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Integrated Data Collection Analysis (IDCA) Program - RDX Standard, Data Set 1

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Integrated Data Collection Analysis (IDCA) Program —RDX Type II Class 5 Standard, Data Set 1

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ABSTRACT

This document describes the results of the first reference sample material—RDX Type II Class 5—examined in the proficiency study for small-scale safety and thermal (SSST) testing of explosive materials for the Integrated Data Collection Analysis (IDCA) Program. The IDCA program is conducting proficiency testing on homemade explosives (HMEs). The reference sample materials are being studied to establish the accuracy of traditional explosives safety testing for each performing laboratory. These results will be used for comparison to results from testing HMEs. 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 results of the study will add SSST testing results for a broad suite of different HMEs to the literature, potentially suggest new guidelines and methods for HME testing, and possibly establish what are the needed accuracies in SSST testing to develop safe handling practices. Described here are the results for impact, friction, electrostatic discharge, and scanning calorimetry analysis of a reference sample of RDX Type II Class 5. The results from each participating testing laboratory are compared using identical test material and preparation methods wherever possible. Note, however, the test procedures differ among the laboratories. These results are then compared to historical data from various sources. The performers involved are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Air Force Research Laboratory/RXQL (AFRL), Indian Head Division, Naval Surface Warfare Center, (IHD-NSWC), 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 test protocols are not identical.

Keywords: Small-scale safety testing, proficiency test, round-robin test, safety testing protocols, HME, RDX.

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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 to span the challenging experimental issues arising 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.

HMEs are often formed by mixing oxidizer and fuel precursor materials. Typically, the solid-solid, liquid-liquid, or solid-liquid mixture precursors are combined shortly before use. For solid-solid mixtures, the challenges associated with producing a standardized inter-laboratory sample 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—and 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 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.

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.

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]	Powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2]	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2]	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 Type II Class 5		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.

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 a well 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 testing laboratory mixes the HME, performs the SSST tests on equipment that is frequently calibrated with the standard(s), and the test results reported as being more or less reactive than the standard. The results are then considered in-house. This 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.

This 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. RDX Type II Class 5 was chosen as the primary standard, and PETN Class 4 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. Here the first testing of RDX Type II Class 5 is reported for each of the participating laboratories and the results are compared and analyzed to set a baseline as well as critique experimental procedures.

The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (IHD-NSWC), and Air Force Research Laboratory/RXQL (AFRL). Sandia National Laboratories (SNL) contributed to the document, but did not perform any SSST testing.

2 EXPERIMENTAL

General information. All samples were prepared according to IDCA methods on drying and mixing procedures[2,3]. 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 RDX Type II Class 5 and was obtained from the Holston Army Ammunition Plant batch # HOL89D675-081 and provided to the participating laboratories test by IHD[4]. 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[5,6].

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

The SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for IDCA—RDX (LLNL)[7], RDX 51088_rev 0 (LANL)[8], RDX Report (IHD)[9], and RDX, Integrated Data Collection Analysis (IDCA) Program, Small Scale Safety Testing (SSST) (AFRL)[10].

Table 2. Summary of conditions for the analysis of RDX (all = All participants)

Impact Testing

1. Sample size—LLNL, IHD, and AFRL 35±2 mg; LANL 40±2 mg
2. Preparation of samples—all, dried per IDCA procedures[3]
3. Sample form—all, loose powder; also LLNL pressed RDX
4. Powder sample configuration—all, conical pile
5. Apparatus—LANL, LLNL, IHD Type 12A; AFRL MBOM with type 12A tooling*
6. Sandpaper—LANL (150 garnet, 180 garnet), LLNL (120 grit S/C paper), IHD (180 garnet), AFRL (180 garnet)
7. Sandpaper size—all, 1 inch square except LANL 1.25 inch diameter disk dimpled (LANL used 1 inch square for the one additional 180 garnet paper test)
8. Drop hammer weight—all, 2.5 kg
9. Striker weight—LLNL, IHD, and AFRL 2.5 kg; LANL, 1.0 kg
10. Positive detection—LANL and LLNL microphones with electronic interpretation and observation; IHD and AFRL, observation
11. Data analysis—all, modified Bruceton, LANL also uses Neyer

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—BAM; IHD, AFRL—ABL*
6. Positive detection—all, by observation
7. Room Lights—LANL and AFRL on, LLNL off, IHD (BAM) on, (ABL) off
8. Data analysis—LANL, LLNL, and IHD, modified Bruceton and TIL; AFRL, TIL

ESD

1. Sample size—all ~5 mg, but not weighed
2. Preparation of samples—all, according to IDCA procedures[3]
3. Sample form—all, powder
4. Tape cover—LANL, scotch tape; LLNL Mylar tape; IHD and AFRL no tape
5. Sample configuration—all, cover the bottom of sample holder
6. Apparatus—LANL, IHD, AFRL, ABL; LLNL has custom built*
7. Positive detection—all, by observation
8. Analysis methods—all, TIL

Differential Scanning Calorimetry

1. Sample size—all <1 mg
2. Preparation of samples—all, according to IDCA procedures[3]
3. Sample holder—all, pin hole; LLNL also hermetic sealed pan
4. Scan rate—all, 10°C/min
5. Range—40 to 400°C
6. Pan hole size—LLNL 50 μm, LANL, IHD, and AFRL 75 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920; IHD, TA Instruments Q1000, AFRL TA Instruments Q2000*
8. Analysis methods—all, TIL

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:* LANL, IHD, AFRL —ABL Electrostatic Discharge Apparatus, LLNL—custom-built Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry:* LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Seteram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

3 RESULTS

3.1 RDX Standard, Data Set 1

LANL, LLNL, IHD, and AFRL participated in this part of the SSST testing of the RDX Type II Class 5 Set 1. This standard will be tested throughout the proficiency test. The RDX standard was selected by participant consensus. A very well characterized RDX was provided by IHD and shipped to all the testing participants using Department of Transportation (DOT) approved shipping containers.

