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Integrated Data Collection Analysis (IDCA) Program - RDX Standard Data Set 2

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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 the RDX Type II Class 5 standard, from testing the second time in the Proficiency Test. This RDX testing (Set 2) compared to the first (Set 1) was found to have about the same impact sensitivity, have more BAM friction sensitivity, less ABL friction sensitivity, similar ESD sensitivity, and same DSC sensitivity.

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 are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), Sandia National Laboratories (SNL), and Air Force Research Laboratory (AFRL/RXQL). These tests are conducted as a proficiency study in order to establish some consistency in test protocols, procedures, and experiments and to compare results when these testing variables cannot be made consistent.

Keywords: Small-scale safety testing, proficiency test, impact-, friction-, spark discharge-, thermal testing, round-robin test, safety testing protocols, HME, RDX, potassium perchlorate, potassium chlorate, sodium chlorate, sugar, dodecane, PETN, carbon.

IDCA Explosives Safety Testing of Homemade Explosives
Integrated Data Collection Analysis Program

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 explosives[1]. 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 gunpowder.

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, RDX Class 5, is the second examination of the standard during the Proficiency Test. The Standard has been scheduled to be tested a minimum of one time and a maximum of four times throughout the Proficiency Test. LLNL, LANL, IHD, and AFRL all have examine the RDX previously[2]. This is the first time for SNL.

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), and Sandia National Laboratories (SNL).

2 EXPERIMENTAL

General information. All samples were prepared according to IDCA methods on drying and mixing procedures[3,4]. Briefly, the sample was dried in an oven at 60°C for 16 h, then cooled and stored in a desiccator until use. The RDX used in this effort is Class 5 Type II RDX and was obtained from the Holston Army Ammunition Plant batch # HOL89D675-081 and provided to the participating laboratories test by IHD[5]. High Performance Liquid Chromatography analysis gave 90% RDX and 10% HMX; Laser Diffraction (Light Scattering method using Microtracs Model FRA9200) gave a particle size distribution of 7.8 to 104.7 micron with a maximum at 31.1 microns[6,7].

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

Impact Testing

1. Sample size—LLNL, IHD, SNL 35 ± 2 mg; LANL 40 ± 2 mg
2. Preparation of samples—All, dried per IDCA drying methods[3]
3. Sample form—All, loose powder; LLNL, pressed
4. Powder sample configuration—All, conical pile; LLNL pellet also
5. Apparatus—LANL, LLNL, IHD, Type 12; SNL, MBOM with Type 12 tooling*
6. Sandpaper—All (180-grit garnet); LLNL (120-grit Si/Carbide)
7. Sandpaper size—LLNL, IHD, SNL, 1 inch square; LANL, 1.25 inch diameter disk dimpled;
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL, IHD, SNL, 2.5 kg; LANL 0.8 kg
10. Positive detection—LANL, LLNL, microphones with electronic interpretation as well as observation; IHD, SNL, observation
11. Data analysis—All, modified Bruceton and TIL before and above threshold; LANL Neyer also

Friction analysis

1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA procedures[3]
3. Sample form—All, powder
4. Sample configuration—All, small circle form
5. Apparatus—LANL, LLNL, IHD, SNL, BAM; IHD, ABL
6. Positive detection—All, by observation
7. Room Lights—LANL and SNL on; and LLNL off; IHD, BAM on, ABL off
8. Data analysis—LLNL modified Bruceton (log-scale spacing) and TIL; LANL, modified Bruceton (linear spacing) and TIL; IHD Neyer and TIL; SNL, TIL

ESD

1. Sample size—All ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA drying methods[3]
3. Sample form—All, powder
4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD, none
5. Sample configuration—All, cover the bottom of sample holder
6. Apparatus—LANL, IHD, ABL; LLNL, custom built*
7. Positive detection—LANL, LLNL, IHD, observation; SNL IR gas (CO_2/CO)
8. Data analysis methods—All, TIL

Differential Scanning Calorimetry

1. Sample size—LLNL, LANL, IHD, ~ <1 mg
2. Preparation of samples—LLNL, LANL, IHD, dried per IDCA procedures[3]
3. Sample holder—LANL, IHD, pin hole; LLNL, pin hole and hermetically sealed
4. Scan rate—LLNL, LANL, IHD, 10°C/min
5. Range—LLNL, LANL, IHD, 40 to 400°C
6. Sample holder hole size—LANL, IHD, 75 μm; LLNL 50 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920 and Setaram Sensys; IHD, TA Instruments Q1000*

Footnotes: *Test apparatus, *Impact:* LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL, SNL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD, SNL—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark:* LLNL, LANL, IHD, AFRL, SNL—ABL Electrostatic Discharge Apparatus, LLNL—custom-built 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.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the RDX Type II Class 5. The SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for RDX (second calibration) (LLNL)[8], 50188 I RDX Second Run (LANL)[9], RDX Report Run #2 (IHD)[10], and SNL Small-Scale Sensitivity Testing Report: RDX (SNL)[11].

3 RESULTS

3.1 RDX Type II Class 5

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 different. These differences are described in the IDCA report on method comparisons[12], which compares the different procedures by each testing category. LANL, LLNL, IHD, and SNL participated in this part of the SSST testing of the RDX.

