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# Integrated Data Collection Analysis (IDCA) Program - Methods Report

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March 25, 2013

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This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

# Integrated Data Collection Analysis (IDCA) Program —SSST Testing Methods

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## ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the methods used for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis during the IDCA program. These methods changed throughout the Proficiency Test and the reasons for these changes are documented in this report. The most significant modifications in standard testing methods are: 1) including one specified sandpaper in impact testing among all the participants, 2) diversifying liquid test methods for selected participants, and 3) including sealed sample holders for thermal testing by at least one participant.

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

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



# 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<sup>1</sup>. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues when dealing with HMEs. Many of these challenges are not normally encountered with military type explosives. To a large extent, the issues are centered on the physical forms and stability of the improvised materials.

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

**Table 1. Materials for IDCA Proficiency study**

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

<sup>1</sup>. Simulates diesel fuel; <sup>2</sup>. Contains 3 wt. % cornstarch; <sup>3</sup>. Sieved to pass through 100 mesh; <sup>4</sup>. Alliant Bullseye<sup>®</sup> smokeless pistol gunpowder.

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

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

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

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

In this report, the IDCA test methods and equipment are documented throughout the Proficiency Test. Changes to the methods and testing have been made during the Proficiency Test to better accommodate specific materials. Changes to these test methods with explanations why are also documented.

This report was prepared after the participants had tested all the materials and after these analysis reports had been issued: RDX first testing<sup>2</sup>, RDX second testing<sup>3</sup>, RDX testing comparison<sup>4</sup>, KClO<sub>3</sub>/sugar (separated with a 100 mesh sieve)<sup>5</sup>, KClO<sub>3</sub>/sugar (as received)<sup>6</sup>, KClO<sub>3</sub>/Dodecane<sup>7</sup>, KClO<sub>4</sub>/Dodecane<sup>8</sup>, KClO<sub>4</sub>/Al<sup>9</sup>, KClO<sub>4</sub>/Carbon<sup>10</sup>, NaClO<sub>3</sub>/sugar<sup>11</sup>, and PETN<sup>12</sup>.

## 2 METHODS

*General information.* The descriptions below capture the standard methods used in the Proficiency Test. These methods evolved during the testing and the rationale will also be given in the Discussion section. The major standard tests are impact, performed by the drop hammer method, friction (BAM and ABL), ESD and DSC. Also, all samples were prepared according to IDCA methods on drying and mixing procedures<sup>13,14</sup>.

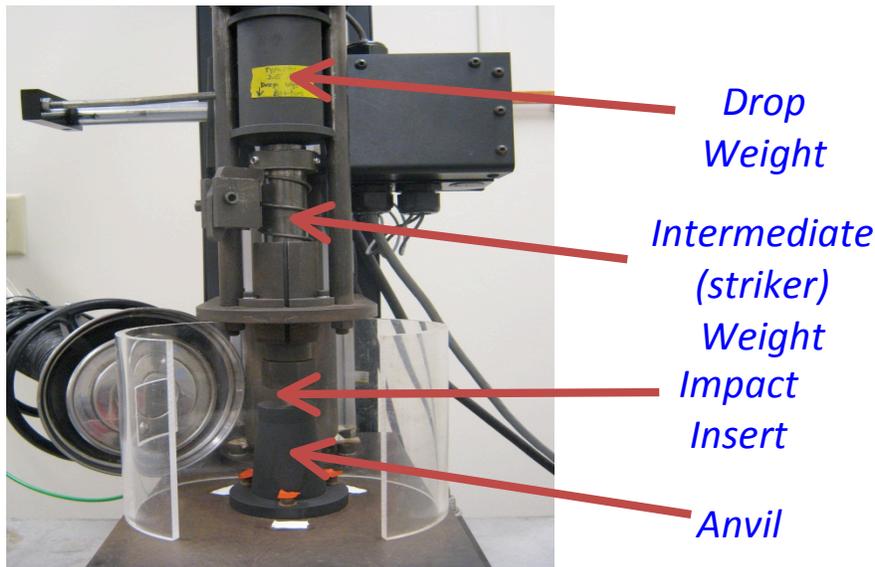
### 2.1 Impact Testing

Impact testing is performed by the drop hammer method. This method is fairly simple and straightforward and has been around in some form since the early 1900s<sup>15</sup>. The test is used to establish the sensitivity to impact either by dropping the material or something dropping on it. There are variations on how this is accomplished, but the primary mechanism for this is to drop a weight on a small sample of the material, and monitor for a reaction.

