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Ignition of Nitrocellulose in Various Atmospheres

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Nitrocellulose (NC) remains an important ingredient in modern gun propellants. For fire safety, the ability to extinguish burning NC, or, at least reduce its rate of combustion, would be highly desirable. Toward this end, a study has been conducted on the effect of various gases and powders on the decomposition and/or burning of NC.

Chemical analyses were performed to determine the nature of the gases emitted by decomposing and burning NC. Nitric oxide (NO) was the principal nitrogen-containing product of NC decomposition in inert atmospheres when NC was heated by a nichrome ribbon. When air was present, some NO_2 was also formed. The amounts of carbon monoxide (CO) and carbon dioxide (CO_2) were also affected by the nature of the gaseous atmosphere.

When certain nonreactive powders were mixed with the NC prior to heating with the nichrome ribbon, the combustion or decomposition of NC was greatly reduced. Maintaining power to the ribbon was necessary for combustion to continue. The formation of NO, NO_2, CO, and CO_2 were all affected by the powders.

When the powders were simply layered on top of NC, and the NC heated, combustion proceeded essentially as if the powders were not present.

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TABLE OF CONTENTS

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LIST OF FIGURES

LIST OF TABLES

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PREFACE

The U.S. Army Ballistic Research Laboratory was deactivated on 30 September 1992 and subsequently became a part of the U.S. Army Research Laboratory (ARL) on 1 October 1992.

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1. INTRODUCTION

Nitrocellulose (NC) is still an important ingredient of modern gun propellants. Unfortunately, like energetic materials in general, NC presents a certain degree of fire danger. The military has experienced all too many circumstances in which propellant fires have caused serious casualties to personnel and loss of equipment. From a safety point of view, the ability to extinguish burning propellants, or, at least reduce the rate of combustion to a low level, would be highly desirable. Unfortunately, the military does not possess the capability of extinguishing propellant fires, except at the ammunition plant level where water deluge systems are available (Loyd 1989).

In an effort to address the problems of hydrocarbon fires, the military sponsored extensive research studying the physical and chemical reactions of various potential fire extinguishing agents (Purdue 1950). This has led to the development of military fire extinguishing systems which control fuel and hydraulic fluid fires. The military now has useful methods of controlling both combat-induced and accidental fires in vehicles (S.A.F.E.).

The present study was conducted to identify materials which have an effect on the combustion of nitrocellulose. Agents which could influence the combustion of a propellant ingredient would be worthy of further study. This could eventually lead to a method of preventing or modifying propellant fires.

NC (properly, but infrequently, called cellulose nitrate) is a polymeric material synthesized by nitration of cellulose, a naturally occurring polymer. If all the hydroxyl groups of the cellulose polymer were nitrated, the nitrogen content of the resulting NC would be 14.11%. In practice, this degree of nitration is never achieved. The NC used in propellants typically has a nitrogen content of 12%–14%. A segment of an NC chain is presented in Figure 1.

2. EXPERIMENTAL

2.1 Materials. This study was carried out using the following materials:

Nitrocellulose (NC) - 13.44% nitrogen content, supplied courtesy of Dr. Robert Fifer, Interior Ballistics Division of the U.S. Army Ballistic Research Laboratory (BRL). This was a fluffy, finely divided material.

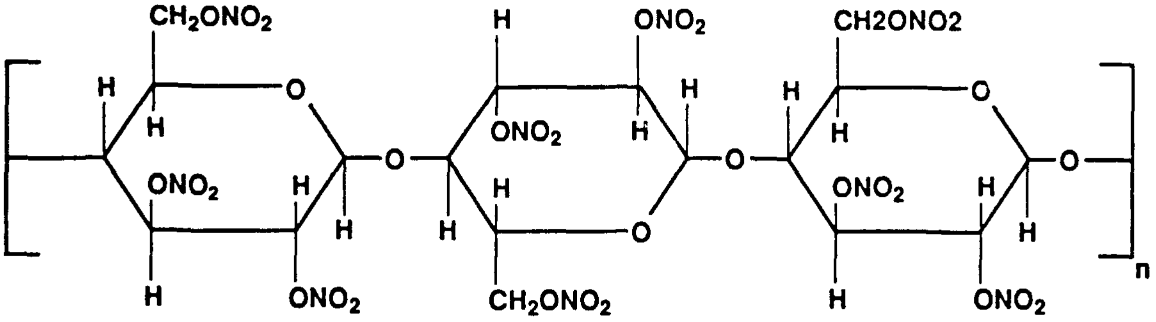

Figure 1. A Segment of a Fully Nitrated Cellulose Chain (Cellulose Nitrate).

Air - breathing quality, Air Products.

Nitrogen (N_2) - Federal Government supply.

Carbon dioxide (CO_2) - Linde Division of Union Carbide.

Carbon monoxide (CO) - Scott Specialty Gases.

Nitric oxide (NO) - in N_2, calibration mixture, Scott Specialty Gases.

Nitrogen dioxide (NO_2) - in air, calibration mixture, Scott Specialty Gases.

CO - in air, calibration mixture, Scott Specialty Gases.

CO_2 - in air, calibration mixture, Scott Specialty Gases.

Argon (Ar) - Airco.

Halon 1301 - Great Lakes Chemical Co.

Sulfur hexafluoride (SF_6) - Scott Specialty Gases.

Molecular Sieve 13X - powder sample, courtesy of Union Carbide.

Monnex - fire extinguishing powder, ICI of America.

Purple K - fire extinguishing powder, Federal Government supply.

2.2 Apparatus. Tests were conducted in a 20-liter stainless steel reaction chamber (Figure 2). It had been purchased on a special order from Fike Metal Products, Division of Fike Co., Blue Springs, MO. The chamber was rated at 1,500 psi (10.2 MPa) and 400° C.

The chamber had a 9-in (23 cm) opening when the lid was locked in the up position. A quartz crucible easily fit inside the chamber. A section of nichrome ribbon was used to heat NC powder in the crucible.