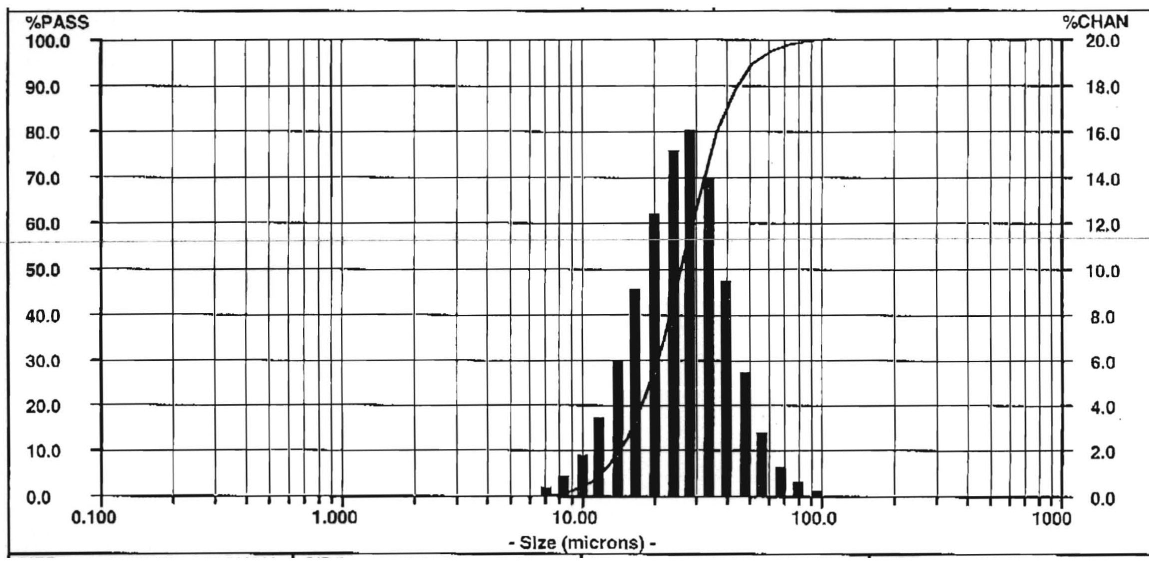

Figure 1. Microtracs laser light scattering particle size distribution for RDX.

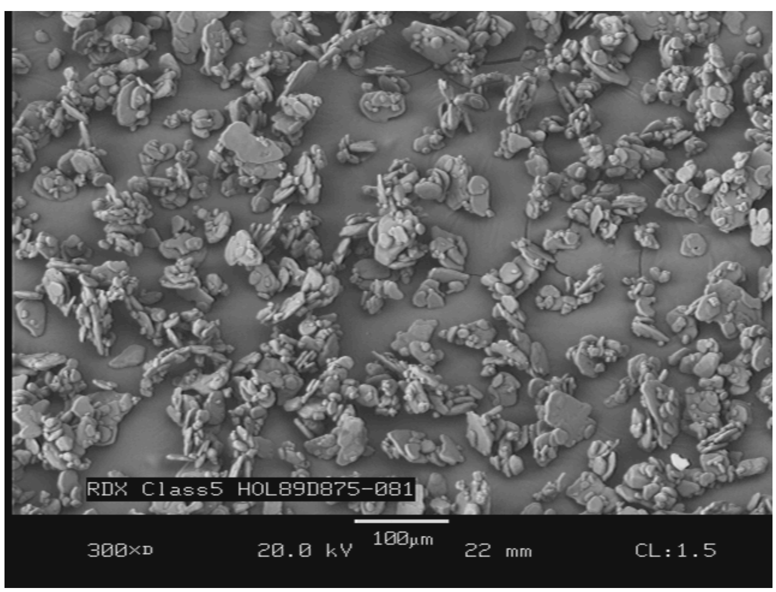

Figure 2. Scanning Electron Micrograph of RDX at 300 X magnification

RDX in this study is Type II was provided and distributed by IHD from inventory. RDX Type II is from the acetic anhydride (Bachman) process and generally contains ~ 10-wt % HMX as a by-product[13]. The HMX content has been verified by HPLC analysis[6]. The Military Specification for RDX Type II Class 5 is that a minimum of 97-wt % of the materials passes through a 325-mesh (44 µm[14]) sieve fraction[15]. Figure 1 shows the particle size distribution. Clearly, some particles are determined to be larger by the Microtracs system than should be passed through the 325-mesh sieve. However, Figure 2 shows a Scanning Electron Micrograph (SEM) of the RDX and clearly indicates particles with aspect ratios of around 0.4, which would pass through the 325-mesh sieve.

3.2 Impact testing results for RDX Type II Class 5 (Set 2)

Table 3 shows the results of impact testing of the RDX Type II Class 5 (Set 2) as performed by LANL, LLNL, IHD, 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. All participants performed data analysis by normal modified Bruceton method[16,17]. All participants found the RDX to be sensitive to impact testing. Most testing was performed using 180-grit sandpaper to hold the sample. Examining all the values in Table 3 generated with 180-grit sandpaper shows wide variation in the DH_{50} values with the average of 20.8 ± 2.8 cm. For the individual performers, the DH_{50} values are, in cm: LLNL 21.8 ± 1.6 (two measurements); LANL 20.8 ± 1.1; IHD 17.7 ± 3.1; SNL 23.3 ± 1.6. LLNL also performed the testing using 120-grit sandpaper on a sample of RDX that was pressed in a pellet. The DH_{50} value is 34.0 cm.