Figure 1 shows the drop hammer equipment used by the participants in the Proficiency Test. The drop hammer used by LANL is the ERL Type 12 Drop Weight, custom built in 1954. The drop hammer used by AFRL is the MBOM modified Type 12 Drop Weight, purchased in 2009. The drop hammer used by SNL is the MBOM modified Type 12 Drop Weight, purchased in 2012. The drop hammer used by LLNL is the ERL Type 12 Drop Weight, custom built in 1956. The drop hammer used by IHD is the ERL Type 12 Drop Weight, custom built in 1945 and is the oldest of the drop hammers used in this study.



**Figure 1. Drop hammer equipment used in Proficiency Test**



**Figure 2. Close up of drop hammer reaction area (AFRL Drop Hammer)**

*Drop hammer experimentation.* In general, the sample size is  $35 \pm 2$  mg. Most samples were prepared as either a loose powder, liquid or loose slurry. LLNL also examined pelletized RDX in a few cases. The

sample is placed on an anvil. If liquid, the containment is by various methods that will be discussed below. Solid and sometime liquid samples are held in place with sandpaper. The drop weight is dropped at variable heights until reaction is noted. For LLNL and LANL, a microphone is used for detection. For IHD, AFRL and SNL, observation is used for detection. A reaction is noted as a pop, flash or smoke and the sensitivity is reported in cm (height of the drop weight). The drop weight is 2.5 kg and the striker weight 0.5 to 2.5 kg (LLNL, 2.5 kg for solids, 1.0 kg for liquids; LANL, 0.8 kg for all; IHD, SNL, 2.5 kg for all; AFRL 1.0 and 2.5 kg).

The experiment for solids is the following: the sample is placed on sandpaper that is on the anvil. The striker weight is lowered onto the sample and secured in place. The drop weight is raised to a beginning height and then dropped. If no reaction occurs, the drop weight is raised to a higher height. If a reaction occurs, the drop weight is raised to a lower height.

**Table 2. Drop hammer conditions for Proficiency Test**

	LANL	LLNL	SNL	IHD	AFRL
Test Apparatus	ERL	ERL	MBOM	ERL	MBOM
Drop hammer weight, kg	2.5	2.5	2.5	2.5	2.5
Striker weight, kg	0.8	1.0 <sup>4</sup> ; 2.5	2.5	2.5	1.0; 2.5
Powder samples <sup>1</sup>					
Sample Size, mg	35 ± 2	35 ± 2	35 ± 3	35 ± 2	35 ± 2
Form	powder	pressed or powder	powder	powder	powder
Powder configuration	conical pile	conical pile	conical pile	conical pile	conical pile
Sandpaper Grit	150 <sup>2</sup> ; 180 <sup>2</sup>	120 <sup>3</sup> ; 180 <sup>2</sup>	180 <sup>2</sup>	180 <sup>2</sup>	180 <sup>2</sup>
Size	1.25" disk dimpled	1" square	1" square	1" square	1" square
Liquid Samples					
Sample size	1 drop	1 drop	1 drop	1 drop	1 drop
Anvil Surface	sandpaper, greased anvil	silicon-greased anvil	not specified	cavity	sandpaper, bare/greased anvil
"Go" "No-Go"	microphone	microphone	observation	observation	observation
Number of Drops	25	15, 20-30	20-25	25	25
Maximum Height, cm	320	177	115	320	116
Data Analysis <sup>5</sup>	B and N	B	B	B	B and N

1. Dried by IDCA procedures; 2. garnet; 3. Si/C; 4. For liquids; 5. B = Modified Bruceton, N = Neyer D-Optimal

*Drop hammer experimental conditions.* Table 2 shows the parameters used in the drop hammer experiments for all participants. The principal differences are:

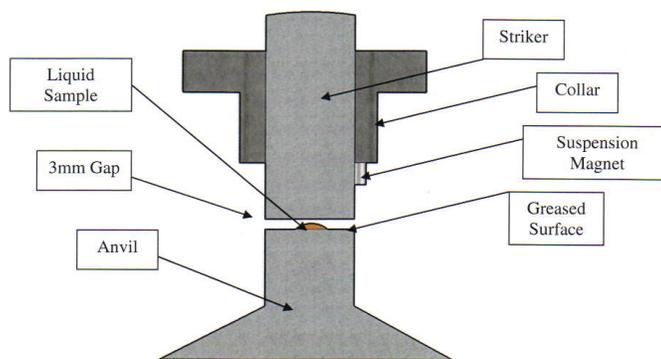
1. Types of equipment (ERL vs. MBOM configured for Type 12)
2. Striker weights (LANL uses 0.8 kg exclusively; LLNL uses 1.0 kg for liquids and 2.5 kg for solids; AFRL can use any between 1.0 and 2.5 kg, but usually uses 2.5 kg)
3. Form (LLNL has done some experiments with pressed—no pellet density recorded)
4. Sandpaper (LLNL and LANL use 120-grit Si/C and 150-grit garnet, respectively for standard method; this changed in the first 3<sup>rd</sup> of tests when everyone decided to use 180-grit garnet from the same source)
5. Tooling for liquids compared to solids (striker weights, anvil configurations)
6. Liquid anvil surface (four types—sandpaper, grease, bare, and cavity)

