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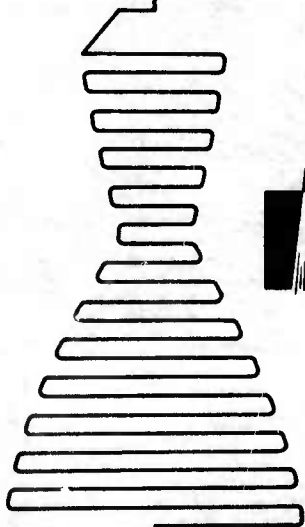
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(Unclassified Title)

QUARTERLY PROGRESS REPORT,  
INORGANIC HALOGEN OXIDIZERS  
(1 June through 31 August 1964)

Group 4  
Downgraded at 3-Year Intervals  
Declassified After 12 Years

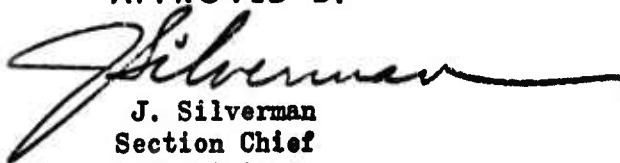
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Sponsored by Advanced Research Projects Agency  
Washington 25, D.C.  
ARPA Order No. 23

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DATE 30 Sept. 1964

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## FOREWORD

The research reported herein was supported by the Advanced Research Projects Agency, Washington 25, D.C., and was monitored by the Office of Naval Research, Power Branch, Code 429, Washington 25, D.C., with Mr. Richard Hanson as Scientific Officer. This report was prepared under G.O. 8614 in compliance with Section H of Contract Nonr 4428(00) under ARPA Order No. 23, and covers the period 1 June through 31 August 1964.

The Responsible Engineer for this program is Dr. D. Pilipovich. Dr. H. F. Bauer and Dr. C. J. Schack are full-time associates.

## ABSTRACT

Preparative routes to new interhalogen compounds and their derivatives are being pursued. Initial approaches utilized discharge techniques to evaluate both fluorination and oxygenation of substrates such as  $\text{ClF}_3$ ,  $\text{Cl}_2\text{O}$ , and  $\text{ClO}_2$ . The reaction of solid  $\text{FNO}$  with excited fluorine was demonstrated at  $-196^\circ\text{C}$  with the production of  $\text{NF}_3\text{O}$ . Similar procedures were used in reacting  $\text{Cl}_2\text{O}$  and  $\text{ClO}_2$  with  $\text{F}_2$ .

(Confidential Abstract)

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**INTRODUCTION**

This report is the first quarterly report on a program directed toward the preparation of new inorganic oxidizers. The particular synthetic approaches to be utilized on this program are diverse, but all will be directed toward the realization of new interhalogen fluorides, oxyhalogen fluorides, and oxynitrogen fluorides. Initial studies to be described herein were designed to prepare new oxychlorine fluorides and utilized discharge techniques. The rationale for the use of the electric discharge is discussed both with respect to probable utility and in relation to other chemical approaches in synthesizing these materials.

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## DISCUSSION

## DISCHARGE REACTIONS

The selection of the electric discharge as a synthesis tool is based, to some extent, upon its successful application in preparing the new inorganic compounds  $\text{ClF}_5$  and  $\text{NF}_3\text{O}$  (Ref. 1 and 2). These compounds are both simple and symmetrical and reflect the type of molecule that will arise from subjecting the proper elemental mixture to a discharge. If more complex molecules are desired it would appear that the electric discharge as a synthesis tool has only a limited utility. This is particularly true if it is realized that the products from a discharge reaction will often be consistent with the most stable species arising from relatively stable radicals. Thus  $\text{N}_2$  and  $\text{F}_2$ , when subjected to a discharge, give only  $\text{NF}_3$  and not  $\text{N}_2\text{F}_4$  or higher NF species such as  $\text{N}_3\text{F}_5$ .