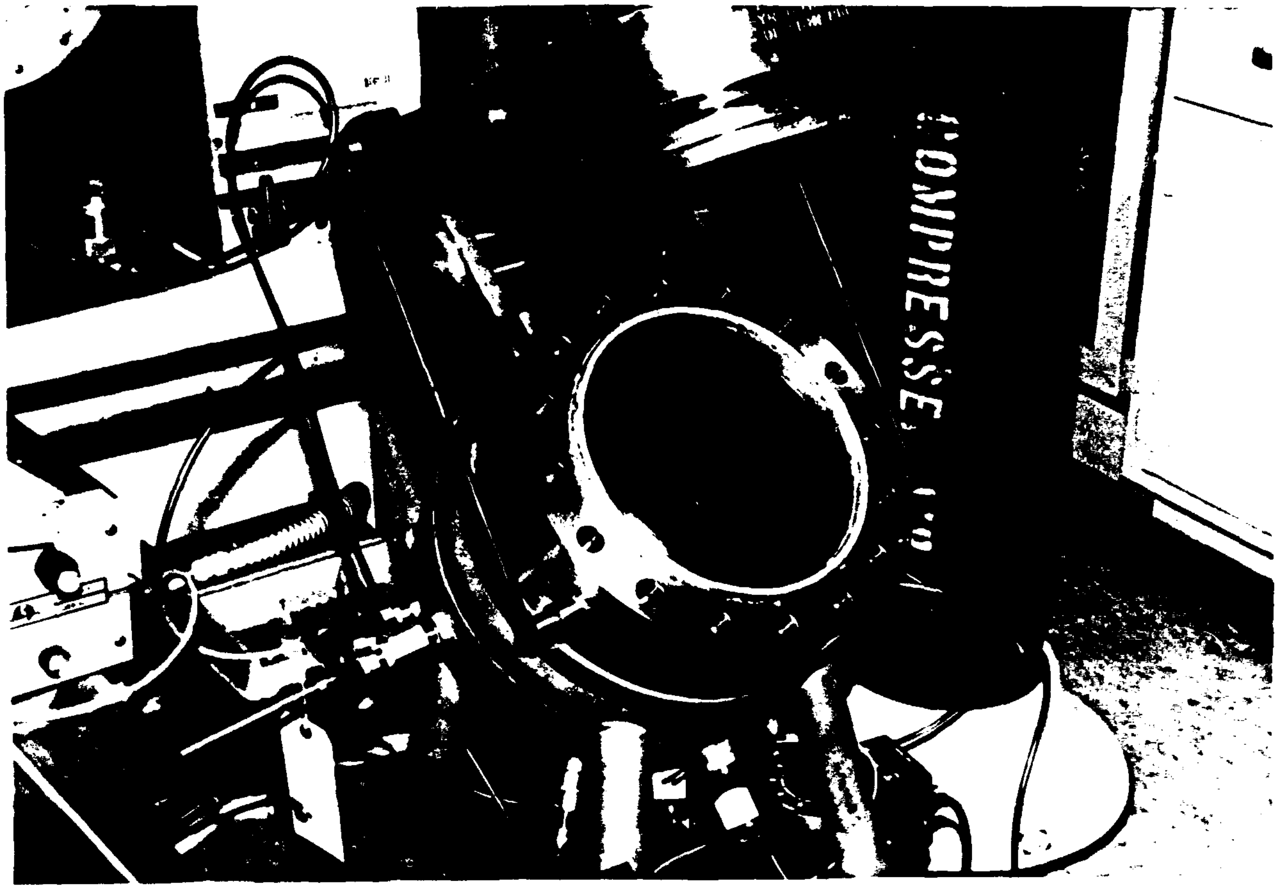

Figure 2. The 20-Liter Chamber.

There were threaded openings in the chamber which allowed sealed electrical leads to pass into the interior to make contact with the nichrome ribbon. A 6.3-V filament transformer, rated at 10 A, was energized by a variable voltage transformer to power the nichrome heater.

Stainless steel vacuum valves were threaded into the chamber using other available openings. Using these valves, gases were added to or removed from the chamber.

A Wallace-Tiernan dial-type pressure gauge, 0- to 800-mm Hg absolute pressure, was used in making up gaseous mixture inside the chamber.

2.3 Analytical Instruments.

2.3.1 BINOSs. BINOS gas analyzers (purchased from Inficon, a Leybold-Heraeus Co. of Germany) were used to analyze combustion gases from NC burned in air or other gaseous atmosphere at 1 atm. The BINOS units used to analyze nitric oxide (NO), carbon monoxide (CO), and carbon dioxide (CO_2) used the principle of nondispersive infrared spectroscopy. The unit used to analyze nitrogen dioxide (NO_2) operated on the principle of nondispersive ultraviolet spectroscopy. All units had both an analog dial output and 0–5 V output terminals for strip chart recording. The analytical ranges of the instruments are given in Table 1.

Table 1. Analytical Ranges of the BINOSs

Gas	Concentration Range (ppm)	Accuracy of Reading (ppm)
NO	0–2,000	±40
NO_2	0–2,000	±40
CO	0–1% [0-10,000]	±200
CO_2	0–6% [0-60,000]	±1,200

The instruments are claimed to be accurate to ±2% of full scale. In practice, using calibration standards, the accuracy appears to be close to ±0.5% of full scale. The BINOS units were checked frequently and calibrated using calibration gas mixtures.

Since NO_2 is a very reactive material and can be absorbed easily on many surfaces, only stainless steel, teflon, and quartz are recommended for contact with NO_2. Therefore, teflon gas lines and stainless steel fittings were used to connect the stainless steel reaction chamber to the BINOS units. The first unit to receive gases from the chamber was the NO_2 unit. All BINOS units had gas flow in series using teflon lines and teflon or stainless steel connectors. Even the gas transfer lines in the BINOS units were teflon. The filters used to protect the BINOS analyzers were made of teflon with teflon membranes. A diaphragm pump was used on the output side of the last BINOS. At this point in the flow, the gases already had been analyzed. The output of the pump was routed to a flowmeter so that flow through the system could be adjusted to the desired value. The BINOS's flowmeter was used to monitor flow of the various gases since the BINOS analyzers are relatively insensitive to variations in flow. The output from the flowmeter went to a chemical fume hood. This ensured that no toxic gases would be present in the laboratory air. Pictures of the BINOSs are presented in Figure 3.

2.3.2 Mettler Differential Scanning Calorimeter. A Mettler TC 10A Process Controller with a DSC 20 cell (Figure 4) was used to obtain data on the heat generated when NC undergoes combustion. Samples of NC in sealed, vented aluminum pans were heated in the DSC in a variety of gases. The temperature at which the maximum heat liberation occurred was determined. The total heat generated by NC in the different gaseous atmospheres was determined. The flow rate of the various gases through the DSC was measured by a bubble flowmeter, since the results would probably be sensitive to the actual flow rate. While the DSC data might be influenced by the different gaseous atmospheres, it was considered unlikely that it would be possible to quench the NC combustion, since the DSC continuously supplies heat to the sample.

2.4 Procedures.

2.4.1 Tests in the 20-Liter Chamber. Typically, the BINOS analyzers were left operational at all times, with no gas flow in the idle mode.

A sample of NC (nominally 100 mg) was weighed into the quartz crucible. The crucible was placed in the bottom of the open reaction chamber (Figure 5). The nichrome heating ribbon was placed in the crucible in contact with the NC. The lid was then bolted on to the chamber. Using the stainless steel valves, the chamber was then flushed with air, nitrogen, argon, carbon monoxide, carbon dioxide, or Halon 1301. When a gas other than air was used, flow continued for approximately 1 hr to give at least four changes of volume. The exit gas line was attached to the BINOS units. When the ignition test was to occur, the gas line into the stainless steel chamber was disconnected from the gas source. The diaphragm pump of the analyzers was started and adjusted to 1.5 liters/minute flow. The valve which had been used to admit gas was left open to the air to ensure no pressure drop in the system. The power to the nichrome heater was activated. Ignition occurred and gases were analyzed by the BINOS units. A small amount of air contaminant was inevitably present.

