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SMALL-SCALE SAFETY TEST REPORT FOR BUTYL NITRATE

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Small-scale Safety Test Report for Butyl Nitrate

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LLNL-TR-

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This report summarizes results of small-scale safety testing (drop hammer, friction and spark) and DSC analysis performed on Butyl Nitrate (BN). Results showed that BN is not sensitive to impact, friction and ESD spark. Also listed in the tables below are FEFO, PETN, RDX and HMX for comparison. Impact sensitivity and friction sensitivity of BN are similar to those of TATB. BN showed some spark sensitivity but still is 5 times less-spark sensitivity than PETN when the resistance of the discharge circuit was set at zero Ohm. In summary, BN is less sensitive than some commonly used high explosives, PETN, HMX, and RDX. Weight loss measurement was made at ambient temperature when the sample bottle was kept open in order to determine whether each sample droplet would maintain its constant weight for testing. Result showed that BN is very volatile, losing 25% weight in a 24-hour period. Test sample would need to be in closed containers until use. DSC analysis was performed with Setaram Instrument and results showed that the peak temperature at 219.4 C but thermal decomposition started at around 151 C. Since BN has a low flash point (50 C), further thermal sensitivity testing with other methods (e.g. ODTX) is recommended to test for its threshold thermal ignition temperature, time to thermal explosion at elevated temperature and thermal explosion violence [1].

Impact Test Result Summary for BN

Sample #	Test Date	Temp., F	Relative Humidity RH, %	Height for 50% Reaction (DH ₅₀), cm	Standard Deviation (σ), log unit	Note
PCH-XIII-113	02/04/13	74	15	> 177	N/A	Bare anvil
FEFO (liquid)	01/25/13	75	30	29.7	0.035	Bare anvil
TMETN (liquid)				15		Bare anvil
PETN				15		120 grit sand paper
HMX				32		120 grit sand paper
RDX				35		120 grit sand paper

BAM Friction Test Result Summary for BN

Sample #	Test Date	Temp., F	Relative Humidity, RH, %	Friction Reaction (TIL) 0/10 @	Friction Reaction 1/10 @	Load for 50%* Reaction (F_{50}), kg	Standard Deviation (σ), log unit
PCH-XIII-113	02/04/13	75	16	36.0 kg	> 36.0 kg	> 36.0	NA
PETN				1/10 @ 6.4 kg	1/10 @ 6.4 kg		
HMX					1/10 @ 11.6 kg		
RDX					1/10 @ 12.4 kg		

Static Spark Test Result Summary for BN

Sample #	Test Date	Temp., F	Relative Humidity RH, %	Spark Reaction
PCH-XIII-113	02/03/13	73	13	0/10 @ 1.0 J @ 510 Ohms resistance; 2/2 @ 0.25 J at 0 Ohms 0/10 @ 0.15 J at 0 Ohms, TIL = 0.15 J
PETN				0/10 @ 1.0 J @ 510 Ohms; TIL = 0.031 J at 0 Ohms
RDX				0/10 @ 1.0 J @ 510 Ohms; TIL = 0.038 J at 0 Ohms
HMX				0/10 @ 1.0 J @ 510 Ohms; TIL = 0.063 J at 0 Ohms

DSC Test Result Summary for BN

Sample #	Test Date	DSC Onset/Peak Temperatures, °C, (ΔH , J/g)	Thermal decomposition process	Note
PCH-XIII-113	02/06/13	198.7/219.4 (16); exotherm, 3081 J/g	Started at 151.3 C and ended at 338.3 C	Setaram DSC

Before SSST testing started, we performed weight loss study for butyl nitrate. Without sample container cap on, BN lost weight very quickly, at the rate of 25% per day. Results are shown in appendix 2. All samples were thus kept in the container with cap on until use.

Appendix 1: SSST equipment description

Appendix 2: Weight loss study result

Appendix 3: SSST data sheets

Appendix 1: Small-scale Safety Test Capability at LLNL

Small-scale sensitivity testing was done to determine material response to various stimuli including impact, friction, static spark and thermal. NMR was also used to analyze for the presence of reaction products if any chemical reaction would occur during the aging process. The SSST tests, briefly described below, provide parameters for determining safe handling practices.