In this proficiency test, all testing participants are required to use materials from the same batch, and materials are to be prepared by the same methods. However, the actual testing procedures can be different. These differences are described in IDCA Program Analysis report on methods[11], which compares the different procedures by each testing category.

3.2 Impact testing results for RDX Type II Class 5, Data Set 1

Table 3 shows the results of impact testing of RDX Standard, Data Set 1 as performed by LANL, LLNL, IHD, and AFRL. Differences in the testing procedures are shown in Table 2. Notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive test. In addition, LLNL performed the test where the sample was pressed, as well as a loose powder. All participants performed data analysis by normal modified Bruceton method[12] and LANL also performed data analysis by the Neyer method[13]. LANL also performed one test using 180-grit sandpaper for comparison between the two grit sizes. This analysis was done with the Neyer method. Note, IHD and AFRL used the 180-grit size paper.

Table 3. Impact testing results for RDX Type II Class 5, Data Set 1

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120-P)	11/19/09	24	18	28.8	2.8	0.042
LLNL (120)	02/08/10	23	22	24.2	0.8	0.015
LLNL (120)	02/16/10	23	23	24.0	1.9	0.035
LANL (150)	11/23/09	21	17	26.5	1.2	0.019
LANL (150)	11/23/09	22	16	25.5	1.1	0.019
LANL (150)	11/23/09	22	16	24.2	1.5	0.027
IHD (180)	11/24/09	26	38	22	8.3	0.16
IHD (180)	01/11/10	26	38	19	8.1	0.18
IHD (180)	01/20/10	26	40	18	10.9	0.25
IHD (180)	01/20/10	26	40	18	4.6	0.11
AFRL (180)	4/29/10	22	43	15.1	3.5	0.10
AFRL (180)	4/29/10	23	45	13.1	5.3	0.17
AFRL (180)	5/4/10	27	57	17.6	3.7	0.09

1. Value in parenthesis is grit size of sandpaper (180 is 180 garnet dry 150 is garnet dry and 120 is 120 Si/Carbide wet/dry); 2 relative humidity; 3. Modified Bruceton method, load for 50% reaction (DH_{50}); 4. Standard deviation; p = pressed into pellet

The test results from the four participating laboratories for impact show a range for DH_{50} from 13.1 to 26.5 cm (not including the results of the pressed sample by LLNL) with an average value of 20.6 ± 4.4 cm. Average values for each participant are, in cm: LLNL, 24.1 ± 0.1; LANL, 25.4 ± 1.3; IHD, 19.3 ± 1.9; AFRL, 15.3 ± 2.3. The standard deviation is in the 0.097 log unit range except for IHD, where the variance is in the 0.25 log unit range. The IHD standard deviations are likely higher because IHD used 0.1 log spaced steps, which is twice what LLNL and LANL used. AFRL used linear steps for the Bruceton testing.

Table 4. Impact testing results for RDX Type II Class 5, Data Set 1 (Neyer or D-Optimal Method)—150- and 180-grit sandpaper

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (150)	12/24/09	20	17	24.0	3.3	0.06
LANL (150)	12/24/09	20	17	24.4	3.4	0.06
LANL (150)	12/24/09	20	17	23.7	2.7	0.05
LANL (150)	4/8/10	24.2	<10	26.7	5.6	0.09
LANL (180)	4/8/10	24.2	<10	20.4	3.3	0.07

1. 150 = 150-grit garnet sandpaper used in test,180 = 180-grit garnet sandpaper used in test; 2. Relative humidity; 3. Modified Bruceton method, load for 50% reaction (DH_{50}); 4. Standard Deviation.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method[13]. Most of the testing was done with 150-grit sandpaper, the standard paper used by LANL in their testing. The DH_{50} values are within the range of the values determined by Bruceton method. The average value from the Neyer method is 24.7 ± 1.4 cm that compares favorably with the average value from the Bruceton method.

Table 4 also shows the impact test results comparing the 180- and 150-grit sandpaper using Neyer or D-Optimal Method to analyze the data. The DH_{50} for the 180-grit paper is several cm lower than for the 150-grit paper. Even though the DH_{50} analyses were not done the same way, this value using 180-grit sandpaper is similar to the IHD and AFRL values for DH_{50} shown in Table 3. As described in Table 2, IHD and AFRL also use 180-grit sandpaper. The DH_{50} value for the 180-grit sandpaper is also closer to the LLNL measured values, although the analyses of the LLNL results are not by the Neyer method. LLNL, however, uses a 120-grit sand paper.

3.3 Friction testing results for RDX Type II Class 5, Data Set 1

Table 5 shows the BAM Friction testing performed by LANL, LLNL and IHD. AFRL does not have a BAM system. The difference in testing procedures by the three laboratories is shown in Table 2. The notable differences are in the methods for positive detection. Analyses were preformed by the threshold (TIL) method[14] by LLNL and IHD, and a modified Bruceton method by LLNL and LANL[12].

