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Surface Catalytic Degradation Study of Two Linear Perfluoropolyalkylethers at 345 °C

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Summary

Thin-liquid-film degradation studies of two commercially available perfluoropolyalkylether fluids (PFAE) were performed at 345 °C, in nitrogen and air atmospheres, on iron and 440 C stainless steel surfaces. It was found that one fluid degraded on both iron and 440 C stainless steel surfaces in an air atmosphere, whereas the other fluid did not degrade. Chemical analysis revealed that the test fluid degraded to lower molecular weight products and that the degradation was accompanied by the formation of a brownish deposit on both the iron and 440 C stainless steel surfaces. Surface analysis of the deposit revealed a substantial amount of iron oxide (Fe_2O_3). It was hypothesized that the fluid which degraded did so because of its acetal structure. The other fluid, lacking the acetal structure, did not degrade.

Introduction

The use of synthetic liquid lubricants evolved from the continuing demands placed on conventional lubricants (petroleum-extracted liquids) to operate successfully in increasingly hostile environments. These hostile environments can be of many types (ref. 1) and can require the use of synthetic lubricants with particular chemical or physical properties. However, the increased operating temperature range of advanced aircraft is the main reason for the development of synthetic lubricants, which have excellent thermal and oxidative properties.

The search for synthetic lubricants that can meet the demands of high-temperature operating conditions (fluid temperatures in excess of 316 °C (refs. 2 to 5)) has led to several classes of synthetics, including phosphate esters, polyglycol ethers, silicate esters, polyphenyl ethers, and halogenated fluids. Of all the synthetic fluids available, the perfluoropolyalkylethers (PFAE) appear to hold the most promise for successful operation in high-temperature applications (refs. 6 and 7).

Two classes of PFAE fluids have been commercially available for some time: PFAE with branched molecules (fluid K) and PFAE with linear unbranched molecules (fluid F) (figs. 1(a) and (b)). Studies conducted on the thermal oxidative degradation of both classes of fluids (ref. 8) showed that the thermal oxidative stability of the linear unbranched PFAE was lower than that of the corresponding branched PFAE. This result was puzzling, since it was believed that the presence of side chains (pendant groups) in the branched fluids would lower their thermal oxidative stabilities (ref. 9).

Recently a new class of linear unbranched PFAE fluid (fluid D) has become commercially available. The general structure of this class is shown in figure 1(c). The availability of this new PFAE fluid D presented an opportunity to conduct further research on the thermal oxidative breakdown of these fluids and to supplement the work performed by Jones, Paciorek, et al. (ref. 8), who studied the degradation of PFAE fluid F and concluded that the structural arrangement of PFAE fluid F made it less oxidatively stable than PFAE fluid K.

This study focuses on the high-temperature (345 °C) catalytic degradation behavior of the two linear unbranched PFAE fluids F and D, in nitrogen and air atmospheres, on 440 C stainless steel and iron surfaces. Fluid degradation was carried out under thin-liquid-film conditions (a large fluid-surface-to-fluid-volume ratio) in order to minimize mass transfer complications.

Experiment

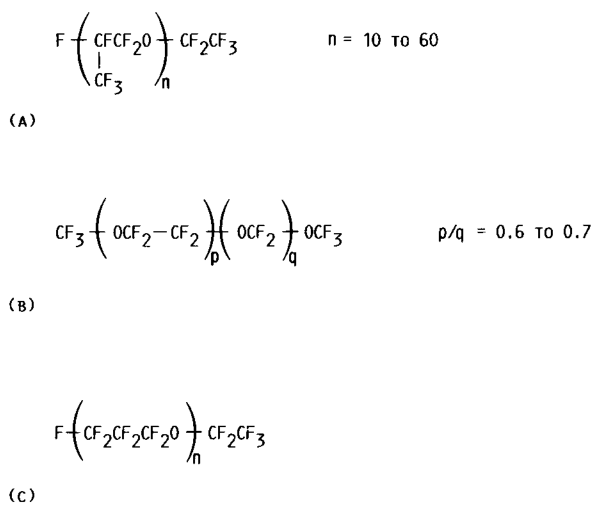
Materials

The PFAE fluids used in this study (fluids F and D) were commercially available products with the structures given by figures 1(b) and (c). Different viscosity grades of both fluids F and D were available (the different grades corresponding to different average molecular weights of the fluids); the grades of fluids F and D were chosen in order to have a minimum average molecular weight difference between the two fluids. Table I lists the properties of fluids F and D.

The metal specimens (fig. 2) used in this study consisted of pure iron (99.8 percent) and 440 C stainless steel.

Thin-Liquid-Film Test Apparatus

A thin-liquid-film test apparatus (fig. 2) was used to study the degradation of fluids F and D. The main feature of this apparatus is the formation of a thin liquid film (a large liquid-surface-to-liquid-volume ratio) on a metal surface. It is hoped that this particular arrangement minimizes any effects of gaseous oxygen diffusion throughout the liquid film (when a test is conducted in an air or oxygen atmosphere) and thus allows the study of a reaction-controlled experiment. Details concerning the origin and concepts behind this particular test



$$F\left(\underset{\substack{|\\CF_3}}{CF}CF_2O\right)_n CF_2CF_3 \qquad n = 10 \text{ to } 60$$

$$CF_3\left(OCF_2-CF_2\right)_p\left(OCF_2\right)_q OCF_3 \qquad p/q = 0.6 \text{ to } 0.7$$

$$F\left(CF_2CF_2CF_2O\right)_n CF_2CF_3$$

(a) Branched PFAE (fluid K).
(b) Linear unbranched PFAE (fluid F).
(c) Linear unbranched PFAE (fluid D).

