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Statistical Analysis of an Inter-Laboratory Comparison of Small-Scale Safety and Thermal Testing of RDX

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Abstract: The Integrated Data Collection Analysis (IDCA) program has conducted a proficiency test for small-scale safety and thermal (SSST) testing of homemade explosives (HMEs). Described here are statistical analyses of the results from this test for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of the RDX Class 5 Type II standard. The material was tested as a well-characterized standard several times during the proficiency test to assess differences among participants and the range of results that may arise for well-behaved explosive materials. The analyses show there are detectable differences among the results from IDCA participants. While these differences are statistically significant, most of them can be justified for comparison purposes to assess potential variability when laboratories attempt to measure identical samples using methods assumed to be nominally the same. The results presented in this report include the average sensitivity results from the IDCA participants and the ranges of values obtained. The ranges represent variation about the mean values of the tests of between 26% and 42%. The magnitude of this variation is attributed to differences in operator, method, and environment as well as the use of different instruments that are also of varying age. The results appear to be a good representation of results generated by the broader safety testing community based on the range of methods, instruments, and environments included in the IDCA proficiency test.

Keywords: Small-scale safety testing · Thermal screening · RDX · Round-robin test · Proficiency test · Statistical evaluation

1 Introduction

The Integrated Data Collection Analysis (IDCA) program is evaluating small-scale safety and thermal (SSST) testing methods as applied to improvised or home-made explosives (HMEs) through a proficiency or round-robin like test. Five laboratories that routinely evaluate the safety aspects of energetic materials are involved in the testing. 16 HMEs and three traditional explosives were tested under essentially the same test methods. The results can be found in IDCA Analysis Reports [1] and publications [2–4]. A description of the SSST testing methods used in the proficiency test has also been reported [2].

The IDCA is also attempting to understand, at least in part, the laboratory-to-laboratory variation that is expected when examining HMEs. 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 testing.

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Evaluation of the results of SSST testing of unknown materials 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, characterized 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 reactivity than conventional military explosives.

The first step in the proficiency test is to have representative data on a standard material to allow for basic performance comparisons. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. RDX was tested in triplicate several times throughout the IDCA proficiency test. In this report, all of the RDX results are analyzed to determine statistical differences among participants, average values, expected ranges, percent variability, dependence on method or environment, and possible causes for the differences that are observed.

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

2 Experimental Section

2.1 Materials

All RDX samples were prepared according to IDCA drying and mixing procedures [5,6]. Briefly, the RDX 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 by IHD. 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 μm with a maximum at 31.1 μm [7,8].

2.2 Bruceton Up-Down Testing

The most useful way to characterize the sensitivity of a material is by measuring the parameters of the statistical distribution that describes its response to an external stimulus. The only way to measure these parameters is by testing at various stimulus levels and interpreting the sequences of various Go (evidence of a reaction) and No-Go (no evidence of a reaction) events based on a statistical model. The method used often for explosives is the Bruceton Up-Down method developed in the 1940's [9]. In this method, the explosive is tested at some initial stimulus level. If a Go is observed, the stimulus level for the next test is decreased by one step, but if a No-Go is observed, the stimulus level is increased by one step. This Up and Down step adjustment continues for a predetermined number of tests to build statistics for the reaction probabilities at a few levels near the mean. If the steps are evenly and linearly spaced with respect to the response of the explosive, if the response is Gaussian, and if the step spacing is close to the standard deviation, then the statistical results can be analyzed with simple algebraic formulas to determine an estimated mean and standard deviation of the probed distribution. This is expressed as a 50 % probability of reaction, DH_{50} (in cm), for impact testing and F_{50} (in kg), for friction testing.

2.3 Neyer D-Optimal Testing

The Bruceton Up-Down method concentrates testing around the mean of the distribution (50 % level) but does not provide an optimum determination of the standard deviation. Neyer developed an alternative method in 1994 [10] using a maximum likelihood approach that concentrates testing at the $+/-\ 1\sigma$ levels. The test design is "D-optimal", meaning that it maximizes the determinant of the information matrix associated with the results. In practice, the testing is carried out via commercial software that monitors analysis and test level changes during testing. The software fits a Gaussian distribution to the final set of test results, providing an estimate of the mean and standard deviation. This is expressed as 50 % probability reaction as with the Bruceton method in this paper.

2.4 Threshold Initiation Level Testing

Threshold Initiation Level testing, or TIL determination, is defined in this context as the method of determining the highest stimulus level at which some predetermined number of No-Go events are observed without any Go events occurring [11]. In the data sets below, the predetermined number of No-Go events is often 10, although occasionally 20 is used. Practically, testing is carried out at a chosen level until either a Go event occurs or the predetermined number of No-Go events is reached. If a Go occurs, the stimulus is decreased one step and the testing is repeated. If the result was all No-Go events, the stimulus is increased one step and testing is repeated. The "TIL" level or "TIL 0" level is defined as the level, at which all No-Go events were observed while at least one Go event was observed at the next highest level. This next highest level can be defined as the "TIL+" level and used for comparison purposes as well. There is no obvious statistical distribution parameter associated with these TIL levels although for a 0/10 result, the TIL 0 level will be an estimate of an upper bound on the 10% reaction probability level.