Table 5. BAM Friction Testing results for RDX Type II Class 5, Data Set 1

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4]	s, kg	s, log unit
LLNL	11/23/09	22.8	18	0/10 @ 19.2	1/10 @ 21.6	25.4	3.2	0.054
LLNL	02/09/10	22.8	23	0/10 @ 21.6	1/10 @ 24.0	24.6	2.8	0.050
LLNL	02/16/10	22.8	30	0/10 @ 16.8	1/10 @ 19.2	26.1	4.2	0.070
LANL	11/23/09	22.0	16.0	NA[5]	NA[5]	20.8	3.4	0.07
LANL	11/24/09	20.0	17.0	NA[5]	NA[5]	23.0	2.1	0.04
LANL	11/24/09	21.0	17.0	NA[5]	NA[5]	18.7	5.2	0.12
LANL	01/11/10	NA[6]	NA[6]	0/10 @ 19.2	1/4 @ 21.6	NA[5]	NA[5]	NA[5]
IHD	11/25/09	26	37	0/10 @ 14.7	1/3 @ 16.3	NA[5]	NA[5]	NA[5]
IHD	01/25/10	27	49	0/10 @ 14.7	1/6 @ 16.3	NA[5]	NA[5]	NA[5]
IHD	01/25/10	27	46	0/10 @ 16.3	1/2 @ 18.4	NA[5]	NA[5]	NA[5]
IHD	01/25/10	27	48	0/10 @ 16.3	1/4 @ 16.3	NA[5]	NA[5]	NA[5]

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, load for 50% Reaction; 5. Not applicable, TIL will be performed on future samples. 6. Not applicable, separate measurement for TIL only. Notes: Testing by LANL for replicates 2 and 3 included a level that was not evenly spaced. Re-evaluation of the actual sequences using the Neyer method showed that this had only a small impact on the results, lowering them by 0.3 kg to 0.5 kg. LLNL uses log-spacing and LANL uses liner spacing for the Bruceton up and down method experimentation and data analysis.

TIL results for LLNL and LANL center around 19 kg (LLNL average is 19.2 kg), while the TIL results for IHD are lower (average value of 15.5 kg). The F_{50} values for LLNL (average value 25.4 ± 0.7 kg) are higher than the F_{50} values for LANL (20.8 ± 2.2 kg).

Table 6 shows the ABL Friction testing performed by IHD and AFRL. As in the case for the BAM friction, originally the only reporting was based on the TIL method[14], but subsequent discussion with the team lead to adapting the Bruceton method[12], and future measurements will include both. The other participants did not have the system in routine performance at the time. For TIL, the IHD and AFRL values overlap (average IHD, 55; AFRL, 56) when considering the 8 fps testing data. For F_{50} (IHD only), the values range from 118 to 163 psig with an average of 155 ± 33 psig at 8 fps.

Table 6. ABL Friction Testing results for RDX Type II Class 5, Data Set 1

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/fps[8]	s, log unit[8]
IHD	11/24/09	26	36	0/20 @ 75/8	1/6 @ 100/8	NA[7]	NA[7]	NA[7]
IHD	01/21/10	27	44	0/20 @ 30/8	1/1 @ 40/8	183/8	175/8	0.37
IHD	01/21/10	26	43	0/20 @ 75/8	1/5 @ 100/8	NA[7]	NA[7]	NA[7]
IHD	01/21/10	27	41	0/20 @ 40/8	1/2 @ 55/8	NA[7]	NA[7]	NA[7]
IHD	01/25/10	27	43	NA[6]	NA[6]	118/8	30/8	0.11
IHD	01/25/10	27	46	NA[6]	NA[6]	163/8	46/8	0.12
AFRL	4/29/10	22.2	43	0/10 @ 56/8	3/5 @ 75/8	NA[7]	NA[7]	NA[7]
AFRL	4/29/10	22.7	45	0/10 @ 56/8	1/1 @ 75/8	NA[7]	NA[7]	NA[7]
AFRL	5/4/10	26.7	57	0/10 @ 100/6	1/4 @ 130/6	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; 5. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 6. Not applicable, will be performed on future samples.7. Not applicable, separate measurement for TIL only; 8. Standard deviation.

3.4 Electrostatic discharge testing of RDX Type II Class 5, Data Set 1

Electrostatic Discharge (ESD) testing of the RDX Standard was performed by LANL, LLNL, IHD, and AFRL. Table 7 shows the results for TIL levels and one above[14]. Differences in the testing procedures are shown in Table 2. Notable differences are the use of tape and what covers the sample. In addition, LLNL uses a custom built ESD system with a 500 ohm series resistor in line to simulate a human body, making a direct comparison of LLNL data with data generated by the other participants challenging. (LLNL has purchased a new ABL spark tester and is being used for the sparking testing on the 3rd RDX calibration run and the remaining IDCA threats.)