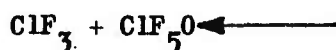
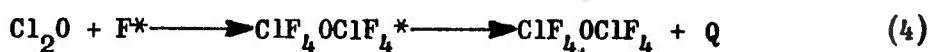
However limited the use of the electric discharge may appear when used in a "conventional" manner, the fact remains that the ternary system  $\text{N}_2\text{-F}_2\text{-O}_2$  does give products containing all three elements, i.e.,  $\text{NF}_3\text{O}$ . The question immediately arises as to whether  $\text{ClF}_3\text{O}$  and  $\text{ClF}_5\text{O}$  can result from the ternary mixture  $\text{Cl}_2\text{-F}_2\text{-O}_2$ . This obvious analogy was tested by subjecting this mixture to a discharge at  $-80^\circ\text{C}$ . No new compounds were observed using a  $\text{F}_2\text{-Cl}_2\text{-O}_2$  ratio of 6:1:1. Products found were  $\text{ClO}_2$ ,  $\text{FClO}_2$ ,  $\text{ClF}_3$ , and  $\text{ClF}_5$ . Due to the accidental presence of air in the starting material,  $\text{NO}_2$ ,  $\text{FNO}$ ,  $\text{FNO}_2$ , and  $\text{ClONO}_2$  also were formed. Additional experiments were carried out at  $-196^\circ\text{C}$ , and in addition to the above products, Compound B (Ref. 1) was found. This compound was identified as  $\text{WF}_6$  which arose from the attack by  $\text{F}_2$  on the tungsten used to achieve the glass-to-metal seals in the discharge apparatus. These results are consistent with those previously obtained in the Rocketdyne laboratories in discharge reactions of  $\text{Cl}_2$  and  $\text{F}_2$  where  $\text{O}_2$  was known, at times, to be an impurity.

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Subjecting mixtures of  $\text{Cl}_2$ ,  $\text{F}_2$ , and  $\text{O}_2$  to the electric discharge does not appear to be promising on the basis of the above results. It appeared that variations in the discharge technique may offer more versatility in product control than the techniques now employed. One method decided upon was to utilize a gas-solid reaction where the gas is some excited species such as  $\text{F}_2^*$  or  $\text{O}_2^*$ . The solid material is in essence a cold wall where excess energy can be readily dissipated. The essential difference from the previous experiments is that specific bond formation is sought by the interaction of an excited species with a readily oxidized material as opposed to an overall excitation followed by random and fortuitous recombinations. The following equations demonstrate the reactions considered:

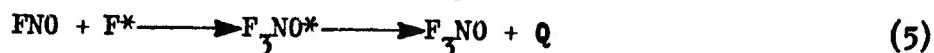


Thus, either  $\text{O}_2$  or  $\text{F}_2$  can be used for the above oxidations depending on the choice of the starting material. It should be noted that the excited species in the above equations are not represented as either radicals or ions. It is suspected that operation of the discharge below the ionization threshold will lead to radicals, and conversely, operation in the glow region will of course yield ions.



The features of the solid-gas reaction at once became obvious for several reasons. First, it can be predicted that a higher degree of control can be exerted with regard to the structure of the products. This is dictated by the fact that one of the reactants, the solid, is the wall at which immediate dissipation of excess energy is anticipated. This is particularly important when it is realized that wall reactions enhance product formation in a discharge reaction. Subsequently, another feature is suggested in that higher yields of complex moieties may be realized. It should be recalled (Ref. 2) that in the discharge preparation of  $\text{NF}_3\text{O}$  from various gas mixtures the same low yields were obtained for the starting material combinations  $\text{NF}_3\text{-O}_2$ ,  $\text{N}_2\text{-O}_2\text{-F}_2$ , and  $\text{N}_2\text{-OF}_2$ . This is no doubt based upon the statistical nature of a favorable combination of the excited F, N, and O to give the desired product.

To test the above assumptions, a model reaction was selected for study at  $-196^\circ\text{C}$ . The desired reaction is:



The use of FNO was selected because it not only has an oxidizable "central" atom but it should give the known compound  $\text{NF}_3\text{O}$ . The apparatus used was a U-shaped Pyrex discharge tube connected to a glass U-trap. Both were maintained at  $-196^\circ\text{C}$ , and a constriction was placed in the trap about 10 inches downstream from the discharge zone. Nitrosyl fluoride was then condensed at the constriction, and  $\text{F}_2$  was introduced into the discharge tube at a pressure below 50 millimeters.

Examination of the products in the trap showed  $\text{NF}_3\text{O}$  to be present, representing a 5-percent conversion of the FNO. A separate experiment, a control without FNO, yielded no  $\text{NF}_3\text{O}$  in the trap. The control was carried out in view of the fact that the  $\text{F}_2$  supply often is contaminated

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with small amounts of air. Subsequent experiments showed the FNO (s)-F\* reaction to be reproducible. In view of the moderate success with the model selected, it appeared desirable to extend it to halogen oxides.

The selection of reactants in seeking routes to new F, Cl, and O species is quite limited. Prime consideration should be given to the valence of the "central" atom. On this basis, then, all chlorine (VII) species do not appear to be desirable since neither oxidations can occur nor is the replacement of O on chlorine probable. Consequently, perchloryl fluoride does not appear to be a suitable precursor for discharge studies. The chlorine compounds which meet the imposed requirements are FC1O<sub>2</sub> (or ClO<sub>2</sub>) and Cl<sub>2</sub>O. Each can be envisioned as giving rise directly to the sought oxychlorofluorides:



The reaction of Cl<sub>2</sub>O as written proceeds through the rupture of a ClO bond which requires little energy in Cl<sub>2</sub>O. The photodecomposition of chlorine monoxide proceeds initially according to the first step of Eq. 6a as does the chlorine-atom sensitized decomposition (Ref. 3).

