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Energetic Materials Center Report-Small-Scale Safety and Thermal Testing Evaluation of Butyl Nitrate

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SUMMARY

Butyl Nitrate (BN) was examined by Small-Scale Safety and Thermal (SSST) Testing techniques to determine its sensitivity to impact, friction, spark and thermal exposure simulating handling and storage conditions. Under the conditions tested, the BN exhibits thermal sensitivity above 150 °C, and does not exhibit sensitive to impact, friction or spark.

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1 INTRODUCTION

BN is a volatile liquid that is of interest for experiments to potentially be conducted in the Energetic Materials Center. As such, the BN has been evaluated for explosive handling safety parameters, to aid in the development of safe handling practices and safety approval. The approached used in the EMC is to conduct SSST testing on the material, simulating as close as possible, energetic insults that may occur while handling the material before, during and after experimentation. The response of the material to stimuli will set the handling and storage conditions. Note, there are procedures that have already been identified by the manufacturer for safe handling and storage of BN as a chemical, found in the MSDS provided by the manufacturer. Presented here is the overall assessment of BN when exposed to impact, friction, spark and thermal insult, combined with the precautions derived from the MSDS.

2 BACKGROUND

Butyl nitrate is a commercially available chemical that is a member of the alkyl nitrate class of compounds. Some of these compounds are used for legal medicinal purposes as inhalants, and some are used for illicit purposes[1]. Figure 1 shows the structure of BN. It is a 4 carbon molecule with a $–NO_3$ moiety capping one end, thus the nitrate nomenclature.

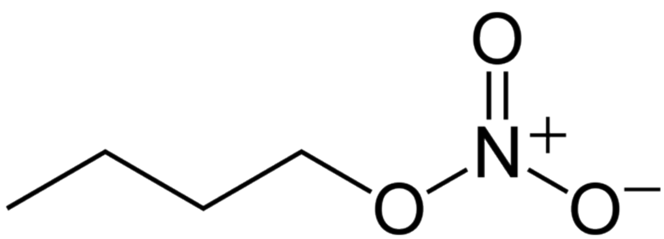

Figure 1. Structure of Butyl Nitrate

Chemical and physical properties. BN has the following properties:

1. Molecular Formula—$C_4H_9NO_3$
2. CAS number—928-45-0
3. Molecular Weight—119.12 g/ml
4. Density—1.047 g/ml
5. Flash Point—49.9 °C
6. ΔH vaporization—35.74 kJ/mol
7. Vapor pressure—9.6 mmHg at 25 °C
8. Melting point—< 0° C
9. Boiling Point—133 °C
10. Hazard classification—flammable

BN has also been identified as explosively decomposing above 150°C. It is an inhalation hazard causing. It "can cause slurred speech, euphoria, nausea, fainting, stupor, rapid heartbeat, anoxia, smooth muscle relaxation, lowered blood pressure, headache, vasodilation, myocardial sensitization, and hallucinations. If used the effects of this can last from 2 to 3 minutes. The effects of overdosing include damage to lungs, liver, kidneys, bone marrow, brain, suffocation, choking, anemia, possible stroke, and, in worst cases, death. Also there is a chance of addiction to BN and common symptoms of withdrawal are runny nose, insomnia, loss of or increased appetite, depression, irritability, headache, cramps, nausea, and tremors[1]."

From the MSDS[2], BN is classified as a combustible material, but not an explosive. It is not a DOT controlled material and is not on the EPA Toxic Substance Control Act (TSCA) inventory. It is an irritant to the eyes, skin and lungs—recommended handling with splash goggles, gloves and lab coat in an approved fume hood. It is labile under excessive heat and light, and is incompatible with oxidizing agents and acids. Respiratory protection, if used, is compliant with OSHA 29 CFR/1910.134 and ANSI Z88.2.

3 EXPERIMENTAL

3.1 Testing Methods

BN was test for impact sensitivity by drop hammer, friction sensitivity by BAM friction, spark sensitivity by ABL electrostatic discharge, and thermal sensitivity by differential scanning calorimetry (DSC). These methods applied at LLNL have been reviewed previously[3-8].

