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A DIVISION OF NORTH AMERICAN AVIATION, INC.
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B-5883-2

(Unclassified Title)

QUARTERLY PROGRESS REPORT,
INORGANIC HALOGEN OXIDIZERS
(1 September through 30 November 1964)

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

Contract Nonr 4428(00)

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

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DATE 31 December 1964

NO OF PAGES 12 & iv.

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FOREWORD

The research reported herein was supported by the Advanced Research Projects Agency 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 September through 30 November 1964.

The Responsible Engineer for this program is Dr. D. Pilipovich. Dr. H. F. Bauer and Dr. C. J. Schack are full-time associates. The work was carried out in the Chemistry Section of the Rocketdyne Research Department.
ABSTRACT

The reaction of solid FC102 with excited fluorine at -196 C gave rise to ClF5. The absence of ClF7 in the reaction products led to the premise that metastable species involving ClOF moieties may have been intermediates in the formation of ClF5.

Solid N2O4 at -196 C reacted with excited fluorine as NO+NO-. The principal products from the reaction were NF3O and NO2F.

A complete examination of the ClF5-NO2 and ClF5-NO systems by nuclear magnetic resonance (n.m.r.) spectroscopy showed conclusively that there were no interactions at -108 to -60 C. Chemical shifts and splitting constants in the mixture were virtually identical to those of the pure components.

The reaction of ClF4+AsF6- with H2O, N2O3, and (CF3CO)2 failed to give ClF3O or ClF2O+AsF6-.

(Confidential Abstract)
CONTENTS

Foreword ........................................ ii
Abstract ....................................... iii
Discussion ..................................... 1
  Discharge Reactions .......................... 1
  Metathetical Reactions ...................... 4
ClF$_5$-Oxynitrogen Fluorides ............... 7
Reactions of ClF$_5$ Complexes ............... 9
Experimental Details ........................ 11
References .................................... 12
DISCUSSION

DISCHARGE REACTIONS

Attempts to prepare new F-C1-O compounds by discharge reactions were continued. The principal effort used FC10₂ as the substrate. The techniques utilized were essentially those evolved earlier in this program, and were described in the first quarterly report (Ref. 1). It was shown previously that the frozen reactant, if much removed from the discharge zone, is not subjected to a high concentration of excited fluorine. Therefore, in these experiments the substrate was condensed immediately adjacent to the discharge zone or even partially in it.

When FC10₂ was fluorinated at liquid nitrogen temperature in this manner, it was observed that the original white to yellow solid developed a purple ring in that portion of the material nearest the discharge. At the conclusion of a run the cooling bath was removed and the volatile materials were immediately recondensed into the vacuum line at -196 °C. Upon warming to room temperature to measure the product gas volume and the recooling, some gases not condensible at liquid nitrogen temperature always remained. Presumably this noncondensible gas is fluorine and/or oxygen. After pumping this gas off, the remaining product gases were allowed to vaporize, and an infrared spectrum was taken.

Along with a trace of SiF₄ produced by attack on the glass, the products were unreacted FC10₂, contaminant ClO₂, and an appreciable concentration of ClF₅. It appears then that the discharge fluorination has resulted in the conversion of FC10₂ to ClF₅. Such a displacement by fluorine of oxygen bonded to chlorine has not been reported previously. Any reasonable interpretation of this result cannot be based upon a direct displacement reaction. Much more likely is the formation of an intermediate...
unstable hypofluorite compound. Such structures, while stable at low temperature, might easily rearrange upon warming to give both the observed ClF and noncondensible gases.