Table 7. Electrostatic discharge testing RDX Type II Class 5, Data Set 1

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL	11/18/09	22.8	18	0/10 @ 1.0[4]	0/10 @ 1.0[4]
LLNL	02/08/10	22.8	23	0/10 @ 1.0[4]	0/10 @ 1.0[4]
LLNL	02/16/10	22.8	30	0/10 @ 1.0[4]	0/10 @ 1.0[4]
LANL	11/24/09	20	17	0/20 @ 0.025	2/11 @ 0.0625
LANL	11/24/09	19	17	0/20 @ 0.025	2/7 @ 0.0625
LANL	11/24/09	19	17	0/20 @ 0.025	2/7 @ 0.0625
IHD	11/24/09	26	36	0/20 @ 0.095	1/7 @ 0.165
IHD	01/15/10	27	40	0/20 @ 0.095	1/7 @ 0.165
IHD	01/15/10	27	40	0/20 @ 0.095	1/14 @ 0.165
IHD	01/19/10	27	40	0/20 @ 0.095	1/12 @ 0.165
AFRL	4/29/10	22.2	43	0/20 @ 0.065	1/1 @ 0.069
AFRL	4/29/10	22.7	45	0/20 @ 0.038	1/3 @ 0.063
AFRL	5/4/10	26.7	80	0/20 @ 0.028	1/6 @ 0.031

1. Relative humidity; 2. Threshold initiation 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 uses a 510-ohm resistor in the discharge unit to mimic the human body.

The testing results from LANL indicate the RDX is more sensitive than the testing results from the IHD and LLNL. Some of the results from AFRL indicate a material that is equally as sensitive indicated by the results from LANL. The average TIL values are, in J/g: LANL, 0.025; IHD, 0.095; AFRL, 0.044.

3.5 Thermal testing (DSC) of RDX Type II Class 5, Data Set 1

Differential Scanning Calorimetry (DSC) was performed on the RDX Standard by LLNL, LANL, IHD and AFRL. All participating laboratories used different versions of the DSC by TA Instruments. However, the scanning conditions employed were the same. Table 2 summarizes the conditions.

Table 8 shows the DSC data from each of the participating laboratories delineated by sample. The data looks almost identical when comparing each laboratory—two endothermic responses, with T_{min} values around 190 and 199°C followed by an exothermic responses with T_{max} 241 to 244°C. The endothermic responses are relatively weak, ΔH ~ 120-150 J/g, compared to the exothermic response, ΔH ~2000 to 2300 J/g.

Table 8. Differential Scanning Calorimetry results for RDX Type II Class 5, Data Set 1 (pinhole vented and hermetically sealed sample holders)

Lab	Test Date	Endothermic, onset/minimum, °C (ΔH, J/g)	Exothermic, onset/maximum, °C (ΔH, J/g)
LLNL[1]	12/01/09	187.5/189.0, 199.2 (143)	203[3]/241.1 (2281)
LLNL[1]	02/04/10	187.8/189.1, 199.3 (139)	203[3]/240.7 (2299)
LLNL[1]	02/04/10	187.8/189.1, 198.8 (136)	203[3]/241.5 (2316)
LLNL[2]	12/01/09	187.4/188.9, 199.2 (125)	205[3]/233.5 (3024)
LLNL[2]	02/04/10	187.7/188.9, 198.8 (144)	205[3]/235.6 (2880)
LLNL[2]	02/04/10	187.6/189.1, 198.8 (125)	203[3]/233.7 (2998)
LANL[1]	11/17/09	188.0/189.1, 199.6 (137)	218.8[3]/242.8 (2205)
LANL[1]	11/24/09	188.1/189.6, 200.7 (135)	220.9[3]/242.8 (2260)
LANL[1]	11/24/09	188.0/189.2, 199.9 (135)	224.8[3]/242.1 (2246)
IHD[1]	11/25/09	188.0/189.2, 199.8 (120)	217.7/242.4 (1947)
IHD[1]	11/25/09	187.8/189.1, 199.4 (122)	218.0/242.3 (2034)
IHD[1]	11/25/09	188.0/189.4, 199.5 (127)	219.2/241.9 (2141)
AFRL[1]	5/5/10	188.1/189.5, 199.9 (141)	216.3/240.5 (2198)
AFRL[1]	5/5/10	188.0/189.5, 199.8 (148)	216.4/242.8 (2250)
AFRL[1]	5/5/10	188.2/189.5, 199.9 (144)	215.3/243.5 (2201)

1. pin-hole vented sample holder; 2. Hermetically sealed sample holder; 3. Onset of exothermic response reported to be obscured by endothermic response as indicated by software.

Table 8 also shows the DSC data, by LLNL only, for the RDX Standard where the DSC sample holder is closed instead of pinhole vented as in the Table 8. The values for the endothermic responses are about the same as the values derived from the pinhole vented pan, but the T_{max} of the exothermic response is a few degrees lower, compared to the same excursion measured with the open pan. As well, the values for the enthalpy of the exothermic excursion measured in the open pinhole pan are lower, possibly due to the loss of heat with the loss of gases, indicating that the closed system has better heat transfer properties.

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 the standard, PETN. The average values are calculated by standard methods for the 50% probability analyses (DH_{50} for drop hammer and F_{50} for friction methods). The average for the TIL values are simply an arithmetic average of the data points. No statistical meaning is given to these values except they help establish trends for comparing each participant. The data for the PETN comes from measurements taken outside of the IDCA Proficiency Test.