7. Positive reaction (LLNL and LANL use microphones, IHD, AFRL and SNL use observation)
8. Maximum height of drop (SNL < AFRL < LLNL < LANL = IHD)
9. Data reduction (LANL and AFRL use modified Bruceton and Neyer D-Optimal)

*Liquid testing.* Table 2 shows four different test methods for liquids. For LLNL and AFRL the configuration is a bare anvil with grease to contain the liquid. For LANL and IHD, the configuration is using a custom-built cell system. The IDCA decided at the beginning of the Proficiency Test that the participants would use their preferred method for analysis. As testing progressed, it became necessary for a selected laboratory to perform multiple testing methods when possible to establish cross performance metrics.

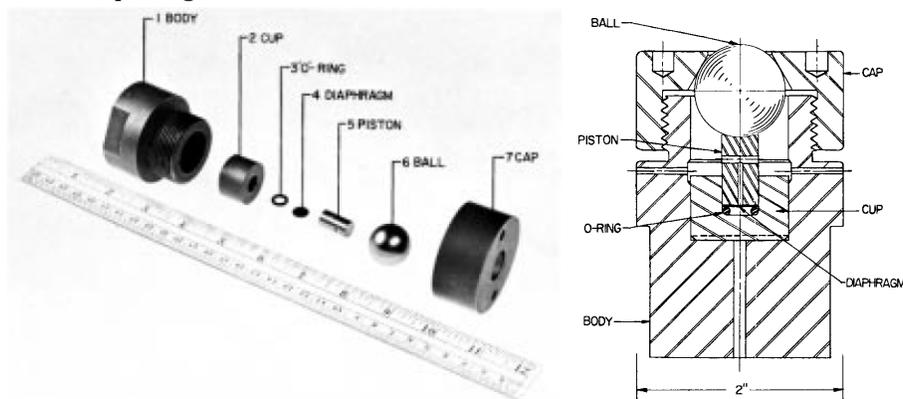
LANL uses an in house designed liquid test configuration. Figure 3 shows a drawing of this test configuration. This is the standard method used by LANL for routine examination of liquids.

**Type 12B Liquid Impact Configuration**



**Figure 3. Liquid test cell used by LANL for the Proficiency Test**

In this test cell, the liquid material is placed on an anvil, and it is held in place by a coating of grease on the anvil (grease must not be soluble in liquid). The striker, which normally is in contact with a solid sample, is set 3 mm above the liquid sample. If it was placed directly on the sample, the liquid could never support the weight, and would be forced off the anvil. The striker weight is kept in position by a magnet attached to the bottom of a collar that goes around the striker. The magnet weakly holds the striker so when the drop weight contacts the striker, the striker will move to the anvil<sup>16</sup>.



**Figure 4. Cavity cell for testing of liquids by IHD in the Proficiency Test (left side, photograph; right side ASTM D 2540-93 drawing)**

IHD employs a cavity cell that was first designed in the 1950s<sup>17,18</sup>. Figure 4 shows the design from the original ASTM specification. The cell allows for the liquid sample to be contained, avoiding leaking problems that occur with a bare anvil. A piston is held in place above the liquid sample. When the drop weight contacts the cell, the piston is released and transfers the energy to the sample. This design has the advantage of containing the sample in a reproducible way. The disadvantage for the testing is that IHD does not run the test with drop heights higher than 50 cm. As well, the sample cell offers containment that could lead to reactions that might not happen in other liquid test designs.



**Figure 5. Far view of drop hammer anvil surface (left side); liquid test surface uses a grease to hold sample in place (right side).**