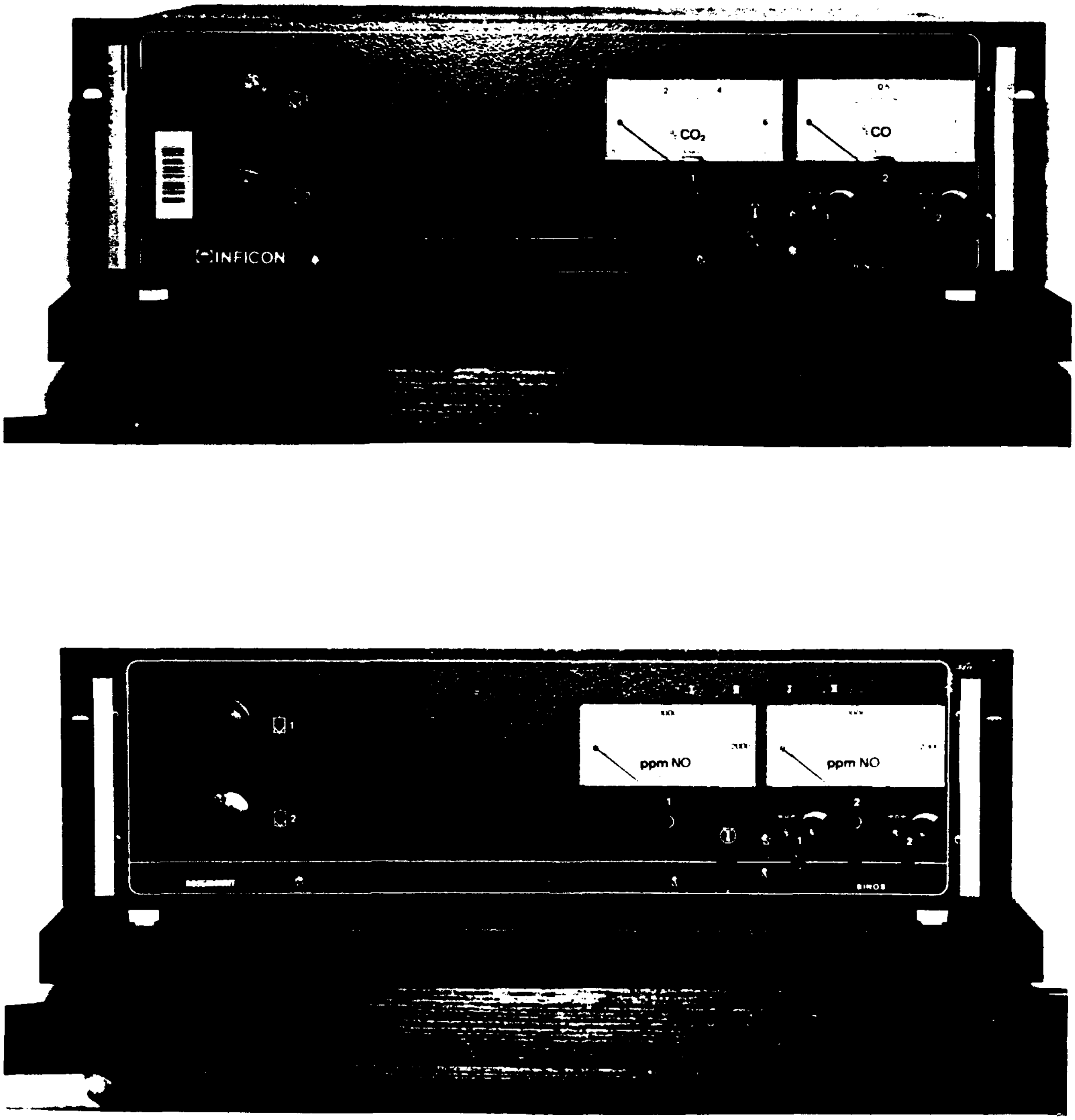

Figure 3. The BINOS Analyzers.

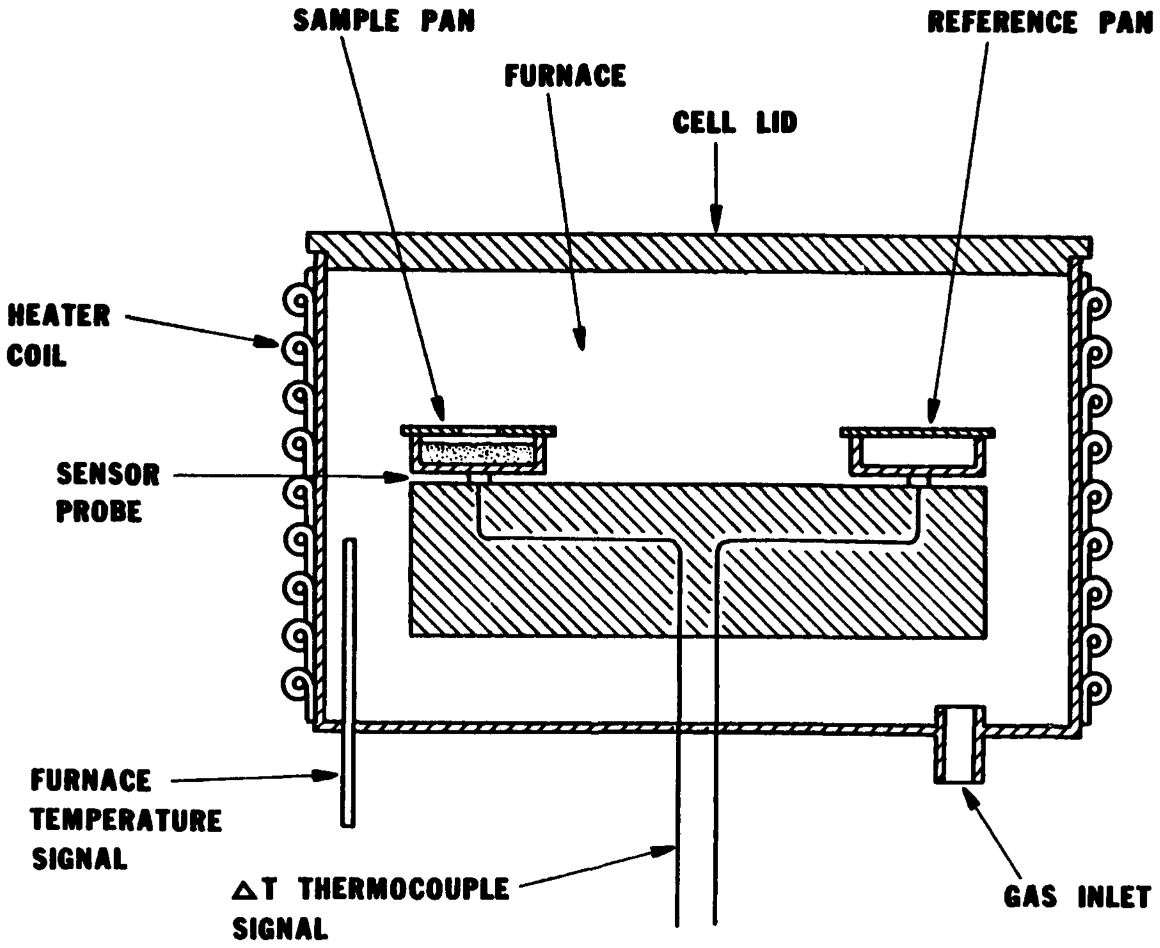

Figure 4. Schematic of the Mettler DSC 20 Cell.

