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Integrated Data Analysis (IDCA) Program - PETN Class 4 Standard

M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, T. J. Shelley, J. A. Reyes, J. J. Phillips, P. C. Hsu, J. G. Reynolds

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Integrated Data Collection Analysis (IDCA) Program —PETN Class 4 Standard

Mary M. Sandstrom¹, Geoffrey W. Brown¹, Daniel N. Preston¹, Colin J. Pollard¹,
Kirstin F. Warner², Daniel N. Sorensen², Daniel L. Remmers²,
Timothy J. Shelley³, Jose A. Reyes⁴, Jason J. Phillips⁵, Peter C. Hsu⁶, and John G. Reynolds^{6*}

¹Los Alamos National Laboratory, Los Alamos, NM USA

²Indian Head Division, Naval Surface Warfare Center, Indian Head, MD USA

³Air Force Research Laboratory, Tyndall Air Force Base, FL USA

⁴Applied Research Associates, Tyndall Air Force Base, FL USA

⁵Sandia National Laboratories, Albuquerque, NM USA

⁶Lawrence Livermore National Laboratory, Livermore, CA USA

ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of PETN Class 4. The PETN was found to have: 1) an impact sensitivity (DH₅₀) range of 6 to 12 cm, 2) a BAM friction sensitivity (F₅₀) range 7 to 11 kg, TIL (0/10) of 3.7 to 7.2 kg, 3) a ABL friction sensitivity threshold of 5 or less psig at 8 fps, 4) an ABL ESD sensitivity threshold of 0.031 to 0.326 j/g, and 5) a thermal sensitivity of an endothermic feature with T_{min} = ~ 141 °C, and a exothermic feature with a T_{max} = ~205°C.

This effort, funded by the Department of Homeland Security (DHS), ultimately will put the issues of safe handling of these materials in perspective with standard military explosives. The study is adding SSST testing results for a broad suite of different HMEs to the literature. Ultimately the study has the potential to suggest new guidelines and methods and possibly establish the SSST testing accuracies needed to develop safe handling practices for HMEs. Each participating testing laboratory uses identical test materials and preparation methods wherever possible. Note, however, the test procedures differ among the laboratories. The results are compared among the laboratories and then compared to historical data from various sources. The testing performers involved for the PETN Class 4 are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), Air Force Research Laboratory (AFRL/RXQL), and Sandia National Laboratories (SNL). These tests are conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to understand how to compare results when these testing variables cannot be made consistent.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX, potassium perchlorate, potassium chlorate, sugar, dodecane, PETN.



1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory Small-Scale Safety and Thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives¹. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues when dealing with HMEs. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are centered on the physical forms and stability of the improvised materials.

Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the mixture precursors are combined shortly before use. The challenges to produce a standardized inter-laboratory sample are primarily associated with mixing and sampling. For solid-solid mixtures, the challenges primarily revolve around adequately mixing two powders on a small scale, producing a mixture of uniform composition—particle size and dryness often being a factor—as well as taking a representative sample. For liquid-liquid mixtures, the challenges revolve around miscibility of the oxidizer with the fuel causing the possibility of multiphase liquid systems. For liquid-solid mixtures, the challenges revolve around the ability of the solid phase to mix completely with the liquid phase, as well as minimizing the formation of intractable or ill-defined slurry-type products.

Table 1. Materials for IDCA Proficiency study

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	Powder mixture
Potassium perchlorate	Charcoal	Powder mixture
Potassium perchlorate	Dodecane ¹	Wet powder
Potassium chlorate	Dodecane ¹	Wet powder
Potassium chlorate as received	Sucrose (icing sugar mixture) ^{2,3}	Powder mixture
Potassium chlorate -100 mesh ³	Sucrose (icing sugar mixture) ^{2,3}	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture) ^{2,3}	Powder mixture
Ammonium nitrate		Powder
Bullseye® smokeless powder ⁴		Powder
Ammonium nitrate	Bullseye® smokeless powder ⁴	Powder mixture
Urea nitrate	Aluminum	Powder mixture
Urea nitrate	Aluminum, sulfur	Powder mixture
Hydrogen peroxide 70%	Cumin	Viscous paste
Hydrogen peroxide 90%	Nitromethane	Miscible liquid
Hydrogen peroxide 70%	Flour (chapatti)	Sticky paste
Hydrogen peroxide 70%	Glycerine	Miscible liquid
HMX Grade B		Powder
RDX Class 5 Type II		Powder (standard)
PETN Class 4		Powder (standard)

1. Simulates diesel fuel; 2. Contains 3 wt. % cornstarch; 3. Sieved to pass 100 mesh; 4. Alliant Bullseye® smokeless pistol gun-powder.

The IDCA has chosen several formulations to test that present these challenges. Table 1 shows the materials selected for the Proficiency Test and the Description column describes the form of the resulting mixture.

Evaluation of the results of SSST testing of unknown materials, such as the HMEs in Table 1, is generally done as a relative process, where an understood standard is tested alongside the HME. In many cases, the standard employed is PETN or RDX. The standard is obtained in a high purity, narrow particle size range, and measured frequently. The performance of the standard is well documented on the same equipment (at the testing laboratory), and is used as the benchmark. The sensitivity to external stimuli and reactivity of the HME (or any energetic material) are then evaluated relative to the standard.

Most of the results from SSST testing of HMEs are not analyzed any further than this. The results are then considered in-house. This approach has worked very well for military explosives and has been a validated method for developing safe handling practices. However, there has never been a validation of this method for HMEs. Although it is generally recognized that these SSST practices are acceptable for HME testing, it must always be kept in mind that HMEs have different compositional qualities and reactivities than conventional military explosives.

