II. - The second formulation consisted of 0.10-weight-percent 2-[hexafluoro-*i*-propoxy] ethyl phenylphosphinate. This multipurpose additive is a boundary lubrication improver as well as a corrosion and oxidation inhibitor, both of which reduce sludge.

Formulation III. - The third formulation contains 0.10-weight-percent *i*-propylphenylphosphinate as the antiwear additive plus 0.05-weight-percent trichloroacetic acid to improve fluid wettability.

Formulation IV. - The fourth formulation contained 0.075-weight-percent *m*-trifluoromethylphenylphosphinic acid, which functions as a boundary lubricator as well as an "antisludge" additive. In a previous study (6) one formulation containing 0.10-weight-percent phenylphosphinic acid gave good bearing lubrication, but large amounts of sludge were formed. This sludge could come from reaction of the C-ether base stock at reactive sites on the metal. To prevent this, the additive was partially fluorinated to form an even more protective absorbed film between the metal and the C-ether.

Formulation V. - The fifth formulation contained 0.10-weight-percent of a commercial acid phosphate mixture (Table 2) and 0.05-weight-percent of dibenzyl disulfide. This formulation is expected to function similar to formulation IV. The difference from formulation IV is that an active sulfur compound is combined with a phosphorous compound to function as the metal deactivator.



## RESULTS AND DISCUSSION

### Steady-State Wear

Typical wear results for all reference and test fluids in Figs. 3 and 4 show a linear relation with sliding distance in the range of about 200 to 1100 m, or 50 to 130 minutes test time. Wear rates were calculated from the slopes of these lines using least-squares estimates. These "steady-state wear rates" are presented in Table 3 for three test runs at each fluid-temperature combination. The correlation coefficient  $R$  is shown in Table 4, where  $R$  is a measure of the degree of fit for a linear relation between rider wear and sliding distance. The consistently high values of  $R$  between 0.995 and 0.999 indicate an extremely high degree of correlation. Good reproducibility of the steady-state wear rate results in Table 3 is shown by the fact that the ratio of standard deviations to mean wear values was generally in the range of 0.1 to 0.2.

Formulated ester. - This reference fluid was used because it represents the most advanced ester lubricant known to date. Although it has not been fully qualified as a MIL-L-27502 specification lubricant, it is considered to be a prime candidate. Typical wear results are plotted in Fig. 3(a), and the average wear rates for three separate test runs are summarized in Table 3 and shown graphically in Fig. 5 for each of the three disk operating temperatures.

The steady-state wear rates are  $15 \times 10^{-14}$  and  $26 \times 10^{-14} \text{ m}^3/\text{m}$  at  $20^\circ$  and  $150^\circ \text{ C}$ , respectively. These wear results and the appearance of the rider wear surfaces indicate that lubrication was comparable to that in prior studies with a MIL-L-23699 formulated type II ester that was considered only marginal (8). However, the wear rate at  $260^\circ \text{ C}$  is  $6.1 \times 10^{-14} \text{ m}^3/\text{m}$ . The

increased effectiveness of the additives at this high temperature is probably due to greater chemical reactivity to form protective surface films.

C-ether base fluid. - Typical wear results for the base fluid appear in Fig. 3(b), and the average steady-state wear rates for each temperature are given in Table 3 and in Fig. 5. Wear rates decreased slightly from  $6.9 \times 10^{-14} \text{ m}^3/\text{m}$  at  $20^\circ \text{ C}$  to  $4.1 \times 10^{-14} \text{ m}^3/\text{m}$  at  $260^\circ \text{ C}$ , indicating that the fluid molecules are more reactive at higher temperatures and apparently follow the general relationship that exists between wear and reactivity as described in Ref. (11). The steady-state wear rates were about one-half the comparable values for the reference ester fluid at temperatures of  $20^\circ$  and  $150^\circ \text{ C}$  and about two-thirds the value at  $260^\circ \text{ C}$ . These results show the inherently better lubricating ability of the sulfur-containing C-ether base fluid as compared to a fully formulated ester.

C-ether formulations. - Typical wear results for formulations I to V appear in Fig. 4, and the average steady-state wear rates for three separate runs for each test condition are shown in Table 3 and in Fig. 5. In general, these fluids follow the same pattern of wear behavior with temperature as the formulated ester fluid: that is, steady-state wear rates were low at  $20^\circ \text{ C}$ , reached maximum values at the intermediate temperature of  $150^\circ \text{ C}$ , and decreased to lower values at  $260^\circ \text{ C}$ . One exception to this general wear trend was observed for formulation I, where wear rate remained essentially constant at about  $7 \times 10^{-14} \text{ m}^3/\text{m}$  at temperatures of  $150^\circ$  and  $260^\circ \text{ C}$ .

Effectiveness of the various additives on wear is shown by the comparisons between the steady-state wear rates for the five formulations and the C-ether base fluid in Fig. 5. Formulations II and III yielded lower wear than the base fluid at all test conditions and gave the best overall wear results. Formulation IV gave somewhat less desirable wear behavior at the

150° C level than the other formulations. Formulation I gave lower wear only at 20° C. The most adverse wear behavior was shown by formulation V, which gave higher wear than the C-ether-base fluid at all test conditions. Formulations II and III, which gave the best wear results, were the only two fluids containing esters of phenylphosphinic acids. Any further efforts in formulating C-ether fluids should include additional studies on these types of antiwear compounds.

Figure 6 shows comparative plots of steady-state wear rate data for the formulated ester, the C-ether base fluid, and C-ether formulations II and III. This figure shows the increased thermal operating potential for formulated C-ethers in relationship to bulk oil temperature limits for MIL-L-23699 and revised MIL-L-27502 type esters. This difference in temperature operation above 240° C is even more significant because of the increasing ester oxidative instability at these higher temperatures.