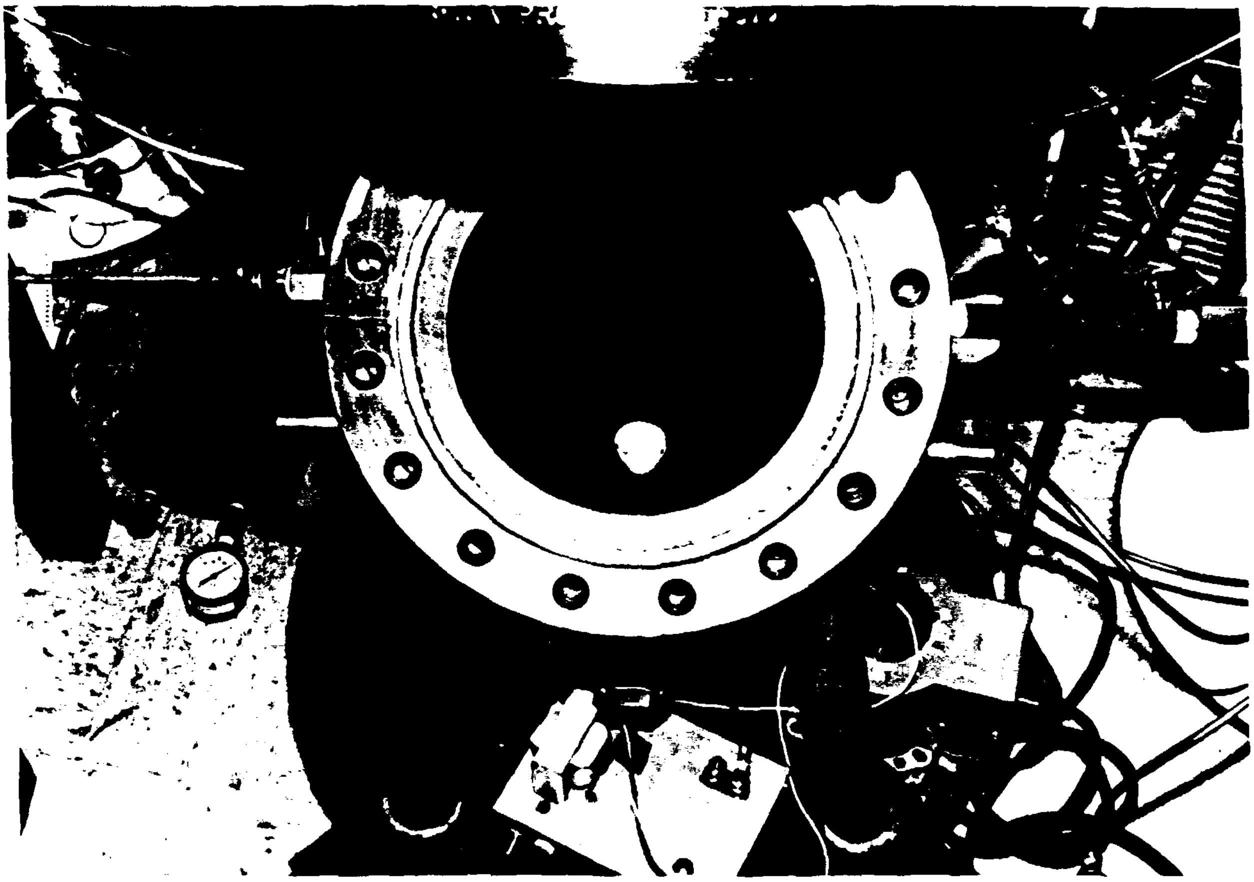

Figure 5. Crucible in the 20-Liter Chamber.

When a mixture of Halon 1301 in air, sulfur hexafluoride (SF_6) in air, or CO_2 in air was used as the gaseous atmosphere, the chamber was initially evacuated with the valve to the BINOS analyzers closed. The proper amount of the Halon 1301, SF_6, or CO_2 was introduced into the chamber and read on the Wallace-Tiernan gauge. Make-up air to 1-atm pressure was then added. A valve was opened to allow gas flow to the BINOSs. The valve to the air was left open to prevent any pressure drop when the diaphragm pump was activated. The nichrome ribbon was heated to ignite the NC and the liberated gases were analyzed.

In the tests in which a powder was added to the NC sample, equal weights of powder and NC either were stirred in the crucible or were layered, with the powder on top of the NC. An air atmosphere was used in all powder tests.

2.4.2 Tests in the DSC. Approximately 1 mg of NC was weighed into an aluminum pan and the lid sealed on top. One pinhole was punched into the top to prevent pressure buildup in the sealed pan. Initial tests had shown that NC samples as small as 0.80 mg in an unvented, sealed aluminum pan would explode upon heating.

A flow of the gas to be used was initiated. All tests were done at a flow rate of 40 cm^3/min. All NC samples were heated from 100° to 300° C at a rate of 20° C/min. The high gas flow and small sample size appear to have effectively diluted the gases evolved from the NC decomposition. No evidence of NC ignition was observed.

3. RESULTS

3.1 NC Combustion in Various Gaseous Atmospheres. Using the BINOS nondispersive spectrometers, analyses were carried out for CO, CO_2, NO, and NO_2 when samples of NC were heated in various gases in the 20-liter chamber. The nominal weight of NC used in each test was 100 mg. The nitrogen content of the NC was 13.44%. The maximum production of NO and NO_2 can be calculated, assuming these are the nitrogen-containing products of NC combustion.

For a 100-mg sample of NC containing 13.44% nitrogen, burned in a 20-liter chamber, the maximum amount of NO plus NO_2 that can be formed would be 1,075 ppm. If some of the nitrogen content forms N_2, or even some nitrous oxide (N_20), then the amount of NO plus NO_2 found would be less than 1,075 ppm.

