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IN SPACE ORDNANCE

Author(s):

Mary S. Campbell, DX-16
Kien-Yin Lee, DX-16
Michael A. Hiskey, DX-16

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NEW HIGH-NITROGEN ENERGETIC MATERIALS FOR GAS GENERATORS IN SPACE ORDNANCE

Mary M. Stinecipher (Mary S. Campbell), Kien-Yin Lee, and Michael A. Hiskey

Los Alamos National Laboratory

Los Alamos, New Mexico

Abstract

High-nitrogen nitroheterocyclic energetic compounds are used as explosives, propellants, and gas generants when safe, thermally stable, cool-burning energetic materials are desired. A series of compounds are compared for sensitivity properties and calculated burn performance. Thermodynamic equilibrium calculations by NASA/Lewis rocket propellant and Blake gun propellant codes gave flame temperatures, average molecular weight, and identity of the equilibrium burn products for ambient, rocket, and gun pressure environments. These compounds were subjected to calculations both as monopropellants and as 50/50 weight ratio mixtures with ammonium nitrate (AN). Special attention was paid to calculated toxic products such as carbon monoxide and hydrogen cyanide, and how these were affected by the addition of an oxidizer AN. Several compounds were noted for further calculations of a formulation and experimental evaluation.

Introduction

Desirable properties for energetic materials for propellants and gas generants include high thermal stability, insensitivity to spark, clean low-molecular-weight gases as the burn products, and low detonability. High-nitrogen heterocycles have a high percentage of nitrogen, low carbon and no halogens and possess desirable stability. Many nitroheterocyclic compounds were synthesized and characterized as to their chemical, physical, and sensitivity properties for use as explosives or gun propellant ingredients by members of the Explosive Technology and Safety Group at Los Alamos.^{1,2,3} Many of these compounds are salts because a nitro group on a five-membered nitrogen-containing heterocyclic ring is stable in the anionic form, which has the correct number of pi-electrons for aromaticity.

Cations selected for stability and ease of synthesis are: ammonium (A), hydrazinium (H), guanidinium (Gu), triaminoguanidinium (TAG), ethylenediammonium (E), and 3,3-dinitroazetidinium (DNAZ, an energetic cation).

Anions used were: nitrate (N), 5-nitro-1,2,4-triazol-3-onate (anion of NTO), 3,5-dinitro-1,2,4-triazolate (DNT), and 5-nitrotetrazolate (NT). Neutral heterocycles studied were RDX, NTO,⁴ 3-amino-5-nitro-1,2,4-triazole (ANTA),⁵ 1-amino-5-nitro-1,2,4-triazole (I-ANTA),^{*} 3,6-diamino-1,2,4,5-tetrazine (DATZ),⁶ 1,4-dioxy-3,6-diamino-1,2,4,5-tetrazine (TZX),⁶ 1,4-dinitroglycoluril (DINGU),^{7,8} 1,1'-dinitro-3,3'-azo-1,2,4-triazole(N-DNAT), and 5,5'-dinitro-3,3'-azo-1,2,4-triazole (DNAT).⁷ ANTA was used as a synthon to prepare 4,6-bis(5-amino-3-nitro-1H-1,2,4-triazol-1-yl)-5-nitropyrimidine (ANTAPM), 3,6-bis(5-amino-3-nitro-1H-1,2,4-triazol-1-yl)-1,2,4,5-tetrazine (ANTATZ), and 2,4,6-tris(5-amino-3-nitro-1H-1,2,4-triazol-1-yl)-1,3,5-triazine (ANTATRZ).⁵

Sensitivity Properties

Sensitivity Test Methods

New energetic materials are tested for safety before making greater than one-half gram by finding impact and thermal sensitivities. The impact sensitivity is found on a ERL-type machine equipped with type-12 tools and a microphone for sound analysis. A 40-mg sample heaped on a

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* Sample from J. Botarro, SRI, International.

circle of sandpaper is placed under an anvil and a 2.5 kg weight is dropped onto the anvil. An arbitrary level of sound is set as "go" to give a 150-cm drop height as a 50% "go" for TNT. The 50% "go" height was determined by the Bruceton Up and Down method.⁹

The thermal sensitivity is measured on a mg of sample by differential thermal analysis (DTA), which indicated a temperature imbalance between the sample and an inert. As the sample begins to decompose it releases heat as noted by the beginning of an exotherm. A larger scale thermal sensitivity test is the modified Henkin method developed by Rogers.¹⁰ A thin slab of material is pressed into a cartridge and confined with a metal seal. The cartridge is placed in an isothermal bath of molten metal and timed until explosion. The temperature of the bath is changed to find the lowest temperature that an explosion will occur. The Henkin critical temperature is the lowest temperature that will cause a 0.7-mm-thick slab to go into a runaway reaction that causes an explosion. This test is especially useful for testing compatibility of formulations.