#### Steady-State Coefficient of Friction

The steady-state friction coefficients for all the test fluids at the three operating temperatures are compared in Fig. 7 and presented in Table 3. During the run-in period the coefficients were higher. As shown in Fig. 7 the general trend of all fluids evaluated was for the values to double when operating temperatures are increased from 20° to 150° C; the average value going from about 0.08 up to 0.16 for most fluids. Further increasing the disk temperatures from 150° C to 260° C resulted in decreases in friction coefficients to 0.14 for the ester fluid and C-ether formulations II, III, and IV, while the values increased to about 0.18 for the C-ether base fluid and C-ether formulations I and V.

A qualitative study of friction can be made by observing the friction traces during steady-state operating periods. Four general types of fric-

tion traces were found. In addition, audible friction noise occurred at high temperatures in some runs when apparent fluid starvation in the system due to evaporation was evident. Representative examples are illustrated and described in Table 5, and a listing is shown in Table 6 of types encountered for each test fluid at each of the three operating disk temperatures. The four types of friction traces are characterized as (1) smooth, wide-band  $S_w$ ; (2) smooth, narrow-band  $S_n$ ; (3) erratic, spiked  $E_s$ ; and (4) erratic, wandering  $E_w$ . Each type of trace is discussed separately below.

Smooth, wide-band. - The smooth, wide-band type friction trace was observed for all fluids only at the 20° C operating level, where lower coefficients of friction (0.06 to 0.09) were measured. Friction trace variation ranged from about  $\pm 0.05$  to  $\pm 0.07$ .

Smooth, narrow-band. - Five of the seven fluids exhibited the smooth, narrow-band type of friction at 150° C, and two of the fluids showed this type at 260° C. Friction trace variation ranged from  $\pm 0.01$  to  $\pm 0.02$ .

Erratic, spiked. - The erratic, spiked friction trace was similar to the smooth, narrow-band trace in that normal variations were about  $\pm 0.02$ . However, there were periodic "spikes" or surges in the friction values to about twice the normal values. This frictional behavior was noted for several of the fluids at the 150° and 260° C operating temperatures. At 260° C it was usually accompanied by audible friction noise in the test components.

Erratic, wandering. - Erratic, wandering friction traces displayed a somewhat larger friction variation (about  $\pm 0.03$ ) than does the erratic, spiked type of friction trace. It is the most erratic or irregular friction that was observed in all the experimental runs. Audible friction noise accompanied the two examples that were noted at the 260° C test conditions.

Based on this qualitative analysis, the formulated ester fluid gave the best results, exhibiting smooth friction traces over the entire temperature range. The C-ether formulation II fluid had erratic, spiked traces with audible noise at the two upper operating temperatures. The remaining fluids showed mixed results, with both smooth and erratic friction. No correlation exists between the type of friction trace and the coefficient of friction values, but there appears to be some relation between trace type and temperature.

All the friction and wear results are summarized in Table 3, including a tabulation of the run-in wear rates. Run-in wear behavior of these lubricants could be important if it is a significant part of the total wear. Over longer running periods, such as these tests, run-in wear is less significant than in previous studies where shorter test durations were used (8), (9).

Two problems observed for the C-ether fluids were (1) the increased volatility of the fluids at the 260° C disk temperature that required additional fluid to be added to the test lubricant reservoir cup during the incremental testing, and (2) the formation of sludge material during friction and wear studies that is a potential filter-clogging agent in system applications [e.g., as encountered in the bearing tests of Ref. (7)]. Sludge formation was probably due to mild corrosive wear in an oxidizing atmosphere. Any adverse effect of this material on abrasive wear must have been minimal because total wear decreased as test temperatures increased from 150° to 260° C.

#### SUMMARY OF RESULTS AND CONCLUSIONS

Steady-state wear and friction measurements were determined under boundary lubrication conditions in a pin-on-disk sliding friction and wear

apparatus at disk temperatures of 20°, 150°, and 260° C with five C-ether formulations containing phosphinic esters or acids and other antiwear additives. Results were compared with those obtained under similar conditions for a fully formulated MIL-L-27502 candidate ester lubricant and the C-ether base stock as reference oils. Test components were annealed, pure-iron riders sliding against rotating, hardened, consumable-vacuum-melted (CVM) M-50 tool steel disks in a dry-air (<100 ppm H<sub>2</sub>O) atmosphere. Other test conditions were a load of 1 kilogram (initial Hertz stress,  $1 \times 10^9$  N/m<sup>2</sup>), a disk speed of 50 rpm, which results in sliding velocities of 7.1 to 9.1 m/min, and time sequences for each test run of 1 to 130 min. The major conclusions are:

1. The susceptibility of C-ethers to selective additive treatment was demonstrated. Two of the C-ether formulations gave somewhat better boundary lubrication than the C-ether base fluid, based on steady-state wear rates and coefficients of friction. A third C-ether formulation gave less desirable wear behavior only at the 150° C test temperature level. These formulations all contained phosphinic acids or esters, including one that was a mixture of phenylphosphinic acid ester and trichloroacetic acid.

2. The other two C-ether formulations yielded higher wear rates and friction coefficients than the C-ether base fluid for most of the temperature range. One of these formulations contained a blend of a glutaric acid and an ester of this acid, and the other one contained an acid phosphate mixture and dibenzyl disulfide.