The IDCA is attempting to evaluate SSST testing methods as applied to HMEs. In addition, the IDCA is attempting to understand, at least in part, the laboratory-to-laboratory variation that is expected when examining the HMEs. The IDCA team has taken several steps to make this inter-laboratory data comparison easier to analyze. Each participating laboratory uses materials from the same batches and follows the same procedures for synthesis, formulation, and preparation. In addition, although the Proficiency test allows for laboratory-to-laboratory testing differences, efforts have been made to align the SSST testing equipment configurations and procedures to be as similar as possible, without significantly compromising the standard conditions under which each laboratory routinely conducts their testing.

The first and basic step in the Proficiency test is to have representative data on a standard material to allow for basic performance comparisons. Table 1 includes some standard military materials. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. These materials are being tested in triplicate and RDX will continue to be tested throughout the IDCA Proficiency test.

The subject of this report, PETN, is the second standard used in this Proficiency Test, with RDX being the primary standard. This material was selected as a standard because of the sensitivity towards impact, friction and spark testing as well as the common use as a sensitivity cut-off point between secondary explosive sensitivity and primary explosive sensitivity².

The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), Air Force Research Laboratory (AFRL/RXQL), and Sandia National Laboratories (SNL).

2 EXPERIMENTAL

General information. All samples were prepared according to the IDCA Program report on drying and mixing procedures^{3,4}. The PETN, C₅H₈N₄O₁₂, CAS # 78-11-5, was obtained from Holston Army Ammunition Plant, lot W923220. Analysis by HPLC shows 100% PETN; water by Karl Fisher Assay is 0.01%; nominal particle size (by Microtrac) of 95% < 348 μm⁵; and passes MILITARY specification (US Standard Sieve amount by weight passing 30 mesh, 100; 100 mesh, 20% maximum; 100 mesh, 5% minimum)⁶.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the PETN.

Table 2. Summary of conditions for the analysis of PETN mixture (All = LANL, LLNL, IHD, AFRL, SNL)

<p>Impact Testing</p> <ol style="list-style-type: none"> 1. Sample size—LLNL, IHD, AFRL, SNL 35 ± 3 mg; LANL 40 ± 2 mg 2. Preparation of samples—All, dried per IDCA drying methods³ 3. Sample form—All, loose powder 4. Powder sample configuration—All, conical pile 5. Apparatus—LANL, LLNL, IHD, Type 12; AFRL, SNL MBOM with Type 12 tooling* 6. Sandpaper—LANL, IHD, AFRL, SNL 180-grit garnet; LLNL 180-grit garnet, 120-grit Si/C 7. Sandpaper size—LLNL, IHD, AFRL, SNL 1 inch square; LANL, 1.25 inch diameter disk dimpled 8. Drop hammer weight—All, 2.5 kg 9. Striker weight—IHD, AFRL, LLNL, SNL 2.5 kg; LANL 0.8 kg. 10. Positive detection—LANL and LLNL, microphones with electronic interpretation as well as observation; IHD, AFRL, SNL observation 11. Data analysis—All, modified Brucceton and TIL before and above threshold; LANL and AFRL Neyer also <p>Friction analysis</p> <ol style="list-style-type: none"> 1. Sample size—All, ~5 mg, but not weighed 2. Preparation of samples—All, dried per IDCA procedures³ 3. Sample form—All, powder 4. Sample configuration—All, small circle form 5. Apparatus—LANL, LLNL, IHD, SNL BAM; IHD, AFRL ABL 6. Positive detection—All, by observation 7. Room Lights—LANL on; AFRL and LLNL off; SNL optional; IHD, BAM on, ABL off 	<ol style="list-style-type: none"> 8. Data analysis—LLNL and IHD, modified Brucceton (log-scale spacing) and TIL; LANL, modified Brucceton (linear spacing) and TIL; AFRL, SNL TIL <p>ESD</p> <ol style="list-style-type: none"> 1. Sample size—All ~5 mg, but not weighed 2. Preparation of samples—All, dried per IDCA drying methods³ 3. Sample form—All, powder 4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD and AFRL, none 5. Sample configuration—All, cover the bottom of sample holder 6. Apparatus—All, ABL 7. Positive detection—LANL, LLNL, IHD, AFRL observation; SNL IR gas (CO₂/CO) 8. Data analysis methods—All, TIL <p>Differential Scanning Calorimetry</p> <ol style="list-style-type: none"> 1. Sample size—LLNL, LANL, IHD, AFRL ~ <1 mg 2. Preparation of samples— LLNL, LANL, IHD, AFRL dried per IDCA procedures³ 3. Sample holder—LANL, IHD, and AFRL pin hole; LLNL pin hole and hermetically sealed 4. Scan rate— LLNL, LANL, IHD, AFRL 10°C/min 5. Range— LLNL, LANL, IHD, AFRL 40 to 400°C 6. Sample holder hole size—LANL, IHD, AFRL 75 μm; LLNL 50 μm 7. Instruments—LANL TA Instruments Q2000; LLNL TA Instruments 2920; IHD TA Instruments Q1000; AFRL—TA Instruments Q2000*
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Footnotes: *Test apparatus, *Impact*: LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark*: LLNL, LANL, IHD, AFRL—ABL Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry*: LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

The SSST testing data for the individual participants were obtained from the following reports: Small Scale Safety Test Report for PETN (LLNL)⁷, PETN Class 4 51088 Q (LANL)⁸, PETN Report (IHD)⁹, PETN—Integrated Data Collection Analysis (IDCA) Program, Small Scale Safety Testing (SSST) (AFRL)¹⁰, and Sandia National Laboratories Small-Scale Sensitivity Testing Report: PETN¹¹.