Figure 2. Drop Hammer at LLNL

Impact Sensitivity (Drop Hammer). ERL Type 12 drop hammer equipment at LLNL, shown in Figure 2, was used to determine the impact sensitivity[3]. The equipment includes a 2.5-kg drop weight, a striker (upper anvil, 2.5 kg for solid samples and 1.0 kg for liquid samples), a bottom anvil, a microphone sensor, and a peakmeter. For each drop, sample (35 mg for solids or 35 microliter for liquids) is placed on the bottom anvil surface and impacted by the drop weight from different heights. Signs of reactions upon impact are observed and recorded. These signs include noises, flashes or sparks, smoke, pressure, gas emissions, temperature rise due to exothermic reaction, color change of the sample, and changes to the anvil surface (noted by inspection). For solid samples, a "GO" was defined as a microphone sensor (for noise detection) response of ≥ 1.3 V as measured by a peakmeter. The higher the DH_{50} values, the lower the impact sensitivity. The method used to calculate DH_{50} values is the "up and down" or Bruceton method[4,5]. PETN and RDX have impact sensitivities of 12 and 24 cm, respectively (powder samples)[9,10]. TATB has impact sensitivity more than 177 cm[11]. For liquid samples, a "GO" was determined by the noise levels as measured by the peakmeter, appearance of flashes, temperature rise of the anvil, and visual inspection of the anvil surface. Two liquid samples TMETN and FEFO have impact sensitivities of 14 and 32 cm, respectively[11].

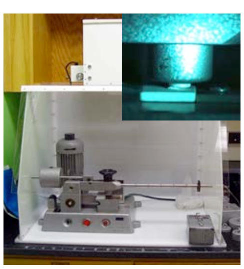

Figure 3. BAM Friction at LLNL

Friction Sensitivity. BAM friction sensitivity test equipment, as shown in Figure 3, was used to determine the sensitivity to friction[6]. The system uses a fixed porcelain pin and a movable porcelain plate that executes a reciprocating motion. Weight affixed to a torsion arm allows for a variation in applied force between 0.5 kg to 36.0 kg. The relative measure of the sensitivity of a material to friction is based upon the smallest load (kg) at which reaction occurs one level above for a 0-in-10 series of attempts, the Threshold Initiation Level (TIL)[12]. The lower the load values, the higher the sensitivity.

PETN has a sensitivity of 6.4 kg[9]. Also F_{50}, in kg, was determined by a modified Bruceton method[4,5].

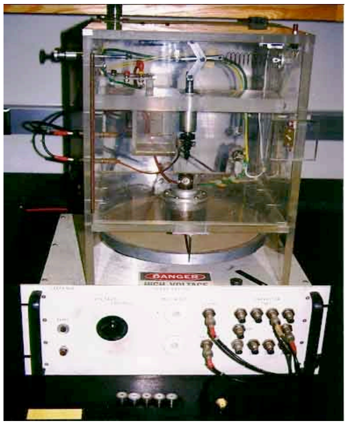

Figure 4. Custom ESD at LLNL

Spark Sensitivity. The static spark equipment at LLNL was used to evaluate the electrostatic discharge hazards (human ESD) associated with the handling of explosives[7]. The machine was custom-built almost 30 years ago and consists of a capacitor bank (up to 20,000 pF), a voltage meter, and a discharge circuit, as shown in Figure 4. An adjustable resistor up to 510 Ω (chosen to simulate human body) is wired to the discharge circuit. A 5-mg sample is placed in a Teflon washer sealed to a steel disc and covered with a Mylar tape. High static voltage (up to 10 kv) is applied and discharged to the sample. Evidence of reaction is judged from the condition of Mylar tape, smokes, and color change of the sample. Voltage, capacitance, and resistance can be adjusted to achieve the desired static energy. The results obtained as TIL and are expressed as a 0/10 or TIL+ as 1/10 at a specific voltage and joules[12]. One reaction in ten trials at ≤0.25 joules is considered spark-sensitive. Primary explosives show reaction at 0.1 joule.

