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COMPATIBILITY TESTING OF HEXANITROETHANE WITH BORON

Anthony E. Finnerty
Michael E. Witt

November 1978

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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The boron-hexanitroethane (HNE) system was examined at room temperature to determine what outgassing problems might exist with this new explosive mixture. It was found that there is a small oxygen consumption with a subsequent evolution of nitrogen. The nitrogen is formed on a one to one basis with the oxygen consumed. Reaction ceases long before the reactants are consumed. There is no evidence of an autocatalytic reaction at room temperature between boron and hexanitroethane, even in the presence of oxygen.

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I. INTRODUCTION

The search for more powerful chemical explosives[1] has recently focused attention on the boron-hexanitroethane (HNE) system as potentially a very high energy explosive. Since a separate fuel and oxidizer are utilized, it is easy to formulate a stoichiometric mixture to maximize energy output. The following equation holds for a mixture of 5.2 grams HNE and 1 gram boron.

$$16\ B + 3\ (NO_2)_3\ C - C\ (NO_2)_3 \rightarrow 8\ B_2O_3 + 6\ CO_2 + 9\ N_2 \qquad (1)$$

It is assumed that B_2O_3 will be a gas since its normal boiling point is 1860°C.

The heat liberated by this stoichiometric mixture is 2.86 kcalories per gram of reactant mixture. The gas evolution is 0.0214 moles of gas per gram reactant. For a standard explosive such as trinitrotoluene (TNT), the heat liberated is 1.41 kcalories per gram with a gas evolution of 0.022 moles gas per gram reactant. Thus, it is seen that the boron-HNE system has the potential of delivering double the energy release of TNT with no penalty in gas production.

However, there is a major problem in the use of two distinct components in an explosive system. Reaction can occur only along the boundary where the two phases are in contact. It is difficult to achieve complete reaction in the short time span of an explosion. One approach being used to overcome this problem is the use of submicron particle size boron which can be intimately mixed with the waxy HNE.

The large surface area associated with submicron size particles magnifies any potential problems of slow reaction at room temperature between components of the explosive. This investigation is designed to determine what, if any, outgassing problems might exist in the submicron boron-HNE system.

II. EXPERIMENTAL

A. Materials

The boron used in these experiments was purchased from Callery Chemical Co., Callery, Pa. It was identified as High Purity, 99 + %, produced by decomposition of boron hydride. It is of sub-micron particle size with an average particle diameter of 433 Å.

The HNE was synthesized under contract by Fluorochem Inc., Azusa, CA. NMR was used to check for the presence of impurities. None were found.

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[1] *J. T. Dehn, BRL IMR No. 425, August 1975.*

The argon used was Matheson High Purity grade.

The oxygen was Matheson Extra Dry grade.

Nitrogen dioxide was also obtained from Matheson, with a purity of 99.5%, liquid phase.

Baker "Analyzed" Reagent grade lead nitrate was used to produce nitrogen dioxide.

The reaction between copper and Fisher Scientific Co. Reagent grade nitric acid also served as a source of nitrogen dioxide.

B. Apparatus

A suitable reaction vessel for use in this study was made from a one quart Mason Jar with a specially modified lid. Four holes were drilled through this lid. Two glass tubes with stopcocks, one iron-constantan thermocouple, and one pressure transducer were sealed into place. A schematic of the lid is given in Figure 1.

The glass tubes extended almost to the bottom of the vessel so that gases could be flushed through the jar and samples taken close to the bottom. The thermocouple was long enough so that its junction could actually be covered by any powders put into the vessel. The thermocouple output was read continuously on an Omega Digital Temperature Indicator. A Tyco Corporation semiconductor strain gauge was used to measure pressure. The output from the Tyco gauge was printed by a Digitec Datalogger to provide pressure readings in digital form. The maximum rate was one pressure reading per second.

For analysis of the gases evolved from the boron-HNE mixture, one of the glass tubes was attached to the inlet system of a Bendix Model MA2 Time-of-Flight Mass Spectrometer. A schematic of the inlet is given in Figure 2. The output of the mass spectrometer was recorded simultaneously in analog form on a Gould Brush 280 recorder and in digital form on an Infotronics CRS-160 Digital Readout System. This gave peak height in digital form and the mass number (actually mass to charge ratio) of each peak in the spectrum to the nearest one-tenth mass number.

C. Procedure

The solid material to be tested was placed in the reaction vessel and the lid sealed in place. Care was taken with each run so that the thermocouple tip was well into the powder. Tests were made on boron, on HNE, and on freshly prepared boron-HNE mixtures.