Table 9. Average RDX Type II Class 5, Data Set 1

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
RDX Type II Class 5[2]	24.1[3,4]	25.4[5,6]	19[7,8]	15.3[5,8,]
PETN[9]	15[4]	14.7[6]	ND[10]	ND[10]
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[13]	19.2[14]; 25.3[14]	21.6[15]; 20.8[14]	16.3[16]; ND[10,17]	ND[10], ND[10]
PETN[9]	6.4, 10.5	ND[10], 9.2	ND[10], ND[10]	ND[10], ND[10]
ABL Friction Testing[18-21]	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[22]	ND[10], ND[10]	ND[10], ND[10]	55[23], 154[24]	71[25], ND[10]
PETN[10]	ND[10], ND[10]	ND[10], ND[10]	ND[10], ND[10]	ND[10], ND[10]
Electrostatic Discharge[26]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
RDX Type II Class 5[27]	0/10 @ 1.0[28,29]	0/20 @0.025[29]	0/20 @ 0.095[30]	0/20 @ 0.044[29]
PETN[9]	0/10 @ 1.0	2/2 @ 0.125	ND[11]	ND[11]

1. DH_{50}, in cm, is by a modified Bruceton method, load for 50% reaction; 2. Temperature and humidity are not listed because the values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23; 22–23), LANL (21–22; 16–17), IHD (26; 33–40), AFRL (22–27; 43–56); 3. Average of two measurements from Table 3, 4. 120-grit Si/C wet/dry sandpaper; 5. Average of three measurements from Table 3; .6. 150-grit garnet sandpaper; 7. Average of 4 measurements from Table 3, 8. 180-grit garnet sandpaper; 9. From data obtained outside of the IDCA Proficiency Test; 10. ND = not determined; 11. 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; 12. F_{50}, in kg, is by a modified Bruceton method, load for 50% Reaction; 13. Temperature and humidity are not listed because the values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.8; 18–30), LANL (20.0–22.0; 16.0–17.0), IHD (26–27; 37–49); 14. Average of three measurements from Table 5; 15. one measurement from Table 5; 16. Four measurements from Table 5; 17. IHD did not perform the modified Bruceton analysis method for this set of measurements; 18. LLNL and LANL did not perform measurements; 19. 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; 20. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 21. Measurements performed at 8 fps; 22. Temperature and humidity are not listed because the values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (26–27; 36–49), AFRL (22.1–26.7; 43–57); 23. Average of four measurements from Table 6; 24. One measurement from Table 6; 25. Average of three measurements from Table 6; 26. 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; 27. Temperature and humidity are not listed because the values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.8; 18–30), LANL (19–20; 17), IHD (26–27; 36–40), AFRL (22.2–26.7; 43–80); 28. LLNL has 500-ohm series resistor in circuit; 29. Average of three measurements from Table 7; 30. Average of four determinations from Table 7.

4.1 Comparison of participating laboratory testing

Effect of density (pressing vs. not pressing). Comparisons of initial results indicated that LLNL was obtaining DH_{50} values (statistically significantly) reasonably higher than the other participating laboratories. This is shown in the pellet data shown in Table 3 for LLNL. Comparisons of methodology among the participating laboratories revealed that for many military explosive materials, LLNL usually presses the material before testing. Upon retesting the RDX Type II Class 5 Standard, the DH_{50} values are much lower (by approximately 5 cm), and similar to the results from the other participating laboratories.

Isolating the pressed value from the rest of the data in Table 3 shows the DH_{50} values from the participating laboratories are reasonably aligned ranging from 13.1 to 26.5 cm. Within a specific laboratory, the order is the following AFRL < IHD < LLNL < LANL. No statistical significance are linked to this trend, but some observations are in order and may be considered in the future. The humidity and temperature inversely follow the ordering. The grit size of the sandpaper is different at each participating laboratory, as well as the methods for evaluating positive responses from the tests.

Effect of grit size of the sandpaper. The DH_{50} values in Table 3, after the adjustment to the sample preparation, show reasonable, but not perfect agreement among the participants. To see if there were other experimental parameters that could affect these DH_{50} values (make them closer), LANL examined the difference in grit size of the sandpaper. As shown in Table 4, the grit size of the sandpaper appears to have an effect—the larger grit number produces a lower DH_{50} results. Note that IHD and AFRL results are based upon 180-grit size sandpaper.

4.2 Neyer method compared to modified Bruceton method

LANL also performed the impact testing using the Neyer (or D-Optimal Method) for testing. Comparing the LANL Bruceton method data in Table 3 and the Neyer method data in Table 4 shows a high level of consistency. The methods are compared briefly below, but a more detailed description is given in IDCA report on methods that compares the testing methodologies for this proficiency test[7].

The basic goal of SSST testing is to determine relative sensitivities of materials, although the IDCA proficiency test hopes to establish some guidelines on determining standard values for HMEs. The approach of this type of testing is to probe reaction probability distributions by predetermined test algorithms. Go/No-Go (positive/negative) is determined at given stimuli levels, and a model is used to determine mean and standard deviation. Some caveats must be realized. Distribution of positive/negative reactions is dependent on many factors, including that different procedural methods and instruments, as well as sample homogeneity issues can cause variations (within and across tests).

The sensitivity of a material is best reported in terms of the probability of reaction as a function of input stimulus level. A detailed mapping of this reaction distribution requires many tests at many different stimulus levels, which can translate to large amounts of time, money, and sample material. More efficient methods to probe the distribution can be applied if it is known to be Gaussian or if the stimulus can be transformed so that the distribution becomes approximately Gaussian (e.g. using the logarithm of the stimulus). The mean (50% cumulative reaction probability) and the standard deviation are then the reported parameters describing the material. For SSST testing purposes often there are limited quantities of many different materials and so it is necessary to use efficient methods.