The spark test subjects a 40-mg sample confined by a 3-mil foil to a spark from a capacitor bank. A "go" is determined if the foil is burst. The maximum energy capacity of a person is 0.18 J.

Sensitivity Results

The results of sensitivity tests for these compounds are shown in Table I along with the weight-percent nitrogen. Note that, with the exception of N-DNAT, DNAT, and DNAZN, the beginning of the exotherm on the DTA for these compounds is greater than 170°C. Not all new compounds have been tested on the Henkin test because it is primarily used after a compound is prepared and formulated on a larger scale.

In the impact sensitivity test, the compounds are all more stable than RDX, except N-DNAT. Lee made only a small amount of this isomer of DNAT, which has the nitro group on the ring nitrogen.¹¹ N-DNAT was found to be more thermally unstable and sensitive to impact than we want to handle. It is included in this table to show the difference in stability of a nitro group bonded to nitrogen vs one bonded to a carbon. Some of the compounds are very insensitive and do not react at the highest drop height of 320 cm.

All the new compounds are less sensitive to spark than RDX. Compounds with the ">" have

had only a limited series of tests, which showed they were safe to handle.

Calculations

Before choosing a new energetic material for a use as a propellant or gas generant, a use in ordnance must be specified. However, many of the new uses need clean burning, low toxicity and corrosivity products and low flame temperatures. Desirable compounds can be chosen for further calculations and experiments by studying the preliminary calculations without specifying a use. Some uses require the material to be burned at high pressures or at variable pressures like in a gun chamber. Calculations at pressures less than 70 mPa were done with the ideal gas equation of state in the NASA-Lewis rocket propellant code. Calculations for constant volume with pressures up to 350 mPa were done with the Blake gun propellant code using the truncated virial equation of state.

NASA-Lewis Calculations

The NASA-Lewis code was used to find the flame temperature (T) and average molecular weight of the product gases (M) at constant pressure and enthalpy. Each compound was calculated as monopropellant and a 50/50 weight mixture with an oxidizer ammonium nitrate (AN). A comparison of the effect of the addition of AN on the T shows that it increased much more if the compound is under oxidized. For RDX and, T decreased when mixed with AN because they are so well balanced. The M generally was higher on addition of AN because less hydrogen was present in the products. Table II shows M and T at both 1 and 68 atm (1000 psi) with and without AN. If an energetic material is desired that produces a low M non-condensable gas, some good choices would be TAGNTO, ENT, GuNT, TAGDNT, HNT0, TZX. Those compounds have M below 21 at rocket motor pressure of 68 atm. An exception to the general rule that M becomes larger on addition of AN is the compound DATZ. Calculations show that a mixture of DATZ and AN has a lower M than DATZ. When a small amount of AN is added to ENT0, M is smaller than the monopropellant M. The formulation could be adjusted for the desired performance.

A second consideration is flame temperature. The compound in the low M group of the previous paragraph with the lowest T is TAGNTO with 1428 K. The one with the highest T is TAGDNT

with 2185 K. Again the use and the sensitivity properties would guide the choice.

The products formed on burning are important, also. Table III shows the calculated major products at 1 atm from the NASA/Lewis code for the pure compounds and their mixtures with oxidizer ammonium nitrate (AN). A desirable product for many uses is nitrogen gas (N_2). It is non-toxic, non-condensable, and cooler than carbon oxides. The compound with the highest calculated amount of N_2 is DNAT. An undesirable product containing nitrogen is hydrogen cyanide (HCN). Some high nitrogen compounds with no oxygen give HCN as a product. Addition of an oxidizer reduces the HCN to a very small amount. In the case of DATZ HCN is reduced from a mole fraction of 2×10^{-3} (3300 ppm) to 1×10^{-7} (0.13 ppm). Some uses such as gas generants need very low condensibles such as solid carbon and water. All the compounds presented have low carbon and DATZ, NTO, TAGNTO, ENT, ANTA, ANTAPM, ANTATZ, DNAT, and TZX have low water in the products. Addition of AN increases the water but not as much for DATZ/AN as the others.