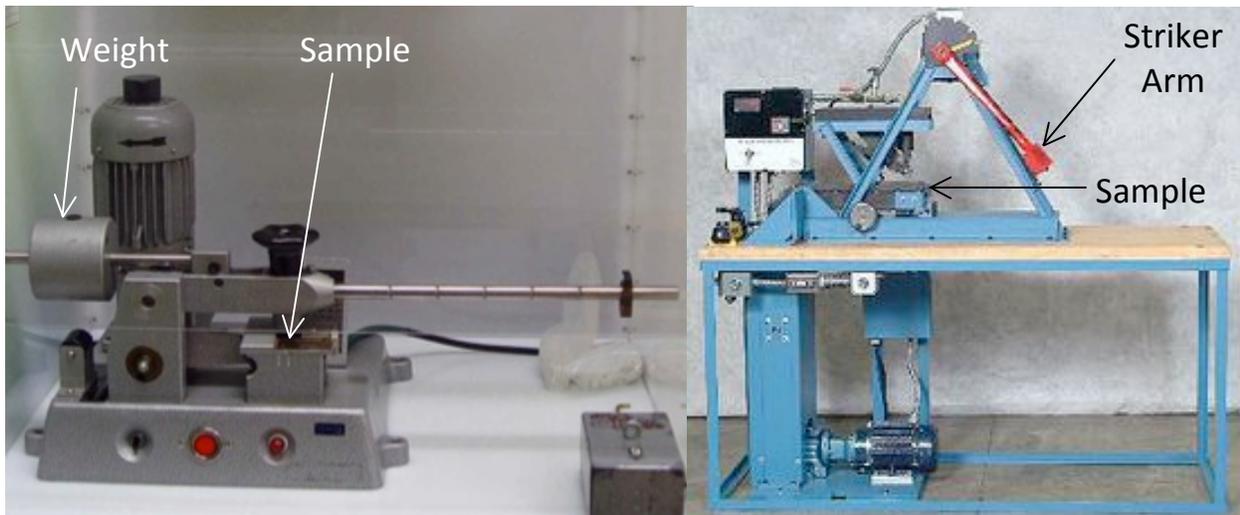
LLNL, AFRL and LANL all use grease to hold the liquid sample in place on the bare anvil. Figure 5 shows on the left side is a photograph of a bare anvil surface for of a Type 12A drop hammer anvil (provided by SMS-ink<sup>19</sup>). The grease is used in a variety of manners. LLNL and AFRL make a ring that surrounds the liquid drop at roughly the diameter of one drop. The grease is placed on the surface at a diameter of roughly one drop (0.5 mm). Usually the grease is Dow Corning High Vacuum Silicon grease (low flammability), but this can be varied depending upon the compatibility with the liquid. LANL coats the entire surface of the anvil. If the liquid does not dissolve in the grease, the droplet shape is retained. Figure 5 on the right side shows a typical configuration for a LANL test.

## 2.2 Friction Testing

Friction testing is performed by using two very old methods—BAM and ABL. The difference between these two methods comes from the design of the equipment, which is based on methods also developed in the early 1900s. The methodologies were brought into official form in the 1950s<sup>15</sup>.

From the onset of the Proficiency Test, LLNL, LANL and IHD have used BAM Friction equipment while IHD and AFRL have used ABL Friction equipment. IHD is the link between the two methods because they test with both types of equipment. SNL joined the testing much later with BAM friction equipment. All the BAM equipment has the same design, just different vintages. The same is true for the ABL equipment. However, the BAM is motor driven via a crank wheel (stretch yoke) and the ABL can either be motor driven (low speed) or impact driven (high speed).

*Friction Experimentation.* The left side of Figure 6 shows the BAM friction test configuration. The test is fairly simple. A sample, 5 to 40 mg, is placed in a ceramic sample anvil and a ceramic pin is dragged across the sample. The force is applied by the weight hanging on the armature, seen in the figure on the far right, and is varied to cause reaction. The reaction is a pop, smoke or jetting and the sensitivity is reported in kg (based on value of the weight). Note: the tooling is not well controlled for BAM.



**Figure 6. BAM friction (left side) and ABL Friction (right side) equipment used in the Proficiency Test**

**Table 3. Friction test equipment configuration for Proficiency Test**

	LANL	LLNL	SNL	IHD	AFRL
Test Apparatus	BAM, ABL	BAM	BAM	BAM, ABL	ABL
Go/No-Go	Observation	Observation	Observation	Observation	Observation
Lights	On	Off	On	On, Off	Off
Sample Size <sup>1</sup> , mg	~ 5	~ 5	5 ± 3	40 ± 2	5 ± 2
Solid Sample	Powder, Circle	Powder, Circle	Powder, Circle	Powder, Circle	Powder, Circle
Liquid Sample	Cup, Moat	Bare	Moat	Cup, Moat	Cup
Data Analysis <sup>2</sup>	B, TIL (0/10)	B, TIL (0/10)	B, TIL (0/20)	BAM TIL (0/10) ABL B, TIL (0/20)	TIL (0/20)

1. All dried per IDCA Drying Procedure; 2. B = Bruceton, TIL = Threshold Initiation Level

The right side of Figure 6 shows the ABL friction test equipment. This test is significantly different than the BAM friction test. A sample, 5 to 40 mg, is placed on a metal anvil surface. A stationary wheel with grooves is placed on the sample. A pendulum arm is released and strikes the anvil, moving the sample. The force of the pendulum arm is applied by pressure at a specific arm velocity and is varied to cause reaction. The reaction is a pop, smoking or jetting and is reported in psig at a pendulum arm velocity.