Figure 1.—Structures of perfluoropolyalkylethers (PFAE).

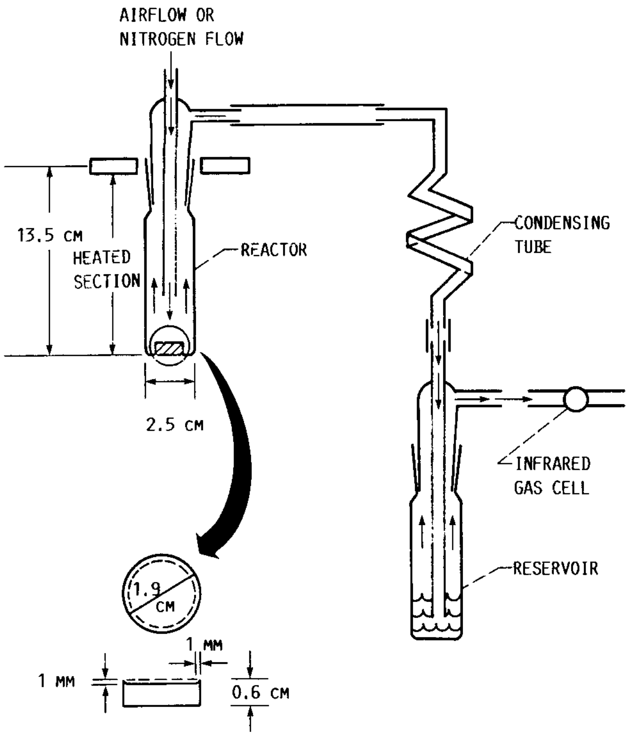


Figure 2.—Thin-liquid-film oxidation apparatus.

apparatus can be found in references 10 and 11 and in D.I.K.A. Ugwuzor, The Effect of Metals on High Temperature Degradation of Ester-Type Lubricants, Masters Thesis, Pennsylvania State University, 1982.

Test Procedure

Three ml each of fluids F and D were degassed at 200 °C and <25 torr for 30 min. The fluids were then eluted through a silica-filled column for further water removal.

TABLE I.—PROPERTIES OF FLUIDS F AND D

Property	Fluid F	Fluid D
Average molecular weight	9500	8400
Kinematic viscosity at 20 °C, cS	255	500 ± 25
Viscosity index	355	210
Pour point, °C	−66	−53
Density at 20 °C, g/ml	1.851	1.894
Surface tension at 20 °C, dyne/cm	25	19
Refractive index, n_D^{20}	1.294	1.298
Vapor pressure, torr		
At 20 °C	2.9×10^{-12}	5×10^{-11}
At 100 °C	1.0×10^{-8}	1.0×10^{-7}

The surfaces of the metal specimens were polished with diamond paste; this was followed by ultrasonic cleaning of the specimens in a beaker of 1,1,2-trichlorotrifluoroethane (TCF). The metal specimens were then rinsed with 200-proof ethanol and given a final polishing with alumina paste. The alumina paste was rinsed off with deionized water, and the specimens were dried in a nitrogen gas stream.

A metal specimen was then placed in the reactor section of the thin-film apparatus, and 50 μl of the test fluid was injected onto the metal surface. The entire apparatus was then purged with nitrogen gas (100 ml/min) for 30 min. This was followed by reducing the nitrogen gas flow rate to 20 ml/min and placing the thin-liquid-film apparatus into a heating unit maintained at 345 ± 1 °C. A 10-min heating period allowed the reactor portion of the thin-liquid-film apparatus to equilibrate at the test temperature. At the end of the 10-min equilibration period, the test atmosphere was either maintained with nitrogen or switched to air (both at 20 ml/min).

During the experiment the outlet of the reactor was attached to a condensing tube (at room temperature), the outlet of which was attached to a reservoir containing 10 ml of TCF. This arrangement allowed the flowing test atmosphere to bubble into the TCF. At the end of a test (30-min test duration, not including the 10-min equilibration period), the thin-liquid-film apparatus was removed from the heating unit and allowed to cool to room temperature. Subsequently, all glassware was disconnected, and the contents of the reactor, the condensing tube, and the TCF reservoir were examined by using high-pressure liquid chromatography (HPLC) in the size-exclusion mode—a separation mode whereby solute molecules are separated according to their molecular size.

Five ml of the TCF solvent was injected into the reactor so that the entire metal specimen was immersed in the solvent. After a few minutes the solvent was drawn off with a syringe and filtered through a 0.45-μm nylon filter; 30 μl of this filtered solution was injected into the HPLC unit for analysis.

The condensing tube contents were extracted with 3 ml of the TCF solvent, and the resulting solution was passed through a silica extraction cartridge. Nonpolar substances were eluted with the TCF solvent, whereas polar substances were adsorbed by the silica. The eluted TCF solution was then stored for HPLC analysis. Five ml of 200-proof ethanol was then passed through the silica extraction cartridge. The first 2 ml of the eluting ethanol was discarded, and the remaining 3 ml was collected and stored for chemical analysis. Polar substances adsorbed on the silica dissolved in the ethanol (a polar liquid) and eluted out of the cartridge. This procedure was repeated for the TCF reservoir.

