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Quarterly Technical Summary Report No. 8  
January 1, 1965 to March 31, 1965

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

ARPA Order No. 410  
ARPA Project Code No. 3730  
Contract No. Nonr-4065(00)

to

Advanced Research Projects Agency  
Washington 25, D. C.

and

Office of Naval Research  
Washington 25, D. C.

from

Kinetics and Combustion Group  
Atlantic Research Corporation  
Alexandria, Virginia

May 11, 1965

Chief Investigator: J. B. Levy  
Internal Consultant: G. von Elbe  
Scientist: J. W. Miller  
Scientist: E. T. McHale

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

**I. ABSTRACT**

The products of the explosive and non-explosive thermal decompositions of 1,2-bisdifluoramino-2-methylpropane (IBA) have been analyzed for hydrogen cyanide and acetylene and products not condensable in liquid nitrogen. The hydrogen cyanide yields for explosion are approximately one mole per mole IBA consumed; for non-explosive decomposition the yields are much lower. Similar results are found for acetylene.

The explosion limit curve for equimolar IBA-NO mixtures intersects that for pure IBA near 415°C. The former curve is lower at temperatures below 415°C.

An analytical method utilizing infrared spectroscopy has been developed to determine the rate of thermal decomposition of 2,2-bisdifluoraminopropane. Preliminary results support the belief that the reaction is homogeneous, first-order and has an activation energy of about 45 kcal/mole.

**II. INTRODUCTION**

This is the eighth quarterly progress report on this research program. In this program we are studying the explosion limit behavior of simple bisdifluoramino compounds. We hope to elucidate the basic nature of the explosive phenomena and to determine whether a relationship exists between explosions, as carried out in these studies, and sensitivity characteristics, as, for example, determined by impact testing. The compounds that are of interest to us are: 1,2-bisdifluoraminopropane, 2,2-bisdifluoraminopropane, 1,1-bisdifluoraminopropane, 1,3-bisdifluoraminopropane and 2-methyl-1,2-bisdifluoraminopropane. These compounds will be referred to in this report as 1,2-DP, 2,2-DP, 1,1-DP, 1,3-DP and IBA respectively.

**III. STATUS OF THE PROJECT AT THE START OF THE PRESENT PERIOD**

The experiments that we have performed to date may be grouped into the following: explosion limit measurements (including effects of geometry and added gases), thermal decomposition studies, product analysis, and temperature profile measurements during the induction period. Very few results have been obtained in the last area because of the experimental difficulties inherent in the fabrication of fine thermocouples into the reaction flasks and the high rate of their destruction during explosions. The results that have been obtained in the above areas will be summarized below.

**A. Explosion Limit Measurements**

We have determined explosion limits-temperature curves for 1,2-DP, 2,2-DP and IBA. The principal features of these results are:

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1. For explosion limit pressures of 5-50 mm the curve for 2,2-DP is about 100°C lower than that for 1,2-DP.

2. Although explosion limit data for 1,2-DP and 2,2-DP could be determined in Pyrex flasks of nominal 200 cc and 1000 cc capacity, explosions could not be achieved with IBA in a 200 cc flask. An explosion limit-temperature curve was determined in a 1000 cc flask and it lay about 20-30°C higher than the comparable curve for 1,2-DP.

3. For 1,2-DP and 2,2-DP, in experiments below the explosion limit, the pressure-time curve was a line of small positive slope. For IBA, in experiments below the explosion limit an abrupt pressure increase of four-fold was observed, followed by a further slow pressure rise comparable to that observed for the other compounds.

4. The presence of nitric oxide in an amount equimolar to the difluoramino compounds lowered the limit curve for 1,2-DP and raised it for 2,2-DP.

#### B. Thermal Decomposition Studies

Manometric measurements of the rate of thermal decomposition have been performed for 1,2-DP and 2,2-DP in Pyrex flasks. The evidence is strong that for the temperature range 250°C-350°C, the former compound decomposed by a heterogeneous mechanism; for the temperature range 276-300°C, the thermal decomposition of the latter compound appears to be homogeneous, first order, and to have an activation energy of about 40 kcal/mole.

#### C. Product Analysis Experiments

##### 1. The Explosion Products

##### a. Qualitative Results

In all the explosion limit experiments to date the products have been in contact with glass either in a reaction vessel itself or in the vacuum lines exterior to the vessel. The absorption peaks of silicon tetrafluoride have been prominent in the infrared spectra of the products of all the compounds studied. An obvious precursor of silicon tetrafluoride is hydrogen fluoride. However the possibility that other fluorine compounds have reacted with the glass to yield silicon tetrafluoride cannot be excluded.