Table 3. Impact testing results for RDX Type II Class 5 (Set 2)

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120-P)[5]	9/8/10	23.9	32	34.0	4.63	0.059
LLNL (180)	9/9/10	23.9	30	22.9	2.22	0.042
LLNL (180)	9/13/10	22.8	23	20.7	4.56	0.095
LANL (180)	12/06/10	22.3	< 16	22.0	1.52	0.030
LANL (180)	12/09/10	21.7	< 16	20.3	2.30	0.049
LANL (180)	12/10/10	21.7	< 16	20.0	2.26	0.049
IHD (180)	3/8/11	28	40	17	4.76	0.12
IHD (180)	3/9/11	24	43	21	1.94	0.04
IHD (180)	3/8/11	29	43	15	3.13	0.09
SNL (180)	5/8/12	21.7	29.9	22.2	0.8	0.016
SNL (180)	5/10/12	20.0	28.2	22.6	1.5	0.023
SNL (180)	5/15/12	22.5	33.6	25.1	1.2	0.021

1. Value in parenthesis is grit size of sandpaper (180 is 180 garnet dry and 120 is 120 Si/Carbide wet/dry); 2 relative humidity; 3. DH_{50}, in cm, by modified Bruceton method, height for 50% probability of reaction; 4. Standard deviation; p = pressed into pellet

Table 4. Impact testing results for RDX Type II Class 5 (Set 2) (Neyer or D-Optimal Method) 180-grit sandpaper

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)	12/06/10	21.8	< 10	23.2	2.5	0.047
LANL (180)	12/09/10	21.8	< 10	21.2	2.3	0.047
LANL (180)	12/10/10	21.7	< 10	20.1	1.3	0.028

1. Value in parenthesis is grit size of sandpaper (180 is 180 garnet dry); 2 relative humidity; 3. DH_{50}, in cm, Neyer method, height for 50% probability of reaction; 4. Standard deviation.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method[18]. The LANL average value for DH_{50} is 21.5 ± 1.6 cm, similar to the average value for DH_{50} determined by the Bruceton method.

3.3 Friction testing results for RDX Type II Class 5 (Set 2)

Table 5 shows the BAM Friction testing performed by LANL, LLNL, SNL and IHD. 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. All participants performed data analysis using the threshold initiation level method (TIL)[19]. LANL and LLNL also used a modified Bruceton method[16,17] and IHD used the Neyer method[18] because their data did not meet Bruceton criteria (analysis performed by LANL). Table 5 shows that data on the sensitivity of the mixture varies depending upon on which participant. The average values and sensitivity ordering for F_{50}, in kg are: IHD 27.8 ± 3.4 > LLNL 24.8 ± 1.5 > LANL 16.3 ± 1.1. The TIL values follow a different trend. The order and average TIL values, in kg, are: LLNL 16.5 > SNL 16.3 > IHD 11.8 > LANL 10.4.

Table 5. BAM Friction Testing results for RDX Type II Class 5 (Set 2)

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	9/08/10	23.9	26	0/10 @ 16.0	1/10 @ 16.8	23.1	1.86	0.035
LLNL	9/09/10	23.9	31	0/10 @ 16.8	1/10 @ 18.0	25.4	3.17	0.054
LLNL	9/09/10	23.9	31	0/10 @ 16.8	1/10 @ 19.2	26.0	3.00	0.050
LANL	12/06/10	22.1	< 10	0/10 @ 9.6	1/8 @ 12.0	NA[7]	NA[7]	NA[7]
LANL	12/08/10	21.1	< 10	0/10 @ 12.0	1/3 @ 14.4	NA[7]	NA[7]	NA[7]
LANL	12/08/10	22.1	< 10	0/10 @ 9.6	1/5 @ 12.0	NA[7]	NA[7]	NA[7]
LANL	12/0610	22.2	< 10	NA[6]	NA[6]	15.1	3.6	0.106
LANL	12/08/10	20.8	< 10	NA[6]	NA[6]	16.7	2.3	0.060
LANL	12/08/10	20.8	< 10	NA[6]	NA[6]	17.1	1.8	0.046
IHD	3/31/11	23	40	0/10 @ 11.0	1/4 @ 12.2	NA[7]	NA[7]	NA[7]
IHD	2/23/11	26	40	0/10 @ 12.2	1/5 @ 14.7	NA[7]	NA[7]	NA[7]
IHD	4/22/11	22	40	0/10 @ 12.2	1/5 @ 14.7	NA[7]	NA[7]	NA[7]
IHD[8]	4/11/11	NA[10]	NA[10]	NA[6]	NA[6]	31.6	7.0	0.098
IHD[8]	4/11/11	NA[10]	NA[10]	NA[6]	NA[6]	24.9	12.0	0.228
IHD[8]	4/11/11	NA[10]	NA[10]	NA[6]	NA[6]	26.9	23.7	0.600
SNL	5/8/12	22.2	31.0	0/20 @ 16.8	1/14 @ 18.0	NA[9]	NA[9]	NA[9]
SNL	5/9/12	22.2	28.1	0/20 @ 16.0	1/2 @ 16.8	NA[9]	NA[9]	NA[9]
SNL	5/10/12	20.4	31.3	0/20 @ 16.0	1/7 @ 16.8	NA[9]	NA[9]	NA[9]

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. F_{50}, in kg, is by a modified Bruceton method, weight for 50% probability of reaction; 5. Standard deviation; 6. Not applicable, separate measurement performed for TIL; 7. Not applicable, separate measurements performed for modified Bruceton analysis. 8. Modified Neyer analysis; 9. Bruceton analysis not performed; 10. Not measured. LLNL uses log-spacing and LANL uses liner spacing for the Bruceton up and down method experimentation and data analysis.