At the start of each run, the system was purged with an argon-oxygen mixture for five minutes. Then both inlet and outlet valves were closed. The outlet tube from the vessel was attached to the sample entrance port

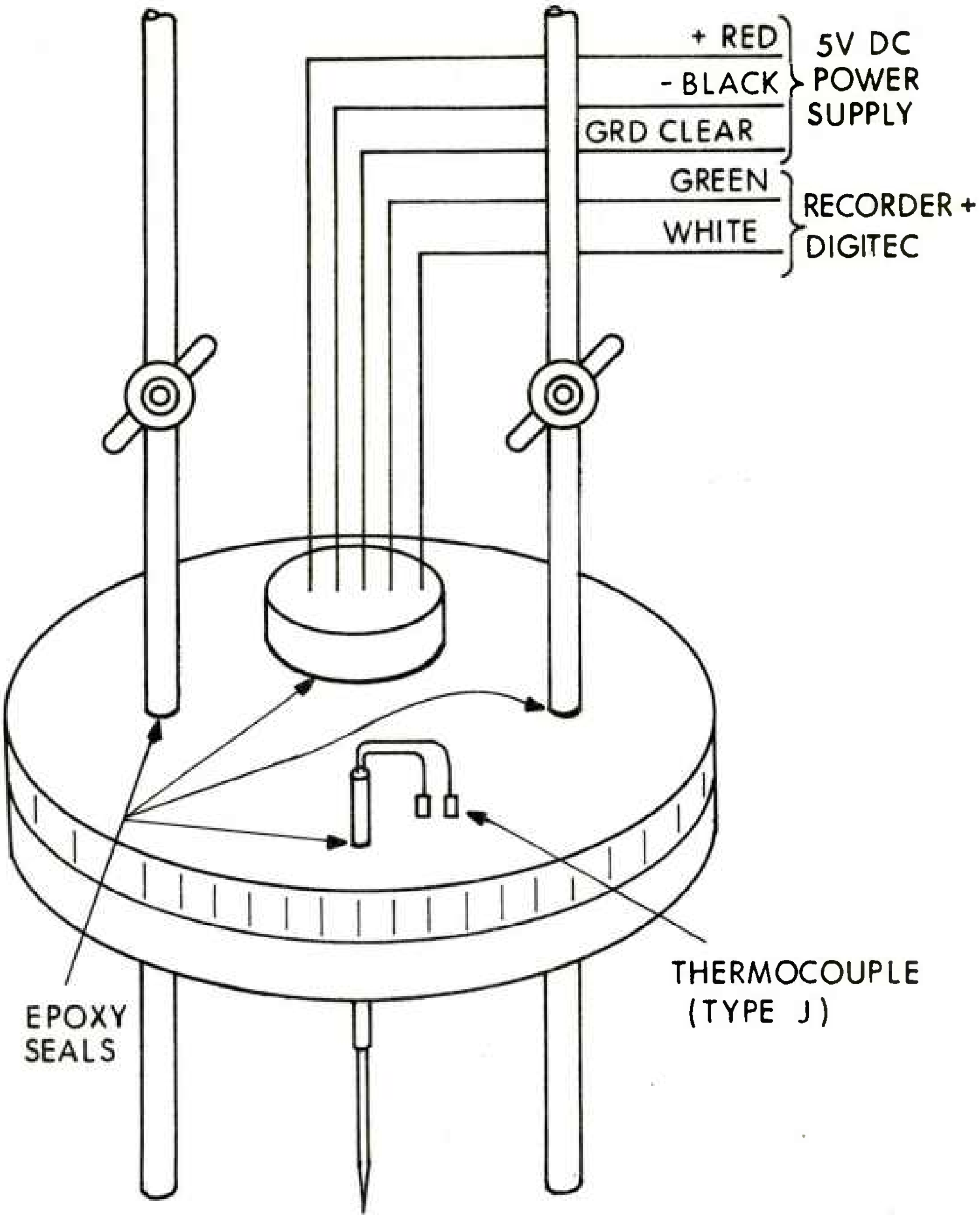

Figure 1. HNE-Boron Sampling System

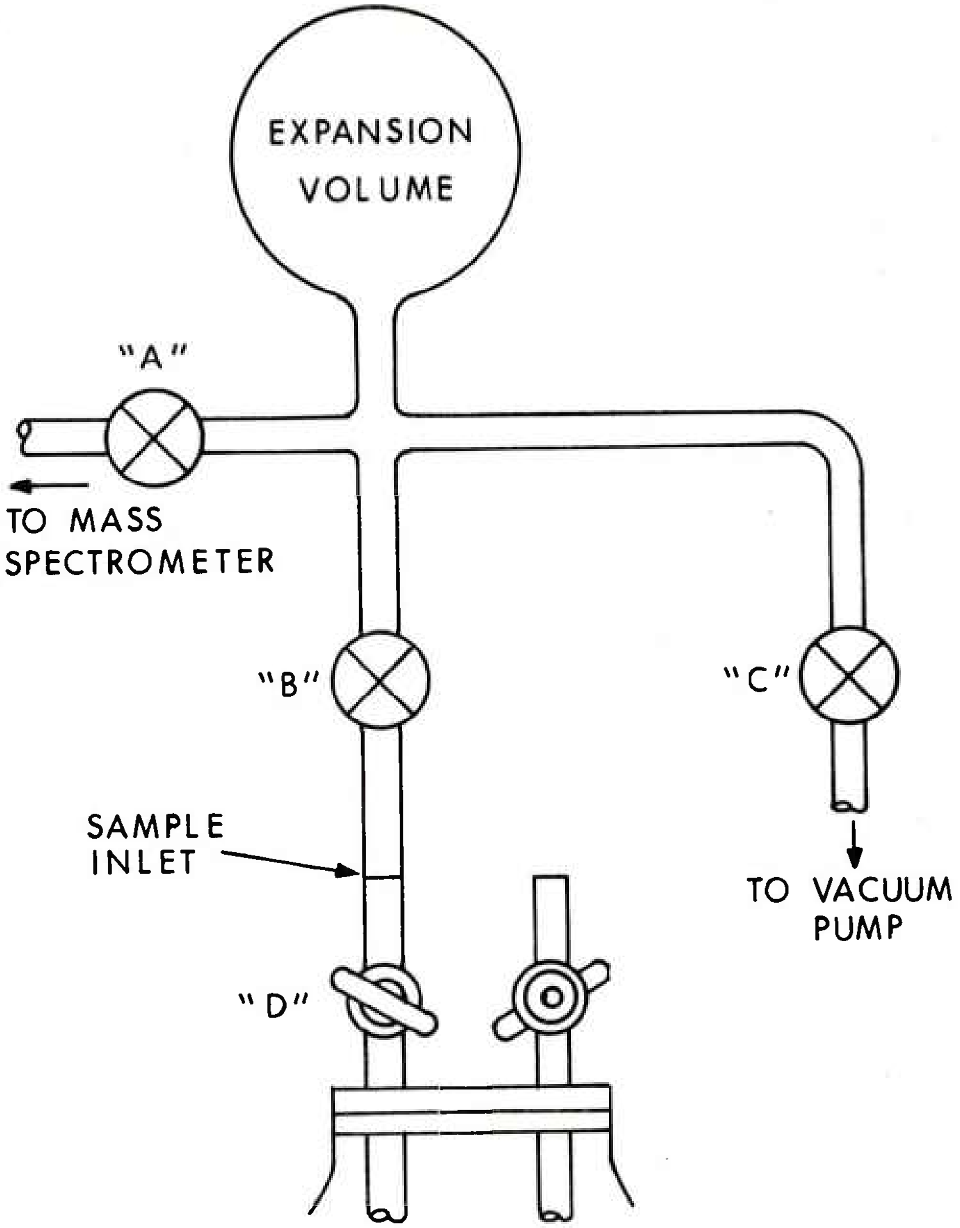

Figure 2. Sample Inlet System

of the mass spectrometer. Small gas samples were periodically put into the spectrometer's sampling system for analysis. The argon was used as an internal standard to quantify results. Two successive scans were made and then averaged for each analysis.

Pressure values were printed once each minute. This was sufficient since the reaction rate was slow, taking several hours for completion.

Temperature readings were recorded from the Temperature Indicator as needed.

In some experiments nitrogen dioxide was added to boron powder in the reaction vessel to determine the extent of direct reaction between the two materials. In these cases, the nitrogen dioxide was added to the argon-oxygen stream.

III. RESULTS

In order to determine the precision of the mass spectrometer analysis and thus its suitibility for use in this study of gas evolution, a blank run was made using an argon-oxygen mixture sealed in the reaction vessel. Two spectrometer scans were made forty minutes apart. The data are given in Table I. The argon to oxygen ratios are quite close, within 1.5 percent on the duplicate analyses. The error in the analysis of residual nitrogen is about 14 percent. This large error is due to the small amount of nitrogen in the samples.

Table I also presents data on argon-oxygen mixtures exposed to boron and to HNE. The results show that there was no consumption of oxygen in these two experiments.

Experiments were then conducted to determine oxygen consumption by mixtures of boron with HNE. A sample of 5.2 grams HNE with 1 gram boron was prepared by grinding the components together using a porcelain mortar and pestle until the boron particles were completely embedded in the waxy HNE. The grinding operation took about four minutes. The resulting black powder appeared to be of uniform consistency. The mixture corresponded to the stoichiometry of Equation 1, which is the reaction mixture of interest. The data of pressure and temperature at different times and analysis of the gases in the sealed reaction vessel are presented in Table II. Similar information, obtained on a 31.6 gram sample of the same HNE to boron ratio, is given in Table III. The pressure data are displayed graphically in Figures 3 and 4. It is readily apparent that oxygen is consumed and nitrogen produced. Over the entire course of reaction, nitrogen production approximately equals oxygen consumption. At the beginning of the reaction, there is an overall drop in pressure while the temperature of the solid is constant or even increasing. As the reaction progresses, the overall pressure increases, while the temperature of the powder falls back to room temperature. The error limits on oxygen consumption and nitrogen production were determined using data from Table I.