*Friction equipment experimental conditions.* Table 3 shows the parameters used in friction testing. The principal differences are:

1. The lights are on or off during detection (for BAM—LANL, SNL and IHD lights on, LLNL lights off)
2. Sample size (IHD uses a much larger amount, concern for edge effects).
3. TIL attempts (for BAM—SNL 0/20; all others 0/10)

### 2.3 ESD Testing

Historically, testing for spark sensitivity was developed later than for impact and friction, but the current test methods were developed in the 1950s<sup>15</sup>. The method is straightforward in concept—

discharge a spark through a material monitoring for a visible or audible indication of a reaction. Unlike the other methods, this testing requires more sophisticated electronics.

For the Proficiency Test, two types of equipment have been used—ABL ESD and LLNL Custom built. The ABL systems of the participants are essentially the same design except for the age (and where produced).



**Figure 7. ABL ESD (left side) equipment and LLNL Custom (right side) equipment used in the Proficiency Test**

*ESD Experimentation.* This left side of Figure 7 shows the ABL ESD testing system. In the center of the system is where the sample is placed. To the inset is an expansion for sample site, showing the discharge and grounding electrodes. The sample size is 5 to 40 mg. Tape is sometimes placed on the sample to keep the discharge from scattering particles. An energy differential is generated between the electrodes and a spark is discharged at a selected energy level. A positive reaction is noted as a pop, puff, smoke or noise. Sensitivity is measured in joules as threshold of reaction. Newer versions of the ABL ESD have more flexibility in setting levels of stimulus—static energy output, 0.0001 J to 38 J, high voltage up to 10 KV and variable resistance: 0-, 50-, 510- $\Omega$ , to mimic many different handling conditions.

The right side of Figure 7 shows the LLNL custom built system. The LLNL custom built system was designed to have a 510- $\Omega$  resistor in the circuit. (Current ABL systems are also designed for selection of resistance levels to mimic various situations.) Built in the late 1970s by LLNL personnel, it was designed specifically to evaluate the electrostatic discharge hazards specifically associated with humans handling of explosives. To accomplish this, a 510- $\Omega$  resistor was permanently inserted into the discharge circuit to mimic the resistance of the human body. The system consists of a capacitor, a voltage control, and a discharge circuit where high static voltage (up to 10 kv) with static energy up to 2.0 J is applied and discharged to the sample.

*False Positives.* Care must be taken in interpreting indication of a positive reaction in this test because some of the component materials may be reactive to spark even though it is not an explosive event. Carbon based materials are particularly sensitive and activated carbon provides an example. In testing the  $KClO_4$ /charcoal mixture, participants found difficulty in discerning visually the carbon burning due

to the spark or the  $\text{KClO}_4$ /charcoal reacting as a mixture. Background subtraction, would possibly solve the problem, but this is difficult to accomplish with detection with observation only.

To more effectively discern the positive and negative reaction, especially when competing oxidation mechanisms may be in play, some of the participants have resorted to using light gas meters in addition to observation for detection. Figure 8 shows an example of the manufacturer provided gas detection system for the ABL equipment during testing of RDX. On the left side of the figure is the readout of the atmosphere in the test chamber before the spark discharge (in ppm)— $\text{CO}_2$ , 581;  $\text{CO}$ , 0;  $\text{NO}_x$ , 9. On the right side of the figure is the readout of the atmosphere in the test chamber after the discharge (in ppm)— $\text{CO}_2$ , 1069;  $\text{CO}$ , 29;  $\text{NO}_x$ , 50. The right side of the figure shows higher values in  $\text{CO}_2$  and to a lesser extent,  $\text{CO}$  and  $\text{NO}_x$ , indicating combustion is occurring (RDX is reacting to spark). With the proper calibration and background tests, changes in the light gas concentrations over background can be very useful in helping confirm detection of a positive and negative event.



**Figure 8. ABL ESD gas analyzer before and after spark initiation of RDX**

**Table 4. ESD test equipment configuration for Proficiency Test**

	LANL	LLNL	SNL	IHD	AFRL
Test Apparatus	ABL	ABL, Custom <sup>1</sup>	ABL	ABL	ABL
"Go" "No-Go"	Observation	Gas detection Observation	Gas detection Observation	Gas detection Observation	Gas detection Observation
Test Range, J	0.00125 to 0.25	ABL: 0.0001 to 38 Custom: 0.001 to 2	0.0013 to 9.4	0.0084 to 8.33	0.0013 to 9.4
Sample <sup>2</sup>	Powder	Powder	Powder	Powder	Powder
Size	~ 5	~ 5	~ 5	~ 5	~ 5
Powder amount	Cover bottom sample holder	Cover bottom sample holder	Cover bottom sample holder	Cover bottom sample holder	Cover bottom sample holder
Tape Cover	Scotch	Mylar	None	None	None
Liquid amount	Cover bottom sample holder	Cover bottom sample holder	Cover bottom sample holder	Cover bottom sample holder	Cover bottom sample holder
Data Analysis	TIL (0/20)	TIL (0/10), TIL (0/10)	TIL (0/20)	TIL (0/20)	TIL (0/20)