Figure 5. New ABL Friction system at LLNL

New ABL spark test equipment, as shown in Figure 5, was procured in 2010. The system is more powerful than the existing custom built system and is capable of discharging static energy 10 μJ to 38 J. The resistance in the discharge circuit can be set at 3 levels (0, 50, or 510 Ω). The new spark tester provides greater flexibility for testing different explosives. A gas detector is connected to the new spark tester to monitor the gas emission (CO_2, CO, and NO_x) from the testing. When a spark reaction occurs, sudden increases in the concentrations of CO_2, CO, and NO_x are seen if molecules of test sample consist of organic carbon and organic nitrogen (e.g. TNT). The gas detector provides the operator a good tool to judge a spark reaction. The results are expressed as TIL and TIL+[12].

Figure 6. TA DSC and Setaram DSC at LLNL

Thermal (DSC) sensitivity. Thermal testing was performed by differential scanning calorimetry (DSC)[8]. The standard system at LLNL is the TA Instrument Q 2920. For volatile liquid samples, the Setaram Sensys DSC is used with a sealed sample holder (this minimizes enthalpy loss due to volatilization). Figure 6 shows these systems. For standard configuration, a small amount of sample, usually less than 1 mg, is placed in the sample holder and the sample is heated at a constant heating rate of 10°C/min. The heat flow is monitored in and out of the sample and presented as a function of temperature. The usual expression of results is temperature of maximum evolution (T_{max}) associated with an exothermic heat flow (ΔH_{exo}) and a temperature of minimum evolution (T_{min}) associated with an endothermic heat flow (ΔH_{endo}). An exothermic event is usually associated with an energetic release (indicating a possible energetic material) and an endothermic event is usually associated with a phase change (such as a melt).

3.2 Materials

The BN used in this study was purchased from Frontier Scientific, Catalog #B 1451, batch HM12-6298, purity > 99% (by NMR, GC, and appearance). A rough evaporation rate of the BN was determined by letting ~ 2.0 g of BN evaporate from a weighing vial with no cap and comparing this behavior to ~ 2.0 g of BN in the same type of vial with the cap not removed.

4 RESULTS

The results shown below come from the testing report Small-Scale Safety and Thermal Testing Report for Butyl Nitrate[13].

4.1 Volatilization of BN

Because BN is a volatile liquid, the evaporation rate was measured before testing to assure that there would be enough material remaining in the testing apparatus to derive high quality data. Figure 7 shows the weight loss of BN in a small weighing vial over time when exposed to the atmosphere.