A similar calculation with respect to CO and CO_2 yields a maximum production of 2,259 ppm or 0.23% CO plus CO_2. This theoretical concentration is based on the assumption that all carbon forms CO or CO_2. The calculation is also based on the theoretical composition of NC, which is $C_6 H_7 N_3 O_{11}$. The 0.23% CO plus CO_2 sum should be close to the real value. Any carbon unaccounted for in the measured concentrations was probably in the form of a carbonaceous solid.

3.1.1 NO and NO_2 Data. The results of a series of tests in which NC was heated by the nichrome ribbon are given in Table 2. In a nitrogen atmosphere, and in other chemically inert gases, the nitrogen content of the NC is virtually quantitatively released as NO. Very little, if any, NO_2 was found. Only a small flame was usually found in the inert atmospheres.

Table 2. Gases Generated When NC is Heated by Nichrome Ribbon in 20-Liter Chamber

Test	Atm	Mass NC (g)	Residue (g)	CO (% max)	CO_2 (% max)	NO (ppm max)	NO_2 (ppm max)	Comments
1	N_2	.100	—	.145	.060	1,380	16	small flash
2	N_2	.100	—	.130	.045	1,060	6	small flash
3	N_2	.100	—	.133	.048	1,140	11	small flash
4	N_2	.100	.004	.180	.066	1,200	0	small flash
5	N_2	.103	.003	.219	.081	1,240	0	small flash
6	He	.100	.004	.130	.045	970	0	—
7	He	.100	.004	.135	.050	970	0	—
8	Ar	.100	.007	.110	.050	1,340	9	—
9	Ar	.100	.003	.135	.050	1,400	16	—
10	CO_2	.100	.004	—	—	810	0	CO and CO_2 analyzers were overloaded.
11	CO_2	.100	.005	—	—	850	17	CO and CO_2 analyzers were overloaded.
12	CO_2	.102	.003	—	—	1,010	16	CO and CO_2 analyzers were overloaded.
13	50% CO_2 50% Air	.100	.006	—	—	1,330	148	CO and CO_2 analyzers were overloaded.
14	50% CO_2 50% Air	.100	.005	—	—	1,110	136	CO and CO_2 analyzers were overloaded.
15	CO	.100	.004	—	.060	1,200	0	—

Table 2. Gases Generated When NC is Heated by Nichrome Ribbon in 20-Liter Chamber (continued)

Test	Atm	Mass NC (g)	Residue (g)	CO (% max)	CO_2 (% max)	NO (ppm max)	NO_2 (ppm max)	Comments
16	CO	.100	.005	—	.030	1,160	0	
17	Halon 1301	.100	.006	.172	.050	1,050	22	Halon 1301 caused positive readings on NO, CO, and CO_2 channels.
18	7% Halon 1301 in Air	.101	—	.119	.060	530	96	Halon 1301 caused positive readings on NO, CO, and CO_2 channels.
19	7% Halon 1301 in Air	.100	.005	.120	.090	670	112	Halon 1301 caused positive readings on NO, CO, and CO_2 channels.
20	21% Halon 1301 in Air	.100	.005	.170	.060	1,190	148	Halon 1301 caused positive readings on NO, CO, and CO_2 channels.
21	7% SF_6 in Air	.100	.005	.060	.140	1,040	142	small flash
22	7% SF_6 in Air	.100	.004	.015	.210	540	40	large flash - similar to air
23	7% SF_6 in Air	.100	.005	.013	.210	550	36	large flash - similar to air
24	21% SF_6 in Air	.100	.006	.118	.045	750	112	small flash
25	21% SF_6 in Air	.100	.004	.135	.090	1,100	164	small flash
26	Air	.100	.006	.004	.210	400	20	large flash
27	Air	.100	.003	.010	.240	500	28	large flash

When air was present, NO_2 was found. The combustion was usually stronger with a larger flame in air than in the inert gases. There was less NO plus NO_2 found for the air combustion than for combustion in inert gas. The low concentration of NO plus NO_2 may well be due to the stronger air flame consuming more of the oxides of nitrogen, forming N_2 gas.

It is interesting that CO acted as an inert gas, at least as far as oxides of nitrogen were concerned. Only NO was formed, no NO_2. As expected, CO_2 also acted as an inert gas, in that no NO_2 was formed, except for the small amounts in tests 11 and 12, probably due to some residual air in the chamber. An atmosphere of Halon 1301 also appeared to act as an inert gas, with only a small flame from the NC. The small quantity of NO_2 formed may also have been due to the presence of some residual air. Halon 1301 caused positive readings on the NO, CO, and CO_2 channels of the BINOS. The readings were subtracted from readings after the NC burns, but may have introduced some errors.

When NC was heated in a He atmosphere, no NO_2 was detected, as expected. However, the concentration of NO found was somewhat lower than found with Ar and with N_2. The low concentration of NO in He may be due to better mixing of product gases with He than with Ar or N_2. The concentrations of NO found with Ar and N_2 exceeded that predicted from the composition and weight of NC samples. Stratification of product gases may occur in the chamber. Since there was no forced convection in the 20-liter chamber, product gases may not have mixed well with the atmosphere originally in the chamber. The scenario of partial mixing of product gases and eventual flame is depicted in Figure 6. Superior mixing of product gases with He is to be expected, based on the small size and mass of the He atom. The NO analysis with He may be more representative of the true situation when NC is heated in an inert atmosphere than the Ar and N_2 data. Incomplete burning of NC in He, caused by the high thermal conductivity of He, is probably not a valid explanation. The solid residues from reactions in He atmospheres were no greater than in other atmospheres. Data on residues from the individual tests are presented in Table 2.