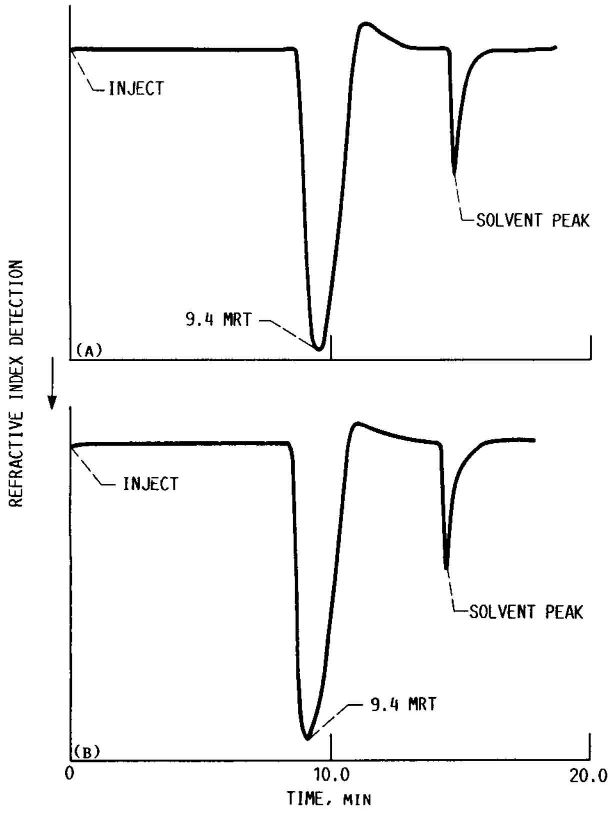
Results

Nitrogen Atmosphere Results For Clean Metal Specimens

Figures 3 and 4 are the HPLC chromatograms of fluids F and D remaining on the surfaces of both the 440 C stainless steel and the iron specimens at the end of the tests. Comparison with the pure, unreacted fluids for both test fluids shows no degradation of either fluid (each test was repeated three times).

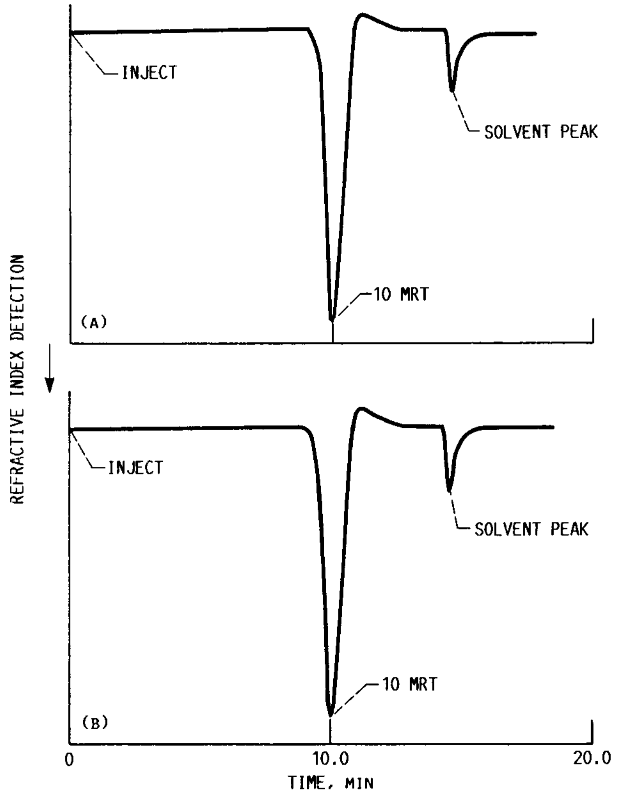
Air Atmosphere Results For Clean Metal Specimens

Fluid F on 440 C stainless steel surface.—In an air atmosphere, fluid F completely degraded; HPLC analysis revealed no fluid left on the 440 C stainless steel surface (fig. 5). The degradation of fluid F was accompanied by the formation of a liquid in the condensing tube and the bubbling of fumes into the TCF reservoir.



(a) 440 C stainless steel.
(b) Iron.

Figure 3.—HPLC chromatograms (size-exclusion mode) of fluid F remaining on surface of clean metal specimens, where MRT is minutes retention time. Test duration, 30 min; atmosphere, nitrogen; temperature, 345 °C.



(a) 440 C stainless steel.
(b) Iron.

Figure 4.—HPLC chromatograms (size-exclusion mode) of fluid D remaining on surface of clean metal specimens, where MRT is minutes retention time. Test duration, 30 min; atmosphere, nitrogen; temperature, 345 °C.

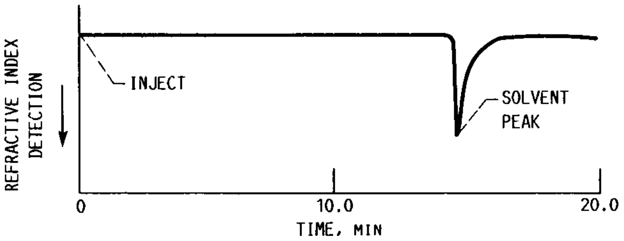


Figure 5.—HPLC chromatogram (size-exclusion mode) of fluid F remaining on surface of clean 440 C stainless steel specimens. Test duration, 30 min, atmosphere, air; temperature, 345 °C.

The liquid product in the condensing tube was extracted with TCF solvent and prepared for analysis as outlined in the subsection **Test Procedure**. HPLC analysis of the eluted TCF extract (fig. 6) revealed a product having a greater retention time (12.2 min) than fluid F. Based on a calibration chart relating the average molecular weights of known fluorinated substances to their retention times, the 12.2-min retention time corresponds to an average molecular weight of 1150.