3 RESULTS

3.1 PETN Class 4

In this proficiency test, all testing participants are required to use materials from the same batch, and mixtures are to be prepared by the same methods. However, the actual testing procedures can be dif-

ferent. These differences are described in the IDCA report on method comparisons¹², which compares the different procedures by each testing category. LANL, LLNL, IHD, AFRL and SNL participated in this part of the SSST testing of the PETN Class 4.

3.2 Impact testing results for PETN Class 4

Table 3. Impact testing results for PETN Class 4

Lab ¹	Test Date	T, °C	RH, % ²	DH ₅₀ , cm ³	s, log unit ⁴	s, cm ⁴
LLNL (120)	4/6/11	23.3	21	12.3	0.042	1.19
LLNL (120)	4/14/11	23.9	15	9.4	0.049	1.06
LLNL (180)	4/14/11	23.9	15	8.9	0.022	0.45
LLNL (180)	4/15/11	23.9	18	7.6	0.035	0.61
LANL (180)	4/12/11	21.8	<10	7.9	0.036	0.66
LANL (180)	4/12/11	21.8	<10	7.7	0.061	1.09
LANL (180)	4/14/11	22.2	<10	8.7	0.051	1.02
IHD (180)	4/18/11	26	42	10	0.10	2.33
IHD (180)	4/19/11	25	44	9	0.06	1.25
IHD (180)	4/19/11	26	44	9	0.10	2.09
AFRL (180)	8/18/11	25	67	6.6	0.13	1.8
AFRL (180)	8/19/11	22.8	53	6.0	0.18	2.4
AFRL (180)	8/22/11	23.9	57	7.9	0.11	1.9
AFRL (180) ⁵	4/3/12	22.2	47	5.2	0.13	1.5
AFRL (180) ⁵	4/6/12	22.2	46	7.4	0.09	1.5
AFRL (180) ⁵	4/6/12	22.2	45	5.3	0.16	1.9
SNL (180)	6/29/12	20.8	32.4	14.4	0.015	0.51
SNL (180)	6/29/12	21.9	32.2	13.2	0.050	1.5
SNL (180)	7/3/12	22.4	45.1	10.3	0.055	1.3
SNL (180)	7/13/12	23.2	34.7	11.2	0.016	0.4
SNL (180)	7/13/12	23.2	31.0	13.3	0.010	0.3

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. Modified Bruceton method, load for 50% probability of reaction (DH₅₀); 4. Standard deviation; 5. PETN re-dried immediately before testing.

Table 3 shows the results of impact testing of the PETN Class 4 as performed by LANL, LLNL, IHD, AFRL and SNL. Differences in the testing procedures are shown in Table 2, and the notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive test. LANL, LLNL, IHD, AFRL and SNL used 180-grit garnet sandpaper, and LLNL also used 120-grit Si/C sandpaper. In addition, AFRL repeated the measurements 9 months later on the PETN that was originally dried for testing then re-dried immediately before the additional testing. All participants performed data analysis by normal modified Bruceton method^{13,14} and LANL and AFRL also performed data analysis by the Neyer method¹⁵.

For the testing results using 180-grit garnet sandpaper, the five participating laboratories show a range for DH₅₀ values from 5.2 to 14.4 cm. The average DH₅₀ values are LLNL, 8.3 ± 0.9; LANL, 8.0 ± 0.6; IHD, 9.3 ± 0.3, AFRL 6.4 ± 1.1 cm (AFRL 1st time 6.8 ± 1.0 cm; AFRL 2nd time 6.0 ± 1.2), and SNL 12.5 ± 1.7. The average DH₅₀ value for all the tests using 180-grit garnet sand paper is 8.9 ± 2.6 cm. The average DH₅₀ value for the tests using 120-grit Si/C sandpaper is 10.8 ± 2.1. The s values from the table for all the data are below 2.5 cm.

Table 4 shows the impact test results from LANL and AFRL using the Neyer or D-Optimal method¹⁵. The DH₅₀ values are about in the same range as the values analyzed by the Bruceton method for the 180-grit sandpaper data, where the average DH₅₀ for the Neyer method by LANL is 10.3 ± 0.4 cm. The standard deviation varies around 2 cm. The AFRL DH₅₀ average value is 5.4 ± 1.2 and is measurement is ~ 50% lower than the corresponding LANL measurement. The AFRL average is also lower than the range of the AFRL values derived by the Bruceton method in Table 3.

Table 4. Impact testing results for PETN (Neyer or D-Optimal Method) 180-grit sandpaper

Lab ¹	Test Date	T, °C	RH, % ²	DH ₅₀ , cm ³	s, log unit ⁴	s, cm ⁴
LANL (180)	4/12/11	21.8	<10	10.0	0.079	1.8
LANL (180)	4/12/11	21.8	<10	10.2	0.118	2.7
LANL (180)	4/14/11	22.2	<10	10.8	0.077	1.9
AFRL (180)	8/22/11	23.9	57	6.2	0.17	2.3
AFRL (180) ⁵	4/9/12	25.0	40	4.5	0.12	1.2

1. Number in parentheses indicates grit size of sandpaper; 2 Relative humidity; 3. Neyer method, load for 50% probability of reaction (DH₅₀); 4. Standard deviation; 5. AFRL re-dried PETN immediately before testing.