Although purely hypothetical, the following reaction scheme indicates some of the possible intermediate hypofluorites and shows how they might give rise to ClF:

\[
\begin{align*}
\text{FCIO}_2 + F_2 & \xrightarrow{\text{discharge}} \text{[F}_2\text{ClO}(0F)] \\
\text{[F}_2\text{ClO}(0F)] + F_2 & \xrightarrow{\text{discharge}} \text{[F}_3\text{Cl}(OF)] \\
\text{[F}_3\text{Cl}(OF)] & \rightarrow \text{ClF} + O_2 \\
2\text{[F}_3\text{Cl}(OF)] & \rightarrow \text{ClF} + \text{FCIO}_2 + O_2 + 2 F_2
\end{align*}
\]

Since no ClF was found, it is probably not involved in the conversion of FCIO2 to ClF. The absence of ClF also precludes Cl2 as an intermediate. In discharge reactions of F2 and Cl2, the ratio of ClF to ClF found was always on the order of 10 to 1.

To determine the generality of displacing oxygen from XO species by discharge fluorination, this reaction was carried out with sulfur dioxide. If any hypofluorites of sulfur are formed, they should be stable to warming (e.g., SF5OF), and thus the intermediate character of Cl-OF species in the overall reaction could be demonstrated. Under the same conditions employed with FCIO2, it was determined that SO2F2 and SF6 were produced. Thus, fluorination and displacement did occur, but intermediate OF species were not isolated.
In earlier experiments with Cl₂O and air-contaminated fluorine, it was noted that this discharge reaction also gave rise to some noncondensible gases upon warming to room temperature. Since in the absence of air no such gases were observed, it is possible that an unstable F-N-O compound such as F₂NO(OF) might be the source. To test this possibility several discharge runs were conducted using solid N₂O₄ as the reactant. The results of these reactions were somewhat ambiguous. In addition to a large amount of unreacted N₂O₄, some FNO₃ always was present with little or no FNO₂ found. This suggested that the N₂O₄ had reacted as though it were present in the ionic form (NO⁺NO₃⁻). However, the fact that NF₃ was at times formed instead of the expected NF₂O is perplexing. If the reaction proceeds through NO⁺NO₃⁻ predominantly, as appears to be the case, then it is not clear why in some instances NF₂O is formed and in others only NF₃ is formed. The predominant reaction path, however, is:

\[
\text{NO}^+\text{NO}^- + 2 \text{F}_2 \xrightarrow{\text{discharge}} \text{F}_3\text{NO} + \text{FNO}_3
\]  

(5)

Although present to a lesser degree than in the case of FClO₂, it was again found that the discharge reaction products upon first warming in the vacuum line gave some gases not condensible by liquid nitrogen.

The reaction of CIF₃ in a discharge at -80 C was examined. It was hoped that CIF₂ or CIF₄ radicals might be generated. Such radicals might react with the glass reactor to form new F-CI-0 compounds or couple to yield compounds such as (F₄Cl)₂. The coupling reaction is analogous to the preparation of B₂Cl₄ in a mercury electrode discharge apparatus (Ref. 2).

In a series of experiments beginning at the lowest voltage at which a discharge could be maintained and then at higher voltages, it was determined that no new species were obtained. At the lower voltages, the
ClF$_5$ was essentially unchanged. At higher voltages, the ClF$_5$ was completely destroyed, giving mainly F$_2$ and Cl$_2$. The other products were ClO$_2$, FCIO$_2$, and SiF$_4$.

The discharge reactions reported have indicated several areas in which new metastable compounds may have been produced. The formation of non-condensable gases during product workup may be indicative of the presence of O$_2$F$_2$. However, the characteristic color of O$_2$F$_2$ was never observed in any of the reaction mixtures. These discharge studies will be resumed when appropriate low-temperature infrared equipment is available for a more complete examination of the reaction products.

**METATHETICAL REACTIONS**

This first of the proposed metathetical reactions to be studied is that involving ClF$_3$ and HNF$_2$. This reaction had been studied at Rocketdyne in the past, but the presence of the impurities FCIO$_2$ and ClO$_2$ in the ClF$_3$ has obscured its course. It had been postulated that the NF$_3$ formed in these early experiments and also the deflagrations often encountered were due to the reaction of these impurities (Ref. 3). Therefore, to ascertain the true course of the ClF$_3$-HNF$_2$ reaction, the preparation of pure ClF$_3$ was undertaken.