Drop Hammer (impact sensitivity)

Figure 1. Drop Hammer at LLNL

ERL Type 12 drop hammer equipment at LLNL, shown in Figure 1, was used to determine the impact sensitivity [1]. 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, a 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 may 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 [2,3]. PETN and RDX have impact sensitivities of 15 and 22 cm, respectively (powder samples), as measured at LLNL on this apparatus. TATB has impact sensitivity more than 177 cm, which is the maximum drop height on the LLNL instrument. 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. For reference, liquid explosives TMETN and FEFO have impact sensitivities of 14 and 32 cm, respectively.

Frictional Sensitivity

A BAM friction sensitivity test machine, as shown in Figure 2, was used to determine the frictional sensitivity [4]. The system uses a fixed porcelain pin and a movable porcelain plate that executes a reciprocating motion. A 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 frictional sensitivity of a material is based upon the smallest load (kg) at which reaction occurs for a 1-in-10 series of attempts. The lower the load values, the higher the frictional sensitivity. PETN has a frictional sensitivity of 6.4 kg.

Figure 3. Custom ESD spark

Spark Sensitivity

The static spark machine at LLNL is used to evaluate the electrostatic discharge hazards (from human electrostatic discharge (ESD)) associated with the handling of explosives [5]. 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 3. An adjustable resistor up to 510 ohms (chosen to simulate the electrical resistance of the 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, evolution of smoke, and color change of the sample. Voltage, capacitance, and resistance can be adjusted to achieve the desired static energy. The results obtained are expressed as a zero-in-10 or one-in-ten at a specific voltage and joules. One reaction in ten trials at ≤ 0.25 joules is considered spark-sensitive. Primary explosives show reaction at ≤ 0.1 joule.

New ABL spark test equipment, as shown in Figure 4, was procured in 2010. The system is more powerful than the existing custom built systems and is capable of discharging static energy up to 38 J and down to 10 mJ. Figure 4 shows the new spark tester. The resistance in the discharge circuit can be set at 3 levels (0 ohms, 50 ohms, or 510 ohms). 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.

Figure 4. New ABL Friction system at LLNL

Thermal (DSC) Sensitivity

Figure 5. TA DSC and Setaram DSC at LLNL

Thermal testing was performed by differential scanning calorimetry (DSC). 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, which minimizes endothermic features due to volatilization. Figure 5 shows these systems. For the 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 $10^\circ\text{C}/\text{min}$. 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).

REFERENCES

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3. Dixon, W.J. and Massey, F.J., "Introduction to Statistical Analysis," 2nd ed., McGraw-Hill, New York, pp 318-327, 1957.
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5. Simpson, L.R. and Foltz, M.F., "LLNL Small-Scale Friction Sensitivity (BAM) Test," UCRL-ID-124563, 1996.
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Appendix 2: Weight Loss Study

Weight loss study for butyl nitrate (BN)				PCH, Jan 23, 2013	PCH		
			updated	28-Jan-13			
Wt. of BN container with cap		19.56437	grams				
Wt. of BG container w/o cap		14.45914	grams				
			With Cap			W/O Cap	
Time	Time duration, hours	Total wt, container, BN, cap, g	BN, net, g	BN wt loss, %	Total wt, container, BN, g	BN, net, g	BN wt loss, %
1/23/2013, 10:15 AM	0	24.1410	4.5766	0.0000	16.5137	2.0546	0.0000
1/23/13 13:20	3	24.1407	4.5764	0.0061	16.4249	1.9658	4.3216
1/23/13, 4:53 pm	6.6	24.1410	4.5766	0.0002	16.3412	1.8821	8.3960
1/24/13, 8:57 am	22.5	24.1402	4.5759	0.0170	15.9798	1.5207	25.9861
1/25/13, 10:37 am	48.4	24.1400	4.5756	0.0229	15.4257	0.9666	52.9544
1/28/13, 10:23 am	120	24.1368	4.5724	0.0922	14.4678	0.0086	99.5804

Appendix 3: SSS T Data sheets for Butyl Nitrate

DROP HAMMER TEST REPORT

Operator: Gary Hust
 Reviewed and approved by: Peter Hsu *PCH*
 Date of Test: 2/4/13

Name of Explosive: Butyl Nitrate liquid
 Identification Number: PCH-XIII-113
 Composition:

Requester: P. Hsu
 Project No: 38086
 Task No: Task 1
 Sample Description: 35 microliter Liquid
 Test Type: 12A
 Temperature: 74 °F
 Humidity: 15%
 Book Reference: 25-36

Number of X's	0		
Number of O's	15		
+/- 0.5	0.5		
Least Height for X's or O's	177.4		
LOG of Least Height	2.2490		
LOG of Interval	0.05		
	NO GO	GO	
# of Events at Least Height	15	0	177.4