3.3 Friction testing results for PETN Class 4

Table 5. BAM Friction Testing results for PETN Class 4

Lab	Test Date	T, °C	RH, % ¹	TIL, kg ²	TIL, kg ³	F ₅₀ , kg ⁴	s, kg ⁵	s, log unit ⁵
LLNL	4/4/11	23.3	15	0/10 @ 7.2	1/10 @ 7.6	11.3	0.7	0.027
LLNL	4/5/11	23.3	21	0/10 @ 6.4	1/10 @ 7.2	9.5	0.6	0.027
LLNL	4/11/11	23.9	21	0/10 @ 5.6	1/10 @ 6.0	10.3	0.4	0.058
LANL	4/12/11	21.2	<10	0/10 @ 4.9	1/2 @ 7.3	NA ⁶	NA ⁶	NA ⁶
LANL	4/12/11	21.1	<10	0/10 @ 4.9	1/3 @ 7.3	NA ⁶	NA ⁶	NA ⁶
LANL	4/12/11	21.2	<10	0/10 @ 4.9	1/2 @ 7.3	NA ⁶	NA ⁶	NA ⁶
LANL	4/12/11	22.0	<10	NA ⁷	NA ⁷	9.8	2.40	0.109
LANL	4/12/11	21.6	<10	NA ⁷	NA ⁷	7.8	1.63	0.090
LANL	4/12/11	21.6	<10	NA ⁷	NA ⁷	8.0	2.85	0.161
IHD	5/25/11	23	43	0/10 @ 3.7	1/9 @ 4.3	NA ⁶	NA ⁶	NA ⁶
IHD	5/31/11	24	45	0/10 @ 4.3	1/3 @ 4.9	NA ⁶	NA ⁶	NA ⁶
IHD	5/31/11	24	45	0/10 @ 4.9	1/1 @ 5.5	NA ⁶	NA ⁶	NA ⁶
IHD	6/7/11	24	42	NA ⁷	NA ⁷	7.0	1.1	0.069
IHD	6/20/11	24	41	NA ⁷	NA ⁷	6.8	2.3	0.153
IHD	6/21/11	23	40	NA ⁷	NA ⁷	7.0	2.4	0.156
SNL	7/3/12	20.8	38.2	0/20 @ 3.6	1/11 @ 4.0	NA ⁸	NA ⁸	NA ⁸
SNL	7/3/12	21.8	37.8	0/20 @ 3.2	1/1 @ 3.6	NA ⁸	NA ⁸	NA ⁸
SNL	7/3/12	20.3	36.6	0/20 @ 3.2	1/7 @ 3.6	NA ⁸	NA ⁸	NA ⁸

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% Reaction (F₅₀), LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. Not applicable, separate sample set used for Bruceton analysis; 7. Not applicable, separate sample set used for TIL determination; 8. Not applicable, Bruceton analysis not performed.

Table 5 shows the BAM Friction testing performed by LANL, LLNL, IHD and SNL on the PETN. The difference in testing procedures by the four laboratories is shown in Table 2, and the notable differences are in the methods for positive detection. LANL, LLNL and IHD performed data analysis using a modified Bruceton method^{13,14} and all four by the threshold initiation level method (TIL)¹⁶. All participants show a reasonably sensitive material to friction. The average testing values for F₅₀ are: LLNL, 10.4 ± 0.9

kg; LANL, 8.5 ± 1.1 kg; and IHD, 6.9 ± 1.2 kg. The average testing values for TIL follow the same trend—LLNL 6.4 kg; LANL 4.9 kg; IHD 4.3 kg; SNL 3.3 kg.

Table 6. ABL Friction testing results for PETN Class 4

Lab	Test Date	T, °C	RH, % ¹	TIL, psig/fps ^{2,3}	TIL, psig/fps ^{2,4}	F ₅₀ , psig/fps ^{2,5}	s, psig ⁶	s, log unit ⁶
IHD	7/18/11	23	44	0/20 @ 5.5/8	1/3 @ 7.5/8	NA ⁷	NA ⁷	NA ⁷
IHD	7/18/11	24	45	0/20 @ 10/8	1/3 @ 15/8	NA ⁷	NA ⁷	NA ⁷
IHD	7/18/11	24	45	0/20 @ 7.5/8	1/7 @ 10/8	NA ⁷	NA ⁷	NA ⁷
IHD	8/11/11	23	40	NA ⁸	NA ⁸	44/8	42	0.37
IHD	8/11/11	23	40	NA ⁸	NA ⁸	45/8	13	0.12
IHD	8/11/11	24	40	NA ⁸	NA ⁸	38/8	16	0.18
AFRL	8/18/11	23.9	62	NA ⁹	20/21 @ 25/8	NA ⁷	NA ⁷	NA ⁷
AFRL	7/28/11	22.8	51	NA ⁹	22/22 @ 25/8	NA ⁷	NA ⁷	NA ⁷
AFRL	7/28/11	25.0	68	NA ⁹	21/22 @ 25/8	NA ⁷	NA ⁷	NA ⁷
AFRL ¹⁰	4/2/12	22.2	47	0/20 @ 13/3	1/2 @ 16/3	NA ⁷	NA ⁷	NA ⁷
AFRL ¹⁰	4/4/12	22.2	46	0/20 @ 10/3	1/3 @ 13/3	NA ⁷	NA ⁷	NA ⁷
AFRL ¹⁰	4/9/12	22.8	40	NA ⁹	2/10 @ 5/3	NA ⁷	NA ⁷	NA ⁷

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) 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; 4. Next level where positive initiation is detected; 5. Modified Bruceton method, load for 50% probability of reaction (F₅₀); 6. Standard deviation; 7. Not applicable, separate sample set used for Bruceton-type analysis; 8. Not applicable, separate sample set used for TIL analysis; 9 Could not determine TIL value, lower than the equipment could measure; 10. AFRL re-dried PETN immediately before determination.

Table 6 shows the ABL Friction testing performed by IHD and AFRL on the PETN. LANL did not have the system in routine performance at the time, and LLNL and SNL do not have ABL Friction. AFRL performed two sets of tests, one with the original material dried by IDCA procedures, and another set from the batch but re-dried and tested 9 months later. IHD performed data analysis using a modified Bruceton method^{13,14} and IHD and AFRL by the threshold initiation level method (TIL)¹⁶.