Two common methods used to probe the reaction distribution are the Bruceton method[12] and the Neyer method[13]. The Bruceton method (or Up-Down testing) has been used for over 60 years and is common in many laboratories. The Neyer method (or D-optimal method) was developed in 1994, with the desired aim of giving a more accurate determination of the DH_{50} value.

In the Bruceton method the distribution is probed by initially choosing a stimulus level near the anticipated 50% reaction point and then adjusting the stimulus level for each test based on the previous outcome—if the material reacts (Go), the stimulus is decreased one step and if the material does not react (No-Go), the stimulus is increased by one step. The mean and standard deviation (m and s) are then calculated from the number of Go's and No-Go's at each level using approximation formulas that assume a Gaussian distribution[15]. The advantages of this method are that it concentrates testing near the mean and that it can be carried out without the use of a computer. The disadvantages are: the formulas for m and s assume that the step size between stimulus levels is $> \frac{1}{2} s$ and $< 2 s$ and this may not be true, the step size must be constant, and it can require a relatively large number of tests.

The Neyer method is based on an algorithm that uses maximum likelihood estimation (MLE) of DH_{50} and s to adjust the stimulus levels during testing so that the estimates of both parameters are optimized simultaneously. The step sizes may change depending on the likelihood function. A commercial software package, allows the method to be computer controlled[16]. The advantages of this method are that DH_{50} and *s* are optimized together to better characterize the distribution, and the adjustable step size also allows the distribution to be probed using typically fewer tests than the Bruceton method. A disadvantage of the method is that it requires a computer to carry out the analysis needed to compute *s* and adjust the step sizes between tests.

4.3 Comparison with reference data

Indian Head Historic Data. One of the best comparisons for data on Holston RDX comes from past data collected at IHD[17]. Although the results are on several different batches of Holston-produced RDX, as well as some different classes, the results are useful. The range of the DH_{50} values for the historical Type 12A data is 10 to 17 cm. The IHD impact data in Table 3 from this report overlap on the high end of this range. Only the 0/10 TIL values are presented in the historical data for BAM friction and the range is 8.6 to 14.7 kg. The IHD BAM friction data shown in Table 5 in this report also overlap on the high end of this range, 14.7 to 19.2 kg. For ABL friction, the range with historical data at the 8 ft/sec rate is 0/20 @ 55 to 420 psig (but more often 55 to 180 psig). The IHD ABL friction data shown in Table 6 of this report overlaps on the more sensitive end 0/20 @ 30 to 75 psig. For the ESD, the historical data varies from 0/20 @ 0.037 to 0.326 J. The IHD ESD data shown in Table 6 of this report falls in that range, 0/20 @ 0.095 J.

LLNL High Explosives Reference Guide. The LLNL High Explosives Reference Guide (HE Reference Guide) also provides historical test data for RDX[18]. In this case, the data are from a variety of sources and institutions where some testing details are not completely described. In addition, the characterization of some of the materials is also not as complete as in the IHD historical data (size range and manufacturer of the RDX is not clearly stated). With these caveats, the best level of comparison is that the results are reasonably close. From the HE Reference Guide for Type 12A, powder analysis, the impact data range for DH_{50} is 28 to 38 cm (4 determinations). The LANL impact data shown in Table 3 in this report comes closest for this range. For the RDX pressed into a pellet, the range in the historical data is a DH_{50} of 23 to 51 cm (44 determinations). The first data set shown in Table 3 in this report clearly falls in that range. The HE Reference Guide has much fewer measurements by BAM friction. The range in the historical data is 1/10 @ 12.4 to 16 kg (5 determinations). The BAM friction data shown in Table 5 of this report reflect a more stable RDX material with 1/X @ 16.3 to 24.0 kg ($X \geq 2$). The ESD historical data for the HE Reference Guide shows RDX having no spark sensitivity that compares with the LLNL ESD data shown in Table 7.

LANL Historical Impact Reference Data. LANL has sporadic archival data dating back to 1961. The characterization of the earliest samples is not stated, however for later samples the lot information has been clearly documented. All the samples were run in loose powder form at ambient conditions and were most likely run on the same instrumentation. The range of DH_{50} values are very wide, 16 cm to 123 cm. The highest values are extreme outliers, without those values, the distribution is approximately normal with a mean of 27.8 cm and standard deviation of 8.5 cm. These values are in agreement with the LLNL High Explosives Reference Guide and the impact data shown in Table 3 in this report.

Miscellaneous. Other data with less detail given than above is also available for comparison. Socorro[19] shows RDX has the following sensitivity, DH_{50} of 23 cm, ABL friction > 1100 psig and ESD with no resistor 0.27 J and 0.10 J.

Differential Scanning Calorimetry. The DSC results for RDX described above in Table 8 show approximately the same behavior (at a 10°C/min heating rate)—weak endothermic responses from 180 to 200°C followed by a strong exothermic response with a T_{max} around 240°C. This behavior has been identified previously on an RDX sample of 100 to 800 micron size range at the 10°C/min heating rate[20]. The endothermic response, with a T_{min} = 204°C, is assigned to the melting, followed by the exothermic response, with a T_{max} = 237°C, assigned to decomposition of RDX. HMX, which comprises 10% by wt of the RDX sample studied here, behaves differently with the endothermic response, with a T_{min} = 195°C, assigned to morphological transformation, followed by melting right before a strong exothermic response, with a T_{max} = 281°C, assigned to decomposition. The ΔH of decomposition at 10°C/min for the RDX has also been assigned as 950 J/g.

