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AD NUMBER
AD384873
CLASSIFICATION CHANGES
TO: unclassified
FROM: confidential
LIMITATION CHANGES
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AD 384873

Semi-Annual Progress Report No. 3

February 1, 1967 to July 31, 1967

A STUDY OF THE EXPLOSION LIMITS OF SIMPLE DIFLUORAMINO COMPOUNDS (U)

Contract No. Nonr-4065(00)

to

Office of Naval Research
Washington, D. C. 20360

CLASSIFICATION UNCLASSIFIED
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DATED
3-20-69 THOMAS F. DAVIS
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from

Kinetics and Combustion Group
Atlantic Research Corporation
Alexandria, Virginia

[illegible] 1967

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September, 1967

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I. ABSTRACT

The explosion products of 1,2-DP were analyzed with the aid of a Beckman GC-2A Gas Chromatograph. For explosive conditions the volume of the major products N_2:C_2H_2: HCN was 2.5:1:1. For non-explosive decomposition conditions N_2 and C_2H_2 appeared in about the same quantities and HCN was slightly less than C_2H_2. The survival of thermodynamically unstable HCN and C_2H_2 indicate primary reaction products, an important consideration in the formulation of the chain reaction mechanism.

F_2 induced explosion and inhibition of explosion by O_2 is described by the reaction scheme

$$
\begin{array}{ll}
(1) & F + DP \rightarrow NF + F \\
 & \qquad\qquad\;\; \lfloor\!\rightarrow \text{destruction} \\
(2) & F + DP \rightarrow NF_2 \rightarrow \text{destruction} \\
(3) & F + DP + F_2 \rightarrow 2F \\
(4) & F + DP + O_2 \rightarrow \text{destruction}
\end{array}
$$

The postulated two modes of reaction of F and DP are reconciled with the high temperature explosion scheme of pure DP vapor.

II. INTRODUCTION

This is the third semi-annual progress report on this research program. In this program we are studying the explosion limit behavior of a series of bis-difluoramino compounds. The compounds that are of interest to us are 1,2-bisdifluoraminopropane (1,2-DP); 2,2-bisdifluoraminopropane (2,2-DP); 2-methyl-1,2-bisdifluoraminopropane (IBA); 1,3-bisdifluoraminopropane (1,3-DP); 1,1-bisdifluoraminopropane (1,1-DP); and 2,3-bisdifluoraminobutane (2,3-DB).

III. STATUS OF THE PROJECT

At the start of the present period the vapor phase explosion limit behavior of 1,2-DP, 2,2-DP, IBA, and 1,3-DP has been characterized. We have studied these limits at pressures of 1-200 torr, at temperatures between 295°C and 450°C, and vessel diameters ranging from 4.6 cm to 17 cm. In addition the effect on the limit of inert gas and the effect of the presence of one DP on the limit of another has been studied. All of the evidence presented by this data indicate that the explosion of 1,2-DP and 2,2-DP is governed by a branching chain mechanism with chain branching occurring in the gas phase and chain breaking occurring in the gas phase and at the wall. In addition, the gas-phase chain-branching and breaking reactions involve collisions of the same molecular species and are of the same order with respect to the chain carrier. The equations expressing the explosion limits are

$$\text{1,2-DP} \qquad p^2d^2 = \frac{1.5 \times 10^{-3} \, \frac{658}{T} \, e^{16,600/RT}}{(2e^{-900/RT} - 1)}$$

$$\text{2,2-DP} \qquad p^2d^2 = \frac{1.5 \times 10^{-3} \, \frac{568}{T} \, e^{14,500/RT}}{(2e^{-782/RT} - 1)}$$

Explosion limit data for 1,3-DP indicate a constant pd^2 product. As has been explained in the previous report, this signifies that the chain-branching step is unimolecular. Furthermore, because of the absence of a low temperature limit within the pressure range studied we must eliminate the gas-phase chain breaking step and depend only on the vessel surface for chain destruction.