2.5 Analysis of Variance

Analysis of statistical measurements from different laboratories can be formally evaluated through Analysis of Variance (ANOVA) [12]. ANOVA is a standard method for assessing agreement among different measurements of mean values by comparing the standard deviations of a set of measurements to the standard deviation of the set averages. For data sets that are statistically equivalent, the standard deviations computed in these two different ways will be similar and their ratio will follow a statistical distribution with known characteristics. If one data set is statistically different from the others, the standard deviations computed in these two different ways will differ, and their ratio will vary from the expected distribution by an amount that is characterized here by what is called a p-value. The p-value ultimately represents the probability that claiming there is a difference between the sets of results will be in error. For example, if $p=0.05$, then one could claim that there is a significant difference in the data sets with a 5% chance that the assessment is incorrect. This is called a Type I error in statistical texts. As the p-value gets larger, there is a greater chance of Type I error and so it is accurate to say that the data sets do not differ. For example, if $p=0.95$, one could claim that the results were different but would have 95% chance of being wrong – the natural interpretation would be to say that the data sets were the same. For very small p-values, the chance of a Type I error is very small and it is accurate to say that the data sets do differ in this report, the ANOVA treatment of the data was carried out using MiniTab 16, a commercially available software package [13].

2.6 Tukey and Fisher Comparisons

The data set or sets responsible for disagreement in ANOVA can be determined using Tukey or Fisher comparison methods [12,14]. In either of these methods, pairwise comparisons of the individual data sets are made and assessed against statistical distributions that are expected to describe their behavior. For the Tukey test, the distribution is called the "studentized range" distribution and for the Fisher test, the F-distribution is used. Disagreement among pairs in the set of results is used to assign groups of results that can be considered to be in agreement. These groups can be used to describe average results and identify outliers.

2.7 Averaging

Almost all determinations (experimental data sets for each data reduction method) were performed in triplicate (or more). The Bruceton and Neyer methods produce a value that represents 50% probability of reaction (midway on a reactivity curve) and a standard deviation. The individual determinations were averaged and these values were used for the graphs and the comparisons in this report. The TIL method determines the onset of the sensitivity (upper limit 10% on the upswing of the reactivity curve). This uses a discrete insult level approach, so only specific levels are recorded in the raw data. These specific levels correlate to limited settings on the equipment. As a result, because each participant had different vintages of the same equipment, each participant reported different, but discrete levels in the raw data. Although somewhat of an inexact method, the three (or more) discrete results for a specific material were averaged for the comparisons. This can result in a value that the participant cannot actually measure. For example, ESD data for HMX by IHD is 0.165, 0.326, 0.165 J, which are discrete settings of the ABL equipment used by IHD for the measurements. In this report, the three values are averaged to 0.219 J, which is not a setting on the IHD equipment. However, this estimates the average insult level determined by IHD in the ESD comparison figure.

Supporting Information (see footnote on the first page of this article): All the individual determinations from each participant that are averaged in this report, the standard deviations (if applicable), environmented condition, dates, and some characterization data.

3 Results

Due to the massive amount of data accumulated, only summary figures and tables are presented herein. A full listing of the data is recorded in the Supporting Information that can be obtained from the corresponding author.

For the statistical analysis of results, the goals are to:

(i) Determine whether all laboratories or a subset of laboratories appear to be making equivalent measurements,

(ii) Determine the expected range of values that might be observed by any laboratory,

(iii) Evaluate possible dependence of the results on method or environment variables, and

(iv) Identify causes for any laboratory-to-laboratory variations.

3.1 RDX Class 5 Type II Characterization

RDX in this study is Type II synthesized by the acetic anhydride (Bachman) process and generally contains ca. 10 wt% HMX as a by-product [15]. The HMX content has been verified by HPLC analysis [7]. The Military Specification for RDX Class 5 Type II is that a minimum of 97 wt% of the materi-

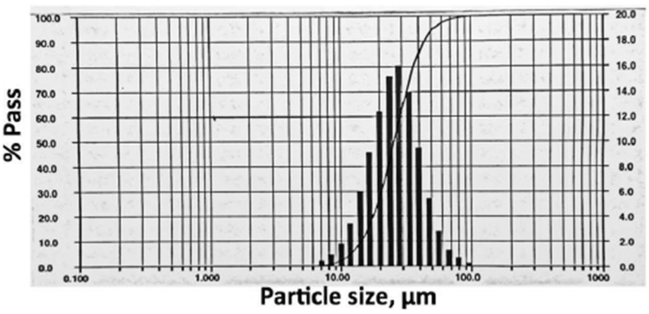

Figure 1. Particle size distribution for RDX Class 5 Type II.

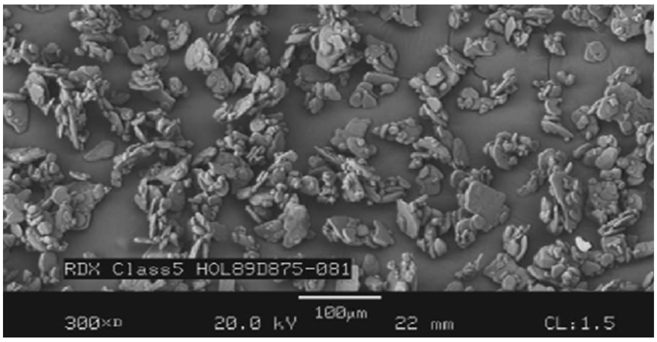

Figure 2. SEM of RDX Class 5 Type II at 300X magnification.

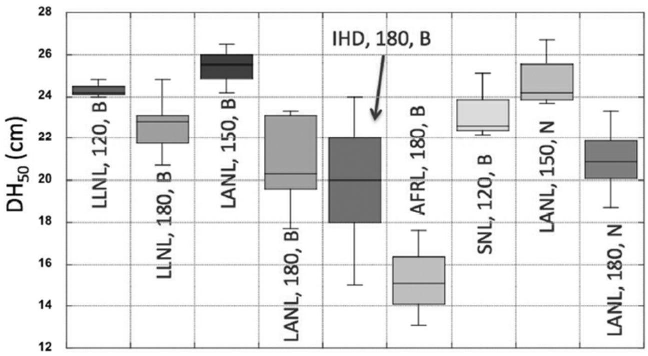

Figure 3. Box plot of the DH_{50} values of RDX Class 5 Type II grouped by participant, sandpaper grit size, and data reduction method.

als passes through 325-mesh (44 µm [16]) sieves [17]. 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 (average size 30 µm), which would pass through the 325-mesh sieve.