The data from IHD show the PETN has friction sensitivity. A TIL, average 7.6 psig at 8 fps, and one level above are established. In addition, the IHD F₅₀ values are average of 42 ± 4 psig at 8 fps. For the original set of AFRL data, tests was not performed below 25 psig at 8 fps, so there is no TIL reported. However, AFRL attempted to find the TIL in the second set of tests by lower the test velocity to 3 from 8 fps. The TIL was found to be around 10 psig/3 fps.

Table 7 shows Electrostatic Discharge (ESD) testing results of the PETN Class 4 performed by LLNL, LANL, IHD, AFRL and SNL. All used the TIL method for analysis¹⁶. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape and what covers the sample. LANL and LLNL show a very sensitive material at about the same level, where as AFRL shows the PETN to be slightly less sensitive. AFRL on the second determination (approximately 9 months after the first and with re-dried PETN) shows the PETN to be as sensitive as what LANL and LLNL determined. IHD shows the PETN to be an order of magnitude less sensitive than what the other participants found and SNL shows the PETN sensitivity to be somewhere between what AFRL and IHD found.

3.4 Electrostatic discharge testing of PETN Class 4

Table 7. Electrostatic discharge testing PETN Class 4

Lab	Test Date	T, °C	RH, % ¹	TIL, Joule ²	TIL, Joule ³
LLNL	4/15/11	23.9	29	0/10 @ 0.038	2/4 @ 0.063
LLNL	4/18/11	23.9	29	0/10 @ 0.031	2/4 @ 0.063
LLNL	4/19/11	23.9	29	0/10 @ 0.031	2/5 @ 0.038
LANL	4/12/11	22.3	<10	0/20 @ 0.025	2/5 @ 0.0625
LANL	4/14/11	21.6	<10	0/20 @ 0.025	1/14 @ 0.0625
LANL	4/14/11	21.6	<10	0/20 @ 0.025	1/11 @ 0.0625
IHD	4/20/11	26	54	0/20 @ 0.165	1/9 @ 0.326
IHD	4/25/11	26	57	0/20 @ 0.326	1/7 @ 0.853
IHD	4/28/11	21	48	0/20 @ 0.165	1/2 @ 0.326
AFRL	8/18/11	25	70	0/20 @ 0.088	2/16 @ 0.13
AFRL	8/19/11	25	66	0/20 @ 0.075	1/6 @ 0.088
AFRL	8/19/11	25	67	0/20 @ 0.065	1/2 @ 0.069
AFRL ⁴	4/3/12	22.2	50	0/20 @ 0.031	1/5 @ 0.038
AFRL ⁴	4/4/12	22.8	44	0/20 @ 0.069	1/6 @ 0.075
AFRL ⁴	4/5/12	23.9	42	0/20 @ 0.028	1/7 @ 0.031
SNL ⁵	7/10/12	23.6	37.9	0/20 @ 0.150	1/10 @ 0.25
SNL ⁵	7/10/12	24.3	38.0	0/20 @ 0.075	1/3 @ 0.150
SNL ⁵	7/11/12	22.0	53.7	0/20 @ 0.150	1/12 @ 0.25

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load (joules) 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. AFRL re-dried PETN immediately before determination; 5. Detection by gas analyzer.

3.5 Thermal testing (DSC) of PETN Class 4

Table 8. Differential Scanning Calorimetry results for PETN Class 4, 10°C/min heating rate

Lab	Test Date	Endothermic, onset/minimum, °C (ΔH, J/g)	Exothermic, onset/maximum, °C (ΔH, J/g)
LLNL ¹	3/24/11	139.9/140.7 (153)	186.6/204.5 (1347)
LLNL ¹	3/30/11	140.7/141.4 (155)	187.2/202.9 (997)
LLNL ¹	3/30/11	140.9/141.6 (155)	187.1/202.8 (1075)
LLNL ²	3/24/11	139.9/141.0 (154)	187.3/204.3 (1194)
LLNL ²	3/24/11	140.0/140.9 (155)	187.2/203.7 (1001)
LLNL ²	3/30/11	140.9/141.6 (157)	186.5/203.4 (932)
LANL ²	4/19/11	140.5/141.3 (155)	185.7/205.7 (1204)
LANL ²	4/19/11	140.4/141.0 (165)	184.4/207.1 (1251)
LANL ²	4/19/11	140.5/141.3 (148)	185.3/206.5 (1081)
IHD ²	4/8/11	141.0/141.7 (164)	185.1/207.2 (823)
IHD ²	4/8/11	141.0/141.5 (159)	185.2/205.9 (1202)
IHD ²	4/12/11	140.8/141.4 (159)	185.1/204.5 (1219)
AFRL ²	8/18/11	140.6/140.9 (170)	187.1/203.7 (941)
AFRL ²	8/18/11	140.5/141.0 (166)	186.3/204.9 (856)
AFRL ²	8/18/11	140.5/141.0 (181)	186.9/203.3 (779)
AFRL ^{2,3}	4/2/12	140.8/141.1 (157)	187.7/204.4 (1197)
AFRL ^{2,3}	4/3/12	140.6/141.1 (158)	185.4/204.4 (1201)
AFRL ^{2,3}	4/4/12	140.6/141.0 (157)	186.5/204.4 (1230)

1. Hermetically sealed sample holder; 2. Open pinhole sample holder; 3. AFRL re-dried PETN immediately before determination.