1. Custom with a 510-Ω resistor in circuit to mimic a human body; 2. Dried according to IDCA drying procedures

*ESD Experimental Testing Configuration.* Table 4 shows the ESD testing configuration used by the participants during the Proficiency Test. The principal differences are:

1. Testing equipment (LLNL used a custom built system for the first ~ 1/3 of the materials tested. After that time, a new ABL system was put into use),
2. Detection of a positive reaction (all used observation but LLNL and SNL also used emitted gas as a confirming technique),
3. Testing parameter range (varies based on vintage of the system),
4. Tape cover (LANL used scotch; LLNL used Mylar; SNL, AFRL, and IHD used none),
5. Data Analysis (all used 0/20 for TIL except LLNL used 0/10).

## 2.4 DSC Testing

Differential scanning calorimetry (DSC) was developed through the concept of isothermal calorimetry first published in the late 18<sup>th</sup> century<sup>20</sup>. Modern instrumentation really came into fruition around the 1970s<sup>21</sup>. Since that time, the equipment has been made dependable and inexpensive. Currently, the most prominent commercial instruments are produced by TA Instruments.

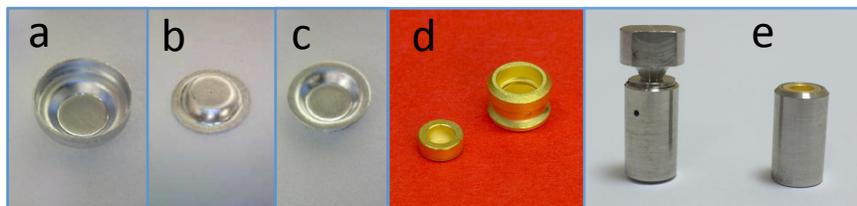
For the Proficiency Test, the TA Instruments DSC was chosen as the standard configuration, because all the participants at the time had one or more versions. As the testing proceeded, however, modifications had to be made because of the nature of some of the test materials. Sample size and sealed sample holders are the key issues requiring modification to the methods. These will be discussed below.



**Figure 9. DSC equipment used for the Proficiency Test (left side-TA Instruments, right side-Setaram Sensys)**

*DSC Experimentation.* The left side of Figure 9 shows the standard DSC system employed during the Proficiency Test. The test procedure is straightforward. A very small sized sample (<1 mg) is placed in sample holder. The standard sample holder has a vented lid. The sample is heated at a constant heating rate of 10°C/min. The starting temperature varies, from ambient, -40°C, or 40°C, depending upon the laboratory. Heat flow in and out of the sample is measured by the system. Heat flow into sample is indicative of endothermic while heat flow out of sample is exothermic and indicates energetic materials. The data output is heat flow as a function of temperature (or time if desired). Sensitivity measured in joules/g.

During the Proficiency Test, conditions for performing DSC on certain materials had to be changed. For these materials, loss of volatile species during heating prevented deriving an accurate assessment of the thermal sensitivity. The standard sample holder is pinhole vented to permit a small amount of pressure release. With military materials, this pressure release was shown to have minimal effect. However, for mixtures with volatile components, initial studies showed that the pinhole vented lid on the sample holder makes a significant difference in the recorded DSC behavior. The solution to this problem (loss of volatiles) was to employ sample holders that remained sealed throughout the measurement.



**Figure 10. Sample holders utilized in the Proficiency Test (a. standard sample holder; b. lid with laser drilled pinhole for the standard sample holder; c. sealed lid for the standard sample holder; d. SWISSI high pressure sample holder; e. Setaram high pressure sample holder)**

Figure 10 shows the different sample holders employed in the Proficiency Test. All participants used the standard sample holder with pinhole in the lid, Figures 10a and 10b. LLNL did all the test materials also with the standard sample holder with a lid that had no hole, Figure 10a and 10c. IHD investigated AN and AN-gunpowder mixtures using the SWISSI sample holder, Figure 10d. This sample holder is rated to withstand 217 bar (3150 psi) at 400°C. It is also gold plated for inertness. A limited number of tests were done by LLNL with the Setaram system, Figure 10e. This sample holder is rated to withstand 500 bar (7250 psi) at 600°C. It is also gold plated on the inside.