Table I. Analyses of Argon-Oxygen Mixtures in Sealed Vessel by Bendix Time-of-Flight Mass Spectrometer

Time	Argon (torr)	Oxygen (torr)	Nitrogen(torr)	$\frac{\text{Argon}}{\text{Oxygen}}$ Ratio
A. Argon plus Oxygen Mixture				
0 minutes	310.6	431	18.1	0.72
40 minutes	314	430	16.4	0.73
B. Argon plus Oxygen Mixture with 0.32 gram Boron				
0 minutes	249	491	19.5	0.51
60 minutes	253	487	19.5	0.52
120 minutes	255	484	20.4	0.53
C. Argon plus Oxygen Mixture with 0.50 gram Hexanitroethane				
0 minutes	254	489	17.4	0.52
60 minutes	258	484	18.5	0.53
120 minutes	250	492	17.0	0.51
1260 minutes	259	483	21	0.54

Table II. Oxygen Consumption by a 6.2 Gram Stoichiometric Mixture of Boron with Hexanitroethane Sealed in an Argon-Oxygen Atmosphere

Time (minutes)	Temp °C	Pressure (torr)	Pressure Argon (torr)	Pressure Oxygen (torr)	Pressure Nitrogen (torr)	$\frac{\text{Oxygen}}{\text{Argon}}$ Ratio	$\frac{\text{Nitrogen}}{\text{Argon}}$ Ratio
0	22	760	323	409	28	1.27	.0867
18	22	756	325	412	19	1.27	.0585
46	22	751	322	410	18	1.27	.0559
100	21	751	322	407	22	1.26	.0683
1064	21	764	320	388	56	1.21	.1750

Total Oxygen Consumption = 21 torr ± 5

Total Nitrogen Production = 28 torr ± 2

Table III. Oxygen Consumption by a 31.6 Gram Stoichiometric Mixture of Boron with Hexanitroethane Sealed in an Argon-Oxygen Atmosphere

Time (minutes)	Temp °C	Pressure (torr)	Pressure Argon (torr)	Pressure Oxygen (torr)	Pressure Nitrogen (torr)	$\frac{\text{Oxygen}}{\text{Argon}}$ Ratio	$\frac{\text{Nitrogen}}{\text{Argon}}$ Ratio
0	22	762	102	630	30	6.18	0.29
37	27	740	102	618	20	6.06	0.20
87	27	723	106	588	28	5.55	0.26
193	25	723	102	566	55	5.55	0.54
229	24	723	103	559	60	5.43	0.58
279	24	727	103	553	71	5.37	0.69
1332	22	758	104	533	121	5.13	1.16
1402	-	757	-	-	-	-	-

Total oxygen consumption = 97 torr ± 5

Total nitrogen production = 91 torr ± 4

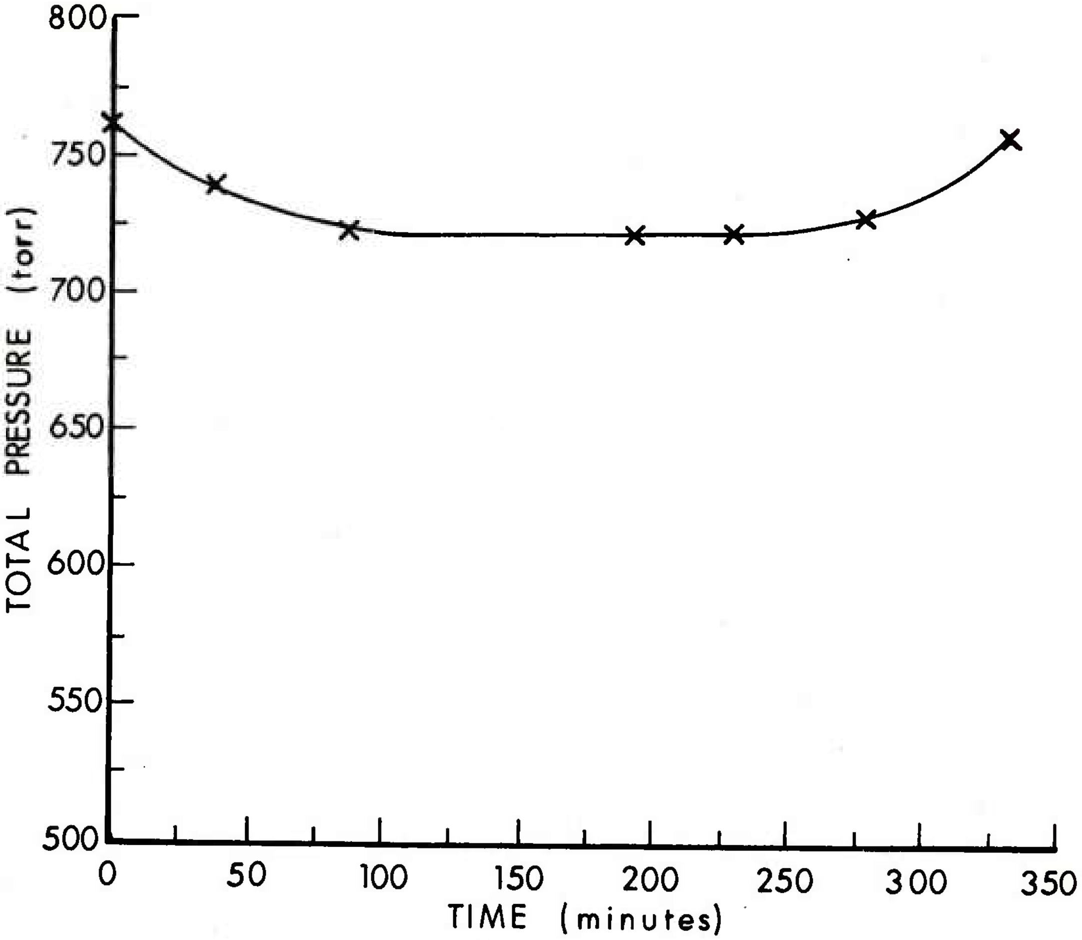

Figure 3. Pressure vs. Time of HNE-Boron (31.6g) Stoichiometric Mixture in Argon-Oxygen Atmosphere