Table 6 shows the ABL Friction testing performed by IHD on RDX Type II Class 5. LANL did not have the system in routine performance at the time. LLNL and SNL do not have ABL Friction. IHD performed data analysis using the threshold initiation level method (TIL)[19] and a modified Bruceton method[16,17]. The data from IHD show that the mixture has some friction sensitivity. A TIL and one level above are established. In addition, IHD calculated F_{50} values from their data. For the ABL data, IHD was able to establish a modified Bruceton F_{50}, unlike for the BAM friction testing.

Table 6. ABL Friction testing results for RDX Type II Class 5 (Set 2)

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[4]	F_{50}, psig/fps[5]	s, psig/fps[6]	s, log unit[6]
IHD	3/31/11	23	40	0/20 @ 75/8	1/1 @ 100/8	NA[7]	NA[7]	NA[7]
IHD	3/16/11	25	44	0/20 @ 100/8	1/9 @ 135/8	NA[7]	NA[7]	NA[7]
IHD	3/31/11	23	40	0/20 @ 100/8	1/6 @ 135/8	NA[7]	NA[7]	NA[7]
IHD	3/31/11	23	40	NA[8]	NA[8]	224/8	57.3/8	0.11
IHD	3/17/11	25	42	NA[8]	NA[8]	196/8	59.4/8	0.13
IHD	3/31/11	23	41	NA[8]	NA[8]	200/8	60.8/8	0.13

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. F_{50}, in psig/fps, is by a modified Bruceton method, force for 50% probability of reaction; 6. Standard deviation; 7. Not applicable, separate measurements done for modified Bruceton analysis; 8. Not applicable, separate measurements performed for TIL analysis.

3.4 Electrostatic discharge testing of RDX Type II Class 5 (Set 2)

Electrostatic Discharge (ESD) testing of the RDX Type II Class 5 was performed by LLNL, LANL, IHD and SNL. Table 7 shows the results. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape and what covers the sample. In addition, LLNL uses a custom built ESD system with a 510-Ω resistor in line to simulate a human body, making a direct comparison of the data from LLNL with data generated by the other participants challenging. SNL did detection of a positive event using two methods, a custom-built high-speed camera system[20] and CO and CO_2 gas analyzer[21]. All participants performed data analysis using the threshold initiation level method (TIL)[19].

Table 7. Electrostatic discharge testing RDX Type II Class 5

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	9/08/10	23.9	26	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	9/08/10	23.9	32	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	9/10/10	23.9	29	0/10 @ 1.0	0/10 @ 1.0
LANL	12/06/10	22.2	< 10	0/20 @ 0.025	1/17 @ 0.0625
LANL	12/08/10	21.0	< 10	0/20 @ 0.0625	1/1 @ 0.125
LANL	12/08/10	20.9	< 10	0/20 @ 0.025	1/13 @ 0.0625
IHD	3/10/11	24	42	0/20 @ 0.037	1/4 @ 0.095
IHD	3/10/11	24	42	0/20 @ 0.037	1/3 @ 0.095
IHD	3/16/11	24	42	0/20 @ 0.0.37	1/16 @ 0.095
SNL[5]	5/14/12	20.0	31.3	0/20 @ 0.15	1/8 @ 0.25
SNL[6]	5/17/12	21.0	24.4	0/20 @ 0.15	1/2 @ 0.25
SNL[6]	5/17/12	20.6	20.5	0/20 @ 0.15	1/3 @ 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. LLNL used a custom built ESD with a 510-ohm resistor in the discharge unit to mimic the human body. 5. Camera system used for go/no-go determination; 6. Infrared Gas Analyzer monitoring CO and CO_2 for go/no-go determination.

For TIL, SNL found the material to be the most stable, while LANL and IHD found about the same sensitivity. The LLNL values using the custom built system show a material with no sensitivity.

3.5 Thermal testing (DSC) of RDX Type II Class 5 (Set 2)

Differential Scanning Calorimetry (DSC) was performed on the RDX Type II Class 5 by LLNL, LANL, and IHD. SNL does not have thermal testing capabilities. All participating laboratories used different versions of the DSC by TA Instruments. Results were obtained at a 10°C/min heating rate.