Because simple physical methods are inadequate, it was necessary to form a complex with ClF$_3$ from which the impurities could be removed. Subsequent removal of the complexing agent would then result in pure ClF$_3$. Three complexes were studied. The complex with BF$_3$ forms readily at -80°C, at which temperature the ClO$_2$ is readily pumped off the solid complex. However, the FCIO$_2$ contaminant also complexes with BF$_3$ and thus is not separated.
It was rather surprising that FC10₂ could not be removed from the mixture since the complex, C10₂BF₄, has been reported to have some volatility at -80 °C (Ref. 4).

The complex with FNO was also formed at -80 °C, and because of the lower volatility of the solid, the impurities could be more readily stripped away at -46 °C:

\[
\text{FNO} + \text{CIF}_3 \xleftrightarrow{\Delta} \text{NO}^+ \text{CIF}_4^- \quad (8)
\]

However, it was then discovered that even though the solid is completely dissociated into its component gases at ambient temperature, these gases regenerate the solid complex despite the most rapid cooling. Therefore, subsequent fractionation fails to secure a separation.

It was finally determined that pure CIF₃ could be derived from the KF complex:

\[
\text{KF} + \text{CIF}_3 \xleftrightarrow{\Delta} \text{KCIF}_4^- \quad (9)
\]

It was formed from solid KF and liquid CIF₃ at room temperature in a stainless-steel cylinder. This technique is analogous to that recently reported by Whitney et al. (Ref. 5). Since the KCIF₄ is nonvolatile at room temperature, the excess CIF₃ and impurities are pumped out of the cylinder readily. By heating the bomb and its solid contents several minutes under vacuum, high-purity CIF₃ is obtained.
When first utilized, it was thought that this method was only upgrading the impure material since some small amounts of ClO$_2$ and FCIO$_2$ were still observed in the infrared spectrum of the ClF$_3$. This difficulty was soon clarified when it was determined that the impurities were arising during the charging of the infrared cell with the ClF$_3$. Decomposition was occurring by reaction of the ClF$_3$ with the inside of the metal ball joints connecting the cell to the vacuum line. This joint must necessarily be exposed to the air when running a spectrum and consequently becomes de-passivated each time the cell is used. When passivated prior to charging with ClF$_3$, no further decomposition was noted, and a true spectrum of the ClF$_3$ was obtained.

Initial experiments using equimolar amounts of ClF$_3$ and HNF$_2$ or excess ClF$_3$ have been conducted without deflagration. When warmed to room temperature and then recooled and fractionated, it was found that all the HNF$_2$ was consumed, giving rise to ClNF$_2$, N$_2$F$_4$, and NF$_3$. These are the same products obtained previously (Ref. 3). Most of the ClF$_3$ remained unchanged. When mixing and while at low temperature, the reactants were kept in a Teflon trap. Over a short period at -80 C there was no detectable increase in vapor pressure; however, warming to ambient temperature with subsequent cooling to -80 C gave higher than predicted vapor pressure. Since the vapor was exposed to metal, it may be that one reaction of the HNF$_2$ was its decomposition on the metal surface. To circumvent this possibility, future reactions of these materials will be conducted in a nonmetal system.
CIF₅-OXYNITROGEN FLUORIDES

Novel routes to new N-O-F, Cl-O-F, or Cl-F compounds may be realized by the fluorination of the hypothetical anions F₂NO⁻, F₂NO₂⁻, ClF₅O⁻, or ClF₅⁻.

Interactions between CIF₅ and either FNO or FNO₂ at low temperatures were considered as possible sources of the desired species:

\[ \text{NO}^+\text{CIF}_5^- \quad \text{or} \quad \text{CIF}_5^+\text{F}_2\text{NO}^- \]

Even slight interactions between these moieties should be detectable by low-temperature n.m.r. F¹⁹ spectra of the mixtures. The results of the n.m.r. study are shown in Table 1. All n.m.r. spectra were taken in quartz n.m.r. tubes flamed out under high vacuum before loading.