Blake Gun Propellant Calculations

Results of the Blake code are in Table IV. Performance parameters calculated are the impetus and flame temperatures of these compounds at a loading density of 0.2 g/cm^3 at constant volume. If we look at the performance of the two salts that we just selected, we see TAGNTO under these conditions has an impetus of 836 J/g $T = 1980 \text{ K}$, and $M = 19.8$; while TAGDNT has an impetus of 1142 J/g, $T = 2798 \text{ K}$, and $M = 20.4$. This illustrates how strongly the performance (impetus) is dependent on the flame temperature. Some uses like emergency air bags may need a cooler gas and lower impetus. Other uses may need more punch. The temperature of TAGDNT is still low compared to some propellant ingredients such as RDX, which has a temperature of 4072 K.

Formulation and Burning Rate Measurement

An important property of an energetic material is its burning rate. If the compound is different from those previously tested, it must be tested in a formulation either as a cylinder in a constant pressure bomb or as grains of known dimension in a closed bomb. During a study of the effect of two of these compounds on the burning rate of a gun propellant formulation, we found in a closed bomb

test that a substitution of only 15% of RDX with ENTO or ENT significantly changed the burning rate of the gun propellant. ENTO slowed the burning rate while ENT increased it. The burning rate of the standard composition 76/12/7.6/4/0.4 wt percent RDX/CAB/ATEC/NC/EC was $3.9 \times 10^{-5} \text{ P}^{1.107}$, the burning rate relation units is in inches per second with respect to pressure in psi. Replacing 15wt% of the RDX with ENTO gave a burning rate of $7.51 \times 10^{-5} \text{ P}^{1.019}$ and with ENT a slight slope break gave $1.81 \times 10^{-4} \text{ P}^{0.977}$ below 15 kPsi and $3.30 \times 10^{-5} \text{ P}^{1.161}$ between 15 and 20 kpsi.¹²

Conclusions

Calculations of the flame temperatures and average molecular weight of the products were done on a series of high-nitrogen heterocycles. Further calculations on formulations of DATZ, ENT, GuNT, HNT, TAGDNT, TAGNTO, and TZX need to be done as well as sensitivity tests on the formulation to select a specific compound for use in space ordnance applications.

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TABLE I
EXPLOSIVES SENSITIVITY OF SELECTED COMPOUNDS

Compound	%N	DTA Exotherm. (°C)	Impact Type 12 (cm)	Spark Sens. 3-mil foil (J)	Henkin Crit. Temp. (°C)
NTO	49	236	280	0.91	238
ENTO	44	250	>320	2.7	232
HNT0	52	170	92	>1.0	----
GuNTO	52	260	>320	>1.0	----
TAGNTO	60	170	103	>1.0	173
ENT	64	216	42	1.7	209
GuNT	64	212	36	1.14	----
ADNT	48	200	58	0.86	225
TAGDNT	59	236	31	>1.0	----
DNAZN	27	150	32 -	0.5	----
ANTA	54	240	>320	>1.0	245
ANTAPM	48	300	>320	>1.0	----
ANTATZ	58	260	>320	>0.5	----
DNAT	55	150	69	0.53	----
N-DNAT	55	130	11	---	----
DATZ	75	280	>320	>1.0	----
TZX	58	220	150	>1.0	235
DINGU	36	220	97	1.2	204
RDX	38	210	22	0.2	217

TABLE II
FLAME TEMPERATURE (T) AND AVERAGE MOLECULAR WEIGHT OF PRODUCTS (M)
CALCULATED USING THE NASA-LEWIS CODE

<u>Compound</u>	<u>1 atm, 0.101 mPa</u>		<u>68 atm, 6.89 mPa</u>	
	<u>T(K)</u>	<u>M</u>	<u>T(K)</u>	<u>M</u>
NTO	1090	23.4	1304	24.7
NTO/AN	2111	24.6	2119	24.6
HNT0	1491	20.3	1498	20.3
HNT0/AN	2220	23.0	2237	23.0
ENTO	1012	20.3	1249	21.8
ENTO/AN(4/1)	1219	20.0	1305	20.6
ENTO/AN	1890	22.1	1891	22.1
GUNTO	948	21.3	1161	22.9
GUNTO/AN	1779	22.1	1780	22.1
TAGNTO	1280	18.1	1428	18.9
TAGNTO/AN	2105	21.5	2113	21.5
ADNT	2747	24.6	2939	25.0
ADNT/AN	2514	24.9	2716	25.3
TAGDNT	2171	20.2	2185	20.2
TAGDNT/AN	2473	22.8	2552	23.0
ENT	1711	19.4	1738	19.5
ENT/AN	2313	22.4	2341	22.4
GuNT	1346	19.4	1453	20.0
GuNT/AN	2163	22.4	2174	22.4
DNAZN	2850	24.9	3211	25.7
DNAZN/AN	2550	25.0	2779	25.5

TABLE II (Continued)