3. The sulfur-containing C-ether fluids were found to be inherently better boundary lubricants compared to the fully formulated ester. Three of the formulated C-ethers as well as the base fluid showed significantly better lubricating ability than the ester over the entire temperature

range. The only C-ether formulation to give consistently higher steady-state wear values contained acid phosphate and dibenzyl disulfide additives.

4. The increased thermal operating potential for the formulated C-ethers was shown in relationship to bulk oil temperature limits for MIL-L-23699 and MIL-L-27502 candidate esters.

5. A qualitative method for comparing friction behavior was devised where friction traces during steady-state testing were designated as one of the following: (a) smooth, wide-band  $S_w$ ; (b) smooth, narrow-band  $S_n$ ; (c) erratic, spiked  $E_s$ ; and (d) erratic, wandering  $E_w$ . No correlation exists between the type of friction trace and the coefficient of friction values, but the trace type seems to be temperature related. It varied from smooth (wide-band) operation at 20° C for all fluids to an erratic (both spiked and wandering) behavior at 260° C for all five C-ether formulated fluids. The ester fluid gave a smooth friction trace over the entire temperature range.

#### REFERENCES

1. Clark, F. S., Miller, D. R., and Reid, S. L., "Development of a Gas Turbine Engine Oil for Bulk Oil Temperatures of -40° to 465° F, Part I," Monsanto Research Corp., MRC-SL-477 (1975). (AFML-TR-74-247, AD-A014808.)
2. Clark, F. S., Herber, J. F., and Reid, S. L., "Development of a Gas Turbine Engine Oil for Bulk Oil Temperatures of -40° to 465° F, Part II," Monsanto Research Corp., MRC-SL-538-PT-2 (1975). (AFML-TR-74-247-Pt. 2, AD-A027068.)
3. Bucknell, R. L., "Influence of Fuels and Lubricants on Turbine Engine Design and Performance," Pratt & Whitney Aircraft, PWA-FR-5673 (1973). (AFAPL-TR-73-52-VOL-2, AD-769309.)

4. Miner, J. R., "Advanced Lubricating Fluids for Turbojet Engines," 1970 Air Force Materials Symposium, Session on Fluids and Lubricants, Air Force Systems Command, Wright-Patterson AFB, Ohio, 1970, Session 1F, Paper 2.
5. McHugh, K. L. and Stark, L. R., "Properties of a New Class of Polyaromatics for Use as High-Temperature Lubricants and Functional Fluids," ASLE Trans., 9, 1, pp 13-23 (1966).
6. Clark, F. S., Green, R. L., and Miller, D. R., "Synthesis and Evaluation of C-Ether Formulations for Use as High-Temperature Lubricants and Hydraulic Fluids," Monsanto Research Corp., MRC-SL-460 (1974). (NASA CR-134643).
7. Clark, F. S. and Miller, D. R., "Formulation and Evaluation of C-Ether Fluids as Lubricants Useful to 260° C," Monsanto Research Corp., MRC-SL-1007 (1980). (NASA CR-159794).
8. Jones, W. R., Jr., "Boundary Lubrication of Formulated C-Ethers in Air to 300 C," Lubr. Eng., 32, 10, pp 530-538 (1976).
9. Jones, W. R., Jr. and Hady, W. F., "Effect of Humidity and a Wettability Additive on Polyphenyl Ether Boundary Lubrication of Steel in Air and Nitrogen to 350° C," NASA TN D-6055 (1970).
10. Loomis, W. R. and Jones, W. R., Jr., "Steady-State Wear and Friction in Boundary Lubrication Studies," NASA TP-1658 (1980).
11. Rowe, C. N., "Wear: Corrosion and Erosion, "Interdisciplinary Approach to Liquid Lubricant Technology, NASA SP-318 (1973), Ed. Ku, P. M., pp 527-568.
12. Thompson, Q. E., Reid, S. L., and Weiss, R. W., "Antioxidants for Ester-Base Functional Fluids," U.S. Patent 3, 684, 711 (August 1972).



TABLE 1. - TYPICAL PROPERTIES OF TEST FLUIDS

Property*	Formulated ester fluid†	C-ether base fluid
Kinematic viscosity, m <sup>2</sup> /sec (cS):		
At 38° C (100° F)	39.6×10 <sup>-6</sup> (39.6)	25×10 <sup>-6</sup> (25)
At 99° C (210° F)	7.02×10 <sup>-6</sup> (7.02)	4.1×10 <sup>-6</sup> (4.1)
At 150° C (302° F)	2.80×10 <sup>-6</sup> (2.80)	1.9×10 <sup>-6</sup> (1.9)
At 260° C (500° F)	1.06×10 <sup>-6</sup> (1.06)	0.76×10 <sup>-6</sup> (0.76)
At 300° C (572° F)	‡0.86×10 <sup>-6</sup> (0.86)	0.69×10 <sup>-6</sup> (0.69)
Pour point, °C (°F)	-51 (-60)	-29 (-20)
Flash point, °C (°F)	274 (525)	239 (445)
Fire point, °C (°F)		285 (540)
Density at 38° C (100° F), kg/m <sup>3</sup> (g/milliliter)	§0.994	1.19×10 <sup>3</sup> (1.19)
Thermal decomposition (isoteniscope), °C (°F) <sup>  </sup>	298 (536)	390 (734)
Vapor pressure, N/m <sup>2</sup> :		
At 220° C (428° F)	1.33×10 <sup>2</sup>	-----
At 371° C (600° F)	-----	1.86×10 <sup>4</sup>
Surface tension at 23° C (73° F), N/cm (dynes/cm)	-----	<sup>  </sup> 4.48×10 <sup>-4</sup> (44.8)

\*Manufacturer's data except where noted otherwise.