Table 8. Differential Scanning Calorimetry results for RDX Type II Class 5, 10°C/min heating rate

Lab	Test Date	Endothermic, onset/minimum, °C (ΔH, J/g)	Exothermic, onset/maximum, °C (ΔH, J/g)
LLNL[1]	8/27/10	187.3/188.3, 199+[4] (126)	213.1[3]/240.1 (2432)
LLNL[1]	8/27/10	187.5/188.6, ~200[4] (129)	215.6[1]/240.6 (2419)
LLNL[1]	8/27/10	187.4/188.4, 199+[4] (135)	217.9[1]/238.7 (2399)
LLNL[2]	8/27/10	187.3/188.3, 199+[4] (126)	215.6[3]/238.0 (3517)
LLNL[2]	8/27/10	187.3/188.3, 199+[4] (132)	214.6[3]/231.2 (3478)
LLNL[2]	8/27/10	187.4/188.3, 199+[4] (114)	215.2[3]/230.6 (3805)
LANL[1]	12/02/10	188.2/189.7, 200.5 (129)	217.0[3]/242.4 (2091)
LANL[1]	12/09/10	188.2/189.6, 200.8 (131)	219.2[3]/243.0 (2138)
LANL[1]	12/15/10	188.0/189.2, 199.3 (140)	218.0[3]/242.1 (2300)
IHD[1]	9/29/09	187.7/189.2, 199.3 (107)	210.9[3]/240.2 (1375)[5]
IHD[1]	9/29/09	188.2/189.5, 199.8 (96)	201.8[3]/244.2 (1038)[5]

1. pinhole sample holder; 2. Hermetically sealed sample holder; 3. Onset of exothermic response reported to be obscured by endothermic response as indicated by software. 4. Visually estimated from hard copy profile; 5. Pan break due to off gases.

Table 8 shows in the DSC data taken with a pinhole or a hermetically seal sample holder. For the pinhole pan data, the data looks almost identical when comparing the contributions from each participant—low temperature endothermic features with T_{min} values around 190 and 199 °C and an intense exothermic feature with a T_{max} 238 to 243 °C. The endothermic responses are relatively weak, ΔH ~ 120-140 J/g, compared to the exothermic responses, ΔH ~ 2000 to 2500 J/g. Table 8 also shows the DSC data using a hermetically sealed sample holder. The endothermic features are essentially the same as in the pinhole sample holder case. However, the exothermic features are slightly different than in the pinhole sample holder data—the T_{max} values are lower and the ΔH values are higher.

4 DISCUSSION

Table 9 shows the average values for the data for RDX Type II Class 5 from each participant and compares it to corresponding data for standards, RDX Type II Class 5 done previously (Set 1) and PETN. The Set 1 data for RDX comes from the IDCA first iterative study of RDX as part of this Proficiency Test[2], and the data for PETN comes from the examination of PETN Class 4 as part of this Proficiency Test[22].

This round of tests is the second time the RDX Type II Class 5 standard has been examined by the IDCA participants. In this round of testing LLNL, LANL, IHD, and SNL participated. LLNL and LANL did not perform ABL Friction testing, and SNL did not perform any thermal testing. This testing is also the first testing contribution from SNL to the IDCA Program and Proficiency Test. In the previous round of testing the RDX Standard, LLNL, LANL, IHD, and AFRL participated in all or some of the tests. Table 9 reflects this with indicating not determined values.

Table 9. Average Comparison values

	LLNL	LANL	IHD	SNL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
RDX Type II Class 5 Set 2[2]	21.8[3,4]	20.8[3,5]	17.7[3,5]	23.3[3,5]
RDX Type II Class 5 Set 1[6]	24.1[7]	25.4[6,8]	19[3]	ND[9]
PETN[10]	10.9[5]	8.0[3]	9.3[3]	ND[9]
BAM Friction Testing[11,12]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
RDX Type II Class 5 Set 2[13,14]	16.5; 24.8	10.4; 11.3	11.8; 27.8	16.3; ND[9]
RDX Type II Class 5 Set 1[6]	19.2; 25.1	19.2; 20.8	15.5; ND[9]	ND[9]; ND[9]
PETN[10]	6.4; 10.5	4.9, 8.5	4.3, 6.9	ND[9]; ND[9]
ABL Friction Testing[15-18]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
RDX Type II Class 5 Set 2[19,20]	ND[9]; ND[9]	ND[9]; ND[9]	92; 207	ND[9]; ND[9]
RDX Type II Class 5 Set 1[6]	ND[9]; ND[9]	ND[9]; ND[9]	74; 154	ND[9]; ND[9]
PETN[10]	ND[9]; ND[9]	ND[9]; ND[9]	7.7, 42	ND[9]; ND[9]
Electrostatic Discharge[21]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
RDX Type II Class 5 Set 2[22,23]	0/10 @ 1.0[24]	0/20 @ 0.0375	0/20 @ 0.037	0/20 @ 0.15
RDX Type II Class 5 Set 1[6]	0/10 @ 1.0[24]	0/20 @ 0.0250	0/20 @ 0.095	ND[9]; ND[9]
PETN[10]	0/10 @ 0.033[25]	0/20 @ 0.025	0/20 @ 0.219	ND[9]; ND[9]

1. DH_{50}, in cm, is by a modified Bruceton method, height for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.8-23.9; 23-32), LANL (21.7-22.3; < 16), IHD (24-29; 40-43), SNL (20.0-22.5; 28.2-33.6); 3. 180-grit sandpaper; 4. Average of two measurements from Table 3; 5. Average of three measurements from Table 3; 6. From reference 2; 7. 120-grit Si/C sandpaper data only; 8. 150-grit garnet sandpaper; 9. ND = Not determined; 10. From reference 22; 11. Threshold Initiation Level (TIL) is the weight (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; 12. F_{50}, in kg, is by a modified Bruceton method, weight for 50% probability of reaction; 13. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 26-31), LANL (20.8-22.2; < 10), IHD (22-26; 40-42), SNL (20.4-22.2; 28.1-31.3); 14. Average of three measurements from Table 5; 15. LLNL and LANL did not perform measurements; 16. 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; 17. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 18. Measurements performed at 8 fps; 19. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (23-25; 40-44); 20. Average of three measurements from Table 6; 21. Threshold Initiation Level (TIL) is the energy (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; 22. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 26-32), LANL (20.9-22.2; < 10), IHD (24; 42), SNL (20.0-21.0; 20.5-31.3); 23. Average of three measurements from Table 7; 24. LLNL has 510-Ω resistor in circuit; 25. ABL ESD apparatus.