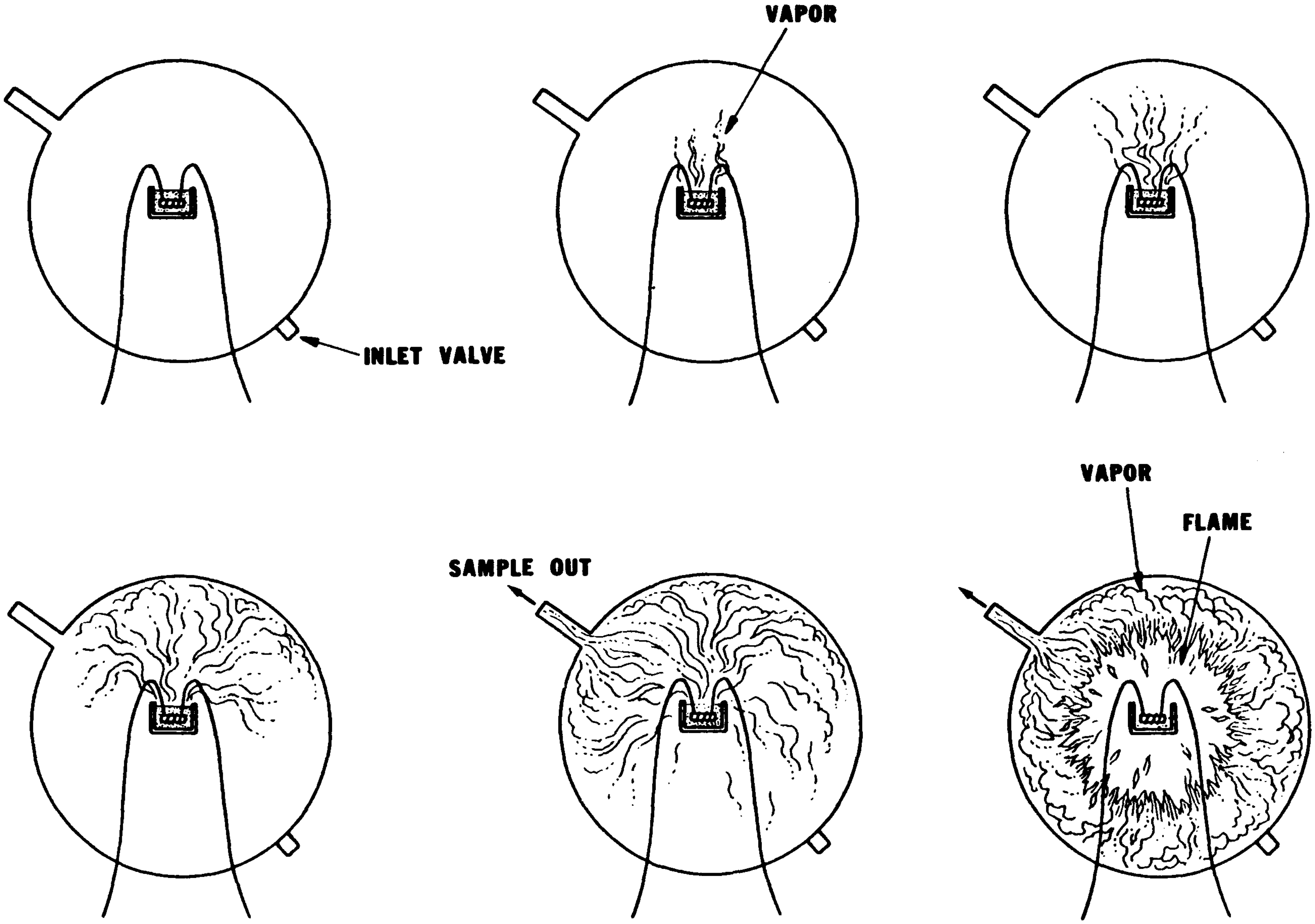

Figure 6. Decomposition of NC Leading to Flame.

Silicone hexafluoride in air gave results similar to those from Halon 1301 in air. Production of NO_2 at the 100 ppm or above level was found for both gases except for two tests of SiF_6 in air. It is interesting to note that in both of these tests, there was a large flash of fire. This large fire probably consumed much of the NO_2 which had formed.

The only instances in which concentrations of NO_2 over 100 ppm were found were when an inert gas was mixed with air. The explanation offered is that when neat air was used, a large flame appeared, and most of the NO_2 that may have formed was consumed. When some inert gas was present, the flame was generally smaller, consuming less of the NO_2. When no air was present, no NO_2 was formed.

3.1.2 CO and CO_2 Data. Table 2 shows that the CO plus CO_2 concentrations were consistently close to the 0.23% sum expected, except in test 5, which had a CO plus CO_2 sum of 0.3%. No explanation can be offered for this single, very high sum.

In a manner similar to the NO and NO_2 measurements in the presence of air, the tendency was to form more CO_2 at the expense of CO if air was present. The increased production of CO_2 was evident even in mixtures of inert gases with air. Mixtures with higher air levels generally had higher CO_2 contents. This trend was not as definitive as in the NO, NO_2 cases.

3.2 Nitrocellulose Mixed With Various Powders. Samples of NC were placed in the quartz crucible with several powders to determine what effect the powders would have on the combustion of NC and the formation of gaseous products. All tests were carried out in the 20-liter vessel in air at 1 atm.

Three powders were chosen. Two of them, Purple K and Monnex, are effective fire extinguishing agents. The third powder, Molecular Sieve 13X, is very effective in absorbing NO_2 (Finnerty et al. 1990).

In tests where the powders were simply layered on top of the NC, with the nichrome ribbon heater in the NC layer, ignition occurred as if no powder were present. Much of the powder was blown out of the crucible. Flame size was comparable to that which was observed when no powder was used.

When the powders were stirred with the NC samples, ignition was different. Power to the nichrome ribbon had to be on for a longer time before a flame appeared. Power had to remain on until combustion was complete. All three powders interfered strongly with ignition and burning of NC.

The results of burning NC with powders are given in Table 3. In general, all powders had similar effects on NC. When well-stirred (equal weights of NC and powder), ignition was difficult and the NC burned slowly. The flames were small and lasted 5 to 14 seconds. The NO and NO_2 production tended to be somewhat higher than in tests of NC burning in air with no powder. The small flames, which appeared after heating the sample for some time, may have consumed less of the oxides of nitrogen than when large flames flashed in the chamber.

When a powder was simply layered on top of the NC, a larger flame occurred and both NO and NO_2 productions were very close to what was found when no powder was used.

The CO production was not very different whether powder was simply layered on top of the NC or stirred with the NC. However, CO production tended to be higher than when no powder was used in air.

The CO_2 production was higher when the powder was layered on the NC than when the two were stirred together. In the layered tests, CO_2 levels were close to what was found using no powders in air. Less CO_2 was found in the tests of stirred material in air.

The most important effects the powders had on NC combustion were: 1) more difficult ignition, 2) lower rate of consumption, 3) smaller flame size, and 4) continued presence of ignition source required.

These effects were especially noticeable in cases when the powders were stirred with NC prior to ignition. The increase in heat capacity of the samples due to the addition of the powders is probably the cause of reduced reactions.

Table 3. Gases Generated When NC, Mixed With Powders, is Heated in 20-Liter Chamber

Test	Atm	Mass NC (g)	Mass Powder (g)	CO (% max)	CO_2 (% max)	NO (ppm max)	NO_2 (ppm max)	Comments
1	Air	.100	.100 13X mixed with NC	.058	.120	730	88	small flame - did not start to burn until ribbon became red hot.
2	Air	.100	.100 13X mixed with NC	.064	.090	635	92	small flame burned 14 s.
3	Air	.100	.100 13X layered on top of NC	.014	.210	670	48	large flame - burned as if no Molecular Sieve 13X were present.
4	Air	.100	.100 13X layered on top of NC	.005	.225	680	52	large flash fire - as if no Molecular Sieve 13X were present.
5	Air	.100	.100 Purple K mixed with NC	.070	.144	600	36	very small flame - incomplete combustion even with power on to heater, flame lasted only 5 s.
6	Air	.100	.100 Purple K mixed with NC	.063	.120	550	34	very small flame - incomplete combustion, power turned off after 5 s since flame went out.
7	Air	.100	.100 Purple K layered on top of NC	.098	.120	1,040	104	much of the Purple K sank down through NC due to greater effective density of Purple K fairly large flame, but smaller than flame of molecular sieve 13X layered experiments.