†A specification MIL-L-27502 candidate lubricant with a base stock mixture of hindered polyol esters, polyester, and dipentaerythritol esters (1), (2).

‡Extrapolated.

§Specific gravity, 15.6° C (60° F).

<sup>||</sup>Measured in author's laboratory.

TABLE 2. - ADDITIVE CONTENTS AND FUNCTIONS OF TEST FLUIDS

Test fluid	Additive	Additive content, wt%	Additive structure	Additive function
Formulated ester* (specification-MIL-L-27502 candidate)	Proprietary metal derivatives consisting of (1) a complexing agent and (2) a metal compound that is complexed by that agent†	(1)	(t)	Deposit inhibitor
	Alkylated amine		(e)	Oxidation inhibitor
	Aromatic amine		(e)	Oxidation inhibitor
	Triphenylphosphine oxide		(e)	Metal passivator (corrosion and oxidation inhibitor)
	Metal deactivator‡		(f)	Magnesium corrosion inhibitor
C-ether base fluid	Dimethyl silicone (Grade, 350 cS at 25° C)	(1)	$(\text{CH}_3)_3\text{SiO}[(\text{CH}_3)_2\text{SiO}]_n\text{Si}(\text{CH}_3)_3$	Antifoaming agent
C-ether** formulation I	Perfluoroglutaric acid	0.07	$\text{HO}_2\text{C}(\text{CF}_2)_3\text{CO}_2\text{H}$	Fast-reacting boundary lubrication
	di (2-ethylhexyl) perfluoroglutarate	.05	$(\text{C}_4\text{H}_9-\underset{\text{C}_2\text{H}_5}{\text{CH}}-\text{CH}_2-\text{O}-\overset{\text{O}}{\parallel}{\text{C}})_2-(\text{CF}_2)_3$	Slow-reacting boundary lubrication
C-ether formulation II	2-(hexafluoro-1-propoxy) ethyl phenylphosphinate	0.10		Boundary lubrication and antisludge agent
C-ether formulation III	i-propylphenylphosphinate	0.10		Boundary lubrication
	Trichloroacetic acid	.05	$\text{CCl}_3-\overset{\text{O}}{\parallel}{\text{C}}-\text{OH}$	Boundary lubrication (wettability agent)
C-ether formulation IV	m-trifluoromethyl-phenylphosphinic acid	0.075		Boundary lubrication and antisludge agent
C-ether formulation V	Commercial acid phosphate mixture	0.10	$\text{C}_9\text{H}_{19}-\text{O}-(\text{CH}_2\text{CH}_2\text{O})_5-\overset{\text{O}}{\parallel}{\text{P}}(\text{OH})\text{OX}$ (X=H or ethylene oxide chain)	Boundary lubrication
	Dibenzyl disulfide	.05		Antisludge agent when combined with phosphorous additive

\*Refs. (1), (2).

†Description of complexing agent given in ref. (12).

‡Proprietary information (1), (2).

§More exact description is proprietary (2).

||Weight percentage of antifoaming agent is proprietary.

\*\*All C-ether formulations contain antifoaming agent.

TABLE 3. - SUMMARY OF FRICTION AND WEAR RESULTS FOR TEST FLUIDS

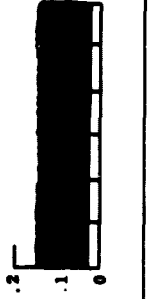
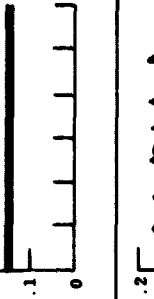
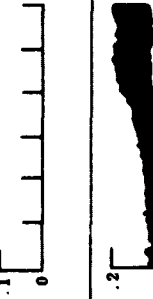
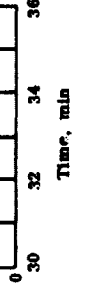
Disk temperature		Test fluid						
		Formulated advanced ester <sup>a</sup>	C-ether base fluid	C-ether formulation				V
°C	°F			I	II	III	IV	
Steady-state coefficient of friction <sup>†</sup>								
20	68	0.09(±0.07)	0.06(±0.045)	0.09(±0.06)	0.08(±0.06)	0.09(±0.06)	0.06(±0.055)	0.07(±0.06)
150	302	.16(±0.02)	.15(±0.02)	.15(±0.015)	.16(±0.025)	.16(±0.02)	.16(±0.015)	.12(±0.01)
260	500	.14(±0.01)	.17(±0.01)	.19(±0.02)	.14(±0.015)	.13(±0.025)	.14(±0.03)	.14(±0.02)
Steady-state rider wear rate <sup>‡</sup> , m <sup>3</sup> /min								
20	68	15(±2.9)×10 <sup>-14</sup>	6.9(±0.21)×10 <sup>-14</sup>	2.1(±0.40)×10 <sup>-14</sup>	1.2(±0.27)×10 <sup>-14</sup>	1.5(±0.21)×10 <sup>-14</sup>	1.9(±1.4)×10 <sup>-14</sup>	8.5(±0.99)×10 <sup>-14</sup>
150	302	29(±24)	6.5(±1.3)	7.1(±0.74)	5.8(±1.4)	6.4(±0.91)	11.8(±1.3)	30(±3.3)
260	500	6(±1.3)	4.1(±1.0)	7.3(±1.2)	2.6(±1.3)	2.9(±0.70)	3.5(±1.7)	12(±1.8)
Run-in wear rate <sup>‡</sup> , m <sup>3</sup> /min								
20	68	31(±10)×10 <sup>-14</sup>	16(±2.4)×10 <sup>-14</sup>	17(±3.8)×10 <sup>-14</sup>	16(±2.3)×10 <sup>-14</sup>	16(±2.8)×10 <sup>-14</sup>	8.4(±2.6)×10 <sup>-14</sup>	28(±3.5)×10 <sup>-14</sup>
150	302	25(±12)	12(±5.0)	15(±4.9)	18(±7.6)	23(±4.8)	18(±3.5)	25(±2.3)
260	500	8.1(±5.2)	3.3(±1.3)	7.1(±0.81)	3.3(±3.7)	8.6(±6.0)	7.5(±1.1)	8.1(±5.6)

<sup>a</sup>A mixture of hindered polyol esters, polyester, and dipentaerythritol esters formulated for use as a specification MIL-L-27502 candidate lubricant (1), (2).