## 2.5 Data Reduction Methods

Tables 2-4 show how the data taken during testing is reduced to an analytical form. For impact testing, two methods are used—modified Bruceton<sup>22,23</sup> and Neyer D-Optimal<sup>24</sup>. For Friction, three methods are used—modified Bruceton, Neyer D-Optimal (rarely), and TIL (threshold)<sup>25</sup>. For ESD, one method is used—TIL. These methods have been reviewed previously and are well established and will only be summarized briefly here.

*Threshold initiation level (TIL).* This method is the simplest of all, and is applied to BAM and ABL Friction methods and to ESD. The method tests at discrete energy levels and determines the level that gives no reaction as well as levels that trigger reactions. The IDCA also reports the level above where there is observed reactions, at least part of the time. For BAM Friction, the 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. In the BAM friction test, the load levels are controlled by the weight placed on the friction arm, as shown in Figure 6. For ABL Friction, the TIL is the load (psig/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. In ABL Friction, the load levels are controlled by the pressure that drives the pendant arm, as shown in Figure 6. This pressure is also modified by the low speed drive. For ESD, the 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. In ESD, this is set by the energy charged to the circuit to generate the spark that passes through the sample, which is converted to joules.

*Probability of reaction.* Both the modified Bruceton and the Neyer D-Optimal are employed to determine 50% probability of reaction. For the Proficiency Test, all laboratories used the modified Bruceton method, and LANL also used the Neyer D-Optimal method. The general approach of the two methods is the same: choose an algorithm for picking test levels and number of tests, carry out testing, following the algorithm, noting the result at each level, and then analyze the results for mean and standard deviation. An optimized algorithm meets laboratory and testing needs: faster determination with fewer tests, better determination of mean (higher confidence), and better determination of standard deviation. However, there are drawbacks in this approach. Attempts to optimize all of these simultaneously usually lead to trading confidence for more rapid testing (fewer tests).

*Modified Bruceton method.* The Bruceton method was developed at ERL, Bruceton PA, in early 1940's, and was optimized by Tukey<sup>26</sup>, Dixon, and Mood<sup>22</sup>. It can be implemented without a computer (free of cost and can be performed with a calculator), and relies on significant simplifying assumptions. It is optimal for determining 50% reaction level, but is not optimal for determining standard deviation without extra testing.

The experimental method is to start by choosing a high insult level, fix the level spacing near the expected standard deviation, and test sample. If a positive reaction, then decrease the insult one level. If negative reaction, then increase the insult one level. Repeat until a positive reaction is followed by a negative reaction. Use those as first two tests of series. Continue testing. If a positive reaction, then decrease insult one level; if a negative reaction, then increase insult one level. After an odd number of tests, tabulate statistics at each level. Apply equations to determine 50% reaction level and standard deviation.

*Neyer D-Optimal Method.* The Neyer D-Optimal method was developed at EG&G Mound, Miamisburg OH in 1980s by Barry T. Neyer<sup>24</sup>, costs around \$2500 for the software and must be implemented on a computer. It does not rely on very strict assumptions, and can run tests optimized for mean or some other probability level—at any positive or negative test level. The result is equal confidence in mean and standard deviation.

The experimental method is to input instrument parameters and choose test level resolution into SenTest software (comes with the Neyer package). Begin testing at computer-chosen level, and input result (positive or negative reaction). The computer applies Maximum Likelihood Estimation, chooses next level, attempts to test at +/- 1 standard deviation, and continues testing at computer-chosen levels until results meet test parameters, such as predetermined number of tests or confidence level. Results are the level for 50% probability of reaction and standard deviation.

## 3 DISCUSSION

The follow section addresses some of the reasons motivating for the changes in methods during the Proficiency Test.

### 3.1 Impact Testing

*Modification—Sandpaper Type.* The first material tested in the Proficiency Test was the RDX standard. The results from testing by LLNL, LANL, IHD and AFRL went as expected, because RDX has been extensively tested over the years. When the solid oxidizer/sugar mixtures were tested, an interesting feature appeared in the impact sensitivity data—the 50% probability of reaction level was sandpaper dependent. Three different sandpapers were being used for the testing, the type depending upon the par-

ticipant. LLNL and LANL were using 120-grit Si/C and 150-grit garnet sandpapers, respectively. IHD and AFRL were using 180-grit garnet sandpaper. In testing RDX, this made little difference. However, in testing most of the HMEs, the difference among results from the different sandpapers was dramatic.

Table 5 shows the DH<sub>50</sub> values of several Proficiency Test materials relative to RDX DH<sub>50</sub> values, in cm, using 120-grit and 180-grit sandpapers. The sandpaper used for the RDX measurements that the mixtures were compared to were the same.