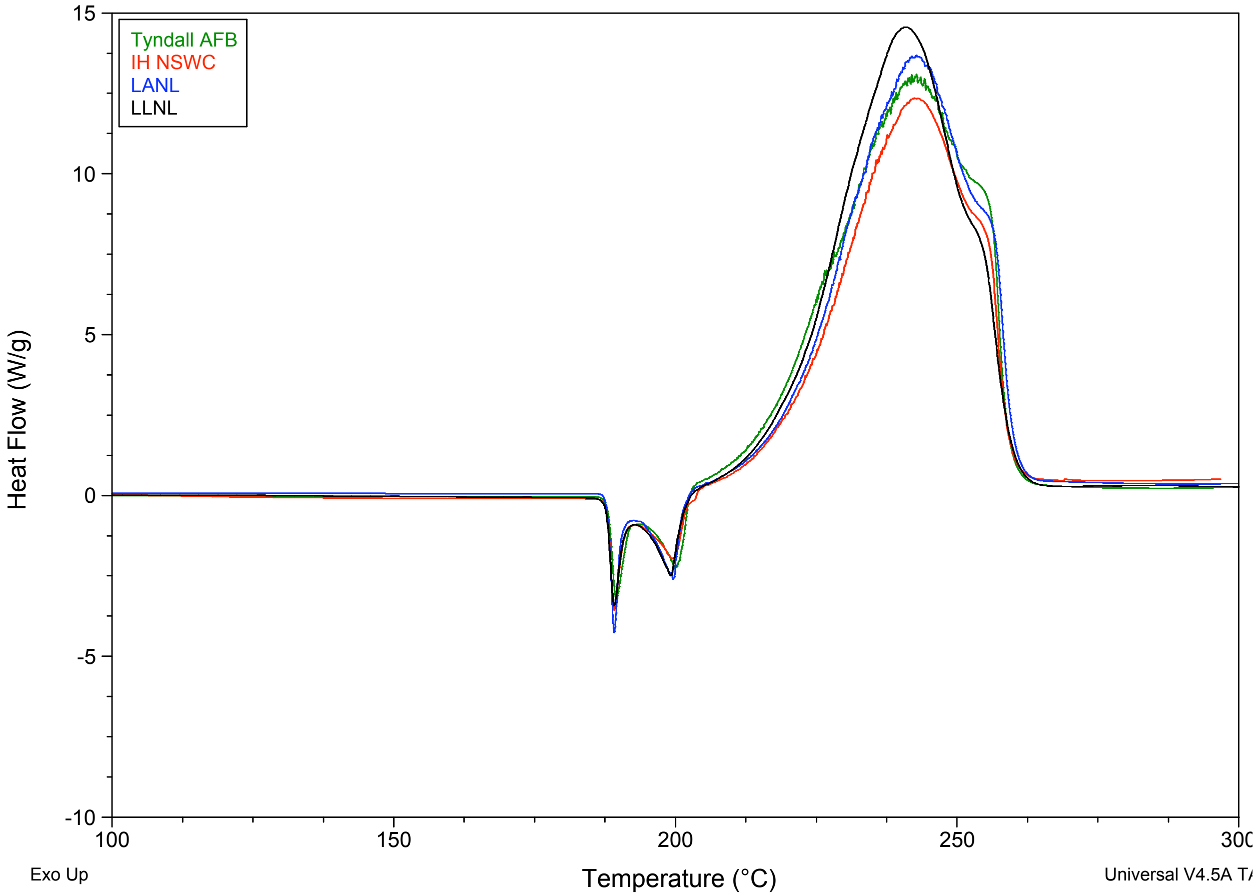

Figure 1. Overlay of DSC profiles for RDX Standard, Data Set 1 from all participants. Heating rates are 10°C/min.

Values in Table 8 elucidate the DSC behavior of RDX that has been seen in other studies. The HE Reference Guide[18] and the URI data base[21] show comparable behavior of RDX. The HE Reference guide sample of RDX, at 10°C/min heating rate with a vented Al sample holder, shows an endothermic response, with a T_{min} = 205°C and ΔH = 135 J/g, and an exothermic response, with a T_{max} = 242°C and ΔH = 2022 J/g. The URI data base sample of RDX, at 10°C/min heating rate with a vented pan, shows an endothermic response, with a T_{min} = 203°C and ΔH = 139 J/g, and the exothermic response, with a T_{max} = 249°C and ΔH = 2237 J/g. These results all agree well with the results reported in this study.

The above comparisons are to demonstrate that SSST data has significant variability when comparing historical data. However, most of the data presented compares to the data presented in this report.

The averages as well as standard deviation and relative standard deviations of the determinations of the DSC for LLNL, LANL, IHD and AFRL data are shown in Table 10. Although the number of determinations from each laboratory is only three, the average and deviations indicate the scatter of the results are small throughout the table. When comparing the average among the participants, the T_{min} values for the first endothermic event (En_1) are within a °C. The T_{min} values for the second endothermic event (En_2) behave the same. The ΔH values for the sum of the endothermic events ($En_1 + En_2$) are within 5 J/g. For the exothermic event, the T_{max} values for Ex_1 are within a degree, and the ΔH values for the exothermic event (Ex_1) are within 100 J/g. Even though the data set is relatively small, the results are remarkably consistent.