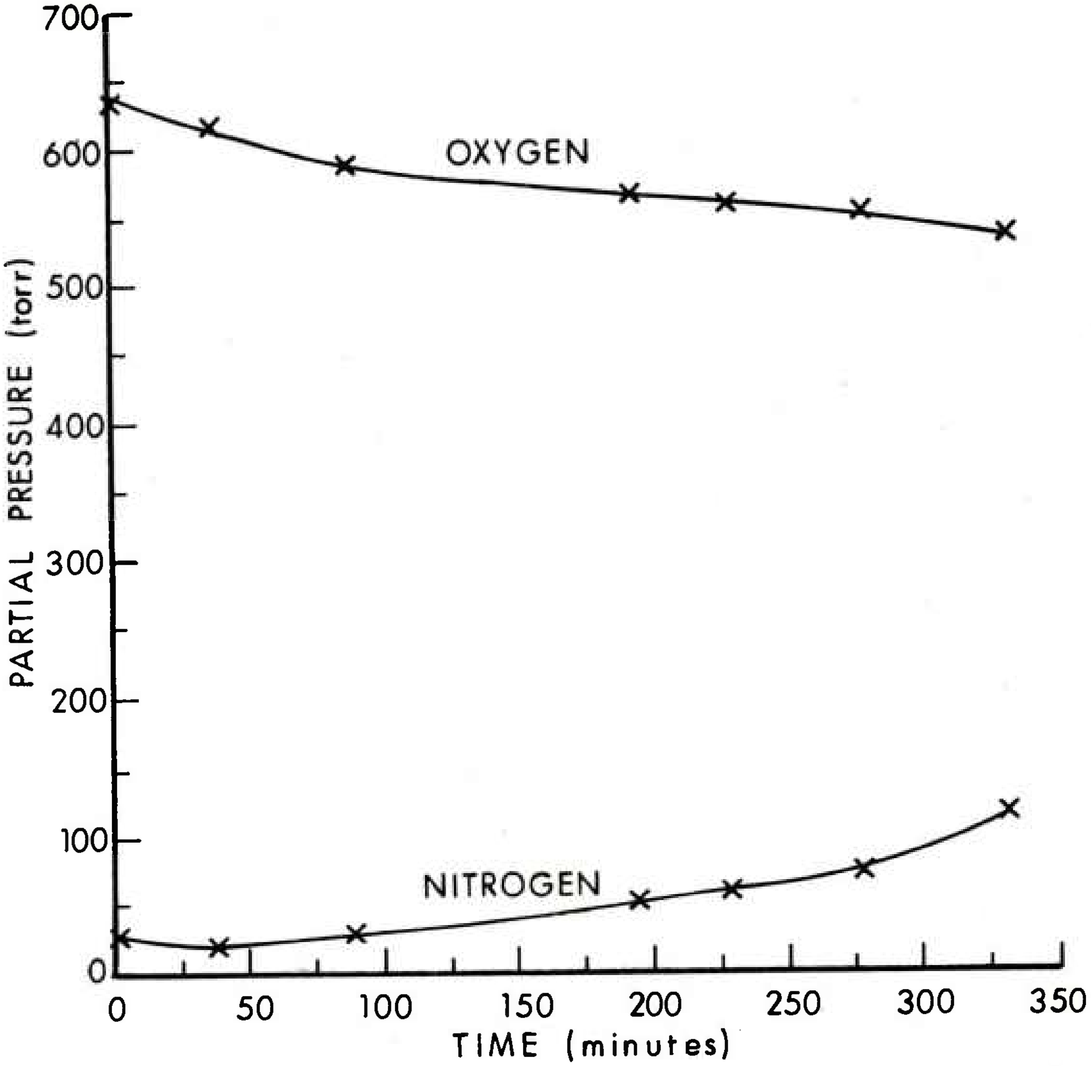

Figure 4. Nitrogen and Oxygen Partial Pressures vs. Time for HNE-Boron (31.6g) Stoichiometric Mixture

It is seen from a comparison of initial oxygen pressure and total oxygen consumption for the two samples that oxygen consumption is dependent more on sample size than oxygen pressure. Reaction ceases when surface sites for reaction are no longer available rather than when the oxygen is consumed.

No mass spectrometer peaks were found at m/e = 46 or 44. Therefore, neither NO_2 nor CO_2 was detected. However, in the case of the larger sample, a red-brown gas was seen at the very bottom of the reactor. It was thought that this was an indicator of nitrogen dioxide production. If the NO_2 was consumed by boron, the NO_2 might not be detected by the mass spectrometer, which can detect only fairly large quantities of nitrogen dioxide since this gas is readily adsorbed by the metal walls of the spectrometer.