HPLC analysis of the TCF reservoir revealed the presence of a product (fig. 7) at 18.4 min, several minutes past the solvent peak (14.4-min retention time); this indicates adsorption interaction with the column packing. By bubbling

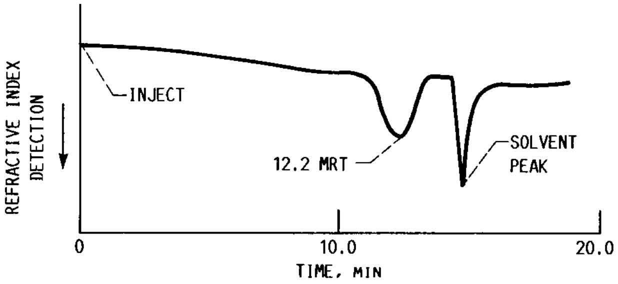


Figure 6.—HPLC chromatogram (size-exclusion mode) of liquid extracted from condensing tube, where MRT is minutes retention time. Fluid F; 440 C steel specimen; test duration, 30 min; atmosphere, air; temperature, 345 °C.

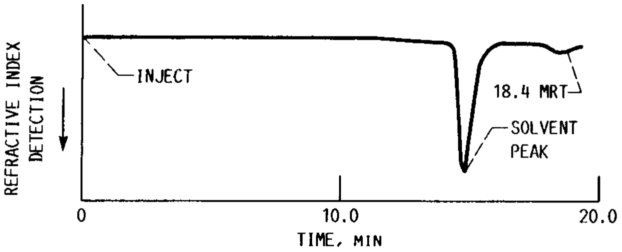


Figure 7.—HPLC chromatogram (size-exclusion mode) of reservoir contents, where MRT is minutes retention time. Fluid F; 440 C steel specimen; test duration, 30 min; atmosphere, air; temperature, 345 °C.

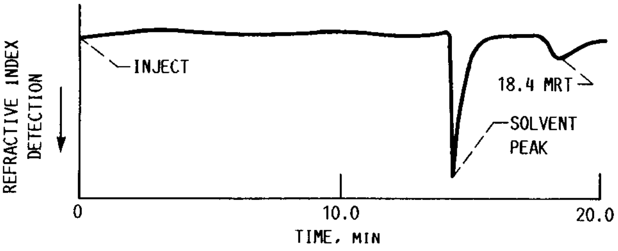
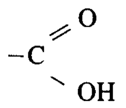


Figure 8.—HPLC chromatogram (size-exclusion mode) of resulting solution obtained by bubbling carbon dioxide gas into 5-ml 1,1,2-trichlorotrifluoroethane (TCF), where MRT is minutes retention time.

carbon dioxide gas into 5 ml of TCF solvent for a few minutes and immediately analyzing the solvent by HPLC (fig. 8), chromatographic evidence was obtained which suggested that the product eluted at 18.4 min was carbon dioxide.

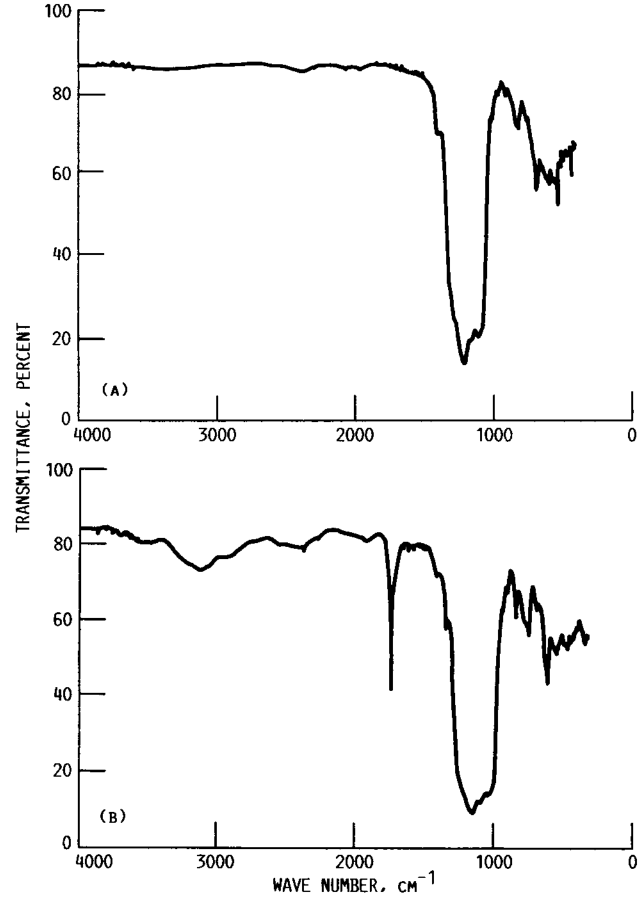
Figure 9 represents the Fourier transform infrared (FTIR) spectra of fluid F and the liquid product formed inside the condensing tube. The FTIR spectrum of the liquid product is similar to the FTIR spectrum of fluid F; however, a strong, sharp peak at 1782 cm^{-1} and a broad peak between 2650 and 3550 cm^{-1} are present in the FTIR spectrum of the liquid product. These bands correspond to carbonyl (C=0) and hydroxyl (OH) groups, respectively. To confirm the presence of these functional groups, the FTIR spectrum of a known diacid of fluid F was taken. Figure 10 gives the molecular structure of the diacid and its FTIR spectrum.