For 1,2-DP and 2,2-DP the procedure of separating the reaction products, by means of a Toepler pump, into a fraction non-condensable in liquid nitrogen and a fraction condensable in liquid nitrogen has been followed. Nitrogen and hydrogen have been identified in the non-condensable fraction. Hydrogen cyanide, acetylene and silicon tetrafluoride have been identified in the condensable fraction. For IBA the only results to date are that silicon tetrafluoride, hydrogen cyanide and acetylene are products.

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In addition, explosion of each of these compounds is accompanied by the deposition of a black solid on the walls of the reaction flask. In the experiments with nitric oxide, the formation of this solid is very substantially reduced.

b. Quantitative Results

Only limited data are available on the quantitative nature of the product distribution. Mass spectrographic analysis has indicated the following result for the products of a 1,2-DP explosion at 414°C.

TABLE I  
The Products of Explosion of 1,2-DP at 414°C

<u>N<sub>2</sub></u>	<u>H<sub>2</sub></u>	<u>CO</u>	<u>HCN</u>	Moles per mole DP	
				<u>C<sub>2</sub>H<sub>2</sub></u>	<u>S<sub>1</sub>F<sub>4</sub></u>
0.22	0.12	0.11	0.98	0.17	0.52

We feel that this area, that of product analysis, deserves more effort than we have given it to date.

2. Products of Non-Explosive Decompositions

The principal differences between the products of explosive and non-explosive experiments are that acetylene is a prominent product in the former and is virtually absent in the latter and that the ratio of product moles to reactant moles is much higher in the former than in the latter.

D. Temperature Measurements During Induction Period

The extent of the temperature rise during the induction time to explosion is an important clue as to the nature of the explosion. For a thermal explosion, thermal theory predicts the magnitude of this rise (1). For branched chain explosions the temperature rises are much smaller than the above prediction (2) since it is chain carrier accumulation that is important, not temperature rise. For this reason we have been most interested in measuring temperature histories during the induction period to explosion. The thermocouples for this purpose must be fabricated from wires that are very fine so that the heat capacities and conduction of the wires do not interfere in the readings. We have made many measurements with .001 inch chromel-alumel and Pt-Pt, 10 per cent Rh thermocouples. However, such a high incidence of breakage occurs that little in the way of useful results have been obtained. At 406°C (3) a temperature rise of ~30°C was found for a 1,2-DP explosion. This indicated a reaction having an energy of activation of 48 kcal/mole.

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IV. PROGRESS DURING PRESENT PERIOD

During the present period we have performed more experiments on IBA and on 2,2-DP. These are discussed below.

A. Experiments with IBA

1. Product Analyses Experiment

In our last report (4) the presence of hydrogen cyanide, acetylene and silicon tetrafluoride in the products of the explosion of IBA was reported. In the present period we have performed experiments whose aim was the quantitative determination of these compounds.

In the procedure used to date, the products of an explosion are transferred to a reservoir bulb, via a liquid nitrogen-cooled trap, by means of a Toepler pump. The material passing this trap exhibits no absorption in the infrared region and is referred to as the non-condensable fraction (n.c.). The liquid nitrogen trap is then replaced by a  $-80^{\circ}\text{C}$  trap and the material that vaporizes is Toepler-pumped to a reservoir flask. Auxiliary experiments with hydrogen cyanide and acetylene showed that hydrogen cyanide is retained in the  $-80^{\circ}\text{C}$  trap while acetylene is transferred to the reservoir. The residual contents of the  $-80^{\circ}\text{C}$  trap are then dissolved in 6N ammonium hydroxide and the cyanide content determined by an argentometric technique (5).

The acetylene is determined by means of its absorptions at  $3230\text{ cm}^{-1}$  and  $1340\text{ cm}^{-1}$ . We hope to determine silicon tetrafluoride by making use of the fact that sodium fluoride pellets (prepared by heating pellets of sodium hydrogen fluoride in nitrogen so that the hydrogen fluoride is driven off) absorb silicon tetrafluoride quantitatively.

We have obtained scattered preliminary results with IBA and these are tabulated below.