3.2 Impact Testing of RDX Class 5 Type II

All participants evaluated the RDX impact sensitivity using the Bruceton Up-Down method [18] to estimate the mean and standard deviation of the response function. In addition to the Bruceton method, LANL also evaluated the RDX using a Neyer D-optimal maximum likelihood method implemented in commercial software [10]. Notable differences in testing protocols among the participating laboratories are variation in sandpaper type, amount of sample, striker mass, and the methods for detection of a reaction.

3.2.1 Equivalency Characterization

Statistically, the question in this type of comparison is whether the impact test results from different participants are in agreement. In other words, given a few test results from each laboratory is there a statistically significant difference among the participants or does the pooled set of results appear to arise from natural sampling error that would occur with repeated identical measurements of a single system? Since the RDX material supplied to the participants was from one batch and prepared the same way, this analysis probes variation in the test methods and testing environments.

Due to significant differences in the test method details among laboratories, only the DH_{50} values are compared – the standard deviations produced by Bruceton analysis are not analyzed. The standard deviations are dependent on the number of tests, the step size, and the type of spacing (linear vs. logarithmic), many of which varied among the participants.

The impact results can be visually evaluated using box plots with the data divided into sets differentiated by the combination of testing laboratory, sandpaper type (as indicated by grit size, 120-, 150-, 180-) and evaluation method (Bruceton, B, or Neyer, N). Figure 3 shows these plots. Box plots are constructed so that the shaded region represents the middle 50% of the data; 25% of the data is found below the box, and 25% is found above the box. The horizontal line is the median of the DH_{50} values. The vertical lines extend to the maximum and minimum DH_{50} values. The mean of the data set is at the midpoint of the shaded area.

In Figure 3, two data points for LLNL 1-kg striker with 180-grit sandpaper were not included in the analysis. Striker mass was constant within each laboratory when not including this data and is not indicated to differentiate sets of results. Visual inspection suggests that the results range from symmetric to skewed and that a subset or subsets of the different groupings are likely in agreement with each other, based on overlap of the shaded regions and to some extent the maximum/minimum bars. The AFRL 180 data appears to be significantly separated from the rest.

Tukey and Fisher comparison analysis results at a 95% confidence level are shown in Table 1. Overall, many of the individual data sets are in agreement but, as expected, the AFRL 180 data set is different from the rest of the sets no matter how they are grouped.

Table 1. Groupings resulting from Tukey and Fisher comparison tests of RDX Class 5 Type II impact data.

Tukey	Data sets in grouping[a)]
Subgroup 1	LANL (150/B), LANL (150/N), LLNL (120/B), SNL (180/B), LLNL (180/B)
Subgroup 2	LANL (150/N), LLNL (120/B), SNL (180/B), LLNL (180/B), LANL (180/N)
Subgroup 3	LLNL (120/B), SNL (180/B), LLNL (180/B), LANL (180/N), LANL (180/B)
Subgroup 4	SNL (180/B), LANL (180/N), LANL (180/B), IHD (180/B)
Subgroup 5	AFRL (180/B)
Fisher	
Subgroup 1	LANL (150/B), LANL (150/N), LLNL (120/B)
Subgroup 2	LANL (150/N), LLNL (120/B), SNL (180/B), LLNL (180/B)
Subgroup 3	SNL (180/B), LLNL (180/B), LANL (180/N), LANL (180/B)
Subgroup 4	LANL (180/N), LANL (180/B), IHD (180/B)
Subgroup 5	AFRL (180/B)

a) Values in parentheses indicate type of sandpaper/analysis method (120 is 120-grit Si/C wet/dry sandpaper, 150 is 150-grit garnet sandpaper, 180 is 180-grit garnet sandpaper, B = Bruceton method, N = Neyer D-Optimal method).

The p-value resulting from the ANOVA treatment of the RDX DH_{50} impact data was 0.000. Based on this at least one of the data sets represented in Figure 3 is statistically different than the others and there is less than 0.1 % chance that this assessment is in error.

3.2.2 Expected Range of Observations

One useful outcome of a proficiency test is an assessment of the expected range of values that might be obtained by other laboratories carrying out nominally the same measurements in the future. This can be used as verification that future laboratories are capable of making this type of measurement adequately, hence the name "proficiency". In the IDCA context, the range of observations is probably more appropriately interpreted as the variability in the observations that may be expected. This can be used as a lower bound for expected variability in materials that are not as well behaved or as well characterized as RDX.

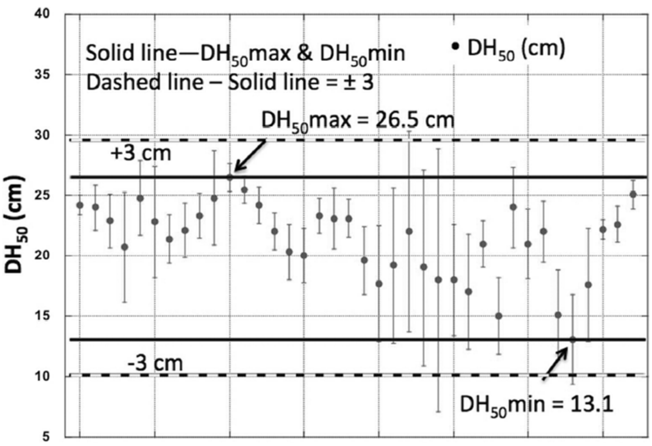

Figure 4. Comparison of individual RDX Class 5 Type II DH_{50} evaluations for all laboratories with estimated errors determined from the Bruceton or Neyer standard deviation values. Test number is arbitrary and for display purposes only (implies no order of testing).