REMARKS: none

Dh₅₀ CALCULATION

Sum ($N_i * i$) 0
 Sum of Events for Calculation 15
 LOG of Dh₅₀

Dh₅₀ >177 cm

STANDARD DEVIATION CALCULATION

Sum ($N_i * i^2$) 0

STANDARD DEVIATION N/A

MAXIMUM Dh₅₀ N/A

MINIMUM Dh₅₀ N/A

FRICION B.A.M. TEST REPORT

Operator: Gary Hust

Test date: 2/4/13

Reviewed and approved by: Peter Hsu

Requester: P. Hsu

Project No: 38086

Book Reference: 3-126

Task No: Task 1

Material: Butyl Nitrate

Identification Number: PCH-XIII-113

Composition:

Temperature: 75 °F

Relative Humidity: 16%

Test Results: 0/10 @ 36.0 kg

Remarks:

ABL Spark Sensitivity Test Report

Operator: Gary Hust

Material: Butyl Nitrate

Reviewed and approved: Peter Hsu

ID/lot #: PCH-XIII-113

Requester: P. Hsu

Date: 2/4/13

Project #/Task # 38086 / Task 1

Resistance ohms 0

: 13

Temp (deg F): 73

ABL ESD Machine	Energy, J	Capacitance	Voltage	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	GO's
Go (X)				X	X																			2
No-Go (O)	0.25	0.02	5000																					2
Go (X)																								O
No-Go (O)	0.15	0.12	5000	O	O	O	O	O	O	O	O	O	O											10
Go (X)																								
No-Go (O)																								
Go (X)																								
No-Go (O)																								
Go (X)																								
No-Go (O)																								
Go (X)																								
No-Go (O)																								
Go (X)																								
No-Go (O)																								
Go (X)																								
No-Go (O)																								
Go (X)	510 Ohm R		10000																					O
No-Go (O)	1.0 J	0.07		O	O	O	O	O	O	O	O	O	O											10

X = reaction

Reference

materials TIL
 PETN 0.031 J
 RDX 0.038 J
 HMX 0.063 J

TIL = 0.15 J at 0 Ohms
 0/10 @ 1.0 J at 510 Ohms

O = no reaction

Gas detection mode (circle): CO, CO2, NOx
 for organic explosives

Observe the electric arc shape and size for reaction; Also CO2 (Gas Analyzer) typically increases by more than 50 ppm for most organic explosives when reaction occurs.

Use Tape ? No
 Yes for inorganic explosives
 No for organic explosives

Sensys 2/6/2013

Experiment : YS13-008_2/6/2013 10:41:13 AM

Mass : 4.9 (mg)

Crucible : Incoloy 2012

Procedure : 15-550 @ 10cpm

Zone name : 1 15-550 @ 10cpm

Butyl Nitrate #PCH-XIII-113

Hsu_Montgomery_5 ul

PCH

DROP HAMMER TEST REPORT

Operator: Gary Hust

Reviewed and approved by: Peter Hsu

PH

Date of Test: 1/25/13

Name of Explosive: FEFO

Identification Number: B-153

Composition: FEFO

Liquid standard

Requester: P. Hsu

Project No:

Task No:

Sample Description: 35 micro-liters

Test Type: Liquid

Temperature: 75 °F

Humidity: 30%

Book Reference: 25-34

Number of X's 10

Number of O's 5

+/- 0.5 0.5

Least Height for X's or O's 25

LOG of Least Height 1.3979

LOG of Interval 0.05

# of Events at Least Height	1	25.0
-----------------------------	---	------

# of Events at Next Height	3	28.1
----------------------------	---	------

# of Events at Next Height	1	31.5
----------------------------	---	------

# of Events at Next Height	0	35.3
----------------------------	---	------

# of Events at Next Height	0	39.6
----------------------------	---	------

# of Events at Next Height	0	44.5
----------------------------	---	------

Dh₅₀ CALCULATION

REMARKS:

Sum ($N_i * i$)	5
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Sum of Events for Calculation	5
-------------------------------	---

LOG of Dh ₅₀	1.4729
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Dh₅₀	29.7 cm
------------------------	----------------

STANDARD DEVIATION CALCULATION

Sum ($N_i * i^2$)	7
---------------------	---

STANDARD DEVIATION	0.035
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MAXIMUM Dh₅₀	2.5	32.2 cm
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MINIMUM Dh₅₀	-2.3	27.4 cm
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