The equation resulting from this data for the 1,3-DP explosion limit is

$$pd^2 = 435/4.8 \times 10^{13} e^{-44,500/RT}$$

IBA explosions do not lend themselves to this type of an analysis because a self-inhibition reaction which occurs prior to explosion obscures the limit.

IV. PROGRESS DURING THE PRESENT PERIOD

A. Analysis of Products of DP Explosions

It has been previously determined that the major gaseous products of the explosion of DP are HCN, N_2, C_2H_2, and HF. During the present period an effort was made to establish what quantitative relationship existed between these products. For this purpose we obtained a Beckman GC-2A Gas Chromatograph. The manifold containing the explosion reactor was attached to the inlet of the chromatograph. Because the pressure in the vessel after explosion is only a small fraction of an atmosphere (∿50 torr) the sample was introduced through a gas-sampling valve placed before the column. The column found to be most satisfactory for this analysis was a stainless steel (thin wall), 6-ft column containing silicone 550, 30% on C-22 firebrick, 42/60 mesh. The program used was 70°C column temperature, He-flow rate 33cc/min for 2 minutes, then 86cc/min for the remainder of analysis, filament current 200 milliamp. 1,2-DP explosion products were used for these analyses. A sampling valve loop with a volume of 9.2cc was used to probe the explosion mixture.

Interpretation of the chromatograph curves proved difficult due to the multitude of small minor peaks, many of which shouldered the larger major peaks. Using the program described above, we obtained peaks for N_2, C_2H_2, and HCN at 1.3, 1.45, and 1.8 minutes respectively. SiF_4 could not be desorbed from the column under conditions that still gave satisfactory results for the N_2, C_2H_2, and HCN retention times. The explosion conditions for 1,2-DP were 1 liter reactor, 18 mm of DP, and 409°C. The final pressure after explosion was 64 mm. The ratio $N_2:C_2{}_2$:HCN was about 2.5:1:1. In addition, unreacted DP residing in the manifold lines outside the reactor was recovered.

When 20 mm of 1,2-DP was decomposed non-explosively in the one-liter reactor at 384°C, N_2 and C_2H_2 appeared in about the same quantities and HCN was slightly less than C_2H_2. We conclude that there is no fundamental difference in the over-all chemistry of the explosive and non-explosive reaction. The significant fact is the survival of C_2H_2 and HCN as major reaction products. Since both compounds are thermodynamically unstable at the temperatures and pressures of these experiments, they evidently represent primary decomposition products of the DP-molecule. This is an important consideration in the formulation of the chain reaction mechanism, as has already been noted in earlier reports. The larger yield of N_2 in the explosion experiments does not seem to be significant. It may be expected that less C_2H_2 and HCN survive in the explosion transient than in the non-explosive reaction, so that more of the stable products N_2, carbon and H_2 are formed. We did not attempt to determine yields of the two latter products. No trace of fluorocarbon was found. It appears that fluorine reacts to form HF which in turn reacts with the glass reaction vessel to form SiF_4.

B. Branched-Chain Explosion of DP-Fluorine Mixtures

It was found that mixtures of DP and fluorine explode spontaneously at low temperatures when the fluorine concentration exceeds a critical limit. This discovery resulted from attempts to prepare mixtures of DP and fluorine at room temperature. For this purpose fluorine was gradually added in small increments to 1,2-DP vapor. The reaction vessel was a 500cc Pyrex bulb and the Pyrex inlet tube was provided with two stopcocks in series, so as to leave a space of 3cc between the stopcocks. Fluorine was admitted to this space at a pressure exceeding the 1,2-DP pressure in the vessel; in this way it was possible to admit a small amount of fluorine to the reactor by closing the upper stopcock and opening the lower stopcock. Table 1 summarizes the data and observations.

TABLE 1

Fluorine-Induced Explosion of 1,2-DP Vapor at Room Temperature (Experiments performed by Mr. Wesley Copeland.)