<sup>†</sup>Mean values. Scatter in parentheses.

<sup>‡</sup>Mean values. Standard deviation in parentheses.

TABLE 3. - EXAMPLES OF FOUR TYPES OF FRICTION TRACES FROM SLIDING FRICTION EXPERIMENTS

Friction type*	Friction trace segment	Test fluid	Disk temperature, °C	Steady-state coefficient of friction†
Smooth, wide band ( $S_w$ )		C-ether formulation III	20	0.09 (±0.06)
Smooth, narrow band ( $S_n$ )		Formulated advanced ester	260	0.14 (±0.01)
Erratic, spiked ( $E_s$ )		C-ether formulation V	260	0.14 (±0.02)
Erratic, "wandering" ( $E_w$ )		C-ether formulation IV	260	0.14 (±0.03)‡

\*Further variations include these types of friction accompanied by audible friction noise.  
 †Mean values. Scatter in parentheses.  
 ‡Mean value obtained over more extended period of time.

TABLE 4. - CORRELATION COEFFICIENTS FOR WEAR RATES AS CALCULATED BY LINEAR REGRESSION ANALYSIS FOR SLIDING FRICTION EXPERIMENTS

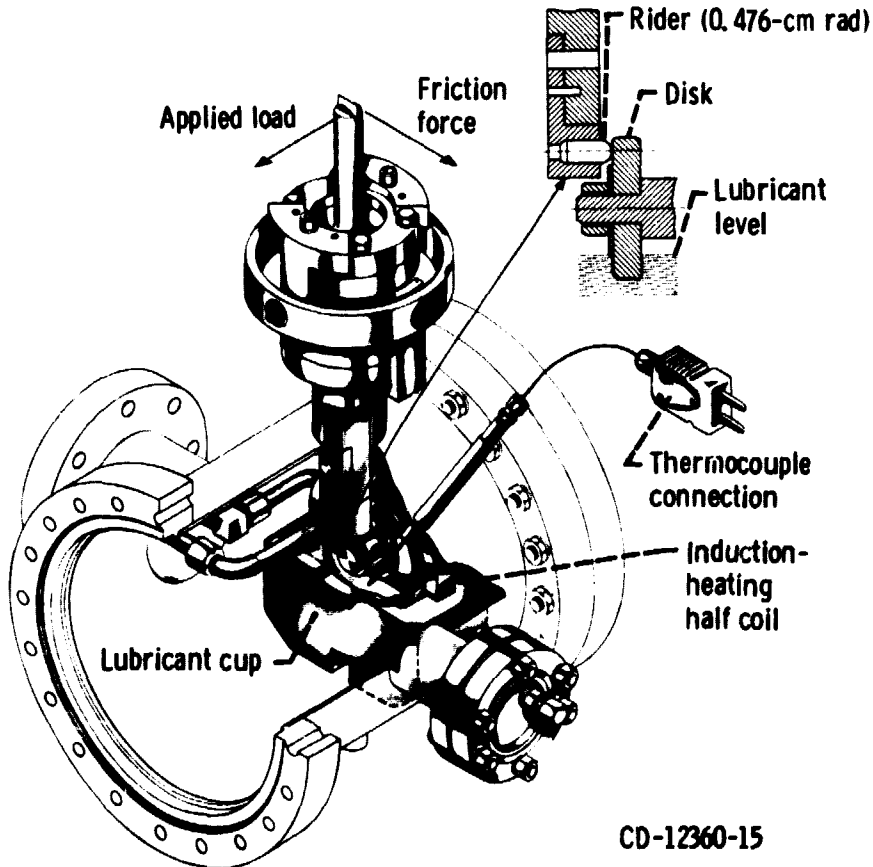
Test fluid	Disk temperature, °C		
	20	150	260
	Correlation coefficient at disk temperature, R		
Formulated ester	0.9987	0.9997	0.9597
C-ether base	.9979	.9960	.9937
C-ether formulation I	.9998	.9973	.9985
C-ether formulation II	.9699	.9660	.9693
C-ether formulation III	.9632	.9975	.9960
C-ether formulation IV	.9920	.9983	.9655
C-ether formulation V	.9998	.9990	.9992

**ORIGINAL PAGE  
BLACK AND WHITE PHOTOGRAPH**

**TABLE 6. - TYPES OF FRICTION TRACES  
FROM SLIDING FRICTION EXPERIMENTS**

Test fluid	Disk temperature, °C		
	20	150	260
	Friction trace type*		
Formulated ester	S <sub>w</sub>	S <sub>n</sub>	S <sub>n</sub>
C-ether base fluid	S <sub>w</sub>	E <sub>s</sub>	S <sub>n</sub>
C-ether formulation I	S <sub>w</sub>	S <sub>n</sub>	E <sub>s</sub>
C-ether formulation II	S <sub>w</sub>	E <sub>s</sub>	E <sub>s</sub> , N
C-ether formulation III	S <sub>w</sub>	S <sub>n</sub>	E <sub>w</sub> , N
C-ether formulation IV	S <sub>w</sub>	S <sub>n</sub>	E <sub>w</sub> , N
C-ether formulation V	S <sub>w</sub>	S <sub>n</sub>	E <sub>s</sub> , N

\*Types of friction traces illustrated in Table 5.