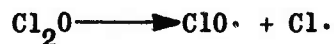
To extend fluorination to halogen oxides, the selected precursors were subjected to conditions similar to those in the FNO(s)-F\* experiments. For example, chlorine monoxide was frozen at -196 C on the reactor wall just downstream from the glow discharge electrodes. Fluorine passing between the electrodes is converted to fluorine atoms. These radicals are then able to bombard the solid reactant and effect fluorination.



By maintaining starting materials and products at  $-196\text{ C}$ , side reactions are minimized and hence the possibility of conversion to new energetic species is maximized.

In extending the solid-gas reaction to chlorine systems, two electrical discharge tubes have been employed. The first design maintained the  $\text{Cl-O}$  compound to be attacked at  $-196\text{ C}$  but left the electrodes above the bath where they were cooled by an air blower.

Chlorine monoxide was reacted twice with essentially the same result. All of the  $\text{Cl}_2\text{O}$  was used up, but the only  $\text{ClO}$  species formed was  $\text{ClO}_2$ . Also formed were  $\text{Cl}_2$  and much  $\text{SiF}_4$ . The chlorine dioxide and chlorine probably arose directly from the decomposition of  $\text{Cl}_2\text{O}$ .



Initiation of this sequence by F atoms comparable to the known Cl atom process is possible. A chain radical reaction promoted by the product Cl atoms makes fluorine necessary only for initiation and not propagation. Thus, small amounts of ClF could have been formed but not detected.

Air contamination in one run was noted by the production of a small quantity of chlorine nitrate. This again is a well-characterized product of ClO radical reactions:



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Due to difficulties with the method originally selected for the preparation of  $\text{FClO}_2$ ,  $\text{ClO}_2$  was present as an impurity. Nevertheless, since  $\text{ClO}_2$  is the precursor of  $\text{FClO}_2$  it was expected that it would not adversely affect the reaction. In fact, fluorination of  $\text{ClO}_2$  would itself indicate that suitable fluorination conditions were being approached.

Using this mixture in the solid phase, it was found that the discharge reaction resulted in consumption of the  $\text{FClO}_2$  but not the  $\text{ClO}_2$ . The products were  $\text{FClO}_3$  and  $\text{SiF}_4$ , in addition to the  $\text{ClO}_2$ . The conversion of chloryl fluoride to perchloryl fluoride is certainly not the result of fluorination, and the failure of the  $\text{ClO}_2$  to be fluorinated leads to the conclusion that fluorination conditions were lacking.

The formation of appreciable amounts of  $\text{SiF}_4$  using this technique can account for the findings. The activated fluorine may react with the glass of the tube around the electrodes too fast to allow sufficient amounts to reach the intended reaction zone. In addition, oxygen that is liberated by the conversion of the silica to  $\text{SiF}_4$  may be responsible for the oxidation of  $\text{FClO}_2$  to  $\text{FClO}_3$ .

So severe was the etching of the apparatus near the electrodes and the thermal strain encountered during operation, that the discharge tube cracked after several experiments. A newly designed system was fabricated in which both electrodes and substrates could be immersed in a liquid nitrogen bath. This tube has shown only limited etching after several experiments.

Again utilizing  $\text{Cl}_2\text{O}$  as the starting material, three experiments were conducted. Two distinct results were obtained depending upon the purity of the fluorine. In the absence of air impurity, most of the chlorine monoxide was reacted after 20 minutes. The yield of  $\text{FClO}_2$ ,  $\text{ClO}_2$ , and

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$\text{ClF}_5$ , along with a trace of  $\text{SiF}_4$ , demonstrated that fluorination had been effected. Chlorine dioxide was formed from  $\text{Cl}_2\text{O}$  as shown earlier. Chloryl fluoride and chlorine pentafluoride resulted from the reaction of fluorine with the  $\text{ClO}_2$  and  $\text{Cl}$  atoms, respectively. Presumably,  $\text{Cl}$  atoms are generated from the decomposition of  $\text{Cl}_2\text{O}$ , as pointed out earlier.

When air-contaminated fluorine passed through the discharge, the product composition was altered. As expected,  $\text{ClO}_2$  and trace amounts of  $\text{SiF}_4$  were formed. However, no  $\text{FClO}_2$  was observed. Furthermore, appreciable yields of  $\text{ClNO}_3$ ,  $\text{ClF}_5$ , and  $\text{NF}_3$  were noted. These molecules are all known to result from the glow discharge reaction of the appropriate elements. The important point derived from the experiment is that fluorination of  $\text{Cl}$  in  $\text{Cl}=\text{O}$  bonds is much more difficult to achieve than is the corresponding reaction of other radical atoms or groups. Thus, although these are available, the active fluorine is so effectively added to them that an insufficient amount remains to promote the desired new species.