The chemical shifts and splitting constants of the mixture components were essentially the same as those of the pure component. The recurrent resonance near 100 ppm was attributed to FC₁₀₂ rather than FC₁₀ since some splitting would be expected from the nonequivalent fluorines predicted for FC₁₀. The appearance of FNO₂ in the FNO-CIF₅ mixture was noted, along with considerable NO and NO₂. The presence of NO caused troublesome bubbling in the tube and necessitated operation at -108 C rather than -80 C. No evidence for interactions leading to stable anions or new species was found. The absence of any interaction between either FNO₂ or FNO with CIF₅ by n.m.r. analysis is in sharp contrast to the reports of other workers. The reaction of CIF₅ and FNO₂ has been reported to yield a complex at -80 C which is capable of a reversible dissociation (Ref. 6). With FNO, CIF₅ has been reported to form a solid complex that is dissociated at -20 C (Ref. 7). Attempts at checking interactions by vapor pressure measurements were unsuccessful due to partial decomposition of
# TABLE 1

NUCLEAR MAGNETIC RESONANCE SPECTRA OF FN0-C1F_5 AND FN0_2-C1F_5 MIXTURES

<table>
<thead>
<tr>
<th>Components</th>
<th>Chemical Shift Relative to F_2, ppm</th>
<th>J Value, cps</th>
<th>Assignment</th>
<th>Temperature, °C</th>
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<tr>
<td>FN0_2</td>
<td>36</td>
<td>118</td>
<td></td>
<td>-41</td>
</tr>
<tr>
<td>FN0</td>
<td>34</td>
<td>116</td>
<td></td>
<td>-59</td>
</tr>
<tr>
<td>C1F_5</td>
<td>-60 broad unresolved peak</td>
<td></td>
<td></td>
<td>-80</td>
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<tr>
<td>FN0_2-C1F_5(1:5)</td>
<td>164</td>
<td>142</td>
<td>doublet</td>
<td>-80</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>142</td>
<td>quintet</td>
<td>-80</td>
</tr>
<tr>
<td></td>
<td>-3.5</td>
<td>133</td>
<td>C1F_5 doublet</td>
<td>90</td>
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<tr>
<td></td>
<td>30</td>
<td>130</td>
<td>C1F_5 quintet</td>
<td></td>
</tr>
<tr>
<td></td>
<td>95  singlet</td>
<td>123</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>167.5</td>
<td>130</td>
<td>C1F_5 doublet</td>
<td>90</td>
</tr>
<tr>
<td></td>
<td>-2.5</td>
<td>130</td>
<td>C1F_5 quintet</td>
<td></td>
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<tr>
<td></td>
<td>31.5</td>
<td>116</td>
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<td>C1F_5 quintet</td>
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<td>31  singlet</td>
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<td>101       singlet</td>
<td>130</td>
<td>C1F_5 quintet</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-62 broad unresolved peak</td>
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the mixtures in metal. When FNO and ClF$_5$ were allowed to combine at ambient temperature in metal, partial reaction gave N$_2$O, FNO$_2$, Cl$_2$, and NF$_3$O as products.

REATIONS OF ClF$_5$ COMPLEXES

Several attempts have been made to react the ClF$_5$-AsF$_5$ complex with oxygen-containing materials in the hope of utilizing the increased electrophilicity of the chlorine atom and thus producing new Cl-O-F compounds. The reaction of ice with the ClF$_5$-AsF$_5$ complex produced ClF$_5$ and AsF$_3$O when the mixture was warmed slowly from -80°C. It is possible that the initial formation of HF converts H$_2$O to H$_2$O$^+$, which makes oxygen attack by ClF$_4^+$ unlikely.