A FTIR spectrum (fig. 11) of the liquid product was also taken after eluting it through the silica extraction cartridge with TCF solvent. This spectrum shows two things: it is nearly identical to the FTIR spectrum of fluid F, and the carbonyl and hydroxyl groups are absent. The evidence suggests that the liquid product formed in the condensing tube was not a single component. Therefore, the FTIR spectrum of the condensing tube product (fig. 9(b)) appears to be a composite spectrum of at least two substances, one substance with a molecular structure very similar to that of fluid F (but with a much lower molecular weight) and a second substance with a carboxylic acid functional group:



$$-\mathrm{C}\begin{matrix}\nearrow\!\!\!\!\nearrow \mathrm{O}\\ \searrow \mathrm{OH}\end{matrix}$$

During these tests the outlet of the TCF reservoir was connected to an infrared (IR) gas cell, which allowed the collection of the fumes. Figure 12 is the FTIR spectrum of



(a) Fluid F.

(b) Liquid product from condensing tube.

Figure 9.—FTIR spectra of fluid F and liquid product extracted from condensing tube.

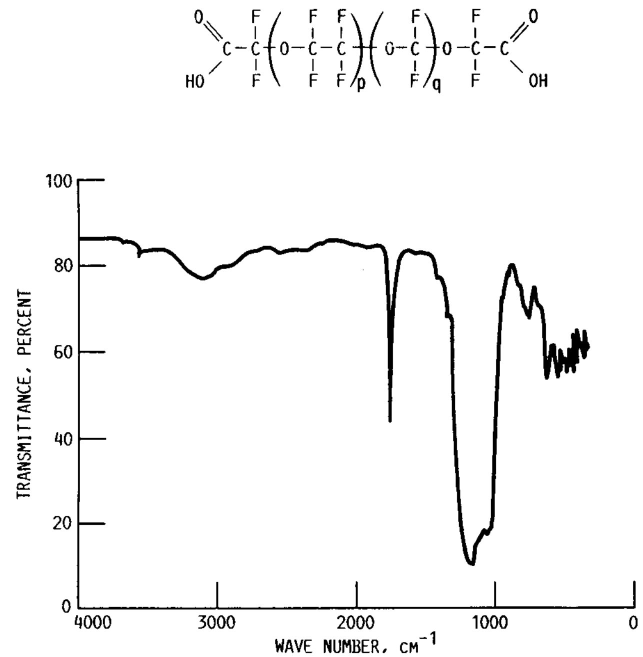


Figure 10.—Molecular structure of fluid F diacid and its FTIR spectrum.

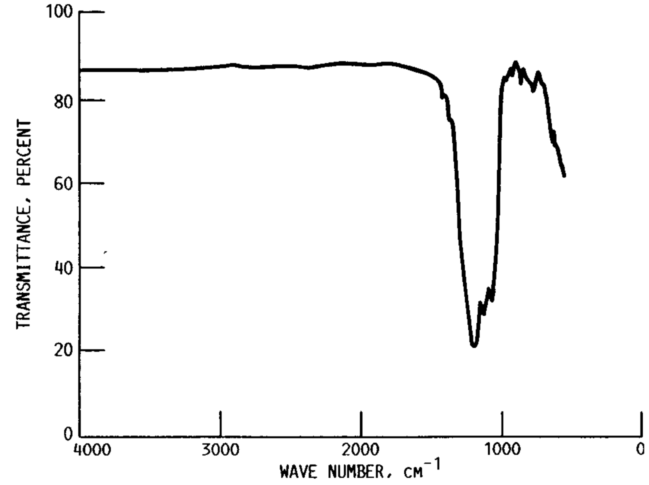


Figure 11.—FTIR spectrum of liquid product (from condensing tube) after elution through silica extraction cartridge.

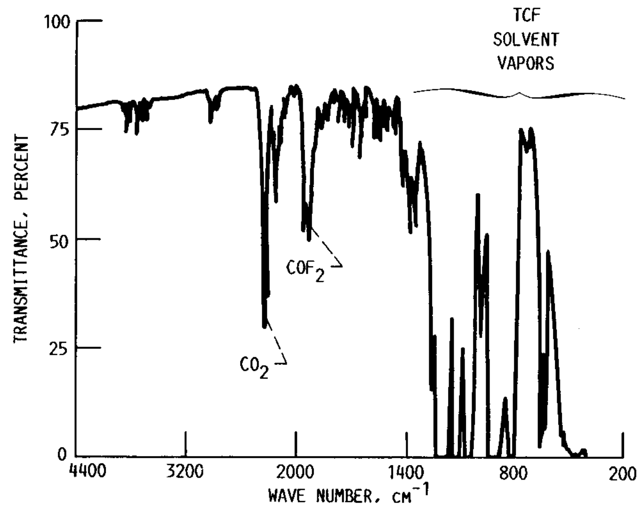


Figure 12.—FTIR spectrum of fumes collected in gas cell, showing 1,1,2-trichlorotrifluoroethane (TCF) solvent vapors, carbon dioxide (CO_2), and carbonyl fluoride (COF_2).

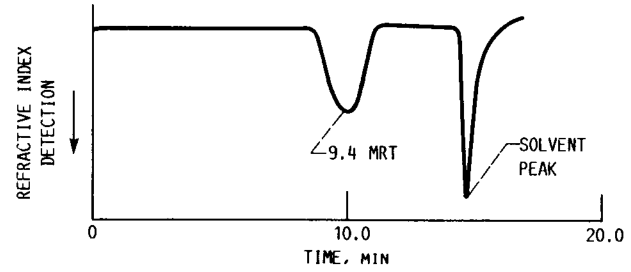


Figure 13.—HPLC chromatogram (size-exclusion mode) of fluid F remaining on surface of clean iron specimen, where MRT is minutes retention time. Test duration, 30 min; atmosphere, air; temperature, 345 °C.

the fumes. In addition to the TCF solvent vapors, bands corresponding to carbon dioxide and carbonyl fluoride are present.