Table 3. Gases Generated When NC, Mixed With Powders, is Heated in 20-Liter Chamber (continued)

Test	Atm	Mass NC (g)	Mass Powder (g)	CO (% max)	CO_2 (% max)	NO (ppm max)	NO_2 (ppm max)	Comments
8	Air	.100	.100 Purple K layered on top of NC	.071	.15	820	68	larger flame than previous experiment. Purple K was added carefully to minimize its sinking through NC.
9	Air	.100	.100 Monnex mixed with NC	.043	.15	495	31	very small flame - power turned off after 7 s since flame went out.
10	Air	.100	.100 Monnex mixed with NC	.040	.13	446	28	very small flame - power turned off after 6 s since flame went out.
11	Air	.100	.100 Monnex layered on top of NC	.040	.21	480	28	not all of NC was covered by Monnex large flame - similar to burning NC in air with no powder.
12	Air	.100	.100 Monnex layered on top of NC	.070	.18	665	49	Monnex was spread more evenly over NC, large flame - similar to burning NC in air with no powder.
13	Air	.100	—	.010	.18	485	37	NC was stirred by itself before burning to compact it, flame similar to burning the more fluffy NC.

The powders, especially the molecular sieve, were expected to reduce the NO_2 to low values by absorption. Previous work (Finnerty et al. 1990) had shown that Molecular Sieve 13X was an effective material for removing this toxic gas. However, even when using the molecular sieve, the NO_2 levels remained higher than were desired.

3.3 Tests in the DSC. Five gaseous atmospheres were used in the DSC. They were: N_2, O_2, SF_6, CO, and air. Each atmosphere was tested five times and the data from individual tests were averaged. The temperature at which the maximum rate of heat liberations from NC occurred was determined by the software of the DSC. The total heat liberated during decomposition of the NC was also determined by the DSC and displayed.

The temperatures at which NC generated heat at the maximum rate were not affected by the nature of the gaseous atmosphere. The maximum rate of heat generation occurred at about 215° C in all gases. The absence of any temperature effect means that the various gases did not influence the solid phase decomposition of NC. The lack of any influence due to the nature of the surrounding atmosphere is consistent with results of NC ignition tests in various gases conducted by Fenimore and Frazer (1944). The temperatures of the maximum rate of heat liberation from NC in the different gases are given in Table 4. The same data, in graphical form, are presented in Figure 7.

The total heat generated by decomposing NC was influenced by the nature of the gaseous atmosphere. The oxidizing gases (O_2 and air) gave larger values of heat generation than did the gases regarded as inert (N_2 and SF_6). CO behaved more like a reactive gas than an inert gas. One explanation is that CO may react with other gases generated by decomposing NC.

The heat liberated per gram of decomposing NC in various atmospheres is given in Table 5. The data are also presented in Figure 8. Only about 1.15 kJ/g is liberated in the inert atmospheres. The DSC experiments were done in vented aluminum pans. The pressure probably did not rise above 1 atm. Frazer and Fenimore (1943) have shown that the heat of explosion of NC decreases with decreasing loading density and decreasing pressure. Combustion is probably incomplete at low pressures. They found, at a calculated pressure of 1.2 atm in their Parr bomb, a heat of explosion of NC of about 2.38 kJ/g, in contrast to a value of about 4.47 kJ/g, at high pressure of inert gas and high loading densities. The DSC

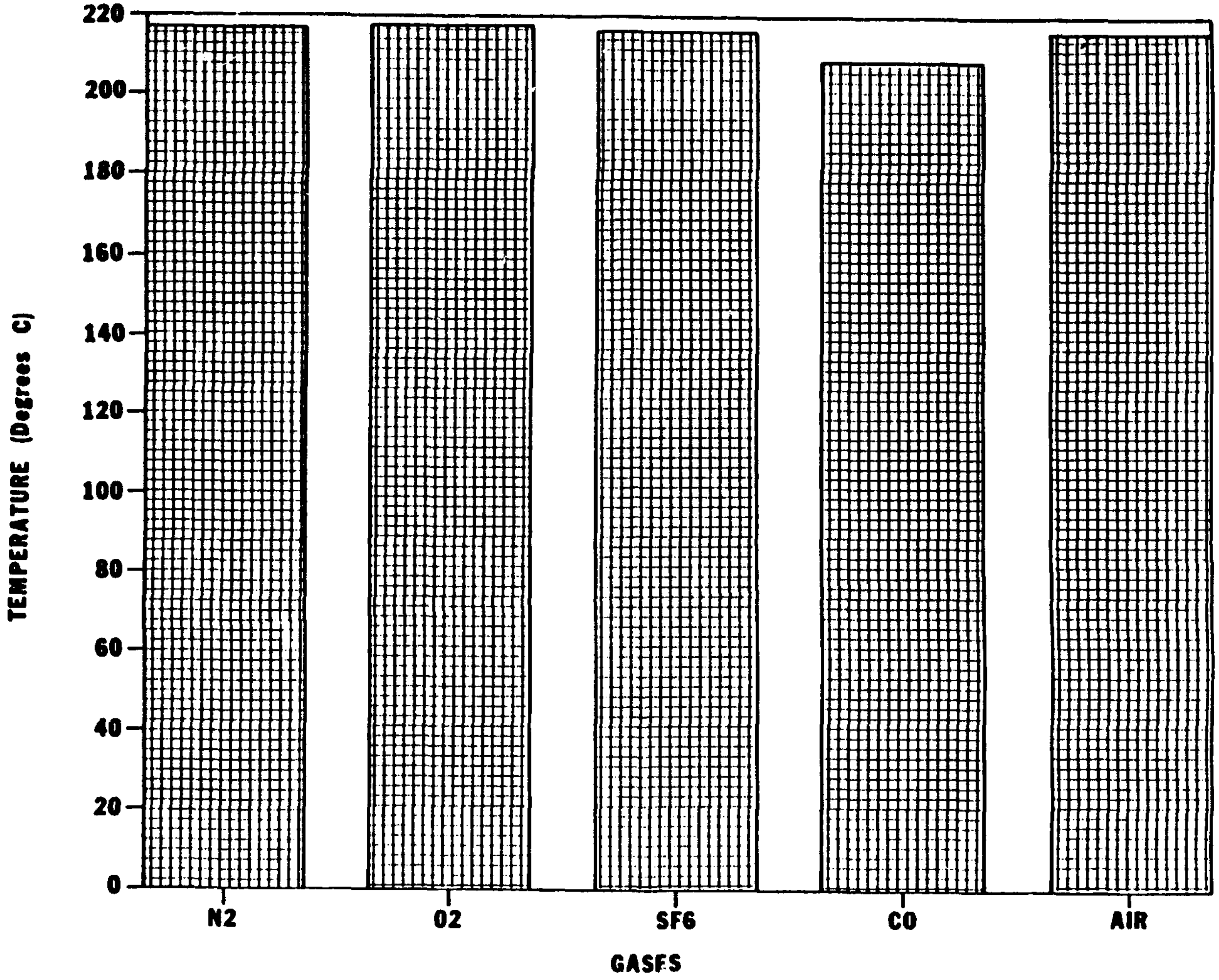

Figure 7. Temperatures of Maximum Rate of Heat Liberation by NC in Different Gases.