Experiment	Pressure, mmHg		Comments
	1,2-DP	1,2-DP + F_2	
1	20mm	20.9	F_2 added in increments of app. 0.25mm, no visible reaction after standing 2 hrs.
2	22	22.9	F_2 added in increments of 0.45mm, no reaction.
		23.5	The addition of 0.6mm more of F_2 produced an explosion. Final pressure 61mm.
3	18.5	19.7	1.2mm of F_2 put in, in one shot. No visible reaction.
		20.15	0.45mm more F_2 added - explosion; final pressure, 51mm +
		51 + .66	0.66mm more F_2 added - another smaller explosion.
4	19.5	21.0	1.5mm of F_2 added in one shot - small flame in bore of stopcock but no explosion. Final pressure 21.0mm
		21.3	0.3mm more of F_2 put in, in 0.15mm shots. No visible reaction.

By infrared analysis it was shown that the explosion products contain HCN and C_2H_2. SiF_4 and CF_4 were also identified.

Numerous additional data were obtained using the following apparatus and experimental procedure (Fig. 1). A vacuum line made up of teflon tubing, fittings, and valves was built to handle the fluorine experiments. Inlets were provided for the DP, the additive gases as well as fluorine. A glass capillary and bulb mercury manometer measured the pressures in the line. The bulb was coated with a layer of Kel-F wax and the mercury with Kel-F oil. A glass reactor was fabricated with separate inlets for DP and for the additive gases in order to prevent mixing of the DP and the additives in the stopcock bore. Pressures in the reactor volume were measured by a U-tube mercury manometer. After the system was completely evacuated a measured amount of DP was led into the reactor with stopcocks (1) and (3) open and (2) closed. Additive gases fill the line with (2) and (3) closed and (1) open to a pressure determined by the ratio of volumes defined by the three stopcocks and the reactor volume according to the formula

$$\%\ \text{additive} = \frac{v}{V}\left(\frac{\frac{p}{P} - 1}{\frac{v}{V}\frac{p}{P} + 1}\right) \times 100$$

Where p and v denote the pressure and volume between stopcocks 1, 2, and 3, and P and V denote the pressure volume of the reactor. Table 2 contains the data obtained in these experiments.

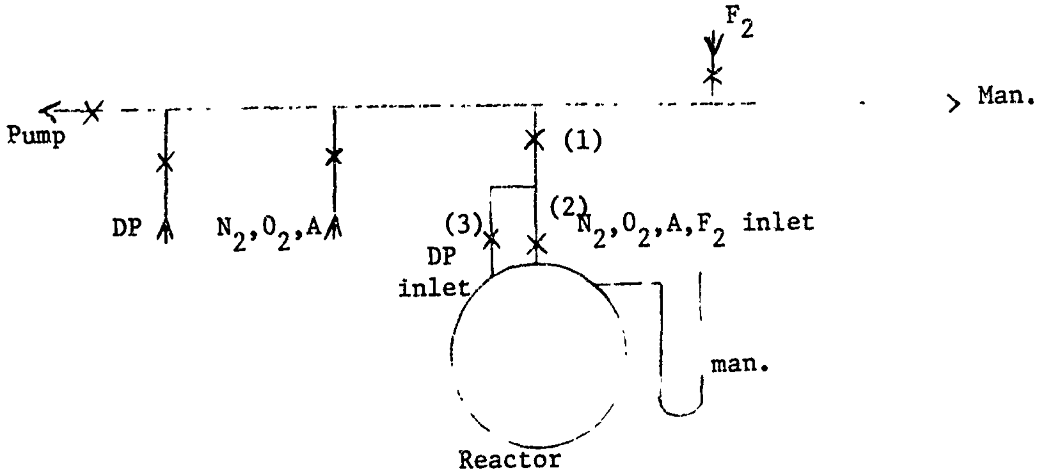


Fig. 1. Scheme of Apparatus for Admitting F_2 to DP Vapor

TABLE 2

Critical Pressure of Fluorine at Fluorine-Induced DP-Explosion Limit. Effect of DP-Pressure, Temperature, Vessel Dimension and Additives

