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RESEARCH MEMORANDUM

MATERIAL COMPATIBILITY WITH GASEOUS FLUORINE

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SUMMARY

Static tests were made on the compatibility of gaseous fluorine with various liquids, solid plastics, waxes, and greases at pressures of 0 and 1500 pounds per square inch gage and atmospheric temperature.

These tests eliminated many materials from further consideration for use in fluorine systems. Several materials were found compatible at atmospheric pressure. Only Teflon and ruby (aluminum oxide) were compatible under the static conditions of the tests at 1500 pounds per square inch gage. Further tests under dynamic conditions are required if the application involves exposure to flowing fluorine.

INTRODUCTION

This report describes a qualitative investigation of various nonmetallic materials for use with fluorine. To use this very active oxidizer, special materials must be examined for application in the flow systems, for example, as seal materials, thread lubricants, and hydraulic fluids for instruments and other equipment.

Work on compatibility of metals with fluorine is reported in references 1 and 2. Reference 2 also discusses the compatibility of several nonmetals.

In searching for nonmetallic materials which may be compatible with fluorine, consideration was first given to fluorinated organic compounds. Among those compounds examined were polytetrafluoroethylene, $CF_3-(CF_2)_x-CF_3$, (Teflon); polychlorotrifluoroethylene, $Cl-(CF_2-CFCl)_x-Cl$, (Kel F and Fluorolube oils, waxes, greases, and solids); and heptacosafluorotributylamine, $(C_4H_9)_3N$, (N-43).

Other materials tested are in common use as solvents for cleaning, pipe-thread lubricants, gasket materials, hydraulic fluids for instruments, brazing flux, bearings, valve packings and seats, leak detection solutions, etc. Many of these have previously been used in fluorine service satisfactorily under carefully controlled conditions.

Exposure to fluorine, under essentially static conditions, was accomplished in two successive phases:

(a) Fluorine gas at atmospheric pressure and temperature

(b) Fluorine gas at 1500-pounds-per-square-inch-gage pressure and atmospheric temperature

APPARATUS AND PROCEDURE

The equipment used for these tests consisted of two separate setups. One was for the atmospheric-pressure tests, and the other was for the 1500-pounds-per-square-inch-gage tests.

The atmospheric-pressure setup consisted of an open, glass test tube fed by a copper tube connected to a fluorine-supply container and to a source of helium for purging. The copper tube reached to within 1 inch of the bottom of the glass test tube. The test was performed by admitting fluorine gas into the test tube which contained the sample. In the case of liquid samples, the fluorine was admitted below the surface and bubbled up through the material.

As each test was performed, visual observations were made to determine whether any reaction took place. Reaction with fluorine was immediately evidenced by vigorous burning or explosion. For tests in which no reaction occurred, the presence of fluorine was verified by the spontaneous ignition of a small amount of jet fuel placed on a wick outside the top of the test tube. After the test, the system was purged with helium and the sample examined.

If a material reacted with fluorine at atmospheric pressure, it was not tested at the higher pressure.

The 1500-pounds-per-square-inch-gage setup consisted of a stainless-steel cylinder 6 inches long made from 3/4-inch heavy-walled tubing. This cylinder was connected to a fluorine container by 100 feet of 1/4-inch copper tubing. Between the fluorine container and the test cylinder, a helium pressurizing source was connected very close to the fluorine source.

The system was first flushed with helium and then with gaseous fluorine. In this manner, it was felt that all air was removed. The system was next charged with fluorine to a pressure of approximately 300 pounds per square inch gage and then pressurized to 1500 pounds per square inch gage with helium by means of a hand-operated loader. The long lead of copper tubing served to prevent any diffusion of the helium into the container to dilute the fluorine during the period of the test. Calculations showed that the fluorine-helium interface should be in the copper tubing 16.5 feet from the test cylinder.

While making the high-pressure tests, a gage in the system was observed as pressure was increased to 1500 pounds per square inch gage. When a reaction took place, a pressure fluctuation was indicated by this gage. If no reaction occurred, the sample was kept at 1500 pounds per square inch gage for approximately 10 minutes. After the test, the system was vented and thoroughly purged with helium. The cylinder was then disconnected and the sample examined.

All samples were selected for high purity, cleaned with organic solvents when applicable, thoroughly dried, and handled carefully with clean rubber gloves to avoid contamination.

RESULTS AND DISCUSSION

Results of the exposure tests are presented in table I.

Because they reacted even at atmospheric pressure, 19 materials were immediately ruled out for fluorine use. The remaining samples did not react at low pressure; however, all but Teflon and ruby did react readily at high pressure.

The difference in behavior between low and high pressure indicates techniques by which such materials may be useful, and explains how some have been used successfully in practice. For example, in fluorine work, pipe-thread lubricants are conventionally applied to the outermost threads only. In such use the lubricant does not experience the full pressure of the fluorine system; exposure, if any, is to fluorine at atmospheric pressure.