The 440 C stainless steel specimen, after being rinsed with the TCF solvent, revealed the presence of a fairly uniform brownish deposit, which was easily scraped off with a knife. It was also noticed that the reactor glassware was etched.

Fluid D on 440 C stainless steel surface.—No degradation of fluid D occurred on the 440 C stainless steel specimen.

Fluid F on iron surface.—Degradation of fluid F occurred in the thin-liquid-film test on the clean iron specimen. Unlike the test involving the 440 C stainless steel specimen, fluid degradation was not complete. Figure 13 represents the amount of fluid F left on the clean iron surface at the end of the test (72 percent of the fluid degraded). Not only was fluid degradation incomplete, but reproducibility of the results could not be obtained: subsequent tests showed 87, 66, and 82 percent of the fluid degraded.

Like the results obtained from the thin-liquid-film test with the clean 440 C stainless steel specimen, a liquid collected in the condensing tube. The HPLC analysis confirmed that the liquid had the same retention time as the liquid from the 440 C stainless steel test. Analysis of the reservoir solution also showed a product (carbon dioxide) with the same retention time as the reservoir product from the 440 C stainless steel test.

The surface of the iron specimen, after being rinsed with TCF, revealed a brownish deposit that did not cover the entire surface of the specimen. This deposit formation was unlike that on the 440 C stainless steel specimen, where the entire surface was covered.

Fluid D on iron surface—No degradation of fluid D occurred on the clean iron specimen.

Further Results For Fluids F and D

The thin-film test was repeated with fluids F and D for the used 440 C stainless steel specimen covered with the brown deposit (after rinsing the specimen with TCF solvent) in a nitrogen atmosphere. Fifty-three percent of fluid F degraded, whereas fluid D did not degrade.

A series of tests involving the use of a special borosilicate glass container (fig. 14) was also performed with fluids F and D. A small quantity (0.5 mg) of a chemical, in powdered form, was introduced into the glass container. Next, 100 μl of the test fluid was injected into the glass container. The stem of the glass container was then connected to a vacuum line and, after evacuation (<10 torr), the stem was sealed. The evacuated glass container was then heated (with a hand-held heat gun, approximate temperature of 300 °C) for 10 min. At the end of the test, the contents of the glass container were extracted with TCF solvent, filtered, and analyzed by HPLC. This test was repeated, but after evacuation the glass container was pressurized (790 torr) with air, sealed, and then heated.

Table II lists the results, whether or not the fluids degraded for these tests. It shows that fluid F degraded in the presence of some chemicals, whereas fluid D did not degrade. No attempt was made to quantify fluid F degradation for these tests.

Analysis of Deposit

Surface analysis (electron spectroscopy for chemical analysis (ESCA)) of the used 440 C stainless steel specimen, with the brown deposit on it, revealed iron and chromium compounds. The iron $2p^3\!/_2$ and chromium $2p^3\!/_2$ binding energies were consistent with the presence of iron oxide and chromium oxide (Cr_2O_3). A flouride 1*s* line was also found in the spectrum. Its binding energy was consistent with a metal fluoride, but not a fluorocarbon.

TABLE II.—COMPARISON OF FLUIDS F AND D DEGRADATION FOR VARIOUS POWDERED CHEMICALS

Material	Atmosphere	Fluid F degradation	Fluid D degradation
α-Fe_2O_3	Vacuum	Yes	No
	Air	Yes	No
γ-Fe_2O_3	Vacuum	No	No
	Air	Yes	No
Cr_2O_3	Vacuum	No	No
	Air	No	No
SiO_2	Vacuum	No	No
	Air	No	No
FeF_2	Vacuum	Yes	No
	Air	Yes	No
FeF_3	Vacuum	Yes	No
	Air	Yes	No

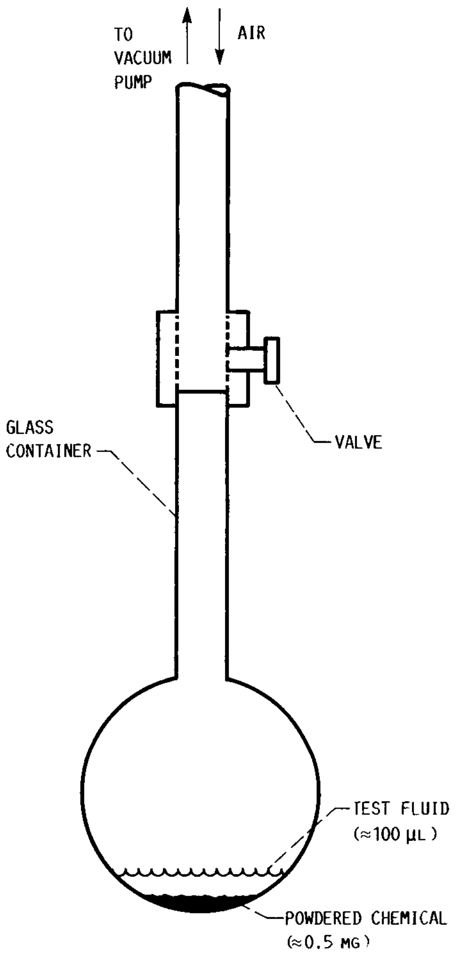


Figure 14.—Glass container containing test fluid and powdered chemical.

The deposit from the 440 C stainless steel specimen was also analyzed by x-ray diffraction. Table III represents the diffraction data. Although interpretation proved to be difficult, the data suggest that part of the deposit may be iron fluoride.

