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Quarterly Technical Summary Report No. 1
April 1, 1963 to June 30, 1963

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

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

to

Advanced Research Projects Agency
Washington 25, D.C.

and

Air Force Office of Scientific Research
Washington 25, D. C.

from

Kinetics and Combustion Division
Atlantic Research Corporation
Alexandria, Virginia

July 30, 1963

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

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

I. ABSTRACT

Apparatus, including an air furnace, has been fabricated for studying the explosion limits of gaseous 1,2-bisdifluoraminopropane. Model experiments have been performed in the apparatus with ethyl nitrate with satisfactory results. Preliminary tests have been performed with a pressure transducer-oscilloscope apparatus with the goal of applying this technique to the detection of explosions.

Pyrolyses of gaseous 1,2-bisdifluoraminopropane at 250-270°C have been performed. The data appear to obey half-order kinetics and to be surface-dependent.

II. INTRODUCTION

This is the first quarterly progress report on this research program. In this program we plan to investigate the explosion limits of certain gaseous difluoramino compounds. Those selected for initial study are: 1,2-bisdifluoraminopropane, 2,2-bisdifluoraminopropane and 1,2-bisdifluoramino-2-methylpropane. We will establish whether the explosions are of the thermal or branched-chain type and will determine the effect on the explosion limits of various chemical species, chosen for their expected inhibiting effect on the reactions involved.

III. PROGRESS DURING PRESENT PERIOD

We have devoted our efforts to date to: (a) fabricating apparatus for the studies; (b) performing model experiments with ethyl nitrate; (c) performing preliminary kinetic experiments on the pyrolysis of 1,2-bisdifluoraminopropane; and (d) testing pressure-sensing equipment for use as a means of demonstrating whether explosion or rapid decomposition occurs.

A. Apparatus

We plan to study explosion limits by the rapid admission of the vapors of the compound to a vessel maintained at some fixed elevated temperature, and to determine under what conditions of temperature and pressure explosion occurs. The first compound we will study is 1,2-bisdifluoraminopropane.

Experiments of this sort are conveniently performed using a glass vacuum line, and we have constructed a conventional type of vacuum system equipped with manometer, a McLeod gage, reservoir bulbs, and various stopcock inlets.

The feature of our equipment that required special design was the furnace to be used. From the fact that thermal decomposition of the species we will be working with occurs at a measurable rate at around 200°C (1) we feel that explosion can probably be observed below 400°C. Although liquid media have been used for constant temperature baths at 400°C and higher, we felt that an air furnace would be much more convenient, and have constructed one. It consists of a cubical aluminum box, twelve inches on a side, which is supported at the center of a cubical transite box, two feet on a side. The interannular space is packed

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with expanded magnesia block insulation. The interior of the aluminum box can be observed visually through glass windows, which are located at the center of two opposite faces of the transite box. Sealed evacuated photometric cells form the paths from the transite walls to the aluminum walls.

The aluminum box is heated by insulated nichrome wire which is cemented in spirals of equal length on each face of the box. We have found that the box can be maintained at $300 \pm 1^\circ\text{C}$ easily by manual adjustment and could undoubtedly be heated several hundred degrees higher without trouble. The box can easily accommodate a two-liter flask and we do not anticipate the necessity of a larger flask at the present time.

The furnace is equipped with a removable section in the top which has vertical ports so that the reaction flask can be conveniently placed in the furnace and its neck can communicate with the vacuum line through one of the ports. Thermocouples can be introduced through the other ports.

B. Experiments with Ethyl Nitrate

We were interested in performing experiments with a model compound whose explosive behavior was well known. Ethyl nitrate seemed to be a very convenient candidate, and one which has been well-investigated (2,3).

In a number of experiments over the temperature range of $250\text{-}300^\circ\text{C}$, we were able to observe explosion in our apparatus as indicated by light evolution. The reaction vessel used here was a 300 cc spherical bulb. The light evolution evidenced itself as a flash of varying intensity. Many investigators have relied on visual observation as evidence of explosion and the ethyl nitrate experiments reinforced our belief that light evolution is a satisfactory indicator for explosions. However it would certainly be desirable to have an objective, somewhat more precise, method for determining whether explosion had occurred and, in the absence of explosion, for giving some indication as to the course of events. Kaufman's (3) elegant experimental technique represents the type of approach that yields a large return of information per experiment. We do not feel that our goals justify the expenditure of time and funds required for instrumentation as elaborate as Kaufman's, but we have devoted some effort to the examination of a fast-response quartz pressure transducer to see if it would be useful for detecting explosions. These experiments have just recently been started and are described in Section C below.