Only Teflon and ruby survived the high-pressure exposures. These tests would indicate the suitability of Teflon in static seals, and of ruby as a bearing material. The tests cannot be taken, however, as conclusive evidence that these materials will be satisfactory in all fluorine applications. Teflon is known to fail under exposure to flowing liquid fluorine.

Of the fluorinated organic compounds, the completely fluorinated polymer exhibited greater stability than did those containing chlorine. Since a strong chlorine odor was observed following reactions of polymers in the group containing chlorine, it is apparent that the carbon-chlorine bond does not withstand fluorine attack.

The area of exposed sample surface was observed to influence reaction. In the high-pressure tests, if the steel test tube were held vertically, samples of polychlorotrifluoroethylene did not react; but, if the tube were inclined to increase the exposed surface of the liquid, reaction occurred. In related work not tabulated, reactions of polychlorotrifluoroethylene and of water with fluorine at atmospheric pressure have been noted when exposed areas of the samples were large. It seems advisable, then, to minimize exposed surface of all nonmetals contacting fluorine in service.

CONCLUDING REMARKS

The qualitative compatibility tests of this work, made under static exposures to gaseous fluorine, have eliminated many materials from further consideration. They have indicated limitations in the use of other materials. The tests, however, have not been decisive in proving unqualified applicability of any nonmetal to fluorine service. Conditions more severe than provided by the present tests remain to be studied with the more promising substances by exposing them to liquid fluorine, and more particularly, to dynamic conditions of fluorine flowing at high velocities.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
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REFERENCES

1. Gundzik, Richard M., and Feiler, Charles E.: Corrosion of Metals of Construction by Alternate Exposure to Liquid and Gaseous Fluorine. NACA TN 3333, 1954.

2. Landau, Ralph, and Rosen, R.: Industrial Handling of Fluorine. Ind. and Eng. Chem., vol. 39, no. 3, Mar. 1947, pp. 281-286.

TABLE I. - SAMPLES EXPOSED TO GASEOUS FLUORINE

	Sample[a]	Sample exposed to F_2 at atm. pressure	Sample exposed to F_2 at 1500 lb/sq in. gage pressure
Liquids	Kel F LO No. 10 (M. W. Kellogg Co.)	No reaction	Reaction[b]
	Fluorolube HO (Hooker Electrochemical Co.)		Reaction[b]
	N-43, $(C_4H_9)_3N$ (Minnesota Mining & Mfg. Co.)		Reaction[c]
	Tap water		Reaction
	Cenco Hyvac Oil (Central Scientific Co.)	Burned	--------
	Glyptol (General Electric Co.)		--------
	Dow Corning 200 Fluid (20 centistokes)		--------
	Water glass		--------
	Safety Solvent 178 (Fine Organics Co.)		--------
	Leak-Tec (American Gas & Chemicals Inc.)		--------
	Leak-Tec with glycerine		--------
	Dry JP-4 fuel		--------
	Carbon tetrachloride	Exploded	--------
Greases	Kel F Med. Wax (M. W. Kellogg Co.)	No reaction	Reaction
	Kel F No. 1 Grease (M. W. Kellogg Co.)		(d)
	Fluorolube LG (Hooker Electrochemical Co.)		(d)
	Fluorolube MG (Hooker Electrochemical Co.)		(d)
	Permatex No. 3 (Permatex Co.)		Reaction
	Q-Seal (Quigley Co., Inc.		
	Blue Goop (Crawford Fitting Co.)		
	Molylube (Bel-Ray Co., Inc.)		
	Plast-O-Seal (The Colonial Plastics Mfg. Co.)		
	Permatex No. 1 (Permatex Co.)	Burned	--------
	Permatex No. 2 (Permatex Co.)		--------
	X-Pando (X-Pando Corp.)		--------
	Tyte Unyte (J. C. Whitlam Mfg. Co.)		--------
	White lead		--------
Solids	Ruby (Al_2O_3)	No reaction	No reaction
	Teflon (E. I. DuPont de Nemours & Co.)		No reaction
	Kel F Solid (M. W. Kellogg Co.)		Reaction
	Kel F Elastomer 5500 (M. W. Kellogg Co.)		
	Graphitar		
	Powdered Graphitar	Burned	--------
	Neoprene covered Fiberglas	No reaction	Reaction
	N-43 plus Neoprene covered Fiberglas		
	Plexiglas (Rohm & Haas Co.)		
	Tygon tubing		
	Vinylite		
	Pennsalt PCC (Pennsylvania Salt Mfg. Co.)		
	Pennsalt PCI (Pennsylvania Salt Mfg. Co.)		
	Flux on silver-soldered copper-to-brass joint		
	Slag on stainless-steel weld joint		
	Flux on silver-soldered stainless-steel joint		
	Dow Corning Elastomer	Burned	--------
	Molylube Type Z Powder		--------
	Litharge and glycerine		--------
	Palmetto (Green, Tweed & Co.)		--------

[a]Samples are listed by trade name in most cases, and the composition was not available.

[b]Reacted when exposed surface area was increased.

[c]Reacted in two out of four tests.

[d]Should give the same reaction as the Kel F Med. Wax.