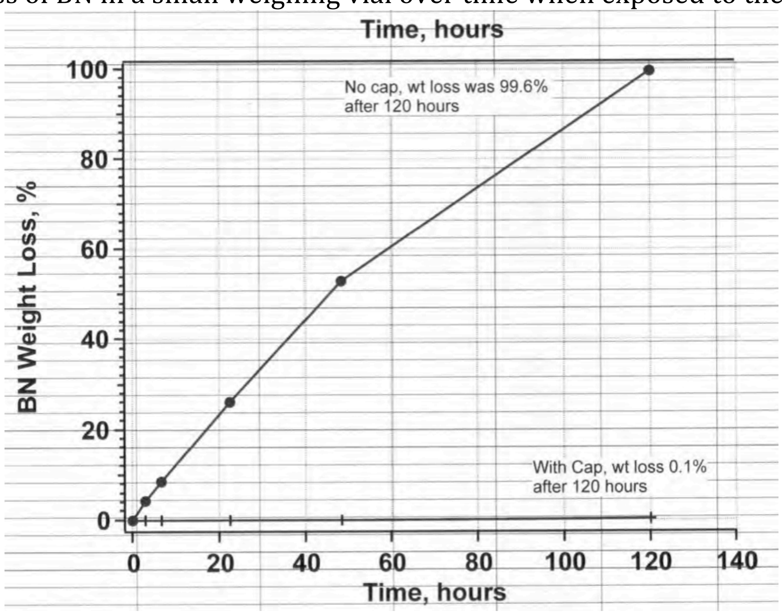

Figure 7. Weight change of BN in vial exposed to air for 120 hours

Over a period of 120 hours, 2.0460 g out of 2.0546 g (99.6%) were lost to evaporation. For the period of operation of the test (less than 8 hours), about 8% of the sample is lost to evaporation. Drop hammer samples are typically 35 mg in size, so there would be roughly 30 mg left on the sample anvil, which is a sufficient amount to complete the test. The caveat is that the test was conducted in a sample vial, not on a flat surface. However, the flat surface had a ring of grease as containment so the values determined above are probably representative of the volatility behavior of the BN.

4.2 Impact sensitivity results

Impact sensitivity is measured on a drop hammer apparatus[3]. This apparatus is simple in design—a weight is dropped onto the sample from a specific height to cause some form of reaction. This height is varied to find the transition between reaction and no reaction. A 50% probability of reaction point (DH_{50}) is derived using a semi-empirical statistical experimental and data reduction method[4,5].

The experimental configuration for liquids is the following: the sample, about 1 drop or 35 mg, is placed on a flat surface (metal anvil) and is contained by a ring of stop cock grease. The striker weigh (1 kg) is lowered to the ring of grease. The drop weight (2.5 kg) is then released from an operator-selected height and the event is monitored for visible and audible signs of a reaction. The experimental configuration for solids is similar: the sample is placed on sandpaper that is on the anvil. The striker weight (2.5 kg) is lowered onto the sample and secured in place. The drop weight (2.5 kg) is raised to a beginning height and then dropped. The operator monitors for reaction. If no reaction occurs, the drop weight is raised to a higher height. If a reaction occurs, the drop weight is raised to a lower height.

The BN was tested in the drop hammer apparatus for impact sensitivity using the bare anvil configurations for liquids. Table 1 shows the DH_{50} values for the BN and other standard materials, both liquid and solid.

Table 1. DH_{50}, in cm, results for BN and selected standards

Sample	Test Date	T, °F	RH, %[1]	DH_{50}, cm[2]	s, log unit[3]	Note
BN[4]	02/04/13	74	15	> 177	N/A	Liquid, Bare anvil[5]
BN[4]	02/01/13	74	15	> 177	N/A	Liquid, 120-grit SP[6,7]
BN[4]	4/08/13	75	18	160	0.022	Liquid, 120-grit SP[5,7]
BN[4]	4/08/13	75	18	167	0.002	Liquid, 180-grit SP[5,8]
FEFO[9,10]	01/25/13	75	30	29.7	0.035	Liquid, Bare anvil[5]
TMETN[10,11]	N/A	N/A	N/A	15	N/A	Liquid, Bare anvil[5]
PETN[12,13]	4/6/11	74	21	12.3	0.042	Solid, 120 grit SP[6,7]
HMX[14,15]	3/17/11	76	18	46	0.088	Solid, 120 grit SP[6,7]
RDX[16,17]	2/16/10	73	23	24	0.035	Solid, 120 grit SP[6,7]

1. Relative humidity; 2. DH_{50}, in cm, is by a modified Bruceton method, height for 50% probability of reaction; 3. Standard deviation; 4. BN = butyl nitrate; 5. 2.5 kg drop weight and 1 kg striker weight, 6. 2.5 kg drop weight and 2.5 kg striker weight, Bare anvil = nothing to hold sample on impact anvil; 7. 120-grit SP = 120-grit Si/C wet sandpaper to hold sample on impact anvil; 8. 180-grit SP = 180-grit garnet dry sandpaper to hold sample on anvil; 9. FEFO = (1,1-[methylenebis(oxy)]-bis-[2- fluoro-2,2-dinitroethane), liquid standard; 10. Data from HE reference Guide; 11. TMETN = (Trimethylolethane trinitrate, CH_3-$C(CH_2$-O-$NO_2)_3$), liquid standard; 12. PETN = pentaerythritol tetranitrate; 13. From reference 9; 14. Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine; 15. Data from reference 14; 16. 1,3,5-Trinitroperhydro-1,3,5-triazine; 17. Data from reference 10.