4.1 Comparison of participating laboratory testing of RDX Type II Class 5

Impact sensitivity. The data in Table 9 for the RDX Type II Class 5 Set 2 is all from using 180-grit garnet sandpaper. LLNL and LANL have very similar results, with both using microphone systems for positive detection. SNL average value is slightly higher (less sensitive). Interestingly, SNL only uses observation. The average value obtained from IHD data indicates a more sensitive material. IHD also uses observation only for positive detection.

Friction sensitivity. For BAM Friction, the average TIL value for LLNL indicates a more stable material than the other participants, consistent with what has been found previously. This is thought to be due to extra safety shielding of the LLNL BAM system[23]. The F_{50} values, however, show a different order where IHD evaluates the RDX Type II Class 5 as being more stable than the other participants. IHD was the only participant to do the ABL friction testing, so there is no comparison to be made.

ESD. LANL, IHD and SNL have similar ABL ESD systems that differ by vintage. This difference is reflected through the ability to set stimulation levels. LANL and IHD testing show the spark sensitivity of RDX Type II Class 5 to be about the same. However, SNL found the material to be somewhat more stable. SNL has the most recently built system of the three participants. LLNL is not compared in this group because, for this testing, LLNL used a custom built system that has a 510-Ω resistor in the circuit, making the direct comparison with other participants difficult.

Thermal sensitivity. All participants found the RDX Type II Class 5 Set 2 material to have essentially the behavior when using the pinhole sample container—two weak low temperature exothermic features just below 200°C and one prominent exothermic feature with a T_{max} near 240 °C. LLNL also used a hermetically sealed sample container and found a slight shift to lower temperatures higher enthalpies for the exothermic feature, compared to the vented system. Figure 3 shows an example of the RDX thermal profile from LLNL using the sealed sample holder. The other participants obtained virtually identical results except for IHD, where the sample cell burst in both cases during the exothermic event.