CD-12360-15

**Figure 1. - Friction and wear apparatus.**

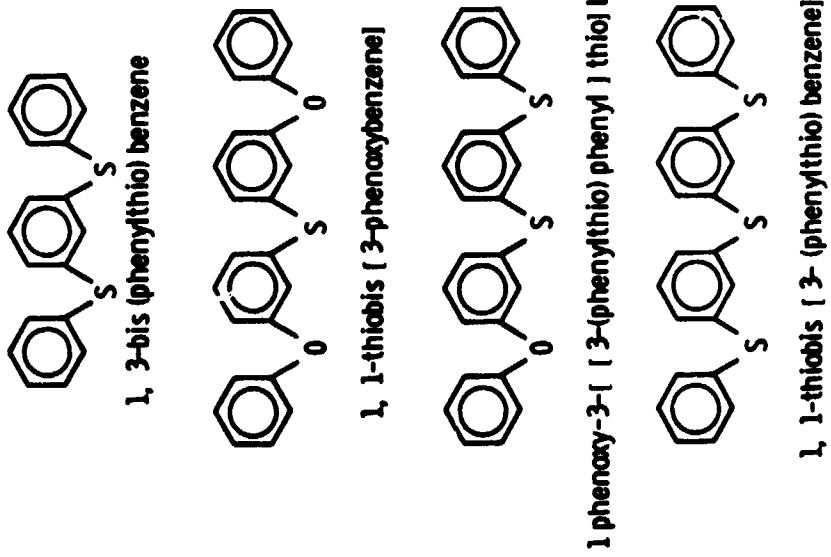


Figure 2. - Structures of C-ether base fluid components.

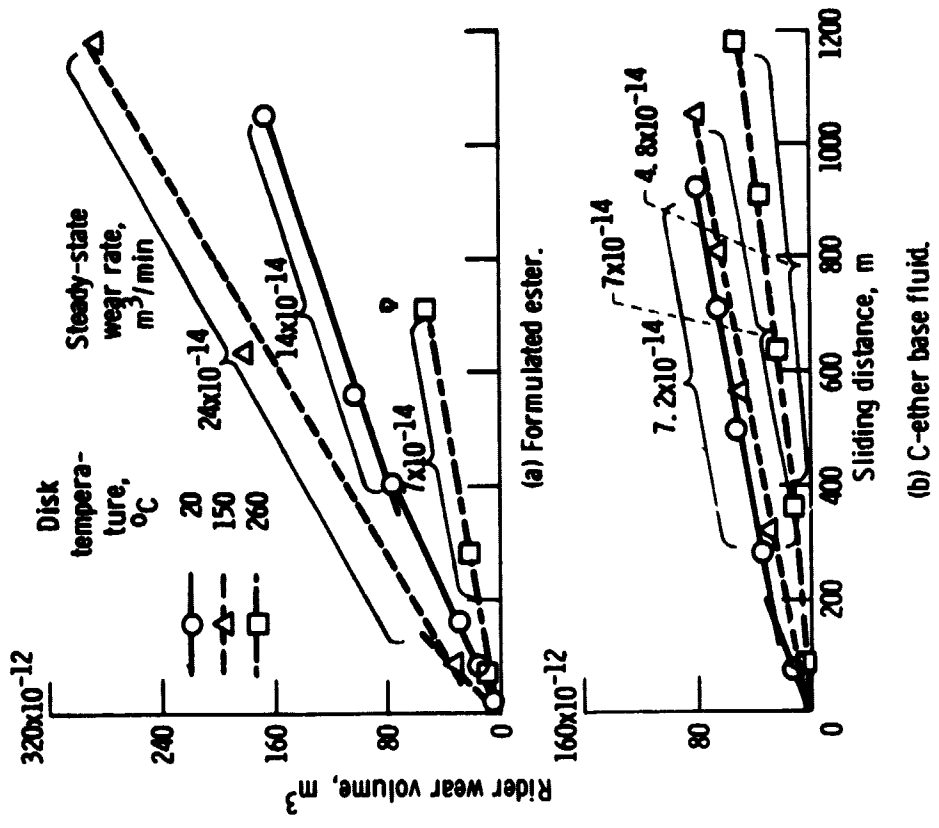


Figure 3. - Typical rider wear as a function of sliding distance for two reference fluids at three disk temperatures.