Experiments were conducted to determine if nitrogen dioxide would react readily with submicron boron. Nitrogen dioxide was produced by reaction of copper with concentrated nitric acid. The NO_2 was mixed with argon and oxygen and allowed to fill the reaction vessel containing 0.5 gram submicron boron powder. The temperature of the boron quickly rose from 22° to 60°C. The mass spectrometer analyses of the main gases are given in Table IV. It can be seen that no oxygen consumption occurs. Much more nitrogen is evolved than would be expected from the consumption of nitrogen dioxide. This together with the rapid temperature increase of the boron powder, leads to the conclusion that nitrogen dioxide is quickly adsorbed onto the boron surface and nitrogen only slowly liberated. The NO_2 was adsorbed before the initial analysis was made. However, the presence of nitrous oxide [N_2O] as a byproduct of the reaction producing NO_2 caused an ambiguity in the interpretation of the reaction between boron and NO_2. Therefore, another experiment was carried out. Nitrogen dioxide was produced by the reaction

$$2\ Pb(NO_3)_2 \xrightarrow{500^\circ C} 2\ PbO + 4NO_2 + O_2\ . \qquad (2)$$

There is no nitrous oxide byproduct in this reaction[2]. The evolved gas was mixed with argon and allowed to flow continuously over 5 grams of submicron boron. The temperature of the boron rose from 22°C to 139°C in 31 minutes. A cautious touch showed that the bottom of the vessel was indeed extremely hot, showing that NO_2 had reacted with the boron powder, not just with the thermocouple metal. It is interesting to note that after peaking at 139°C, the temperature steadily fell, although fresh NO_2 was available and over four grams of the boron were still unreacted. Therefore, it is probable that only the boron on the surface of the particles is available for reaction with NO_2. A layer of boron

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[2] *T. L. Brown, General Chemistry, Second Edition, Charles E. Merrill Publishing Company, 1968.*

Table IV. Mass Spectrometer Analyses of Argon-Oxygen-Nitrogen Dioxide Mixture in Contact with 0.5 Gram Submicron Boron

Time (minutes)	Temp °C	Pressure (torr)	Pressure Argon (torr)	Pressure Oxygen (torr)	Pressure NO_2 (torr)	Pressure Nitrogen (torr)
0	22	735	613	65	32	25
2	46	-	-	-	-	-
81	60	735	608	68	28	29
141	-	741	612	69	28	31
261	-	745	609	69	27	39

oxide is formed, preventing further reaction between the remaining boron and NO_2.

$$8B + 6\ NO_2 \rightarrow 4\ B_2O_3 + 3\ N_2 \qquad (3)$$

When a cylinder of nitrogen dioxide became available, an attempt was made to react NO_2 with boron with no other chemical components present. However, the initial surge of gas from the cylinder into the reactor quickly decreased to no flow. Since the normal boiling point of nitrogen dioxide is 21°C, there was not enough vapor pressure to overcome the atmospheric pressure in the reactor. The powder temperature rose to only 49°C then fell. The cylinder was not able to supply enough fresh NO_2 to the boron powder to sustain reaction. Liquid N_2O_4 was seen to condense in the transparent transfer line.

IV. DISCUSSIONS

The explosive system of HNE and boron will, when detonated, react according to the following equation[1]

$$3\ \mathrm{HNE} + 16\ B \rightarrow 8\ B_2O_3 + 6\ CO_2 + 9\ N_2. \qquad (1)$$

However, the low temperature outgassing reaction which we investigated follows a different chemistry. This is shown by failure to detect carbon dioxide in the gas above the HNE-boron powder even though nitrogen gas is a product of our outgassing system. The consumption of oxygen by the stoichiometric mixture, which we found, would not be predicted on the basis of Equation 1. It has been proposed that HNE decomposes thermally by the loss of NO_2 to form a subsequent free radical[3,4]. The following equations represent possible initial steps in the decomposition of HNE in both the solid phase and inert solvents, forming nitrogen dioxide[4]. Since NO_2 is an odd electron molecule, its loss by HNE produces a free radical. When two molecules of nitrogen dioxide are lost, a diradical or olefinic compound is formed. Free radical species are represented by dots.

$$\mathrm{HNE} \rightarrow (NO_2)_3\ C - \overset{\bullet}{C} - (NO_2)_2 + NO_2 \rightarrow \text{products} \qquad (4)$$

$$\mathrm{HNE} \rightarrow (NO_2)_3 - C - \overset{\bullet}{\underset{\bullet}{C}} - NO_2 + 2\ NO_2 \rightarrow \text{products} \qquad (5)$$

---

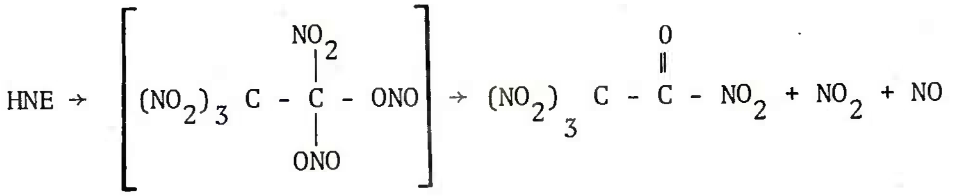
[3] *G. M. Nagin, et. al., Combustion and Flame, 12(1), 102, (1968).*