Table 10. Selected averages and deviations and (relative deviations) for DSC of RDX Type II Class 5, Data Set 1

Participant	T_{min} of En_1^1,°C	T_{min} of En_2^2,°C	ΔH of En_{1+2}^3, J/g	T_{max} of Ex_1^4,°C	ΔH of Ex_1^1, J/g
LLNL	189.1 ± 0.1 (0.1)	199.1 ± 0.3 (0.1)	139 ± 3 (2)	241.3 ± 0.6 (0.2)	2298 ± 18 (1)
LANL	189.3 ± 0.2 (0.1)	200.1 ± 0.5 (0.3	136 ± 1 (1)	242.1 ± 0.6 (0.2)	2237 ± 29 (1)
IHD	189.0 ± 0.1 (0.1)	198.9 ± 0.2 (0.1)	131 ± 11 (8)	242.2 ± 0.3 (0.1)	2041 ± 97 (5)
AFRL	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. En_1 is the first endothermic event in Table 8; 2. En_2 is the second endothermic event in Table 8; 3. ΔH for endothermic events 1+2 in Table 8; 4. Ex_1 is the first exothermic event in Table 8.

4.4 Pin hole vs. no pin hole DSC

For the DSC of these materials, LLNL used two types of sample holders—pinhole (aluminum with laser drilled hole, 50 microns in diameter) and hermetically sealed (pressure rated). These two types of holders were selected because of various reasons. For volatile samples the sealed holders are rated not to rupture when volatile species are in the sample. Although RDX Type II Class 5 is not considered a volatile material, it does produce significant amount of gas during the exothermic event. As well, other materials in the Proficiency test will be examined that are volatile, as seen in Table 1. Closed holders allow for the use of less sample size (LLNL, 0.25 mg for closed pans, 0.35 mg for pinhole pans). Closed holders may not lose mass during the exothermic events. As a result, these holders capture energy release more efficiently and therefore more give a more accurate evaluation of the enthalpies.

For LLNL data in Table 9, the T_{min} values for the endothermic events (~188°C) are comparable for the two different types of holders. 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 holders are most likely due to heat retention and pressure build up causing the reaction to occur more rapidly. This will be studied in more detail elsewhere.

Table 9 shows that the closed holders exhibit different enthalpies for all events—both exothermic and endothermic (closed pans for RDX ~ 3000 J/g, pinhole pans for RDX ~2300 J/g). The impact this difference has on the Proficiency testing has yet to be determined.

4.5 Comparison with PETN Standards

For the purposes of comparisons with future testing in the Proficiency test, the following tables are the average values for RDX Test Standard derived from the tables in the text. These values are only to be used for cursory comparisons because the averaging is of data that was taken under differing environmental conditions. Detailed comparisons will be made in future reports. PETN data, when available, is also included to give a relative sense of where these average values fall compared to that standard. Note: the results for the PETN came from testing done out side of the Proficiency test.

5 CONCLUSIONS

Conclusions from the data:

1. The impact sensitivity of RDX is measured to be about the same by each laboratory when the samples are in the powder form
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 slightly less sensitive from LLNL measurements.
5. Grit size of sandpaper for impact test shows a dependence that could be related to size of the grit. More experimentation has to be done to validate.

Conclusions from the testing methods:

1. For solid materials, only powder form will be tested (one pressed sample exhibited much more impact stability)
2. TIL levels and the levels before threshold will be reported for friction and spark
3. Modified Bruceton method will be used for impact and friction
4. Neyer method will be used for impact along with modified Bruceton (currently only LANL has software)
5. 180-grit sandpaper distributed by one participant will be used by all participants.

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ACRONYMS AND INITIALISMS

ABL	Allegany Ballistics Laboratory Friction Apparatus
AFRL	Air Force Research Laboratory
AN	ammonium nitrate
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
Class 5	97+% passes through 325 mesh sieve
DH_{50}	Modified Bruceton analysis method, load for 50% reaction
DOT	Department of Transporation
DSC	Differential Scanning Calorimetry
EGDN	ethylene glycol dinitrate
ESD	electrostatic discharge
HE	High Explosives
HME	homemade explosives
HMX	cyclotetramethylene-tetranitramine
HP/F	hydrogen peroxide/fuel
IDCA	Integrated Data Collection Analysis
IHD	Indian Head
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
m	Mean
MBOM	Modified Bureau of Mines
ME	mechanical energy
MLE	maximum likelihood estimation
NSWC	Naval Surface Warfare Center
P	detonation pressure
PETN	Pentaerythritol tetranitrate
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
s	Standard deviation
SNL	Sandia National Laboratories
SO/F	solid oxidizer/fuel
Socorro	New Mexico State University, Socorro New Mexico, Energetic Materials Testing Center
SSST	small-scale safety and thermal testing
TATP	triacetone triperoxide
TIL	Threshold Initiation Level, the level before positive reaction
TSA	Transportation Security Administration

Type II	Bachmann Process RDX using acetic anhydride
Type 12A	Impact testing configuration using sandpaper to hold sample
UN	urea nitrate
US/EU	United States/European Union

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