BN shows no sensitivity towards impact (177 cm is the maximum height of the drop weight). As a comparison value, RDX is considered a very stable explosive to impact, with a DH_{50} of 24 cm[10]. The BN DH_{50} value indicates a material that is much more stable.

The BN was also tested in the drop hammer apparatus for impact sensitivity using sandpaper to hold the sample, a non-standard configuration for liquids. Sandpaper is usually used to hold solids in place, but not liquids. Sandpaper and bare anvil surfaces are hardly similar and represent completely different hazard scenarios. This choice of this configuration was operational driven. The sandpaper (120-grit Si/C wet and 180-grit garnet dry) represents a rough surface that the BN may come in contact with during operations. Table 1 shows essentially no impact sensitivity when using sandpaper.

4.3 Friction sensitivity results

The BAM friction apparatus is used for this test. Friction sensitivity is measured by placing a sample on a surface and dragging a sharp pointed object through the material. The force on the pin is varied to stimulate reaction. The reaction is measured by 50% probability of reaction, as in the drop hammer case, by a modified Bruceton method[4,5] or by threshold initiation level (TIL)[12] which is the threshold weight before a positive reaction.

Experimentally, the BAM method uses ceramic plates (surface) and ceramic pins. The force on the pins is varied by a weight on the systems. The reaction is a pop, smoke or jetting and the sensitivity is reported in kg (based on value of the weight).

Table 2 shows the friction sensitivity for BN and selected reference compounds. The TIL and the F_{50} values are at the highest weight possible for the equipment, indicating the BN is simply not friction sensitive.

Table 2. BAM friction sensitivity results for BN and selected reference materials

Sample	Test Date	T, °F	RH, %[1]	TIL, kg[2]	TIL+, kg[3]	F_{50}, kg[4]	s, log unit[5]
BN[6]	2/14/13	75	16	0/10 @ 36.0	0/10 @ 36.0	> 36.0	NA[7]
PETN[8]	4/5/11	74	21	0/10 @ 6.4	1/10 @ 7.2	9.5	0.027
HMX[9]	3/17/11	74	18	0/10 @ 12.8	1/10 @ 14.4	20.3	0.042
RDX[10]	11/23/09	73	18	0/10 @ 19.2	1/10 @ 21.6	25.4	0.054

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. Next level where positive initiation is detected; 4. Modified Bruceton method, load for 50% probability of reaction (F_{50}), LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. BN = Butyl nitrate; 7. Not applicable, outside the experimental conditions; separate sample set used for Bruceton analysis; 8. PETN = pentaerythritol tetranitrate, data from reference 9; 9. Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine, data from reference 14; 10. 1,3,5-Trinitroperhydro-1,3,5-triazine, data from reference 10.

4.4 Static Discharge sensitivity

Sensitivity of the material to spark is measured by placing the material between two electrodes and discharging a spark through the material monitoring for a visible or audible indication of a reaction. Unlike the other methods, this testing requires more sophisticated electronics. Two different sets of equipment were used for the measurements in this report—ABL ESD and LLNL custom built. The difference between the two is that the LLNL custom system has a 510-Ω resistor permanently in the circuit, while the ABL ESD has the ability to vary this resistance.

Table 3. ESD sensitivity of BN and selected standard materials

Sample	Test Date	T, °C	RH, %[1]	TIL, J 0-Ω[2]	TIL+, J 0-Ω[3]	TIL, J 510-Ω[4]
BN[5]	2/3/13	23.9	20	0/10 @ 0.15	2/2 @ 0.25	0/10 @ 1.0
PETN[6]	4/18/11	23.9	29	0/10 @ 0.031	2/4 @ 0.063	0/10 @ 1.0
HMX[7]	4/16/11	23.3	29	0/10 @ 0.065	1/10 @ 0.075	0/10 @ 1.0
RDX[8]	4/26/11	23.9	16	0/10 @ 0.038	1/3 @ 0.063	0/10 @ 1.0

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level, no resistance in circuit, ABL ESD system; 3. Next level where positive initiation is detected, no resistance in the circuit, ABL ESD; 4. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level, 510-Ω in circuit, LLNL custom built ESD system; 5. BN = Butyl nitrate; 6. PETN = pentaerythritol tetranitrate, data from reference 9; 7. Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine, data from reference 13; 8. 1,3,5-Trinitroperhydro-1,3,5-triazine, data from reference 10.