Discussion

These studies showed that specific conditions existed that led to the breakdown of fluid F, whereas fluid D was unaffected by these conditions. It was observed that fluid F did not degrade in the presence of either clean metal specimen in a nitrogen atmosphere at 345 °C but that the fluid degraded when the tests were repeated in an air atmosphere, with the generation of a brownish deposit on both the 440 C stainless steel and iron surfaces. Subsequent tests revealed that fluid F then degraded in an nitrogen atmosphere, in the presence of the deposit. These results will be discussed in detail later in this section.

As described in the section **Experiment**, the cleaning of the metal specimens involved a procedure where the specimens were first rinsed with the TCF solvent, then with 200-proof ethanol, and finally with deionized water after being scrubbed

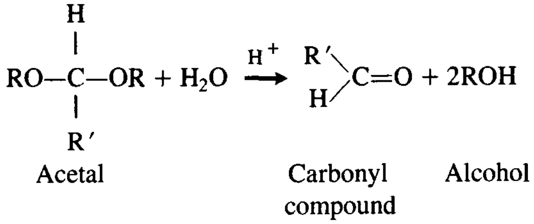
TABLE III.—X-RAY DIFFRACTION DATA FOR DEPOSIT FORMED ON 440 C STAINLESS STEEL SPECIMEN DURING FLUID F DEGRADATION, AND FOR IRON FLUORIDE

Relative intensity	Diffraction, Å	
	Deposit	Iron fluoride
(a)	4.27	---
7	3.73	---
10	3.34	3.32
4	2.69	2.70
1	2.34	2.34
(a)	2.29	---
(a)	2.18	---
3	2.04	---
2	1.77	1.77
(a)	1.67	1.67
(a)	1.64	---
(a)	1.54	---

[a] Weak.

with alumina paste. Several thin-liquid-film tests were conducted with iron specimens for which the rinsing procedure was reversed: the specimens were scrubbed with alumina paste and then rinsed with deionized water, with ethanol, and finally with the TCF solvent. Table IV compares fluid F degradation for tests with a final TCF solvent rinse and for tests with a final water rinse. The table shows that fluid F degradation was always greater when the iron specimens were rinsed last with water. This result led to consideration of two mechanisms that could account for the breakdown of fluid F.

A comparison of the general molecular structures of fluids F and D reveals that the repeating structure —O—C—O— occurs only in fluid F. This is the structure of an acetal (ref. 12). The chemistry is similar to the chemistry of ethers (—C—O—C—) cleaved by acids; however, acetals undergo acidic cleavage with ease:



$$RO-\underset{R'}{\overset{H}{C}}-OR + H_2O \xrightarrow{H^+} {}^{R'}_{H}\!\!>C=O + 2ROH \qquad (1)$$

One hypothesis for the breakdown of fluid F is that degradation occurs by an acetal-type cleavage mechanism with residual water, adsorbed on the metal oxide surface, as the source of protons. Evidence to support this hypothesis was given by the analysis of the degradation products and the results obtained by reversing the metal specimen cleaning procedure.

Figure 15 represents a chemical model depicting the possible cleavage of fluid F by a proton. The fluid, at 345 °C, is in contact with a metal oxide surface. In step 1, a proton coordinates with an acetal oxygen. This leads to cleavage of the molecule, with the subsequent formation of a carbonium ion and an unstable intermediate (step 2). The intermediate rapidly decomposes to carbonyl fluoride (COF_2) and hydrogen fluoride (HF) (step 3). Further cleavage of the carbonium ion (step 4) leads to further generation of carbonyl fluoride. Cleavage of the carbonium ion stops when acetal oxygens are no longer present. Possible termination reactions are shown by steps 5 and 6.

If residual water is the source of protons that leads to fluid F degradation by acidic cleavage, then the degradation process should (1) occur in either the air or nitrogen atmosphere, and (2) not depend on the nature of the metal oxide surface that fluid F is in contact with. However, as was mentioned in the section **Results**, fluid F did not degrade in a nitrogen atmosphere on either clean metal specimen, whereas fluid F degraded in an air atmosphere, with the formation of a deposit on the metal surface. It was discovered that fluid F then degraded in a nitrogen atmosphere in the presence of this deposit. These results, in conjunction with the results shown in table II, lead to the following consideration: if fluid F degradation is due to the fluid's acetal structure, then it appears that proton release from adsorbed residual water is actuated only on certain metal oxide surfaces.

The stable oxide phase on an iron metal at room temperature (≈ 25 °C) is γ-Fe_2O_3 (ref. 13). Heating the metal specimens up to 345 °C in a nitrogen atmosphere does not change the γ-Fe_2O_3 surface oxide. It appears from preliminary lab work, however, that upon switching to an air atmosphere the following transformation occurs:

$$\gamma\text{-}Fe_2O_3 \xrightarrow[345\ ^\circ C]{air} \alpha\text{-}Fe_2O_3 \qquad (2)$$

TABLE IV.—THIN-FILM TESTS FOR A CLEAN IRON SPECIMEN

[Test duration, 30 min; atmosphere, air; temperature, 345 °C.]

(a) With final TCF[a] solvent rinse

Test	Fluid F degradation, percent
1	29
2	37
3	6
4	24

(b) With final water rinse

Test	Fluid F degradation, percent
1	72
2	87
3	66
4	82

[a] 1,1,2-Trichlorotrifluoroethane.