<u>Compound</u>	<u>1 atm. 0.101 mPa</u>		<u>68 atm. 6.89 mPa</u>	
	<u>T(K)</u>	<u>M</u>	<u>T(K)</u>	<u>M</u>
ANTA	1884	21.5	1894	21.6
ANTA/AN	2406	23.7	2456	23.8
I-ANTA	2352	21.5	2386	21.6
I-ANTA/AN	2551	23.5	2661	23.8
ANTAPM	2260	25.2	2276	25.3
ANTAPM/AN	2424	24.2	2473	24.3
ANTATZ	2565	25.7	2615	25.8
ANTATZ/AN	2526	24.2	2611	24.3
DNAT	2205	25.4	2216	25.4
DNAT/AN	2500	25.7	2629	26.0
DATZ	1780	22.4	1796	22.6
DATZ/AN	1741	20.0	1742	20.0
TZX	1984	20.6	1994	20.7
TZX/AN	2428	23.1	2483	23.2
DINGU	2384	23.3	2419	23.4
DINGU/AN	2528	24.6	2672	24.9
RDX	2925	23.5	3284	24.3
RDX/AN	2632	24.4	2905	25.0

TABLE III
CALCULATED MOLE FRACTION OF MAJOR BURN PRODUCTS
AT ONE ATMOSPHERE
FROM NASA-LEWIS ROCKET PROPELLANT CODE

<u>Compound</u>	<u>N₂</u>	<u>H₂</u>	<u>HCN</u>	<u>H₂O</u>	<u>CO</u>
NTO	0.401	0.190	5.2 x10-6	0.0074	0.364
NTO/AN	0.369	0.066	5.3 x10-9	0.348	0.104
HNT0	0.375	0.280	3.8 x10-7	0.095	0.220
HNT0/AN	0.356	0.073	3.1 x10-9	0.426	0.067
ENTO	0.299	0.312	1.9 x10-6	0.029	0.264
ENTO/AN(4/1)	0.300	0.304	4.5 x10-7	0.096	0.246
ENTO/AN(1/1)	0.310	0.144	2.1 x10-8	0.338	0.132
GuNTO	0.361	0.281	6.7 x10-7	0.049	0.160
GuNTO/AN	0.343	0.137	1.6 x10-8	0.344	0.105
TAGNTO	0.386	0.382	6.3 x10-5	0.001	0.230
TAGNTO/AN	0.364	0.141	1.4 x10-8	0.356	0.091
ENT	0.400	0.332	2.5 x10-4	0.0002	0.266
ENT/AN	0.371	0.110	1.0 x10-8	0.360	0.097
GuNT	0.445	0.332	1.0 x10-4	0.0005	0.222
GuNT/AN	0.397	0.105	7.7 x10-9	0.367	0.076
ADNT	0.419	0.078	2.7 x10-8	0.187	0.207
ADNT/AN	0.365	0.018	1.7 x10-10	0.424	0.029
TAGDNT	0.423	0.278	5.4 x10-7	0.066	0.220
TAGDNT/AN	0.381	0.074	3.7 x10-9	0.499	0.069
DNAZN	0.235	0.061	1.0 x10-8	0.267	0.219
DNAZN/AN	0.272	0.018	1.7 x10-10	0.458	0.036

TABLE III (continued)

<u>Compound</u>	<u>N₂</u>	<u>H₂</u>	<u>HCN</u>	<u>H₂O</u>	<u>CO</u>
ANTA	0.417	0.250	2.5 x10 ⁻⁴	0.0002	0.333
ANTA/AN	0.377	0.074	6.0 x10 ⁻⁹	0.356	0.101
I-ANTA	0.415	0.246	2.4 x10 ⁻⁴	0.0002	0.332
I-ANTA/AN	0.374	0.073	6.2 x10 ⁻⁹	0.349	0.104
ANTAPM	0.381	0.142	8.4 x10 ⁻³	<10 ⁻⁵	0.356
ANTAPM/AN	0.358	0.086	1.4 x10 ⁻⁸	0.292	0.162
ANTATZ	0.465	0.119	1.9 x10 ⁻²	<10 ⁻⁵	0.272
ANTATZ/AN	0.402	0.077	1.1 x10 ⁻⁸	0.290	0.134
DNAT	0.500	0.099	1.6 x10 ⁻⁴	0.0001	0.399
DNAT/AN	0.412	0.032	1.4 x10 ⁻⁹	0.333	0.074
DATZ	0.429	0.285	2.0 x10 ⁻³	0	0
DATZ/AN	0.393	0.260	1.3 x10 ⁻⁷	0.169	0.151
TZX	0.428	0.285	2.4 x10 ⁻⁴	0.0002	0.285
TZX/AN	0.385	0.084	6.7 x10 ⁻⁹	0.360	0.093
DINGU	0.299	0.153	1.3 x10 ⁻⁷	0.143	0.343
DINGU/AN	0.310	0.042	1.7 x10 ⁻⁹	0.413	0.082
RDX	0.316	0.091	3.3 x10 ⁻⁸	0.196	0.244
RDX/AN	0.314	0.030	6.0 x10 ⁻¹⁰	0.423	0.051