TABLE II  
Products of IBA Explosion and Thermal Decomposition

Expt.	Temp.	Expl.	Moles per Mole IBA			Pf/P <sub>o</sub>
			n.c.	HCN	C <sub>2</sub> H <sub>2</sub>	
258-2	407	+	.72			4.4
259	406	+	.66			4.8
259	406	+	.70			4.8
259	406	+	.70			4.8
260	405	+	.61			4.3
260	405	+	.61			4.3
261-4	401	+	.68		.42	4.3
261-1	401	+	.72	1.07		4.7
262-2	396	+		0.95		4.9
263	408	+	.60	.98	.32	4.2
264-1	415	+	.65		.38	4.4
265-1	415	+	.78	1.29	.44	5.0
266-1	418	+	.77	1.26	.44	5.0
268	416	-		0.31		3.3

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Expt.	Temp.	Expl.	n.c.	HCN	C <sub>2</sub> H <sub>2</sub>	Pf/P <sub>0</sub>
	420	-				1.9
269	411	-	0.35	0.33	absent	
270	405	-	0.35	0.51		
259	406	-	0.41			3.3
263-2	408	-		0.11		2.7

These initial results for the explosion products are quite consistent. The acetylene yield is fairly constant at about 0.3 - 0.4 moles per mole IBA and the HCN yield is close to unity. When we are able to determine the silicon tetrafluoride we will know how much of the products we can identify.

A few experiments have been performed for non-explosion experiments and these are included in the table. The results are somewhat more erratic but they show markedly lower HCN yields. In experiment 269 where the product infrared spectrum was examined for acetylene, no absorption at 725 cm<sup>-1</sup> was observed--i.e., acetylene was absent.

## 2. Explosion Limit Measurements with Equimolar IBA-NO Mixtures

The effect of nitric oxide on the explosion limit curve of 1,2-DP was to lower it--i.e., the mixture was more explosive than pure 1,2-DP at the same partial pressure. For 2,2-DP the addition of nitric oxide raised the limit. We have performed a number of experiments with equimolar IBA-NO mixtures over the temperature range 393-450°C in a nominal one liter bulb. These results compared to the results for pure IBA in Fig. 1. The curve for IBA-NO is lower than the curve for pure IBA above about 410°C and higher below this temperature. The situation is evidently more complex for IBA than for either of the other two compounds studied to date.

## B. Further Measurements of the Kinetics of the Thermal Decomposition of 2,2-DP

In the preceding report (4), we reported that manometric measurements of the rate of thermal decomposition of 2,2-DP in Pyrex vessels of differing surface area-to-volume ratio indicated that the reaction was homogeneous in the temperature range 276-300°C. We felt that it would be desirable to investigate the kinetics of this reaction by a method that would be more reliable than the manometric method. The general method we have considered is that of withdrawal for analysis of relatively small samples from a rather large reaction volume. We have considered the use of gas chromatography and infrared spectroscopy as analytical techniques and have performed preliminary experiments with both. The results using infrared spectroscopic analyses have been the more satisfactory and we have concentrated on this technique.