The reaction of ClF$_5$-AsF$_5$ with trifluoroacetic anhydride was attempted in the hope of obtaining an extension of the known reaction:

$$\text{NF}_3\text{OBF}_3 + (\text{CF}_3\text{CO})_2\text{O} \rightarrow \text{NO}_2^+\text{BF}_4^- + 2 \text{CF}_3\text{CFO}$$

(10)

Although ClF$_5$ reacted smoothly with (CF$_3$CO)$_2$O at 20°C to produce FCIO$_2$ and CF$_3$CFO, the ClF$_5$-AsF$_5$ complex reacted with sufficient vigor below 0°C to pop open the all-Kel-F/Teflon reactor so that no products were recovered.

The low-melting compound N$_2$O$_3$ was combined with ClF$_5$-AsF$_5$ at -80°C in the hope of producing the following reactions:

$$\text{N}_2\text{O}_3 + 2 \text{ClF}_5\cdot\text{AsF}_5 \rightarrow 2 \text{NO}^+\text{AsF}_6^- + [\text{ClF}_4\text{OCIF}_4]$$

(11)

$$[\text{ClF}_4\text{O ClF}_4] \rightarrow [\text{F}_3\text{CIO} + \text{ClF}_5]$$

(12)
The volatile products found were Cl₂ and ClF₅, but no FClO₂ or ClO₂.
Pumping on the solid residue, with warming, liberated some FNO₂. Analysis
of the residue showed the absence of chlorine. Fluorination of the
N₂O₃ appears to have occurred according to the following equation:

\[
5 \text{N}_2\text{O}_3 + 10 \text{ClF}_5 \cdot \text{AsF}_5 \rightarrow 5 \text{NO}_2^+ \text{AsF}_6^- + \]
\[
5 \text{NO}_2^+ \text{AsF}_6^- + \text{Cl}_2 + 8 \text{ClF}_5
\]  

(13)

The reactions of ClF₅ with Ag₂O or HgO have consistently produced good
yields of FClO₂. Since in the synthesis of Cl₂O from Cl₂, yellow HgO
is successful while red HgO is not, it was felt that the reaction of
ClF₅ with yellow HgO under mild conditions might produce the intermediate
product F₃ClO rather than FClO₂. A slow stream of ClF₅ diluted
with N₂ was passed through a Kel-F U-tube packed with a powdered mixture
of yellow HgO and Kel-F at 0 °C.

Reaction took place with the Kel-F, giving Cl₂, CF₄, CF₃Cl, and
CF₃CFCl₂Cl, as well as other unidentified halocarbons. No Cl-O-F
products were detected. The substitution of copper for Kel-F powder as
an HgO diluent is planned.
EXPERIMENTAL DETAILS

Chloryl fluoride was prepared from silver oxide and ClF₅ (Ref. 1). Nitrogen tetroxide, sulfur dioxide, and boron trifluoride were obtained from the Matheson Company. Nitrosyl fluoride was prepared fresh from the reaction of 1 mole fluorine with 2 moles of nitric oxide at -196 C. Difluoramine was prepared by acidification of an aqueous solution of N,N-difluorourea and fractionated before being used.

All discharge experiments were carried out using the previously mentioned apparatus (Ref. 1) with a 6-inch electrode gap. All runs had an approximate duration of 1 hour except those with ClF₅. In these runs the time was varied from a few minutes to 2 hours. In all cases, the fluorine pressure was maintained at 50 to 60 millimeters throughout the experiment.

The preparation of KClF₄ from ClF₃ and KF was not quantitative. Depending partly upon the length of mixing and also the amount of excess ClF₃, conversions of 50 to 80 percent were achieved. In addition to the long reaction room temperature, satisfactory yields of KClF₄ could be obtained by heating the reactants for 2 hours at 50 to 60 C. It was essential that the reaction cylinder be shaken during the reaction to achieve good yields.
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