Figure 4 shows the individual DH_{50} impact results from Bruceton or Neyer analysis. Included are the LLNL data taken using the 1-kg striker and 180-grit sandpaper and the AFRL data taken with 180-grit sandpaper, but not LLNL data from pressed pellets. Each point is the mean value for a particular data set and the error bars are the standard deviation calculated in the Bruceton or Neyer methods (complete listing of individual data sets are found in the Supporting Information). The standard deviations are not easily compared for reasons noted above. They are, however, often larger than the scatter observed in repeated measurements that produce the DH_{50} values and so they are a good representation of a worst-case estimate.

The range is illustrated in Figure 4 using solid horizontal lines that pass through the maximum and minimum observed mean values. This range is 13.1 (AFRL 180-grit sandpaper, Bruceton analysis) to 26.7 (LANL 150-grit sandpaper, Neyer analysis) cm. Because there were not very many tests leading to some of the data points in Figure 4, it is appropriate to take into account the standard deviations, and report a broadened range of the expected values. As an estimate, using the average standard deviation of all of the measurements, which is around 3 cm, broadens the range to 10.1 to 29.5 cm. This broadened range is highlighted using the dashed horizontal lines. Based on these results, a future laboratory using any instrument from very old to brand new and methods of detection ranging from operator to microphone, should expect results for this particular RDX to fall between 10.1 and 29.5 cm.

The mean of the values presented in Figure 4 is 21.0 cm and half of the un-broadened range is 6.7 cm or approximately 30 % of the mean, setting an expectation to observe similar percentage variability in testing other materials that have higher or lower mean DH_{50} values (such as HMEs).

Based on the ANOVA results presented above, it is appropriate to evaluate the same ranges and percent variability after removing the AFRL 180 data since it is statistically different from all of the other sets based on both Tukey and Fisher comparisons. With the AFRL data removed, the range of means is 15 to 26.5 cm with an average of 21.5 cm and a percent variability of 27%. The broadened range would be 12 to 29.5 cm.

3.2.3 Dependence on Method or Environment Variables

When replicate measurements are available, it is possible to compare the results against other parameters and look for relationships that suggest an influence due to a variable in the test method or in the local environment. For the RDX impact data set, this is complicated by the variability among laboratories. Fortunately, moving between any adjacent Tukey subgroups 1 through 4 includes all laboratories (except AFRL most of the time) and therefore it is appropriate to use all of the data (except the AFRL 180 set) to examine dependence on method or environment variables. The variables that were tracked by each laboratory include striker mass, sandpaper type (identified by grit size but includes other sandpaper properties), temperature, and relative humidity. Figure 5 shows individual DH_{50} values (determined by Bruceton or Neyer methods) as a function of specific variables. None of the variables showed an influence on the DH_{50} with the possible exception of sandpaper type, which has been shown to matter in studies of other materials [2]. The data set is not large enough to be conclusive about the dependence at this point.

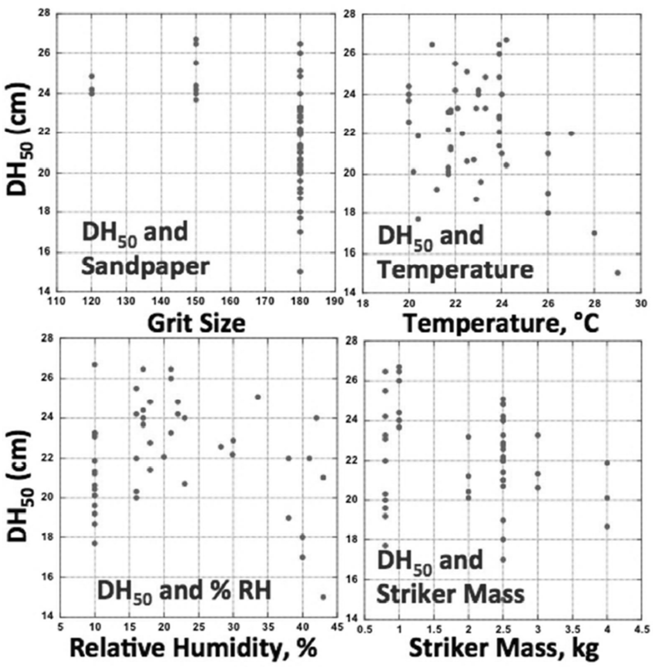

Figure 5. Comparison of RDX Class 5 Type II DH_{50} values with various method and environment variables.

3.3 Analysis of BAM Friction Testing of RDX Class 5 Type II

The BAM friction testing of RDX was performed by LANL, LLNL, IHD, and SNL (AFRL does not have BAM friction). The notable differences in test methods among the participants are the methods for positive reaction detection and the environment surrounding the instrument. LANL, LLNL, IHD, and SNL performed data analysis using the TIL method [11]. LANL, LLNL, and IHD also used a modified Bruceton method [9,18] and IHD used the Neyer method [10] on Data set 2 because their data did not meet Bruceton criteria (analysis performed by LANL). SNL did not carry out a Bruceton evaluation with their instrument.

3.3.1 Equivalency Characterization

Figure 6 (left side) shows the BAM friction data for RDX for each participant, presented as box plots. Box plots are constructed so that the shaded region represents the middle 50% of the data; 25% of the data is found below the box, and 25% is found above the box. The horizontal line is the median of the F_{50} values. The vertical lines show the maximum and minimum F_{50} values. The mean of the data set is at the midpoint of the shaded area. The smaller number of sets visually might imply better agreement between the participants as compared to the impact (DH_{50}) results, however, the bulk of the LANL and LLNL data are still significantly offset from each other.