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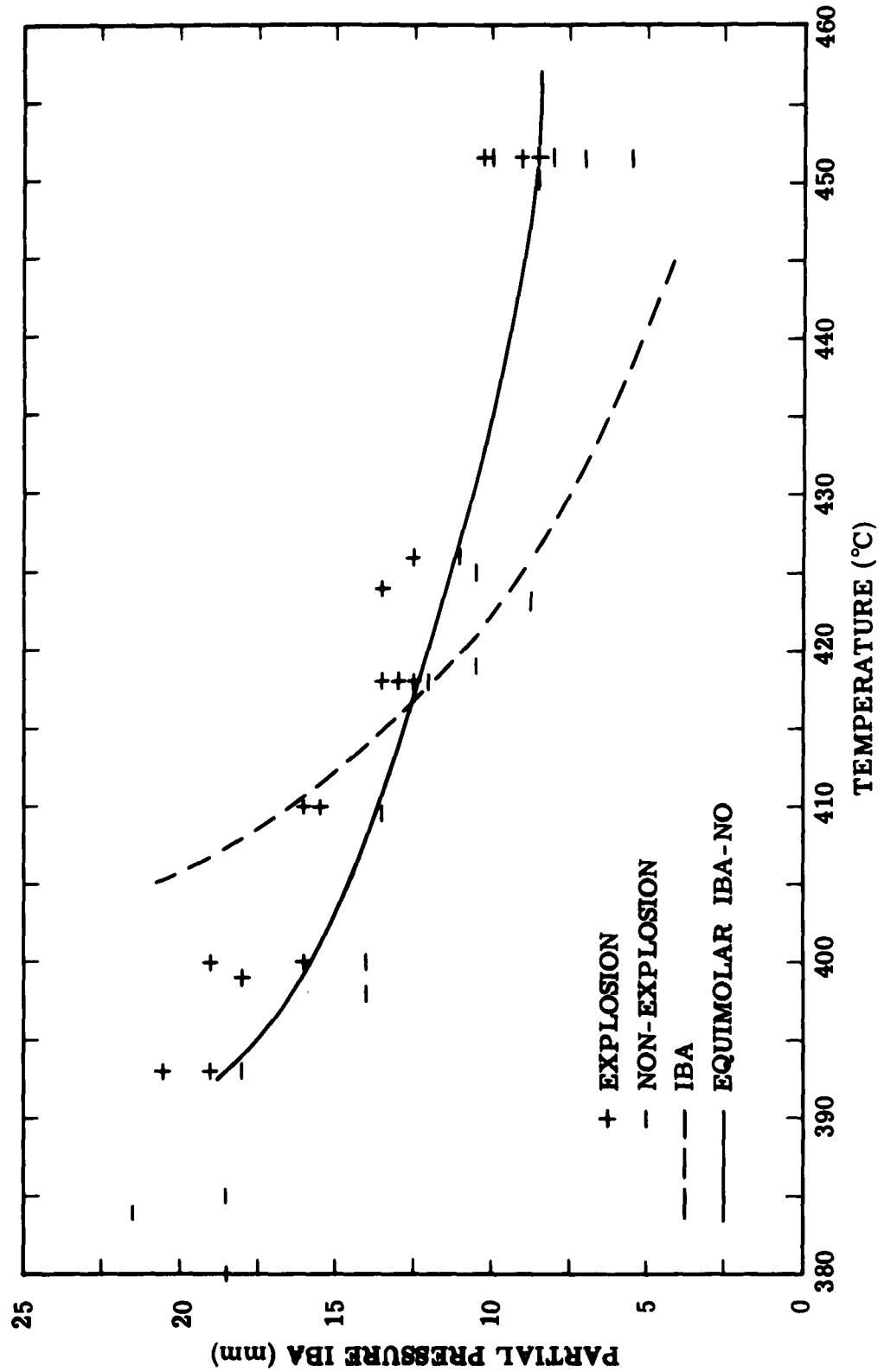


Figure 1. Explosion Limit Curve for IBA and Equimolar IBA-NO in 1090 ml Pyrex Bulbs.

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An apparatus was fabricated consisting of a 600 cc Pyrex bulb connected to a manifold so that samples could be withdrawn by expansion into a small-volume (8.7 cc) Pyrex infrared cell. Rock salt windows were used. After examination of the infrared spectra of 2,2-DP and its decomposition products, the absorption band at  $980\text{ cm}^{-1}$  was selected for use. A major difficulty associated with the use of this band is the strong adjacent silicon tetrafluoride absorption at  $1020\text{ cm}^{-1}$ . It was found that silicon tetrafluoride could be quantitatively removed by passage of the vapors through a tube containing sodium fluoride pellets. Experiments with pure 2,2-DP showed that it was not absorbed.

A series of measurements of optical density at  $980\text{ cm}^{-1}$  vs pressure indicated that Beer's law was not obeyed in the 0-30 mm range and it was necessary to construct a calibration curve. By means of the curve it was possible to convert optical density readings into concentration units.

A few experiments have been performed in the 240-260°C temperature range where the reaction proceeded at a rate convenient for this method of determining the kinetics. Our initial conclusions, which are tentative because comparatively few runs have been made, are that the reaction has an activation energy in the vicinity of 45 kcal/mole and that it is first order. An activation energy of this magnitude is strong evidence for a homogeneous reaction.

We hope during the next period to get precise kinetic data on the thermal decomposition of this compound.

#### V. PLANS FOR THE FUTURE

Our belief that the thermal decomposition of 2,2-DP is a homogeneous first-order reaction in the temperature range studied is reinforced by the results obtained in this period. We intend to perform further experiments to test this.

The product analyses experiments have yielded consistent results. We will perform such experiments on the products for 2,2-DP. We hope to be able to determine silicon tetrafluoride as well.

Explosion limit experiments with 1,1-DP and 1,3-DP will be performed.

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