	DP Pressure torr	Temp. °C	Volume cc	Pressure O_2 torr	Pressure N_2 torr	Pressure A torr	Pressure F_2 at limit torr
1,2 DP	8	25	200	-	-	-	2.3,2.5→2.8,2.3
	20	25	200	-	-	-	2.0
	8	55	200	-	-	-	2.5
	8	1	200	-	-	-	3.5
	8	8	200	-	-	-	3.0
	8	100	200	-	-	-	1.24
	8	25	1000	-	-	-	1.40
	8	25	200	2.2	-	-	6.6→6.9,4.8
	8	25	200	2.2	-	9.8	7.0→7.3
	8	25	200	-	2.3	-	2.3
	8	25	200	1.1	-	-	3.5
	8	25	200	3.6	-	-	8.0
2,2 DP	8	25	200	-	-	-	2.8→3.4
IBA	8	25	200	-	-	-	<0.2
	8	25	1000	-	-	-	0.58
	8	25	200	-	2.3	-	0.4
	8	25	200	5.0, 7.4, 2.3	-	-	0.4
1,3 DP	8	25	200	-	-	-	0.4
	8	25	200	2.2	-	-	3.0→3.3,4.6
	8	25	200	2.2	-	9.2	4.1
	8	25	200	2.2	2.1	-	3.9
	8	25	200	1.1	-	-	1.0

From the data in Tables 1 and 2 we draw the following general conclusions:

(1) Within the range of experimental scatter the critical fluorine pressure is independent of the pressure of DP vapor, the vessel dimension and inert-gas addition.

(2) There is a small decrease of the critical fluorine pressure over the temperature range from 1° to 100°C.

(3) Addition of oxygen causes a substantial increase of the critical fluorine pressure; this increase is unaffected by simultaneous addition of inert gas.

The experimental facts rule out prima facie any thermal interpretation of the explosion phenomenon, which is thus identified as a branched-chain explosion. In performing these experiments we have unfortunately not been aware of an obvious inference from fact 3; namely, since oxygen is an inhibitor in these explosions, and since elementary fluorine may react slightly with the glass wall of the apparatus to liberate oxygen, there exists the probability that all these data are slightly falsified by traces of oxygen. Experiments are planned to test this point; we feel, however, that despite this uncertainty the present data are sufficiently definitive to permit valid conclusions concerning the kinetics and chemical mechanism of the fluorine-induced explosive reaction.

At the explosion limit the rate of chain breaking equals the rate of chain branching. Fact 1 signifies that both chain breaking and chain branching occur in the gas phase; that both rates are of the same order with respect to the DP-concentration; and that chain branching involves collisions with F_2-molecules. Thus, if X denotes a chain carrier, the reaction scheme might be of the general type

$$\text{(a)}\quad X + DP \rightarrow \text{destruction}$$

$$\text{(b)}\quad X + DP + F_2 \rightarrow nX$$

so that the explosion condition becomes

$$k_a\,[DP][X] = k_b\,[DP][F_2][X]$$

or

$$[F_2] = k_a/k_b \qquad (1)$$

Alternatively, the scheme might be

$X + DP \rightarrow R$ (an intermediate species)

(a) $R \rightarrow$ destruction

(b) $R + F_2 \rightarrow nX$

which yields again $[F_2] = k_a/k_b$. The difference is that in the former case reaction (a) is bimolecular and reaction (b) is trimolecular; in the latter case the reactions are unimolecular and bimolecular, respectively.

Typical or "ballpark" values of the pre-exponential factors of rate coefficients may be quoted as

10^{12} sec^{-1}	for unimolecular reactions
10^{-11}cm^{3}sec^{-1}	for bimolecular reactions
10^{-34} cm^6 sec^{-1}	for trimolecular reactions

Thus, for both cases, roughly,

$$[F_2] \approx 10^{23}\ e^{-(E_a - E_b)/RT} \qquad (2)$$

and since for 1,2-DP at room temperature according to Tables 1 and 2,

$$[F_2] \approx 2\text{mmHg} \approx .7 \times 10^{17}\ \text{molecules/cm}^3,$$

one calculates the activation energy $E_a - E_b$ to be approximately 8,500 cal/mole. On this basis, $[F_2]$ should increase over the temperature range from 1°C to 100°C by a factor of approximately 80. However, according to fact 2 $[F_2]$ does not increase at all but decreases instead over this temperature range.