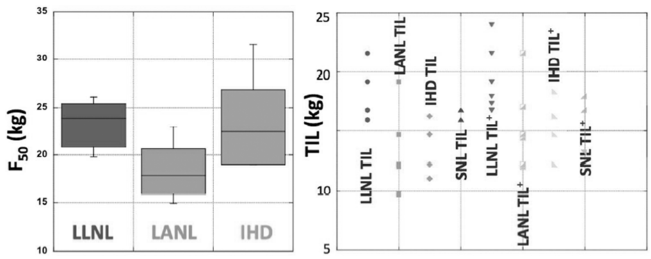

Figure 6. Left side: comparison of F_{50} results for RDX Class 5 Type II represented as box plots; right side: comparison of RDX Class 5 Type II TIL results. All data acquired using BAM friction apparatus.

The right side of Figure 6 shows the TIL results. TIL data is more difficult to compare because not all the participants used equal numbers of test events (trials and No-Go events) to evaluate the threshold levels. As a result, directly comparing the TIL values and the level above TIL (TIL+) better analyzed for possible trends in the data. Figure 6 compares the TIL and TIL+ values determined by BAM friction for LANL, LLNL, IHD, and SNL data. The first four point types are TIL (TIL 0) data and the last four are TIL+ (TIL 1) data.

Although the TIL data sets overlap, the LLNL data points appear higher than the others, implying that LLNL found the RDX less sensitive than the other participants. The same holds true for the TIL+ values, LLNL appears to find the RDX less sensitive than the other participants.

Table 2 shows the ANOVA results for F_{50} and both TIL data sets along with the various subgroups determined by

Table 2. ANOVA and subgrouping results for BAM friction testing of RDX Class 5 Type II.

	ANOVA p-value	Tukey subgroups	Fisher subgroups
F_{50}	0.000	1. IHD, LLNL 2. LANL, IHD	1. IHD, LLNL 2. LANL, IHD
TIL	0.000	1. LLNL, SNL 2. SNL, IHD, LANL	1. LLNL, SNL 2. IHD, LANL
TIL+	0.001	1. LLNL, SNL 2. SNL, IHD, LANL	1. LLNL, SNL 2. SNL, IHD, LANL

Tukey and Fisher analysis methods that are in apparent agreement. For the F_{50} determination, LANL appears to differ from both LLNL and IHD with a p-value of 0.000 although IHD forms a subgroup with either. For TIL values, the participants differ with a p-value of 0.000 and the subgroups each include at least two participants. For TIL+, the participants differ with a p-value of 0.001 and subgroups are similar to those for TIL 0. In both TIL 0 and TIL+, SNL is on the borderline between two groups and may be included in both. These p-values results indicate that there is a data set or sets that are different with a 0.1 % chance that this assessment is a Type I error.

3.3.2 Expected Range of Observations

For the BAM friction F_{50} data, because there were only three participants contributing, all of the data was evaluated together to assign a range of expected values from 14.9 to 31.6 kg. The mean value of all of the F_{50} measurements is 21.0 kg so that half of the range represents a variability of 40 %. The standard deviation of the F_{50} measurements is only 4 kg, which is 19 % of the mean. The 40 % variability is roughly equal to two standard deviations.

For the TIL 0 data, the range runs from 9.6 to 21.6 kg. The mean TIL 0 value is 14.2 kg with a standard deviation of 3 kg. Using half of the range, the expected variability among the participants is 6 kg, or about 42 % of the mean, and again about 2 standard deviations.

For the TIL+ data, the range is from 12 to 24 kg. The mean TIL+ value is 16.0 kg with a standard deviation of 2.9 kg. Using half of the range, the expected variability among the participants is also 6 kg, or about 38 % of the mean, and again close to 2 standard deviations.

3.3.3 Dependence on Method or Environmental Variables

There are sufficient data to be able to compare the results against other parameters and look for relations that suggest an influence due to a variable in the test method or in the local environment. The variables considered were temperature and humidity. Because all of the instruments and friction pin/plate surfaces were the same (not accounting for near due to use), no other variables were considered. Figure 7 shows the comparison of the F_{50}, TIL, or TIL+ as a function of temperature and relative humidity. No dependence on either variable is evident in Figure 7. This im-

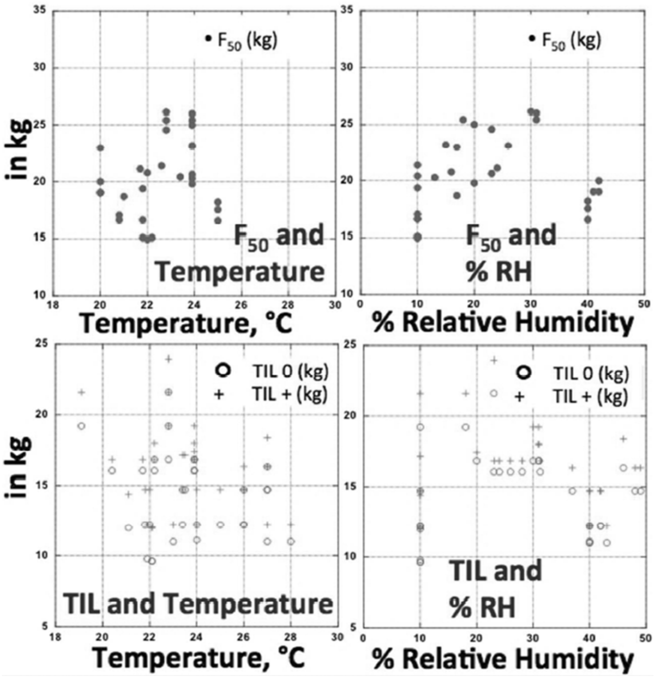

Figure 7. Comparison of F_{50} and TIL results for RDX Class 5 Type II with various method and environment variables.

plies that there is no dependence of these measurements on method or environment variables except for those variables that are inherent to each laboratory and therefore captured in the ANOVA analyses. For BAM, these inherent variables are the operator, background environment (including insulation), and method of reaction determination, which are all interrelated.