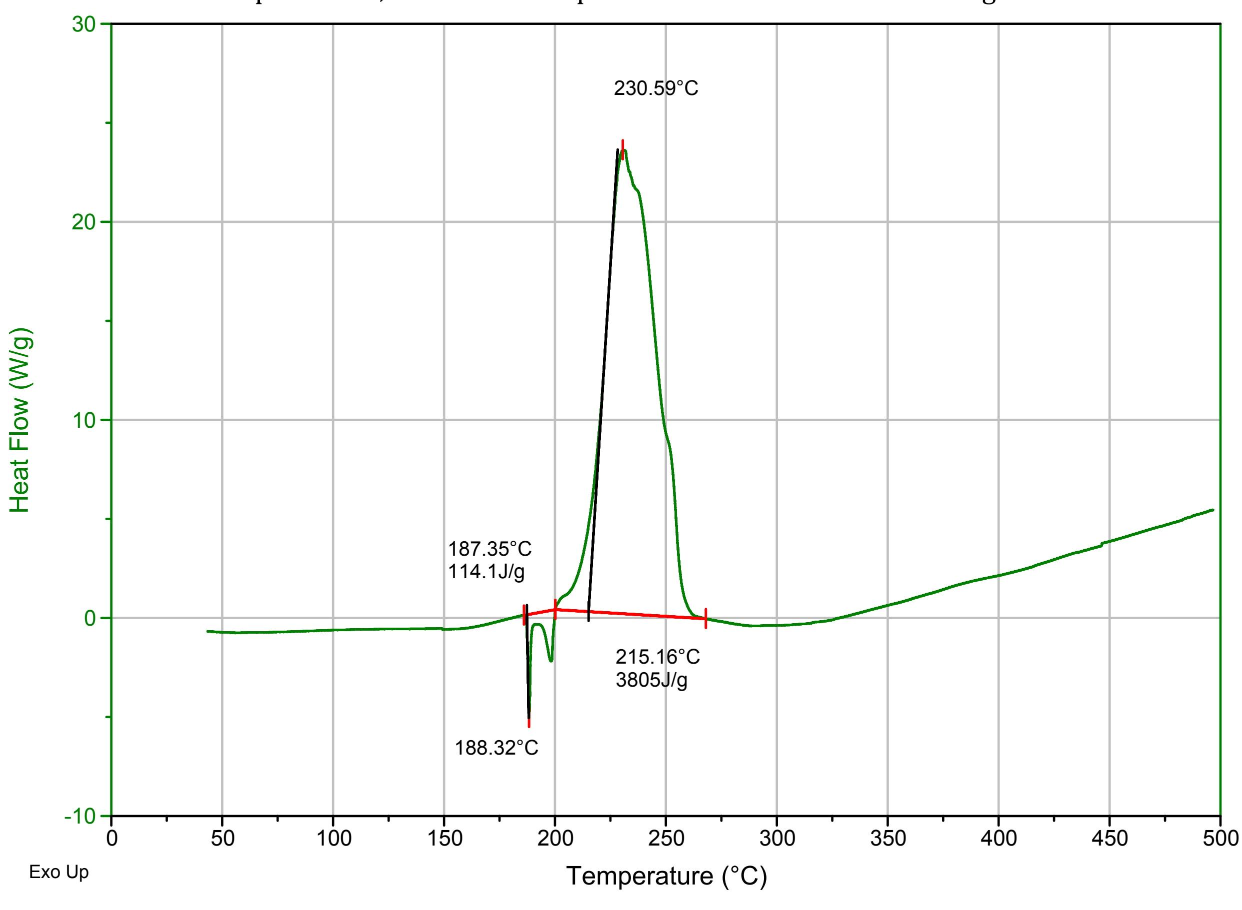

Figure 3. DSC profile of RDX Set 2 by LLNL using a sealed sample holder.

4.2 Comparison of average values for RDX Type II Class 5 Set 1 and 2

Impact sensitivity. Comparing the average values of RDX Type II Class 5 Set 2 with the averages from previously obtained data for RDX Type II Class 5 Set 1 shows similar trends. The average values obtained from LANL data are identical in both cases. Although lower than the average values for LANL, the average values for IHD are very close for the two sets. LLNL cannot be compared directly because the Set 2 data was obtained with 180-grit garnet sandpaper, and the Set 1 data was obtained with 120-grit Si/C sandpaper. Although the values are reasonably close, the IDCA has documented the effect sandpaper grit-size can be severe[23]. SNL average values cannot be compared because this is the first data set taken by SNL using IDCA protocols and materials. AFRL did not participate in this round of testing.

Effect of density (pressing or not pressing). LLNL also tested the RDX Type II Class 5 in pellet (pressed) form in both the first (Set 1) and second (Set 2) testing. In both cases, 120-grit Si/C sandpaper was used—Set 1, DH_{50} = 28.8 cm; Set 2, DH_{50} = 34.0 cm. It is not clear why the values between Set 1 and Set 2 are different. It could be a difference in densities of the pellets because this is not measured. However, both values reflect more stability than the corresponding values obtained from testing the powder form.

Neyer method for 50% probability of reaction. LANL has performed Neyer (or D-Optimal) Method of analysis for both RDX Type II Class 5 Set 1 and Set 2, using 150-grit and 180-grit garnet sandpapers for Set 1 and 180-grit sandpaper for Set 2. For the 180-grit sandpaper, the Set 1 value (only 1 test) is 20.4 cm and the Set 2 average value (three determinations) is 21.5 ± 1.6 cm—very similar and similar to the corresponding Bruceton analyses. For the 150-grit garnet sandpaper, from Set 1 only, the average value is 24.7 ± 1.5 cm, exhibiting the same sandpaper grit-size effect as seen previously.

Friction sensitivity. Comparing RDX Type II Class 5 Set 1 and 2, for BAM friction, LLNL found the material to have about the same sensitivity for both sets. LANL and IHD, however, found Set 2 to show more sensitive than Set 1. For ABL Friction testing, IHD found the opposite, where the average values indicate Set 1 data less sensitive than Set 2.

Spark sensitivity. RDX Type II Class 5 Set 2 testing by LLNL and LANL shows the material to be about the same sensitivity as shown by the corresponding Set 1 testing. Set 2 testing by IHD shows the material to be more sensitive than from Set 1 testing.

Table 10. Comparison of DSC data for RDX Type II Class 5 Set 1 and Set 2 data

Participant[1,2]	T_{min} of En_1[3], °C	T_{min} of En_2[4], °C	ΔH of En_{1+2}[5], J/g	T_{max} of Ex_1[6], °C	ΔH of Ex_1[6], J/g
LLNL Set 1	189.1 ± 0.1 (0.1)	199.1 ± 0.3 (0.1)	139 ± 3 (2)	241.3 ± 0.6 (0.2)	2298 ± 18 (1)
LLNL Set 1H	189.0 ± 0.1 (0.1)	198.9 ± 0.2 (0.1)	131 ± 11 (8)	234.3 ± 1.2 (0.5)	2967 ± 77 (3)
LLNL Set 2	188.4 ± 0.2 (0.3)	199 ± 1 (0)[7]	130 ± 5 (4)	239.8 ± 1.0 (0.4)	2417 ± 17 (0)
LLNL Set 2H	188.3 ± 0.0 (0.0)	199 ± 0 (0)[7]	124 ± 9 (7)	233.3 ± 4.1 (1.8)	3600± 179 (5)
LANL Set 1	189.3 ± 0.2 (0.1)	200.1 ± 0.5 (0.3	136 ± 1 (1)	242.1 ± 0.6 (0.2)	2237 ± 29 (1)
LANL Set 2	189.5 ± 0.3 (0.1)	200.2 ± 0.8 (0.4)	133 ± 6 (4)	242.5 ± 0.5 (0.2)	2176 ± 110 (5)
IHD Set 1	189.0 ± 0.1 (0.1)	198.9 ± 0.2 (0.1)	131 ± 11 (8)	242.2 ± 0.3 (0.1)	2041 ± 97 (5)
IHD Set 2	189.4 ± 0.2 (0.1)	199.6 ± 0.4 (0.0)	102 ± 8 (8)	241.2 ± 1.4 (0.6)	1207 ± 238 (20)
AFRL Set 1	189.2 ± 0.6 (0.3)	199.1 ± 0.1 (0.1)	144 ± 3 (2)	242.3 ± 1.5 (0.1)	2216 ± 29 (1)