TABLE IV
BLAKE CALCULATIONS OF GUN PROPELLANT PERFORMANCE

Compound	Formula	ΔH_f^* (kcal/mole)	Impetus (J/g)	Temp. (K)	Ave.MW (A.M.U.)
NTO	C2H2N4O2	-28.1 $\pm$ 0.9	933	2920	25.0
HNT0	C2H6N6O3	-38 $\pm$ 5	799	1988	20.8
ENTO	C6H12N10O6	-112 $\pm$ 1	608	1638	22.5
GuNT0	C3H7N7O3	-71 $\pm$ 3	531	1489	23.4
TAGNT0	C3H10N10O3	+14 $\pm$ 4	836	1980	19.8
ADNT _r	C2H4N6O4	+0.6 $\pm$ 2.0	1205	3628	25.0
TAGDNT _r	C3H9N11O4	+59.8 $\pm$ 0.2	1142	2797	20.4
ENT	C4H10N12O4	+56 $\pm$ 3	960	2332	20.3
GuNT	C2H6N8O2	+17 $\pm$ 2	802	2000	20.8
DNAZN	C3H6N4O7	-64 $\pm$ 2	1274	3956	25.8
ANTA	C2H3N5O2	+21 $\pm$ 2	911	2421	22.1
I-ANTA	C2H3N5O2	+47.0 $\pm$ 0.8	1166	3072	21.9
ANTAPM	C8H5N13O6	+103 $\pm$ 5	806	2477	25.7
ANTATZ	C6H4O4N14	+175 $\pm$ 1	918	2870	24.3
DNAT	C4H2N10O4	+76 $\pm$ 3	1057	3256	25.6
DATZ	C2H4N6	+71.2 $\pm$ 0.7	532	1677	26.2
TZX	C2H4N6O2	+39.2 $\pm$ 0.4	1029	2621	21.2
DINGU	C4H6N6O6	-42.3 $\pm$ 0.1	1049	3249	25.8
RDX	C3H6N6O6	+14.7 $\pm$ 0.3	1391	4072	24.3

* Solid compounds at 298 K

Glossary

ADNT - ammonium salt of 3,5-dinitro-1,2,4-triazole
AN - ammonium nitrate
ANTA - 3-amino-5-nitro-1,2,4-triazole
ANTAPM- 4,6-bis(5-amino-3-nitro-1*H*-1,2,4-triazol-1-yl)-5-nitropyrimidine
ANTATZ - 3,6-bis(5-amino-3-nitro-1*H*-1,2,4-triazol-1-yl)-1,2,4,5-tetrazine
ATEC - acetyl triethylcitrate
CAB - cellulose acetate butyrate
DATZ - 3,6-Diamino-1,2,4,5-tetrazine
DINGU - 1,4-dinitroglycoluril
DNAT - 5,5'-dinitro-3,3'-azo-1,2,4-triazole
DNAZN - 3,3-dinitroazetidinium nitrate
EC - ethyl centralite
ENT - ethylenediammonium salt of 5-nitrotetrazole
ENTO - ethylenediammonium salt of 3-nitro-1,2,4-triazol-5-one
GuNTe - guanidinium salt of 5-nitrotetrazole
GuNTO - guanidinium salt of 3-nitro-1,2,4-triazol-5-one
I-ANTA - isomer of ANTA, 1-amino-5-nitro-1,2,4-triazole
N-DNAT - 1,1'-dinitro-3,3'-azo-1,2,4-triazole
HNTO - hydrazinium salt of 3-nitro-1,2,4-triazol-5-one
NC - 12.6%N nitrocellulose
NTO - 3-nitro-1,2,4-triazol-5-one
RDX - 1,3,5-trinitro-1,3,5-triazacyclohexane
TAGDNT - triaminoguanidinium salt of 3,5-dinitro-1,2,4-triazole
TAGNTO - triaminoguanidinium salt of 3-nitro-1,2,4-triazol-5-one
TZX - tetrazine explosive, 3,6-Diamino-1,2,4,5-tetrazine 1,4-dioxide