3.4 Analysis of ABL ESD Testing of RDX Class 5 Type II

Electrostatic Discharge (ESD) testing of the RDX Class 5 Type II was performed by LLNL, LANL, and IHD. Differences in the testing procedures among the participants are the use of tape and other sample containment. All participants performed data analysis using the TIL method [11]. LLNL used a custom built ESD system with a 510-Ω resistor in line to simulate a human body for data sets 1 and 2, and a new ABL system for data sets 3 and 4. Other participants used older ABL systems.

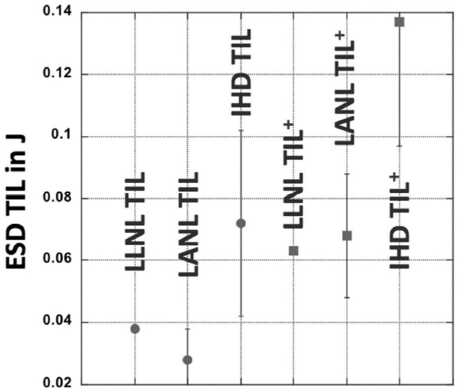

Figure 8. Average TIL and TIL+ values for RDX Class 5 Type II ESD results.

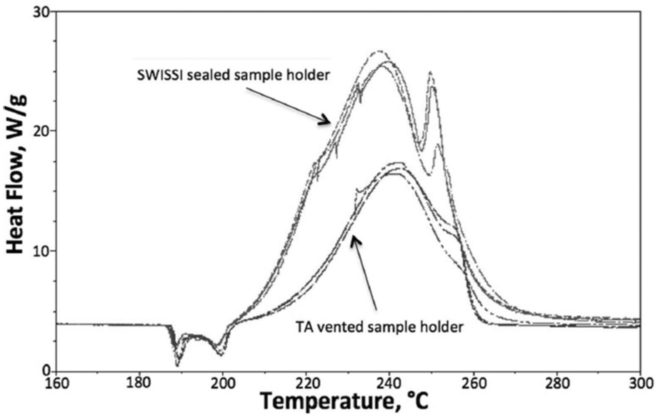

Figure 9. Example DSC scans of RDX Class 5 Type II in typical pinhole hermetic pans and one type of sealed pan.

The ESD results from the participating laboratories are much more coarse grained than the data sets for impact or friction because of the step levels used in the testing, therefore it is not informative to create scatter or box plots for this data. ANOVA analysis is also not useful because of the discreteness and clustering of the TIL and TIL+ levels. The LLNL data taken with the custom built system was not included, but was the obvious main difference due to the 510-Ω resistor in the circuit, while the ABL systems were operated with no resistors in the circuits. This has been analyzed previously [2].

Figure 8 shows a comparison of average TIL values with standard deviations and still illustrates a difference, which is the higher TIL or TIL+ values from IHD compared to the corresponding values from LANL and LLNL. This difference shows the degree of separation between IHD and other participants. In each case, the standard deviations do not even overlap. This shows that the measurements made by IHD are not equivalent to those made by the other participants.

For this data set, there is a possible link between an environment variable and the results. The IHD results were also obtained at roughly twice the relative humidity compared to the humidity at LLNL and LANL. Without more testing at different humidity levels, it is not possible to definitively say that this leads to the higher TIL values, but it is an understandable correlation since electrostatic effects are greatly influenced by humidity [19].

Assuming that the IHD results are due to humidity variation, it is natural to group all participants together to assess the range of possible values that might be obtained by other laboratories attempting to carry out nominally the same type of ESD testing without a tightly controlled laboratory environment. In this case, the TIL range is from 0.025 to 0.095 J with an average of 0.051 J and a standard deviation of 0.030 J. Using either the range or the standard deviation implies greater than 50% variability, which is not very useful as a metric. For TIL+, the range is from 0.0625 to 0.165 J with a mean of 0.099 J and a standard deviation of 0.046 J. The range and standard deviation again imply a very large variability. For reporting and comparison purposes, the range itself is a more useful metric to assess any future measurements.

3.5 Analysis of Thermal Testing of RDX Class 5 Type II

Differential Scanning Calorimetry (DSC) was performed on the RDX Class 5 Type II by LLNL, LANL, and IHD. All participating laboratories used different versions of the DSC by TA Instruments. Results were obtained at a 10 °C min^{-1} heating rate.

Figure 9 shows typical DSC scans for RDX Type II Class 5 using the pinhole hermetic pans and one type of sealed pan. The principal features of the DSC examinations are essentially the same from all participants – two overlapping low temperature endothermic features near 200 °C and a major exothermic feature near 240 °C. LLNL and IHD were able to examine the RDX using both open sample holders and sealed sample holders.

Some insight into the results can be gained by grouping the data into categories and calculating simple averages and standard deviations. Table 3 summarizes these calculations. The data were grouped in the following manner:

(1) The IHD enthalpy data was not used from the measurements when pinhole sample holder ruptured.

(2) Some temperatures were approximated directly from the hard copy of the profile. For the calculations the number was taken at the face value.

(3) Some data were grouped as LLNL pinhole data ("old" for older TA Instruments equipment).

(4) Some data were grouped as IHD and LANL pinhole data ("new" for newer TA Instruments equipment).

(5) Some data were grouped as LLNL sealed sampled holder data (this is really a TA instruments sample without the pinhole – it is not really meant for high pressure work).

(6) Some data were grouped as IHD sealed (which is the SWISSI sample holder, which is meant for high pressure work).