We may introduce a "steric" factor to reduce the value of the pre-exponential factor of reaction (a); but in order to accommodate the theory to fact 2 the steric factor would have to be absurdly small, so as to be totally implausible on the basis of collision concepts.

We resort instead to an interpretation which not only removes the present difficulty but also another hitherto unacknowledged difficulty which is to be found in our previous reaction scheme for the branched-chain explosion of DP at high temperature. There we have proposed the chain-branching reaction

(1) $NF_2 + DP \rightarrow HCN + N_2 + C_2H_2 + 3HF + 3F$

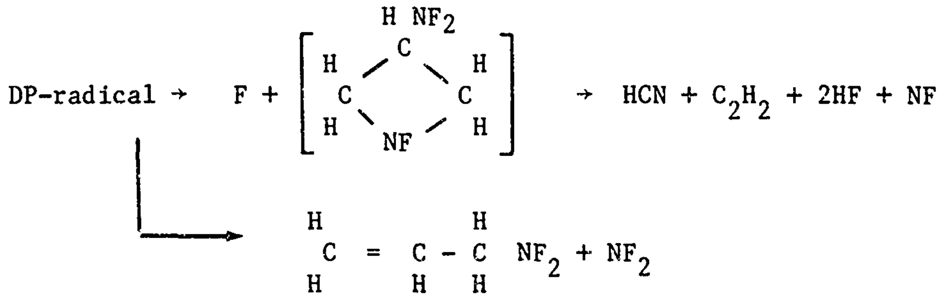
and the chain-breaking reaction

(2) $NF_2 + DP \rightarrow HCN + N_2 + C_2H + 4HF + F_2$

with a difference of activation energy $E_1 - E_2$ of only 900 cal/mole. This is an absurdly small energy difference, considering that reaction (1) yields three free atoms F and reaction (2) none. (The radical C_2H results in this concept from the exothermic reaction $C_2H_2 + F = C_2H + HF$.)

We propose that the key to the situation is to be found in the internal molecular rearrangements that occur upon attack of a DP-molecule by a fluorine atom. This attack results in bonding of F with one of the H-atoms of the DP-molecule, with a net exothermicity of approximately 55 kcal/mole. The free C-bond thus generated will then preferentially engage one of the two NF_2-groups, forming C-N of bond energy ≈55 kcal/mole and breaking one of the stronger N-F bonds (≈70 kcal/mole) by partial utilization of the 55 kcal/mole available from the F-atom attack. Alternatively, but much less frequently, the free C-bond engages the adjacent C-atom, forming a double bond C=C and releasing an NF_2-group. Thus,

$$F + DP = HF + DP\text{-radical}$$



The argument continues from here along the lines developed in the previous reports, except that now the dominant chain carrier of the high-temperature explosion is not NF_2 but NF. Thus, the high-temperature explosion scheme becomes

(1) $NF + DP \rightarrow HCN + C_2H_2 + N_2 + 3HF + 2F$

(2) $NF + DP \rightarrow HCN + C_2H_2 + N_2 + 3HF + F_2$

(3) $NF \xrightarrow{wall}$ destruction

$NF_2 \xrightarrow{wall}$ destruction

(4) $F + DP = HF +$ DP-radical

(5) DP-radical $\rightarrow HCN + C_2H_2 + 2HF + NF + F$

(6) DP-radical $\rightarrow HCN + C_2H_2 + 2HF + NF_2$

and the explosion boundary condition becomes (with $k_5 >> k_6$)

$$\frac{2\,k_1}{k_2}\,\frac{k_5}{k_6} - 1 = \frac{K_3}{k_2\,[DP]} \qquad (3)$$

which is of the same form as the former equation (1) in Semi-Annual Report No. 1, and permits substantially the same analysis of the data. We have merely removed the logical difficulty of attributing nearly identical activation energies to the collision processes (1) and (2) and have introduced instead the concept of two different modes of break-up of the energized DP-radical, one mode being much more frequent than the other.