Experimentally, a 5 to 40 mg the sample is put on a stage, and tape is placed on the sample to keep the discharge from scattering it. An energy differential is generated between the electrodes and a spark is discharged at a selected energy level. A positive reaction is noted as a pop, puff, smoke, noise or increase in gas concentration. Sensitivity is measured in joules as threshold of reaction (TIL)[12].

Table 3 shows the ESD sensitivity of BN and some selected standard materials. The measurements with 0-Ω values were measured with the ABL system and the 510-Ω values were measured with the LLNL custom built system. With the LLNL custom built system, the BN, as the other reference materials show no sensitivity to spark. With the ABL system, the BN shows some sensitivity, but this is at the high-energy limit of the equipment. The PETN standard is about 5 times more sensitive, indicating BN has very little spark sensitivity.

4.5 Thermal sensitivity

Differential scanning calorimetry (DSC) is used to determine the thermal sensitivity of the BN. The test procedure is straightforward. A very small sized sample (<1 mg) is placed in sample holder. The standard sample holder has a vented lid for non-volatile materials and a sealed sample holder for volatile materials. The sample is heated at a constant heating rate of 10°C/min. The starting temperature varies, from ambient, -40°C, or 40°C, depending upon the laboratory. Heat flow in and out of the sample is measured by the system. Heat flow into sample is indicative of endothermic while heat flow out of sample is exothermic and indicates energetic materials. The data output is heat flow as a function of temperature (or time if desired). Sensitivity measured in joules/g.