[4] *H. P. Marshall, et. al., Journal of Physical Chemistry, 69(1), 25, (1965).*

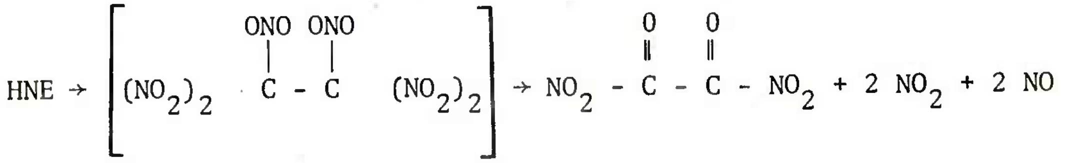
$$\mathrm{HNE} \rightarrow (\mathrm{NO_2})_2 - \dot{\mathrm{C}} - \dot{\mathrm{C}} - (\mathrm{NO_2})_2 + 2\ \mathrm{NO_2} \rightarrow \text{products} \tag{6}$$

HNE does not exhibit loss of NO_2 at room temperature by itself. The submicron boron may act as a catalyst, lowering the activation energy so that reaction can occur at room temperature at the interface between boron and HNE. However, Equations 4, 5, or 6, when coupled to Equation 3, predict reaction of HNE with boron without consumption of oxygen. Yet we have established oxygen uptake by this system. Therefore we must look further for an explanation of the chemistry of this system at room temperature.

We considered the following routes to production of NO_2 by the HNE-boron system at room temperature.



$$\mathrm{HNE} \rightarrow \left[(\mathrm{NO_2})_3\,\mathrm{C} - \overset{\mathrm{NO_2}}{\underset{\mathrm{ONO}}{\mathrm{C}}} - \mathrm{ONO} \right] \rightarrow (\mathrm{NO_2})_3\,\mathrm{C} - \overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}} - \mathrm{NO_2} + \mathrm{NO_2} + \mathrm{NO} \tag{7}$$



$$\mathrm{HNE} \rightarrow \left[(\mathrm{NO_2})_2\ \overset{\mathrm{ONO}}{\mathrm{C}} - \overset{\mathrm{ONO}}{\mathrm{C}}\ (\mathrm{NO_2})_2 \right] \rightarrow \mathrm{NO_2} - \overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}} - \overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}} - \mathrm{NO_2} + 2\ \mathrm{NO_2} + 2\ \mathrm{NO} \tag{8}$$

Equations 7 and 8 would occur at the HNE-boron interface. Both equations show the production of nitric oxide in a one to one ratio with nitrogen dioxide. The nitric oxide would readily react with oxygen to produce additional nitrogen dioxide.

$$2\ \mathrm{NO} + \mathrm{O_2} \rightarrow 2\ \mathrm{NO_2} \tag{9}$$

This scheme predicts a loss of oxygen and an initial decrease in overall pressure if the oxides of nitrogen remained on the boron surface.

The nitrogen dioxide can then react with boron according to

$$8\ \mathrm{B} + 6\ \mathrm{NO_2} \rightarrow 4\ \mathrm{B_2O_3} + 3\ \mathrm{N_2} \tag{3}$$

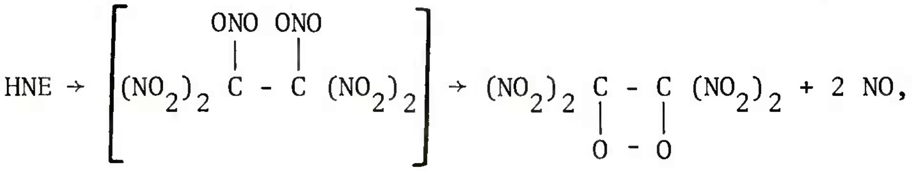
which explains the formation of nitrogen and the heat production in this system.

According to Equation 8, one mole of HNE produces two moles of NO and two moles of NO_2. The two moles of NO will combine with one mole of O_2, by Equation 9, forming a total of four moles of NO_2. Upon reaction with boron, two moles of nitrogen are formed from the four moles of NO_2. Equation 3 may be rewritten as

$$4\ NO_2 + \frac{16}{3}\ B \rightarrow \frac{8}{3}\ B_2O_3 + 2\ N_2\ . \qquad (3a)$$

Therefore, two moles of nitrogen should be formed for each mole of oxygen consumed. Equation 7 predicts the same nitrogen production to oxygen consumption ratio. But, as indicated from Table II and shown more clearly by Table III, oxygen consumption is approximately equal to nitrogen production, rather than being only one-half the nitrogen formed. The return of total pressure to the approximate starting value also indicates equivalence of the moles of gaseous product to moles of gas consumed.