With respect to the F_2-induced low-temperature explosion, we admit the additional ternary reaction

(7) $F + DP + F_2 \rightarrow HCN + C_2H_2 + 3HF + NF_2 + 2F$

which implies that the transient (DP-radical + F_2) collision complex is sufficiently energized to permit the exchange $NF + F_2 = NF_2 + F$. Furthermore, the kinetic analysis of the high-temperature explosion limit leads to the conclusion that at the low temperatures of the fluorine experiments the radicals NF and NF_2 do not significantly react with DP. From literature data (ref. 1) it can also be shown that under the conditions of these experiments these radicals do not significantly react in collisions with F_2. Thus, the fluorine-induced explosions are described by reactions (3), (4), (5), (6) and (7), yielding the explosion boundary condition

$$[F_2] = \frac{k_4}{k_7} \frac{k_6}{k_5} \quad (4)$$

On the basis of the arguments presented in connection with equations (1) and (2), we conclude that reactions (4) and (7) have very low activation energies and that the ratio k_5/k_6 is of the order of 10^6.

Returning to equation (3), if the difference of activation energies between reactions (1) and (2) were of the order of the dissociation energy of F_2, i.e., 35.6 kcal/mole, k_5/k_6 would be of the order of 10^{11}. The temperature is here about 400°C as compared to room temperature in the fluorine experiments. This temperature difference may be a factor in the disparity of the values of the ratio k_5/k_6; it is, however, reasonable to assume that the difference of activation energies between reaction (1) and (2) is considerably less than the F_2-dissociation energy, owing to intramolecular energy available in the break-up of the collision complex (NF + DP); and hence the disparity of the values k_5/k_6 is considerably diminished.

The inhibition of the fluorine-induced explosion by oxygen is explained quite simply by the additional ternary reaction

(8) $F + DP + O_2 \rightarrow$ products other than F

whose detailed chemistry may be imagined in many ways. The explosion condition becomes

$$[F_2] = \frac{k_4}{k_7} \frac{k_6}{k_5} + \frac{k_8}{k_7} [O_2] \quad (5)$$

which is of a form that appears to be consistent with the data.

V. PLANS FOR THE FUTURE

Further chemical and kinetic studies are planned with a view of refining the concept of free-radical reactions and intramolecular rearrangements of difluoramino compounds, to yield active and inert molecular fragments. Attention will be given to effects of molecular structure, including structures comprising more than three carbon atoms in the molecule, and to coordination of our data and concepts with work performed elsewhere, particularly at S.R.I. Additional experiments on fluorine-induced explosions are included in this program. A special effort will be made to eliminate any fortuitous inhibition of the reaction by oxygen contamination.

We expect that during the current period the work on difluoramino propanes will be substantially completed and significant data on the butane members of the series will be obtained.

VI. REFERENCES

1. J. B. Levy and B. K. Wesley Copeland, J. Phys. Chem. 69, 3700 (1965).

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1 ORIGINATING ACTIVITY *(Corporate author)*
Atlantic Research Corporation
Shirley Highway and Edsall Road
Alexandria, Virginia

2a REPORT SECURITY CLASSIFICATION
CONFIDENTIAL

2b GROUP

3 REPORT TITLE
A STUDY OF THE EXPLOSION LIMITS OF SIMPLE DIFLUORAMINO COMPOUNDS (U)

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*
Semi-Annual Progress Report No. 3, February 1, 1967 to July 31, 1967

5 AUTHOR(S) *(Last name, first name, initial)*
von Elbe, Guenther
Levy, J. B.
White, G.

6. REPORT DATE
September, 1967

7a. TOTAL NO OF PAGES
13

7b. NO. OF REFS
1

8a CONTRACT OR GRANT NO.
Nonr-4065 (00)

b. PROJECT NO.

c.

d

9a. ORIGINATOR'S REPORT NUMBER(S)

9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

10 AVAILABILITY/LIMITATION NOTICES

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY
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DD FORM 1 JAN 64 1473

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	ROLE	WT	ROLE	WT	ROLE	WT
bisdifluoraminopropane						
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