Table 9. Average Comparison values

	LLNL	LANL	IHD	AFRL ¹	SNL
Impact Testing ²	DH ₅₀ , cm	DH ₅₀ , cm	DH ₅₀ , cm	DH ₅₀ , cm	DH ₅₀ , cm
PETN Class 4 ³	8.3 ^{4,5}	8.0 ^{4,6}	9.3 ^{4,6}	6.8 ^{4,6}	12.5 ^{4,7}
RDX Class 5 Type II	24.3 ^{8,9}	25.4 ^{8,10}	19 ^{4,8}	15.3 ^{4,8}	23.3 ^{4,11}
BAM Friction Testing ^{12,13}	TIL, kg; F ₅₀ , kg	TIL, kg; F ₅₀ , kg	TIL, kg; F ₅₀ , kg	TIL, kg; F ₅₀ , kg	TIL, kg; F ₅₀ , kg
PETN Class 4 ¹⁴	6.4 ¹⁵ ; 10.4 ¹⁵	4.9 ¹⁵ ; 8.5 ¹⁵	4.3 ¹⁵ ; 6.9 ¹⁵	ND ¹⁶ ; ND ¹⁶	3.3 ¹⁵ ; ND ¹⁶
RDX Class 5 Type II	19.2 ⁸ ; 25.1 ⁸	19.2 ⁸ ; 20.8 ⁸	15.5 ⁸ ; ND ¹⁶	ND ¹⁶ ; ND ¹⁶	16.3 ¹¹ ; ND ¹⁶
ABL Friction Testing ¹⁷⁻¹⁹	TIL, psig; F ₅₀ , psig	TIL, psig; F ₅₀ , psig	TIL, psig; F ₅₀ , psig	TIL, psig; F ₅₀ , psig	TIL, psig; F ₅₀ , psig
PETN Class 4 ²⁰	ND ¹⁶ ; ND ¹⁶	ND ¹⁶ ; ND ¹⁶	7.7 ²¹ ; 42 ²¹	ND ¹⁶ ; ND ¹⁶	ND ¹⁶ ; ND ¹⁶
RDX Class 5 Type II	ND ¹⁶ ; ND ¹⁶	ND ¹⁶ ; ND ¹⁶	74 ⁸ ; 154 ⁸	93 ⁸ ; ND ¹⁶	ND ¹⁶ ; ND ¹⁶
Electrostatic Discharge ²²	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
PETN Class 4 ²³	0/10 @ 0.033 ²⁴	0/20 @ 0.025 ²⁴	0/20 @ 0.219 ²⁴	0/20 @ 0.076 ²⁴	0/20 @ 0.125 ²⁴
RDX Class 5 Type II	0/10 @ 1.0 ^{8,25}	0/20 @ 0.0250 ⁸	0/20 @ 0.095 ⁸	0/20 @ 0.044 ⁸	0/20 @ 0.15 ¹¹

1. AFRL values from measurements on the original sample only; values from measurements on re-dried sample not included; 2. DH₅₀, in cm, is by a modified Bruceton method, load for 50% probability of reaction; 3. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 15-18), LANL (21.8-22.2; < 10), IHD (25-26; 42-44), AFRL (22.8-25.0; 53-67), SNL (20.8-23.2; 31.0-45.1); 4. 180-grit sandpaper; 5. Average of two data points in Table 3; 6. Average of three measurements from Table 3; 7. Average of five measurements from Table 3; 8. From reference 19; 9. 120-grit sandpaper data only; 10. 150-grit sandpaper data only; 11. From reference 20; 12. 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; 13. F₅₀, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 14. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3-23.9; 15-21), LANL (21.1-22.0; < 10), IHD (23-24; 40-45), SNL (20.8-21.8; 36.6-38.2); 15. Average of three measurements from Table 5; 16. ND = Not determined; 17. LLNL, LANL and SNL did not perform measurements; 18. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) 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; 19. F₅₀, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 20. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (23-24; 40-45), AFRL (22.8-25.0; 51-68); 21. Average of three measurements from Table 6; 22. Threshold Initiation Level (TIL) is the load (joules) 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; 23. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 29), LANL (21.6-22.3; < 10), IHD (21-26; 48-57), AFRL (25.0; 66-70), SNL (22.0-24.3; 37.9-53.7); 24. Average of three measurements from Table 7; 25. LLNL has 510-Ω resistor in circuit.

Differential Scanning Calorimetry (DSC) was performed on the PETN by LLNL, LANL, IHD, and AFRL. All participating laboratories used different versions of the DSC by TA Instruments. Table 8 shows the DSC data is almost identical from each of the participating laboratories. For all participants there is observed a sharp, low temperature endothermic feature with T_{\min} value around 140°C. This is assigned to the melting of PETN from previous work on the thermal behavior of PETN by TGA, DTA, and DSC^{17,18}. Also observed by all participants is a broad exothermic feature with a T_{\max} range of 202.8 to 207.2°C which is assigned to a very complicated decomposition that starts after the PETN starts to melt¹⁷.

4 DISCUSSION

Table 9 shows the average values for the data from each participant and compares it to corresponding data for RDX Class 5. The data for RDX comes from the IDCA first iterative study of RDX as part of this Proficiency Test¹⁹. Table 9 allows the comparison of the average results on PETN with RDX to obtain relative sensitivities. Note for the impact testing, the RDX was tested using different grit size sandpaper in the LLNL and LANL cases. Also note that the average values for AFRL are only taken from their first round of testing.

4.1 Sensitivity of PETN Class 4 compared to RDX

Impact sensitivity. Table 9 shows that all the participants found the PETN much more sensitive than RDX. Note that the RDX data was not all taken with 180-grit garnet sandpaper for LLNL and LANL. At the time of the RDX determinations, both LLNL and LANL had not reconfigured their sandpaper for the drop hammer test.