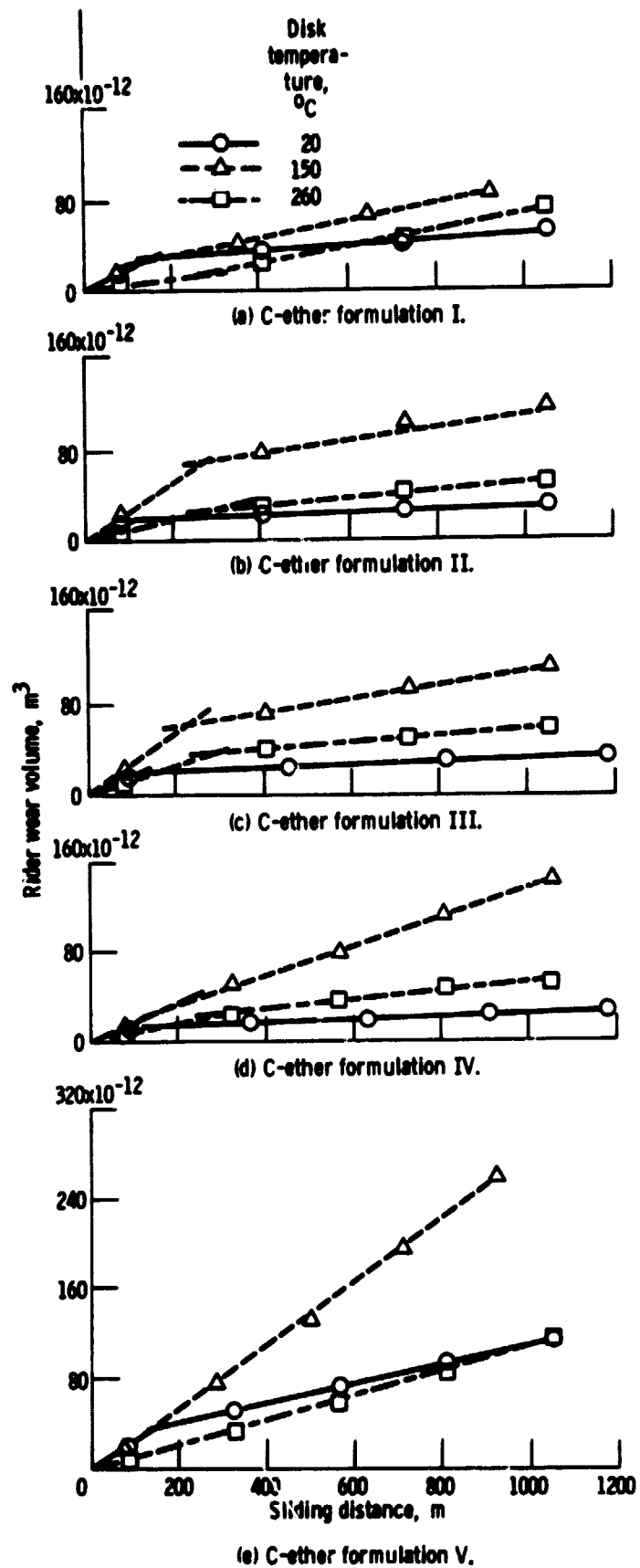


Figure 4. - Typical rider wear as a function of sliding distance for five C-ether formulations at three disk temperatures.

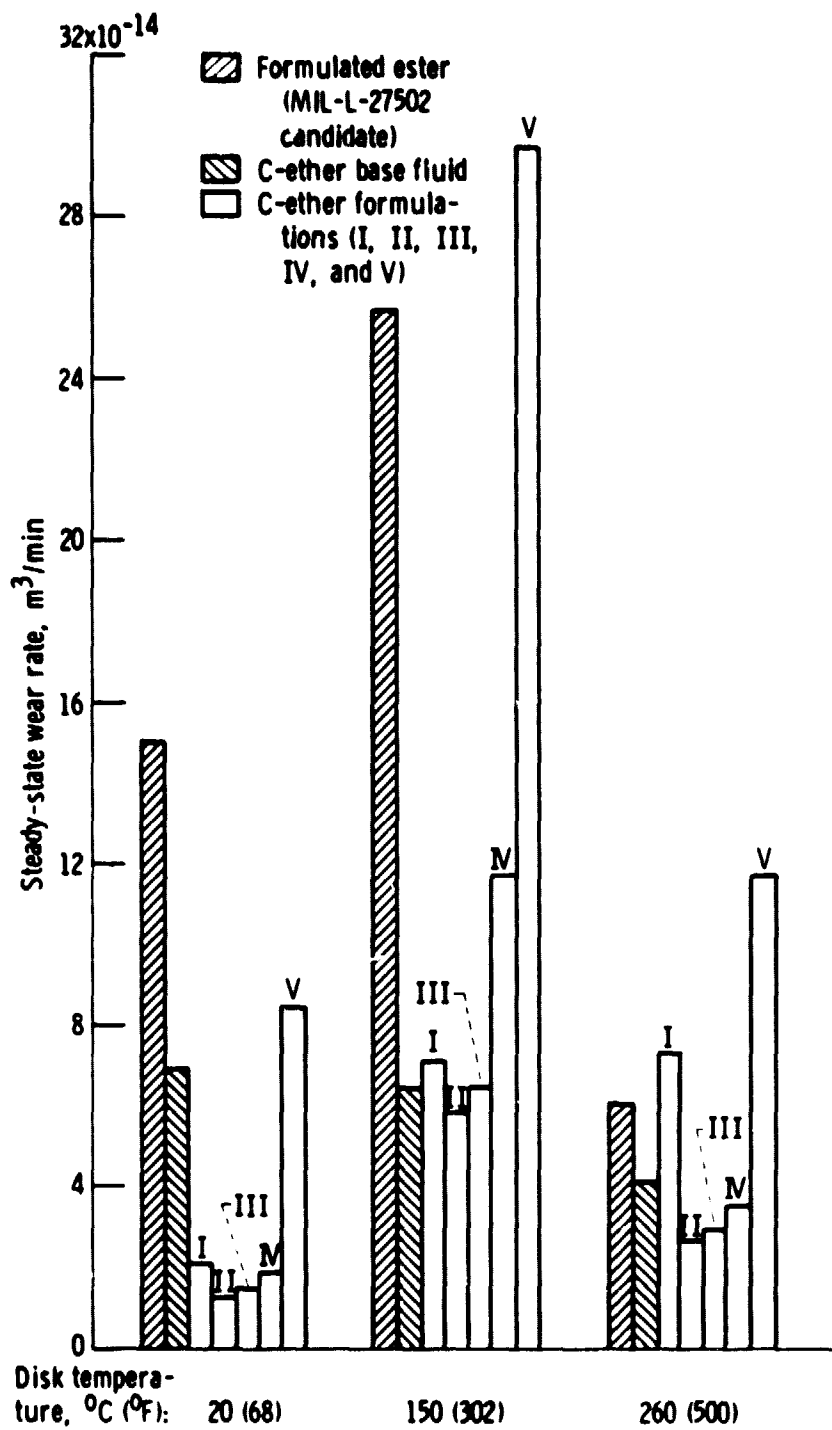


Figure 5. - Steady-state rider wear rate at three disk temperatures for formulated ester, C-ether base fluid, and five C-ether formulations.