Pure  $\text{FClO}_2$  and pure  $\text{F}_2$  now appear to give the most promise of attaining the proposed objective via discharge reaction. Experiments with recently available chloryl fluoride of the required purity are in progress.

In both reactions of  $\text{Cl}_2\text{O}$  with air-contaminated fluorine it was observed that  $\text{NF}_3\text{O}$  was produced from the reaction products during a second warming from liquid nitrogen temperature. This was accompanied by the production of some noncondensable gas. It appears probable that the  $\text{NF}_3\text{O}$  results from the interaction of a trace of  $\text{FNO}$  and the decomposition products of  $\text{O}_2\text{F}_2$ , which is known to form in an  $\text{F}_2-\text{O}_2$  discharge reaction (Ref. 3). Further clarification of this point is necessary.

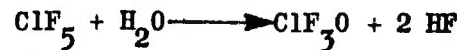
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Other Routes to FC10 Species

It was proposed that high-pressure fluorinations would be carried out in attempting to prepare compounds such as  $\text{ClF}_3\text{O}_2$ ,  $\text{ClF}_5\text{O}$ , etc. For example, the direct fluorination under pressure of  $\text{FC10}_2$  and  $\text{ClO}_2$  had been proposed. Since the proposal was submitted, these pressure fluorinations have been attempted without spectacular results (Ref. 4). Therefore, no pressure fluorinations of ClO species are contemplated at the present time.

The hydrolysis of  $\text{ClF}_5$  has been shown at Rocketdyne not to lead to new ClFO species, particularly  $\text{ClF}_3\text{O}$  (Ref. 5). This was somewhat surprising in that the reaction was assumed to be a stepwise process, i.e.,



It would appear that either  $\text{ClF}_3\text{O}$  is more reactive to  $\text{H}_2\text{O}$  than  $\text{ClF}_5$  or that it readily disproportionates to other products such as  $\text{ClF}_5$  and  $\text{FC10}_2$ . This species will be sought with attention to mode of reaction in the case of pure  $\text{ClF}_5$  or by studying the hydrolysis of  $\text{ClF}_4^+$  salts.

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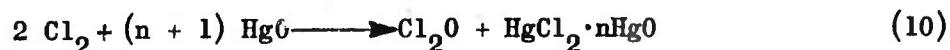


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## EXPERIMENTAL

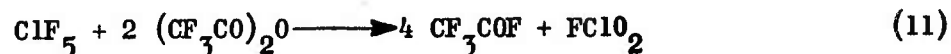
### PREPARATION OF $\text{Cl}_2\text{O}$ AND $\text{FClO}_2$

Chlorine monoxide was prepared fresh from the reaction of chlorine gas diluted with nitrogen and freshly precipitated red mercuric oxide (Ref. 6).



Storage of  $\text{Cl}_2\text{O}$  in glass ampoules at  $-196^\circ \text{C}$  led to the slow formation of  $\text{ClO}_2$ .

Chloryl fluoride was at first synthesized by the reaction of perfluoroacetic anhydride and chlorine pentafluoride.



The reaction was carried out in a stainless-steel bomb at ambient temperature. However, the slow rate of the reaction was found to limit the yield of  $\text{FClO}_2$ . The chloryl fluoride produced was partially decomposed upon prolonged contact with the metal to give  $\text{ClO}_2$ . Also, the large amount of byproduct perfluoroacetyl fluoride required a lengthy separation from the  $\text{FClO}_2$ .

A more practical synthesis of this material is the reaction of silver oxide and chlorine pentafluoride. Again, using a stainless-steel reactor at ambient temperature, it has been possible to obtain a 55-percent yield of  $\text{FClO}_2$ . The only other volatile products are noncondensable at  $-196^\circ \text{C}$  and hence easily removed.

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#### **DISCHARGE APPARATUS**

The glow discharge tube was constructed of Pyrex with vacuum stopcocks at inlet and outlet sides. The electrodes were copper and were sealed in the glass with gaps of 2 and 6 inches, respectively, in the two reactors. With the outlet stopcock barely opened, fluorine was passed through the fully opened inlet stopcock and maintained at 20 to 50 millimeters pressure. Duration of the discharge in all cases was approximately 20 minutes. Power was supplied by a 15-kilovolt luminous-tube transformer.

At the termination of each discharge reaction, the cooling bath was removed from the tube, and all volatile material was transferred to the vacuum line. Vacuum fractionation was conducted in a metal-Teflon system. Infrared spectra were taken on a PE 127 Infracord with the gaseous samples in a 5-centimeter monel cell fitted with silver chloride windows.

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