C. Experiments with the Pressure-Sensing Apparatus

The apparatus available to us for measurement of pressure transients consists of a Tektronix 535A Oscilloscope and an SIM Pressure Indicator (Kistler Instrument Company). The pick-up element is a quartz crystal transducer, the signal from which is displayed on the oscilloscope. We have performed a few experiments wherein air was admitted to an evacuated 300 cc spherical bulb to which the gage was connected, and the oscilloscope trace photographed by means of a Polaroid camera. We have observed rise times of about 30 milliseconds when the stopcock has been opened as rapidly as possible. At the highest sensitivity setting available, the oscilloscope deflection has been about 1 mm per mm Hg. This should be adequate for our explosion limit experiments.

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D. Preliminary Experiments with 1,2-bisdifluoramino propane

We have received (4) 100 g of 1,2-bisdifluoramino propane dispersed as a 20% solution in methylene chloride from the du Pont Eastern Laboratories. We have removed the methylene chloride by distillation at atmospheric pressure, using samples containing no more than 3 g of the difluoramino compound. We have tested the purity of the difluoramino compound by passing it through a gas chromatograph containing a column packed with dinonyl phthalate on fire brick. The results indicate that the compound is at least 99% pure. A sample of this compound has been furnished to Dr. Jerome K. Rosen of the U. S. Naval Ordnance Laboratory for testing in his sensitivity program.

We have performed a number of experiments to determine approximate rates of pyrolysis of the vapor of this material. The experiments have been performed by admitting the vapor to a 300 cc spherical glass bulb located in the furnace and equipped with a mercury manometer external to the furnace. The mercury has been protected by a layer of Kel-F oil. We have performed five experiments in which we have measured the rate of the reaction manometrically. We have found that good straight lines have been obtained when the reaction is plotted as if the kinetics were half order in difluoramino compound. It is premature to draw any mechanistic conclusions from this kinetic behavior, but since the curves are linear, they offer a convenient method for comparing different experiments. In the table below, as a measure of the rate we have listed the rate constants obtained from the half-order plots and the half-lives. We have listed the latter in order to compare the results with those reported by Mill (1). Since in all these experiments the initial reactant concentrations were approximately the same ($P \approx 30$ mm, or $n/v \approx 1.7 \times 10^{-3}$ moles l^{-1}), the half lives and the rate constants are approximately proportional.

TABLE I

No.	Temp. ($^{\circ}$ C)	P_f/P_i	k (mm Hg $^{1/2}$ min $^{-1}$)	$t^{1/2}$ (min)
1	270	2.2	0.53	3.5
2	270	2.0	0.49	3.7
3	260	2.3	0.25	7.0
4	250	1.7	0.017	58
5	262	2.0	0.052	28

The experiments are listed in Table I in chronological order. This is done because the first three experiments gave rates much higher than the last two. We do not know why this is so. We have observed a tarry black coating on the flask surface after experiment 1; we left it on to see if it would affect the kinetics, and after experiment 2 it was gone. The flask was not examined again until after experiment 5 when the coating was again present (cf. ref. 1).

When activation energies are calculated from experiments 1-3 and 4 and 5, the resultant values are 43 kcal/mole and 47 kcal/mole; in other words, the temperature dependences of the two sets of data are quite close to each other.

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Extrapolation of the data of entries 1-3 to 187°C leads to a half-life of about 2500 hours while extrapolation of the data of entries 4 and 5 leads to a half-life of about 10,000 hours. Mill (1) observed half-lives of about 55 hours in glass vessels at 187°C. The present results lead to the conclusion that at 187°C the reaction is, as suggested in ref. (1), probably heterogeneous. However the present results also suggest that the reaction is probably at least partly homogeneous in the 270°C temperature region and they support our original belief that temperatures of about 400°C would be sufficient for explosion at moderate pressures.

Table I shows that the pressure increases observed corresponded to the formation of approximately two moles of products per mole of reactant. We have so far done little to establish the nature of the reaction products. Preliminary examination of these products suggests the presence of nitrous oxide and silicon tetrafluoride indicating reaction with the glass walls.

IV. PLANS FOR THE FUTURE

In the next quarter we plan to: (a) perform further experiments with the pressure gage with air, with ethyl nitrate, and, if these are satisfactory, with 1,2-bisdifluoramino propane; (b) investigate further the pyrolysis of 1,2-bisdifluoramino propane at around 300°C; (c) investigate the nature of the products of pyrolysis and of explosion.

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2. P. Gray and A. D. Yoffe, Proc. Roy. Soc., A 200, 114 (1949).
3. F. Kaufman and N. J. Gerri, Eighth Symposium (International) on Combustion, Williams and Wilkins, Baltimore, Md. (1962, p. 619.
4. We are grateful to Marion E. Hill of the Stanford Research Institute for kindly making an arrangement with the du Pont Company to divert a part of his order of this material to us.

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