- FULLY FORMULATED ESTER (MIL-L-27502 CANDIDATE)
- - - - C-ETHER BASE FLUID
- ==== } 2 C-ETHER FORMULATIONS

BULK OIL TEMPERATURE LIMITS:

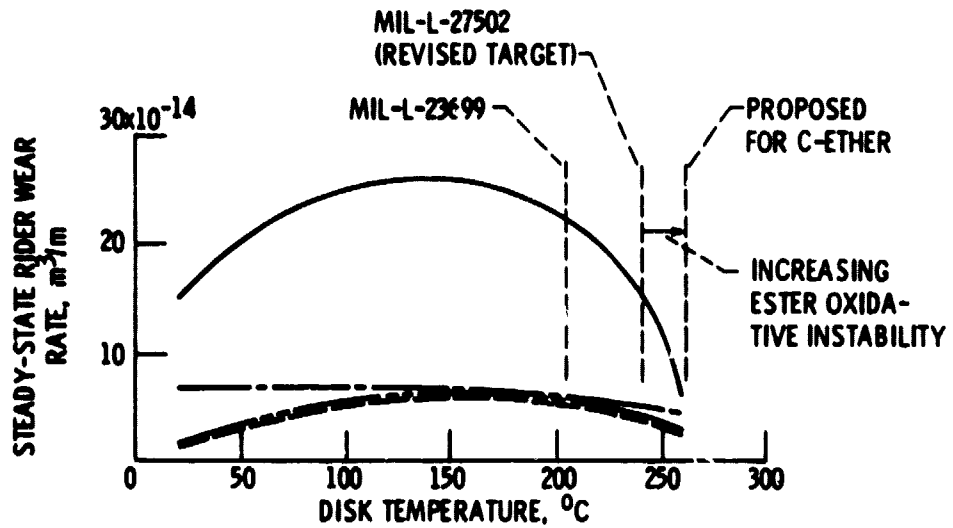


Figure 6. - Steady-state wear as a function of disk temperatures from 20° to 260° C for a formulated ester and C-ether fluids.

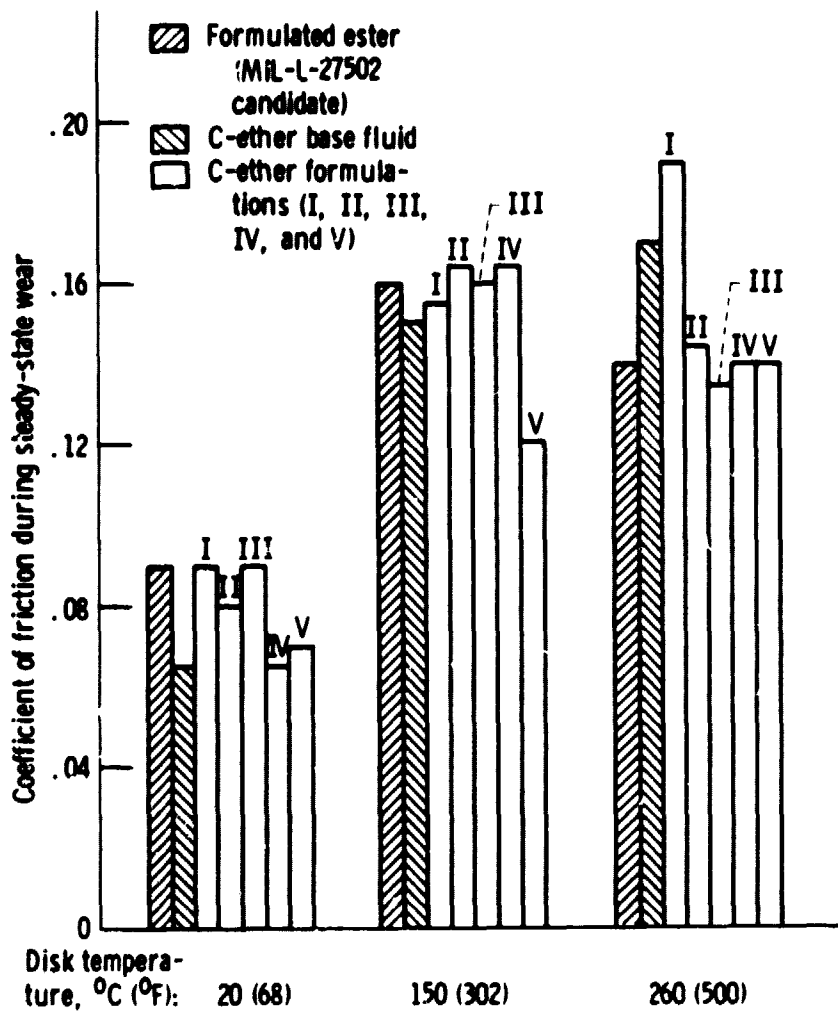


Figure 7. - Coefficient of friction during steady-state wear at three disk temperatures for formulated ester, C-ether base fluid, and five C-ether formulations.