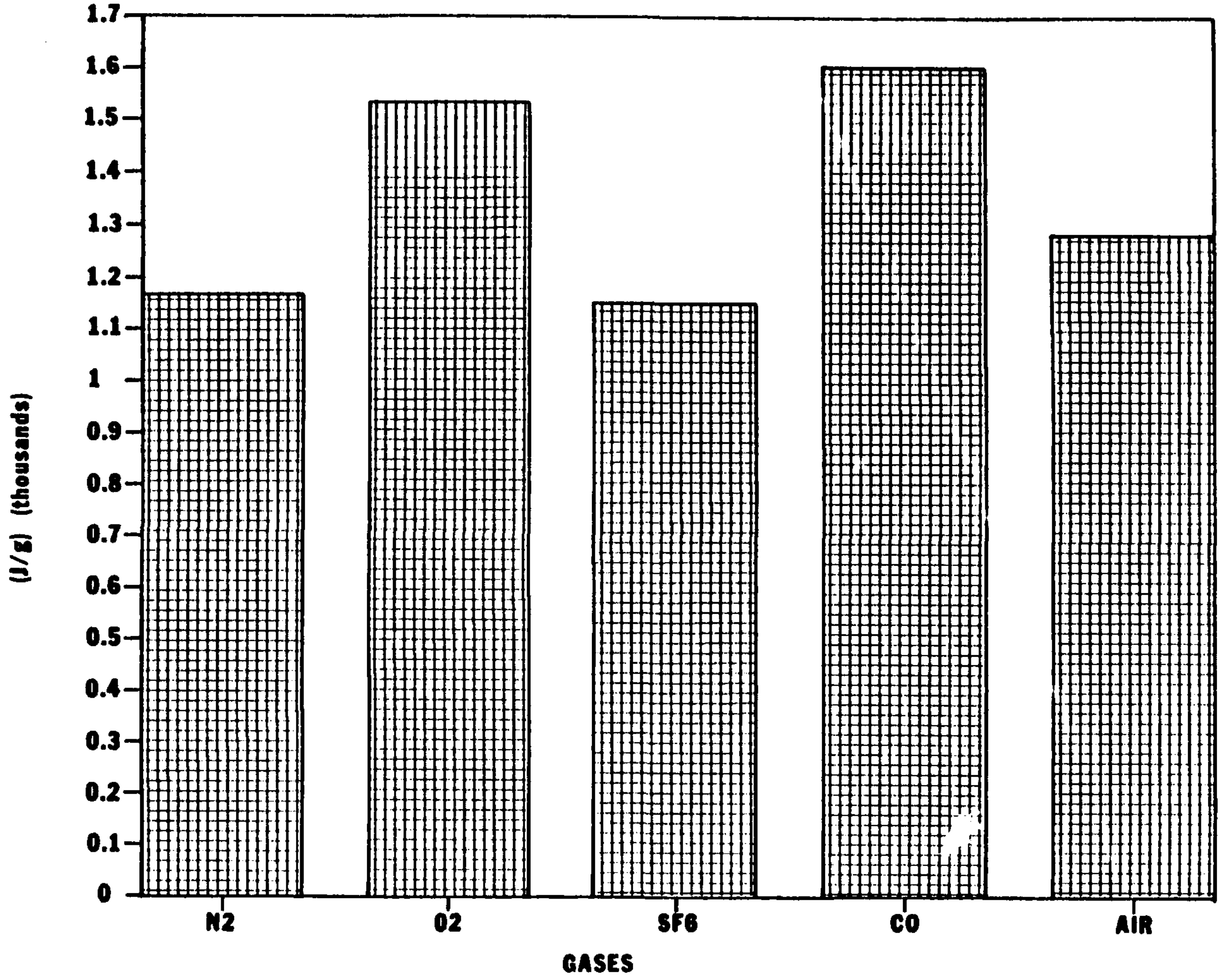

Figure 8. Heat Liberated by Decomposing NC in Various Atmospheres.

Table 4. Temperature of Maximum Rate of Heat Liberation by NC in Vented Pan in DSC

Gas	Flow (cm^3/min)	Temperature (°C)
N_2	40	217.5
O_2	40	218
SF_6	40	216.8
CO	40	210
Air	40	216.8

Table 5. Heat Liberated Per Gram of Decomposing NC in Vented Pan in DSC

Gas	Flow (cm^3/mm)	Heat (kJ/g)
N_2	40	1.17
O_2	40	1.53
SF_6	40	1.13
CO	40	1.61
Air	40	1.26

experiments in inert gases probably gave low values of the heat of explosion of NC due to incomplete combustion. The evolved gases did not react with themselves and/or solid material. Some of the evolved gases escaped from the vented pan before complete reaction occurred. The evolved gases probably underwent more extensive reactions in the oxidizing atmospheres. In O_2, the heat of reaction of NC was about 1.53 kJ/g.

4. CONCLUSIONS

The following conclusions can be drawn from our experiments on the combustion of NC at one atmosphere pressure:

(1) Combustion in an inert atmosphere produces only small, weak flames.

(2) In inert atmospheres, combustion products are low in oxygen. NO is formed in preference to NO_2. Any NO_2 present may well be due to residual air. CO is formed in preference to CO_2 by a factor of about 2.4.

(3) Combustion in an oxidizing atmosphere produces a strong flame.

(4) In an oxidizing atmosphere, less NO is formed than in the inert atmosphere case. However, not much NO_2 is found. Most of the NO_2 that did form was probably consumed in the strong flame which accompanied combustion in air. The CO-CO_2 balance changes to favor CO_2 when air is present.

(5) Upon heating, NC undergoes exothermic decomposition which is much less energetic when the evolved gases are allowed to escape from contact with the solid NC sample.

(6) The solid phase decomposition of NC is affected by temperature but not affected by the nature of the surrounding gas.

(7) Total heat liberated by decomposing NC is greater in an oxidizing atmosphere than in an inert atmosphere.

(8) Mixing a nonreactive powder with NC makes the NC harder to ignite and requires continued application of heat for continued burning. NC probably would not be self-sustaining under conditions of added nonreacting powder.

(9) The NO_2 levels with powders present, even Molecular Sieve 13X, were not below what was found with no powders present.

(10) The most promising method of extinguishing burning NC is to add a non-energetic material of high heat capacity in close contact in order to absorb the heat evolved from decomposing NC.

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