Friction sensitivity. For BAM friction, LLNL, LANL, IHD and SNL performed this testing and found the PETN more sensitive than the RDX. For ABL friction, IHD found the PETN to be more sensitive than RDX. AFRL found that to be the case also at their standard testing conditions for ABL friction, but they did not test low enough to establish a TIL. However, subsequent examination of the PETN that had been re-dried immediately before testing, using lower appendage velocity, AFRL established a very low TIL (very sensitive material).

Spark sensitivity. For ESD testing, LANL and SNL found the PETN to be about the same sensitivity as RDX. AFRL and IHD found the PETN to be less sensitive than RDX. LLNL found the PETN to be much more sensitive than RDX, but the RDX data was taken on the custom built system that has a 510- Ω resistor in the circuit, while the PETN data was taken with an ABL system. Subsequent testing of RDX by LLNL using the ABL system shows a TIL for RDX to be 0/10 @ 0.038 J²¹. This will be reported elsewhere in a more detailed comparison.

Thermal sensitivity. Thermally, PETN melts at a lower temperature than RDX. An exothermic feature is seen in both the PETN and the RDX DSC profiles, with the PETN (due to decomposition) maximum at a lower temperature than RDX. This latter feature indicates the PETN is more thermally sensitive than the RDX.

4.2 Comparison of results based on participants

There are differences in methodologies and equipment configurations among the participating laboratories, so comparison of results for the same material is useful to highlight any differences in SSST testing methods. Using the average values shown in Table 9, although not statistically precise, at least allows for a qualitative comparison of any trends that may be seen among the participants.

For impact testing average results listed in Table 9, LLNL and LANL data are similar results while the IHD data is about 15% and the SNL data is about 50% higher indicating a more stable material and the AFRL data is about 15% lower indicating a less stable material. All these are 180-grit garnet sandpaper results.

For BAM Friction, LANL and IHD data indicate a material that is about the same sensitivity, while LLNL values indicate the PETN is more stable. This has been seen in previous measurements (KClO₃ (-100 mesh)/icing sugar²², KClO₃ (as received)/icing sugar)²³ where LLNL values indicate a material that is more stable than the corresponding values indicate from LANL and IHD. It is thought that the safety housing around the LLNL equipment inhibits detection of a positive reaction event because of sound shielding²⁴. SNL found the PETN to be the most sensitive of all the participants.

For ESD testing, the LANL and LLNL data show about the same sensitivity for the PETN, while both IHD, AFRL and SNL data show a more stable material. Experimental configuration accounts for LANL not finding a level above TIL. Some of these differences may be accounted for by the experiment configuration because of the various vintages of the ABL ESD equipment.

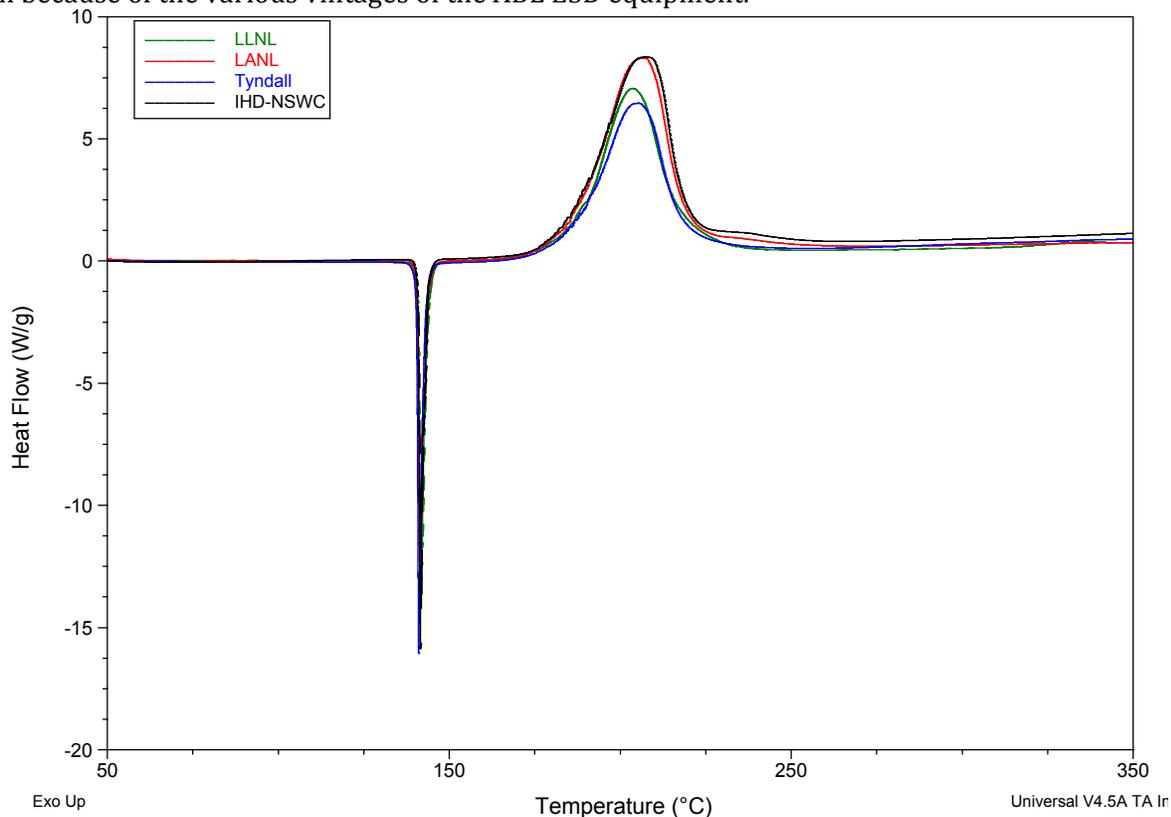


Figure 1. DSC profile at 10°C/min heating rate of PETN Class 4 from LLNL, LANL, IHD, and AFRL.