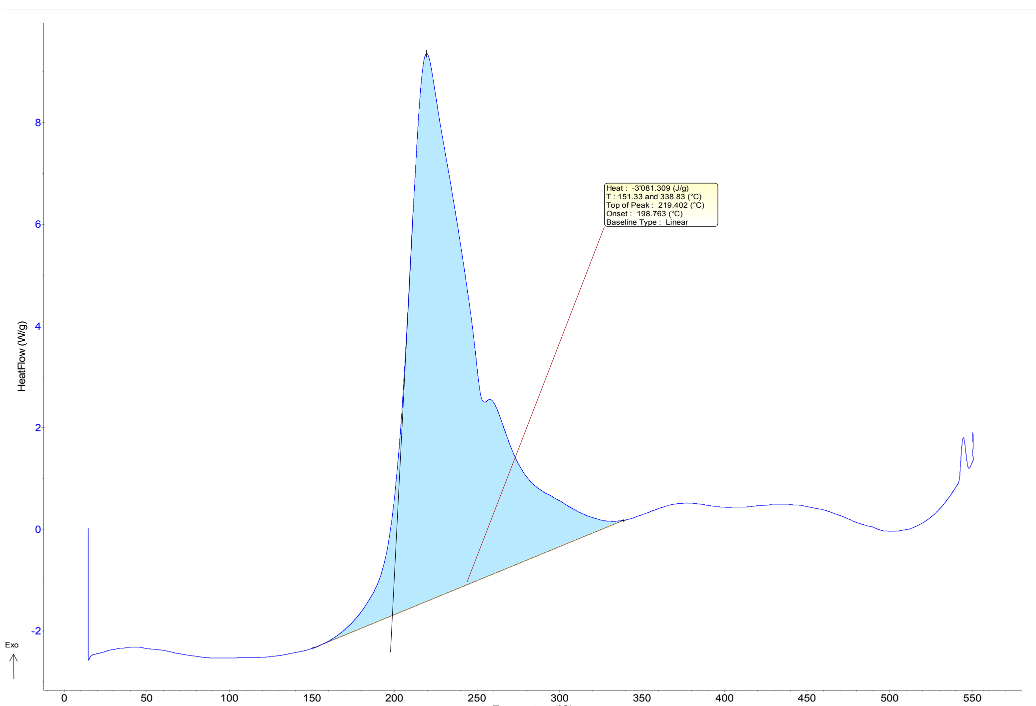

Figure 8. DSC profile of BN at 10°C/min heating rate in sealed sample holder

Figure 8 shows the DSC profile of BN taken on the Setaram Sensys system with a heating rate of 10°C/min in a sealed sample holder. The profile shows a large exothermic feature starting at ~150°C with a T_{max} of 219.4 °C. This exothermic feature overlaps with a very broad exothermic feature that spans at least the 150 to 550 °C scanning range. Because of this overlap, it is hard to assign an exact enthalpy value to the feature at 212.4 °C. The combined ΔH_{exo} output for the temperature range of 198.8 to 383.8 °C is 3081.3 J/g.

5 DISCUSSION

With the equipment used for impact and friction testing, BN showed no reactivity even at the high testing limits of the equipment. Even when using sandpaper in the drop hammer experiment, BN exhibited essentially no sensitivity.

With the ESD testing, the BN exhibited a slight sensitivity, but several times less than the standards.

DSC shows thermal sensitivity, with exothermic decomposition starting at ~ 150 °C. For reference, RDX[10] and PETN[9] show exothermic decomposition temperatures starting ~ 200 °C and 175 °C, respectively. Although not taken with the same sample holder, RDX has and exothermic enthalpy of ~ 3500 J/g, similar to the BN (RDX sample holder was SWISSI high pressure rated; BN sample holder was Setaram high pressure sealed sample holder).

6 CONCLUSIONS

BN was examined by Small-Scale Safety and Thermal testing and was found to be:

- Insensitive to impact, friction and spark insult,
- Thermally sensitive to decomposition leading to energy release starting around 150 °C.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

°C	degrees in centigrade
ABL	Allegany Ballistics Laboratory
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
BN	butyl nitrate
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
CO	carbon monoxide
CO_2	carbon dioxide
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DSC	Differential Scanning Calorimetry
ΔH	Enthalpy change
ΔH_{endo}	Enthalpy change for endothermic feature in DSC profile
ΔH_{exo}	Enthalpy change for exothermic feature in DSC profile
EMC	Energetic Materials Center at LLNL
ERL	
ESD	Electrostatic Discharge
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
FEFO	1,1-[methylenebis(oxy)]-bis-[2- fluoro-2,2-dinitroethane, liquid standard
GC	Gas chromatography
H	Chemical symbol for hydrogen
HE	high explosive
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
j	joules
kg	kilograms
kv	kilovolts
LLNL	Lawrence Livermore National Laboratory
mg	milligram
μj	microjoule
MSDS	Material Safety Data Sheet
N	Chemical symbol for nitrogen

NMR	Nuclear Magnetic Resonance Spectrometry
NO_x	Oxides of nitrogen, NO, NO_2, and N_2O
O	Chemical symbol for oxygen
Ω	ohms, unit for resistance
PETN	Pentaerythritol tetranitrate
pF	picofarad
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
s	Standard Deviation
Si/CV	silicon carbide
SSST	small-scale safety and thermal
SWISSI	Swiss Institute of Safety
TATB	2,4,6-triamino-1,3,5-trinitrobenzene
TIL	Threshold level—level before positive event
TIL+	One insult level above TIL
T_{max}	Maximum temperature of exothermic feature in DSC profile
TMETN	Trimethylolethane trinitrate, CH_3-C(CH_2-O-NO_2)$_3$, liquid standard
T_{min}	Minimum temperature of endothermic feature in DSC profile
TNT	Tri-nitrotoluene

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APPENDICES

A.1. MSDS from Frontier Science for butyl nitrate
A.2 Small-Scale Safety and thermal Testing Report for Butyl Nitrate, P. C. Hsu and J. G. Reynolds, Lawrence Livermore National Laboratory Report LLNL-TR-633652, April 8, 2013.

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