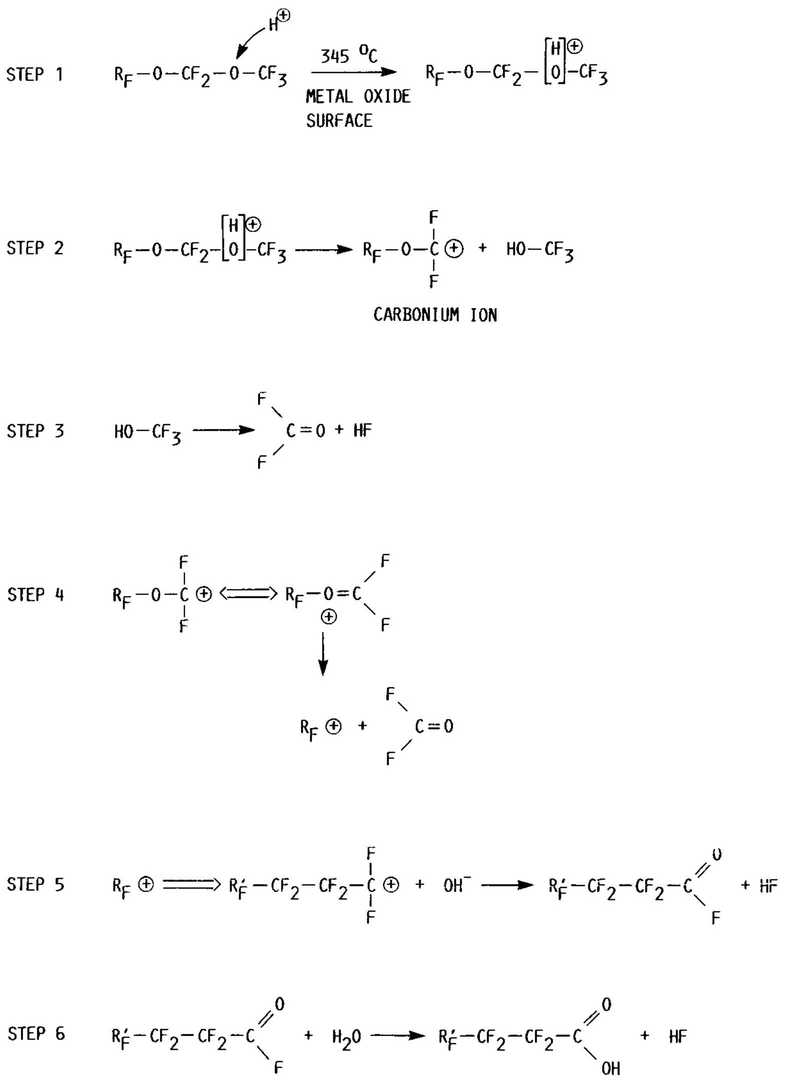


Figure 15.—Chemical model for possible fluid F cleavages by a proton.

The results (table II) show that fluid F, under evacuated conditions, did not degrade in the presence of γ-Fe_2O_3 but degraded in the presence of α-Fe_2O_3.

The second hypothesis for the breakdown mechanism for fluid F also involves the fluid's acetal structure. The possibility exists that coordination from the acetal oxygens to iron cations (from the surface α-Fe_2O_3 metal oxide) can occur. This coordination would serve to lower the activation energy needed to break the acetal bonds, with subsequent degradation of the molecule. At this time it cannot be explained why coordination would occur on the reactive sites of the α-Fe_2O_3 metal oxide surface and not on the γ-Fe_2O_3 surface. This hypothesis also takes into account that the presence of residual water may serve to accelerate reaction (2), thus explaining the results shown in table IV.

The presence of CO_2 can be attributed to two reactions:

$$2COF + SiO_2 \longrightarrow SiF_4 + CO_2 \tag{3}$$

$$COF_2 + H_2O \longrightarrow 2HF + CO_2 \tag{4}$$

Reaction 3 accounts for the etching of the glassware.

Summary of Results

The results from this study are summarized as follows:

1. In a nitrogen atmosphere (345 °C test temperature) fluids F and D did not degrade on clean 440 C stainless steel or iron metal specimens.

2. In an air atmosphere, fluid F degraded on both clean 440 C stainless steel and iron metal specimens. Fluid D did not degrade.

3. Fluid F decomposition products consisted of gaseous carbonyl fluoride, a liquid product (room temperature) having a chemical structure similar to that of fluid F (but with a molecular weight much lower than the molecular weight of fluid F), and a third product having a carboxylic acid functional group.

4. The formation of a brown deposit on the metal specimens resulted from the degradation of fluid F in the air atmosphere. The thin-liquid-film tests were repeated with these deposit-covered metal specimens (after rinsing the specimens with TCF solvent), and it was observed that fluid F then degraded in a nitrogen atmosphere. Fluid D did not degrade.

5. The experimental evidence suggests that fluid F degradation occurs as a result of its acetal structure.

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Cleveland, Ohio, August 25, 1987

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16. Abstract

Thin-liquid-film degradation studies of two commercially available perfluoropolyalkylether fluids (PFAE) were performed at 345 °C, in nitrogen and air atmospheres, on iron and 440 C stainless steel surfaces. It was found that one fluid degraded on both iron and 440 C stainless steel surfaces in an air atmosphere, whereas the other fluid did not degrade. Chemical analysis revealed that the test fluid degraded to lower molecular weight products and that the degradation was accompanied by the formation of a brownish deposit on both the iron and 440 C stainless steel surfaces. Surface analysis of the deposit revealed a substantial amount of iron oxide (Fe_2O_3). It was hypothesized that the fluid which degraded did so because of its acetal structure. The other fluid, lacking the acetal structure, did not degrade.

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