Table 3. Ranges of DSC parameters for RDX Class 5 Type II.

Parameter[a)]	Pinhole old[b)]	Pinhole new[b)]	Sealed LLNL[c)]	Sealed IHD[d)]
Endothermic onset [°C]	187.3–187.8	187.4–188.6	187.3–187.8	186.1–187.8
Range (average)	(187.7 ± 0.2)	(188.0 ± 0.2)	(187.6 ± 0.2)	(187.4 ± 0.6)
Endothermic minimum [°C]	188.3–189.2	188.7–189.9	188.3–189.1	188.3–190.0
Range (average)	(188.9 ± 0.3)	(189.4 ± 0.3)	(188.8 ± 0.3)	(189.3 ± 0.8)
Endothermic minimum [°C]	198.8–200.0	198.6–200.8	198.8–199.4	198.1–199.8
Range (average)	(199.2 ± 0.3)	(199.9 ± 0.5)	(199.0 ± 0.2)	(199.1 ± 0.8)
Endothermic enthalpy [$J\,g^{-1}$]	126–181	92–146	114–144	92–123
Range (average)	(142 ± 15)	(128 ± 14)	(133 ± 9)	(102 ± 11)
Exothermic onset [°C]	203–219	201–225	203–220	209–215
Range[e)]				
Exothermic maximum [°C]	238.7–243.5	239.8–244.2	230.6–244.0	237.4–241.9
Range (average)	(241.6 ± 1.4)	(242.3 ± 1.0)	(235.3 ± 3.9)	(239.8 ± 1.8)
Exothermic enthalpy [$J\,g^{-1}$]	1890–2432	1947–2385[f)]	2003–3805	4203–4662
Range (average)	(2244 ± 177)	(2174 ± 120)	(3108 ± 495)	(4423 ± 179)

a) Onset is the beginning of the maximum or minimum as automatically identified by the equipment, endothermic mininmum is the minimum temperature of the endothermic feature, endothermic enthalpy is the overall enthalpy of the two overlapping endothermic features, exothermic maximum is the maximum of the exothermic feature. b) TA Instruments vented sample holders. c) TA Instrument hermetically sealed sample holder (not pressure rated). d) SWISSI high-pressure sample holder (rated 217 bar at 400 °C). e) Range given only because the transition between the endothermic and exothermic features overlap. f) Two values from IHD Set 2 discarded due to sample holder rupturing during experiment.

The transition between the endothermic and exothermic features is only listed with temperatures because the transitions overlap (exothermic onset in Table 3).

Examination of Table 3 shows that the pinhole and the sealed sample holders show differences in exothermic enthalpies – pinhole different than sealed; LLNL sealed different than the SWISSI sample holder (see Ref. [20] for description). The sealed sample holders show higher enthalpies of decomposition in each case because they do not allow gas to escape. The pinhole sample holders allow gas to escape at a controlled rate that removes heat from the system, lowering the total observed enthalpy. Comparing the two sealed sample holders shows that they are distinctly different [20]. IHD used the SWISSI sample holder; the values for the enthalpy assigned to the exothermic feature are much higher than with the corresponding enthalpy for samples measured in the sealed sample holder that LLNL uses. This is probably due to the fact that the SWISSI sample holder is rated to hold 217 bar (3150 psi) at 400 °C, whereas the LLNL sealed sample holder is not pressure rated. As a result, the LLNL sample holder probably allows some volatile gases escape, therefore cooling the sample.

The endothermic enthalpy is less for the SWISSI sample holder than for the TA sample holder – probably due to the more massive SWISSI sample holder. The SWISSI sample holder weighs more (ca. 1 g) and the mass is distributed differently because of the sealed design than the TA sample holder (ca. 0.1 g). As a result there is a shift of the response to slightly later times that affects the endothermic response (because of overlap with the much larger exothermic feature), so some of the response is lost in the exothermic transition. This has been seen before in DSC profiles of ammonium nitrate [21].

The temperature of maximum exothermic enthalpy is lower for the sealed pans than for the open pans. Two mechanisms occur in the pinhole sample holders during this heating period that are counter to each other – evaporation, which is endothermic, and decomposition, which is exothermic. These mechanisms compete causing the temperature of maximum exothermic enthalpy to be higher in the pinhole sample holder.

4 Discussion

4.1 Statistical Analysis Results

The analyses above allow an assessment of the statistical differences among participants, average values, expected ranges, percent variability, dependence on method or environment, and possible causes for the differences that are observed. These are summarized in Table 4 for the various sensitivity and thermal tests. DH_{50} data from AFRL was excluded from this table since it showed up as a separate group in both Tukey and Fisher comparisons. No other data was excluded from Table 4.

The information in Table 4 shows that there is a statistically significant difference among the participants in all of the tests, whether evaluated by ANOVA or inferred by examination of the specific test results presented above. This is not pointing out deficiencies in the test methods but is highlighting the variability that can result from individual laboratories implementing detailed procedures within bounding facility conditions and within testing guidelines established from previous experience. This variability can be detected and quantified and may be used to determine

Table 4. Results of statistical analyses of IDCA Small-Scale Safety Testing of RDX Class 5 Type II.

	Equivalent results?	p-value from ANOVA	Average	Range	Percent variability	Dependence on method or environment variables	Possible causes of differences
Impact DH_{50}	No	p = 0.000	21.5 cm	15–26.5 cm	27	Possibly grit	Operator, Detection method
BAM friction F_{50}	No	p = 0.001	21.0 kg	14.9–31.6 kg	40	No	Operator, Background noise
BAM friction TIL	No	p = 0.000	14.2 kg	9.6–21.6 kg	42	No	Operator, Background noise
BAM friction TIL+	No	p = 0.004	16.0 kg	12–24 kg	38	No	Operator, Background noise
ESD TIL	No	p = N/A	0.051 J	0.025–0.095 J	N/A	Possibly RH	Detection method, Age of instrument
ESD TIL+	No	p = N/A	0.099 J	0.0625–0.165 J	N/A	Possibly RH	Detection method, Age of instrument
DSC thermal	Yes	p = N/A	See Table 3	See Table 3	N/A	No	Sample holder type

whether a new laboratory is capable of making equivalent measurements if that becomes a goal for future efforts.