If we consider another possible reaction taking place on the boron surface



$$HNE \rightarrow \left[(NO_2)_2\ \overset{\overset{ONO}{|}}{C} - \overset{\overset{ONO}{|}}{C}\ (NO_2)_2 \right] \rightarrow (NO_2)_2\ \begin{array}{c} C - C \\ |\quad\ | \\ O - O \end{array}\ (NO_2)_2 + 2\ NO, \qquad (10)$$

just nitric oxide and an epoxy compound are the initial products of this reaction. The nitric oxide would react with oxygen

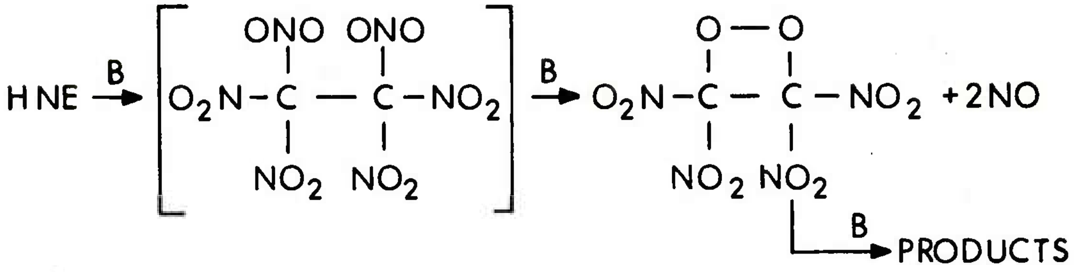
$$2\ NO + O_2 \rightarrow 2\ NO_2 \qquad (9)$$

followed by

$$2\ NO_2 + \frac{8}{3}\ B \rightarrow \frac{4}{3}\ B_2O_3 + N_2 \qquad (3b)$$

which is another form of Equation 3. This reaction sequence predicts one mole of nitrogen will be produced for each mole of oxygen consumed, in agreement with Tables II and III.

The evidence favors reaction 10 being the boron surface catalyzed first step of the room temperature HNE decomposition followed by a rapid reaction of nitric oxide with oxygen. The nitrogen dioxide formed by this reaction then reacts with the boron producing nitrogen gas. This reaction sequence is given in Figure 5. Oxygen consumption starts before nitrogen evolution begins. Therefore the consumption of oxygen is out of phase with nitrogen formation, even though consumption and evolution are on a one to one basis. This accounts for the initial



$$2\,NO + O_2 \longrightarrow 2\,NO_2$$

$$2\,NO_2 + \frac{8}{3}\,B \longrightarrow \frac{4}{3}\,B_2O_3 + N_2$$

ONE MOLECULE OF NITROGEN IS PRODUCED FOR EACH MOLECULE OF OXYGEN CONSUMED.

Figure 5. Proposed Mechanism of Oxygen Consumption and Nitrogen Production

pressure drop followed by eventual return to starting pressure. Reaction will cease when either all available oxygen is consumed or B_2O_3 forms at the HNE-boron interface.

Outgassing is not a severe problem with this system since there is a built-in limitation on the extent of reaction. When the active sites on the boron surface are destroyed by formation of B_2O_3, reaction must cease. Outgassing does not lead to a runaway reaction. Nevertheless, the goal of future research will be the complete elimination of room temperature outgassing from the HNE-boron system. Four approaches will be used.

1. Air will be removed from both HNE and boron before mixing by subjecting them to vacuum conditions. The materials will be stored under argon, an inert atmosphere. The mixture will be compounded and sealed in a gas-tight container under argon. Since no oxygen will be available, there should be no nitrogen production.

2. Attempts will be made to analyze the HNE for impurities. When the impurities have been identified, steps will be taken to eliminate them. High purity HNE may not exhibit nitrogen formation when mixed with submicron boron.

3. Attempts will be made to destroy the catalytic activity of the boron surface. Simple exposure of the submicron boron to nitrogen dioxide or sulfur dioxide may be sufficient to remove the active sites from the boron surface. This would eliminate room temperature reaction with HNE.

4. Tetranitromethane (TNM), a liquid which is chemically similar to HNE, will be used as a supplement to the HNE-boron system. HNE is soluble in TNM. It is expected that excellent physical contact between boron and the oxidizer solution can be achieved since TNM readily wets the submicron boron. Excess TNM will be removed by evaporation. Substitution of some residual TNM for some of the HNE will have virtually no effect on the energetics or the gas production of the system. The outgassing tendency of this modified system may be less than that of HNE-boron since the liquid wets the boron surface, excluding air from the interface.

V. CONCLUSIONS

The following conclusions may be made concerning the hexanitroethane-boron system at room temperature.

1. There is no evidence of an autocatalytic reaction at room temperature between HNE and boron even in the presence of oxygen.

2. The system consumes oxygen from the atmosphere and evolves nitrogen gas on a one mole to one mole basis.

3. Immediately after mixing of components, the main reaction is oxygen consumption. After a period of time, nitrogen liberation is faster than oxygen uptake.

4. If the HNE-boron system is sealed immediately after mixing of components, pressure will drop slightly below atmospheric, only to return to the original starting pressure in time. This would effectively prevent pressure buildup, eliminating any potential outgassing problem.

5. If the HNE-boron system is not sealed immediately after mixing of components, nitrogen production may cause a pressure increase after sealing.

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