Figure 1 shows selected DSC profiles from each of the participants. These profiles virtually overlap with the same features—an endothermic feature followed by an exothermic feature. Table 10 shows average values, deviations and relative deviations (in parentheses) of the temperature minima and maxima along with the corresponding enthalpies of the features. The average values are similar among

the participants, although the enthalpy measured by AFRL on the original dried PETN for the exothermic feature is lower than the corresponding measurements from LLNL, LANL and IHD. The re-dried sample examined by AFRL exhibited essentially all the same properties as measured by LANL, LLNL, and IHD.

Table 10. Selected averages and deviations and (relative deviations) for DSC of PETN Class 4

Participant	T_{\min} of E_n , °C ¹	ΔH of E_n , J/g ²	T_{\max} of E_x , °C ³	ΔH of E_x , J/g ⁴
LLNL ⁵	141.2 ± 0.4 (0.3)	155 ± 2 (1)	203.8 ± 0.5 (0.2)	1042 ± 136 (13)
LLNL ⁶	141.2 ± 0.5 (0.3)	154 ± 1 (1)	203.4 ± 1.0 (0.5)	1140 ± 184 (16)
LANL ⁵	141.2 ± 0.2 (0.1)	156 ± 9 (6)	206.4 ± 0.7 (0.3)	1179 ± 88 (7)
IHD ⁵	141.5 ± 0.2 (0.1)	161 ± 3 (2)	205.9 ± 1.4 (0.7)	1081 ± 224 (21)
AFRL ⁵	140.8 ± 0.3 (0.2)	172 ± 8 (5)	204.0 ± 0.8 (0.4)	859 ± 81 (9)
AFRL ^{5,7}	141.6 ± 0.1 (0.0)	157 ± 1 (0)	204.4 ± 0.0 (0.0)	1209 ± (18)

1. E_n is the endothermic event in Table 8; 2. ΔH for E_n in Table 8; 3. E_x is the exothermic event in Table 8; 4. ΔH for E_x in Table 8; 5. Open pin-hole pan; 6. Hermetically sealed pan; 7. AFRL re-dried PETN immediately before determination.

4.3 Comparison with historical PETN data

Indian Head Historic Data. One of the best comparisons for data on Holston PETN comes from past data collected at IHD²⁵. Although the results are on few different batches produced by Holston and others, as well as some different classes, the results are useful to compare. The range of the DH_{50} impact values for the historical Type 12A data is 10-14 cm. The IHD impact data in Table 3 from this report overlap on the low end of this range. Only the 0/10 TIL values are presented in the historical data for BAM friction and the range is 3.7 to 5.5 kg, which overlap with the BAM friction data shown in Table 5 of this report. For ABL friction, the range with historical data at the 8 ft/sec rate is 0/20 @ 55 or less psig (but more often < 30 psig), in agreement with the data in Table 6. For the ESD, the historical data varies from 0/20 @ 0.037 to 0.326 J. The ESD data shown in Table 7 of this report falls in that range, 0/20 @ 0.095 J.

An interesting subset of the IHD historical data is a short study on the effects of humidity on the sensitivity of PETN. Results showed a slight decrease in sensitivity of all tested properties with increase of relative humidity: relative humidity, % (15, 58, 84); DH_{50} , cm (11, 11, 14); BAM Friction 0/10 TIL, kg (3.7, 3.7, 5.5); ESD 0/20 TIL, joules (0.165, 0.165, 0.326).

LLNL Historic Data. Recent LLNL testing²⁶ of PETN has been focused on impact (pressed into pellet form) and ESD. However a few examples have been studied for impact testing both in the pressed and powder form. The pressed PETN DH_{50} values average 16.4 ± 2.7 cm for 50 determinations and the powder PETN DH_{50} values average 12.0 ± 1.7 cm for 6 determinations. Generally, the pressed form is less sensitive than the powder form, which has also been observed for RDX by the IDCA¹⁹. Much less BAM friction data is available which ranges from 6.4 to 13.4 kg for 1/10 or more positives (one level above TIL). The PETN did not exhibit any ESD sensitivity. However, the historical ESD results were taken with the custom made ESD with the 510- Ω resistor in-line to mimic the human body.

LANL Historic Data. LANL has been using the same batch of PETN as a standard for many decades²⁷. Impact data since 2000 shows an average DH_{50} of 13.1 ± 1.9 cm with high and low values of 17.3 and 6.6 cm, respectively. The BAM friction data over a similar period shows an average F_{50} of 7.2 ± 1.6 kg with high and low values of 12.4 and 4.8 kg, respectively.

5 CONCLUSIONS

PETN was found through SSST testing to be a sensitive material toward impact, friction, and spark handling conditions—generally more sensitive than RDX. Standard thermal testing by DSC shows a more thermally sensitive material than RDX.

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ABBREVIATIONS, ACRONYMS AND INITIALISMS

ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
CAS	Chemical Abstract Service chemical registry number
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
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
E _n	Endothermic
ESD	Electrostatic Discharge
E _x	Exothermic
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
fps	feet per second
ΔH	Enthalpy of reaction
H ₂ O	Water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
HPLC	High pressure or high performance liquid chromatography
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
IR	Infrared Spectroscopy
j	joules
KClO ₃	Potassium Chlorate
KClO ₄	Potassium Perchlorate
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
Microtrac	Brand of particle size determination equipment that uses laser light scattering
ND	No data
NSWC	Naval Surface Warfare Center
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL

s	Standard deviation
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event
TR	LLNL designation for technical report used for document release

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