For present IDCA purposes, the statistical difference also implies that it may be possible to ultimately determine the cause of variability in the results if enough details about the testing are tracked during future round-robin examinations. All of the test parameters, instrument details, sample characteristics, and environment conditions will be important to track if this goal is undertaken.

The information in Table 4 also shows the ranges that can be expected for other materials with DH_{50}, F_{50}, or TIL values near those for RDX. Translated to percent variability, these can suggest what might be expected when testing material at much higher or lower sensitivity values. This will be important to help understand whether differences observed with HME materials in future reports are truly significant.

The largest factors causing differences among participants appear to be the operator, method of detection, and testing environment. Sometimes these factors are inextricably linked, such as when the operator *is* the method of detection and the perception (recognition of a positive event) is limited by background noise in the laboratory. This is the case for BAM friction in which LANL vs. LLNL differences are due to operator and environment. For these tests there is no transducer, the LLNL friction machine has more shielding, and it is operated with a vent fan during use (see Ref. [2] for photo). In other cases, such as with the DH_{50} differences between LLNL and LANL, only detection method and environment play a role since both participants use threshold sound levels to make Go vs. No-Go determinations. Differences in how these threshold levels are chosen may create an offset between DH_{50} values. The issues associated with operator-influenced results are being addressed informally at various testing laboratories through implementation of transducer-driven Go/No-Go discrimination and more formally by commercial entities, which are developing full systems that integrate the test instrument, electronic detection methods, and result analysis.

4.2 Comparison with other Results

Table 5 compares selected impact data on RDX with some average results from the proficiency test. Selected were data on Type II RDX taken with Explosives Research Laboratory (ERL) or Modified Bureau of Mines (MBOM) equipment and evaluated by the Bruceton method. The 50 % value is the 50 % probability of reaction or H_{50} or DH_{50}. These 50 % probability of reaction data fall within the expected range of 15 to 26.5 cm discussed in this report. This range is also similar to the 16 to 25 cm range found previously in the reduced sensitivity RDX (RS-RDX) study limited to ERL and MBOM equipment on Type II RDX [22].

Data for other types of sensitivity are not so forth coming for comparison except for thermal (DSC) data. The RS-RDX round robin also studied the thermal behavior of various preparations of RDX [23]. The results in Table 3 for the low temperature transitions are consistent with the RS-RDX results on Dyno Type II and OSI Type II preparations when corrected for heating rate, and also are consistent with the phase diagrams in HMX and RDX mixtures pub-

Table 5. Selected RDX impact data (RDX Type II; ERL or modified to ERL equipment only).

Source[a)]	50 % value [cm]
RS-RDX Laboratory 5 (ERL)[b)]	19[c)]
RS-RDX Laboratory 5 (ERL)[b)]	20[d)]
RS-RDX Laboratory 6 (ERL)[b)]	22[d)]
RS-RDX Laboratory 6 (ERL)[b)]	21[e)]
RS-RDX Laboratory 7 (MBOM)[b)]	19[c)]
RS-RDX Laboratory 7 (MBOM)[b)]	18[d)]
NSWC Kamlet (ERL)[e)]	25[f)]
LANL Gibbs and Popolato (ERL)[g)]	22.2[f)]
Proficiency Test LLNL (ERL)[h)]	22.6[i)]
Proficiency Test LANL (ERL)[h)]	20.9[i)]
Proficiency Test IHD (ERL)[h)]	19.7[i)]
Proficiency Test SNL (MBOM)[j)]	23.3[k)]

a) 1. RS-RDX is the reduced sensitivity RDX round robin study of the early 2000s. b) Ref. [22]. c) RDX Source Ordinance Systems Inc. d) Dyno Nobel. e) Ref. [23]. f) RDX source not specified. g) Ref. [24]. h) Average of all RDX data taken during the proficiency test by LLNL or LANL or IHD. i) Ref. [25]. j) SNL single determination. k) Ref. [26].

lished previous [27–30] – the endotherm at ca. 188 °C is probably a RDX/HMX eutectic and the endotherm at ca. 199 °C is RDX melting. However, this study did not see the variability and reproducibility issues for these low-temperature transitions that the RS-RDX study observed.

The more prominent transitions are consistent with previous studies on Type II RDX from the RS-RDX round robin also [31–33], where the exothermic transition at ca. 240 °C is due to RDX and the shoulder at ca. 250 °C is due to HMX. However, in these studies, the high temperature shoulder is not nearly as well resolved as in Figure 9 above.

5 Conclusions

The RDX results of this paper validate the former assessment that HME materials evaluated by SSST testing are sensitive to the differences in the test methods and equipment employed by each laboratory. This further accentuates the expectations that differing evaluations of sensitivity are significant from a safety standpoint. Some of these differences can be eliminated by standardization, but others are inherent in the configurations and environments each laboratory has established to safely test energetic materials. Elimination of the differences will require further research, however. This work has shown that, even when a specific standard is carefully tested, variation in results occur and that it is important to be able to test materials under a variety of conditions because of the multiple types of insults possible to these materials. Exploring a range of variables provides the best chance of probing the particular set of test parameters that highlight the extent of sensitivity of the material. Sandpaper properties, striker mass, and the method of detecting the generated sound or reaction are all examples of important variables, and parameter variation is the topic of subsequent papers.

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