1. Set 1, Set 2 are from data using pinhole sample holder from reference 19 and this report, respectively; 2. Set 1H, Set 2H are from data using sealed sample holder from reference 19 and this report, respectively; 3. En_1 is the first endothermic feature as seen in Table 8; 4. En_2 is the second endothermic feature as seen in Table 8; 5. ΔH for endothermic features 1+2 as seen in Table 8; 6. Ex_1 is the exothermic feature as seen in Table 8; 7. Visually estimated from hard copy printout.

Thermal sensitivity. Table 10 compares DSC average values for RDX Type II Class 5 Set 1 and Set 2. The table includes average values from data taken with the pinhole sample holder and the hermetically sealed sample holder. All participants determined the RDX behavior to be about the same, with minor variations in maximum or minimum temperatures and enthalpies. Overall, comparing the average values obtained from data using the pinhole sample holder, the values are consistent for the endothermic features—T_{min} values are within about 1 degree for the respective endothermic features; enthalpies range about 15 J/g throughout the set. Likewise, the exothermic features are also consistent—T_{max} values vary 2.7 °C at the most; enthalpies vary less than 400 J/g.

There are bigger differences in the average values when comparing data from the pinhole sample holders with the sealed sample holders—the enthalpies for the endothermic features are higher, the T_{max} values for the exothermic features are higher, and the enthalpies for the exothermic features are lower.

The behavior seen in Table 10 has been seen previously for RDX Type II Class 5 Set 1 and seems to be characteristic of the two types of sample holders—pinhole (aluminum with laser drilled hole, 50 microns in diameter) and hermetically sealed (pressure rated). These two types of sample holders have been compared throughout the Proficiency Test and the comparison has shown that the hermitically sealed holders have some advantage over the vented holders when examining mixtures with a volatile component[24,25]. Use of the hermetically sealed holders does have some advantages for use on some non-volatile materials. Although Class 5 Type II RDX is not considered a volatile material, it does produce significant amount of gas during the exothermic event. Sealed holders allow for the use of less sample size (LLNL, 0.25 mg for closed pans, 0.35 mg for pinhole pans); may not lose mass during the exothermic events; resulting in capture of energy release more efficiently and therefore more give a more accurate evaluation of the enthalpies. The T_{max} values for the higher temperature exothermic event do differ by around 9°C, where the sealed holder shows the T_{max} at a lower temperature. It is not clear what is causing this, but the exothermic decomposition is autocatalytic, the earlier onset and maximum in the sealed holder are most likely due to heat retention and pressure build up causing the reaction to occur more rapidly.

It should be noted that using the different types of sample holders for this material causes minor differences in DSC behavior, which probably has little impact on the results. For the materials studied so far in the IDCA Proficiency test, this has been the case for $KClO_3$/sugar[26,27], $NaClO_3$/sugar[28], $KClO_4$/Al[29], $KClO_4$/C[30], and PETN[22]. The hermetically sealed sample holder does make a significant difference for $KClO_3$/dodecane[24] and $KClO_4$/dodecane[25]. However, it should be noted *with caution* that standard DSC methods may not be adequate to describe the thermal behavior of the latter two mixtures, and can give misleading results.

4.3 Comparison of RDX Type II Class 5 with PETN Standard

Table 9 compares the RDX Type II Class 5 Set 1 and Set 2 average values with those of PETN, also obtained in this Proficiency Test[22]. For impact and friction sensitivity, all the participants found the PETN more sensitive. The results for spark sensitivity depend upon the participant. LLNL found the PETN to be more sensitive, but the comparison is for results obtained by the custom built system (RDX) and the new ABL system (PETN). LANL found the RDX to be about the same sensitivity as the PETN, while IHD found the PETN to be much less sensitive. All found the RDX to be less thermally sensitive than PETN.

5 CONCLUSIONS

Conclusions from the data for RDX Type II Class 5 Set 2:

1. The impact sensitivity is measured to be
    a. about the same by LLNL, LANL and SNL when the samples are in the powder form
    b. about 50% more sensitive by IHD than LLNL, LANL, and SNL.
2. The impact sensitivity appears less when samples are pelletized.
3. All participants reported almost identical results for the DSC of RDX
4. The friction sensitivity as measured by BAM appears less sensitive from LLNL and SNL .
5. The ESD sensitivity varies among participants
    a. LANL and IHD measured comparable sensitivities,
    b. SNL measured a less sensitive material,
    c. LLNL found the RDX to be insensitive.

Conclusions from comparison of Set 2 with previous testing of Set 1 for a specific participant:

1. Impact sensitivity is about the same.
2. For BAM friction, Set 2 appears more sensitive than Set 1.
3. For ABL friction (for IHD only), Set 1 appears more sensitive than Set 2.
4. For ESD, LANL finds Set 2 slightly less sensitive, IHD finds Set 1 slightly less sensitive, and LLNL finds both insensitive.
5. For DSC, all participants find Set 2 to be essentially the same as Set 1.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
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
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
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
j	joules
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
$NaClO_3$	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

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