**Table 5. DH<sub>50</sub> values<sup>1</sup>, in cm, of selected test materials, relative to DH<sub>50</sub> values, in cm, of RDX<sup>2</sup>**

Grit <sup>3</sup>	KClO <sub>3</sub> /Dodecane	KClO <sub>4</sub> /Al	KClO <sub>4</sub> /Dodecane	PETN	Ammonium Nitrate	Ammonium Nitrate/Gunpowder	HMX
120	14.4	152.9	152.9	-13.3	131.9	61.7	21.9
180	-12.5	-4.9	8.7	-13.5	60.2	25.0	16.4

1. DH<sub>50</sub>, in cm, by modified Bruceton method, height for 50% probability of reaction; 2. All RDX testing was performed using the same sandpaper as used for the compared material, formula, DH<sub>50</sub> material – DH<sub>50</sub> RDX; 3. 120 is 120-grit Si/C wet/dry sandpaper, 180 is 180-grit garnet dry sandpaper.

In the table, a 0 value would indicate that the material is sensitive as RDX. A “+” value indicates the materials are less sensitive than RDX. A “-” value indicates the material is more sensitive than RDX. The table shows obvious discrepancies in evaluating the sensitivity of a material. For example, KClO<sub>4</sub>/Al is completely non-sensitive relative to RDX when using 120-grit sandpaper, but more sensitive relative to RDX when using the 180-grit sandpaper.

Table 6 shows the DH<sub>50</sub> values, in cm, relative to RDX DH<sub>50</sub> values, in cm, taken under the same testing conditions for several of the Proficiency Test materials using 150-grit and 180-grit sandpapers. The differences in the sensitivity of the materials relative to RDX determined using 180-grit and 150-grit sandpapers are not as dramatic as in Table 5. In addition, in both tables, the differences in the results for military standards among all the sandpapers are very little.

**Table 6. DH<sub>50</sub> values<sup>1</sup>, in cm, of selected test materials, relative to DH<sub>50</sub> values, in cm, of RDX<sup>2</sup>**

Grit <sup>3</sup>	KClO <sub>3</sub> /Sugar (100)	KClO <sub>3</sub> /Sugar (AR)	KClO <sub>3</sub> /Dodecane
150	-8.4	-10.4	-14.2
180	-10.2	-10.4	-12.2

1. DH<sub>50</sub>, in cm, by modified Bruceton method, height for 50% probability of reaction; 2. All RDX testing was performed using the same sandpaper as the compared material, formula, DH<sub>50</sub> material – DH<sub>50</sub> RDX; 3. 120 is 120-grit Si/C wet/dry sandpaper, 180 is 180-grit garnet dry sandpaper.

The variability in these results highlighted a real issue in standardization of the test method. As a result, the IDCA decided to use 180-grit garnet sandpaper as the standard testing configuration. LANL purchased a large quantity and distributed that sandpaper to all the participants in the Proficiency Test.

*Modification—Liquid testing standard.* Isopropyl nitrate (IPN) was suggested as a potential liquid test standard in SSST testing, after the Proficiency Test began because the IDCA only had chosen solid materials as standards. This material was examined by several of the laboratories for impact sensitivity as well as feasibility as a liquid standard.

Table 7 shows the results. The sensitivity of the IPN ranges from a little less than PETN (DH<sub>50</sub> 8.0 cm, 180-grit sandpaper, LANL data<sup>12</sup>) to insensitive. This sensitivity range of the IPN is really dependent

upon the drop hammer technique. The technique is related to the preferred method of testing by the specific laboratory. The cavity drop experiment most closely matched the UN values<sup>27</sup>, but it is unclear how accurate are the UN values. The rest of the results show a fairly insensitive material. Some can be explained by the configuration of the technique. For example, the bare anvil (without grease) is expected to be variable because the IPN disperses quickly on the anvil surface due to no containment. As a result of the multiple, ambiguous results, the IDCA chose not to use IPN as a standard. However, in the process of testing the IPN, several issues around the design of the liquid test equipment were realized, so the IDCA made a modification to the methodology to incorporate liquid-testing results from multiple methods by each laboratory if possible, and that there were no explicit standards for testing liquids.

**Table 7. Impact Data (DH<sub>50</sub>) for IPN by various test methods**

Lab	DH <sub>50</sub> , cm <sup>1</sup>	Test Conditions
IHD	14.3	Cavity, un-dried IPN, 2 kg drop weight
SNL	> 115	Brass cup, un-dried IPN, 1 kg drop weight, 1.5 kg striker <sup>2</sup>
SNL	> 115	Brass cup, un-dried IPN, 1 kg drop weight, 2 kg striker <sup>2</sup>
SNL	> 115	Brass cup, un-dried IPN, 2 kg drop weight, 2 kg striker <sup>2</sup>
SNL	> 115	Brass cup, un-dried IPN, 2.5 kg drop weight, 2 kg striker <sup>2</sup>
LLNL	137	Bare anvil (no grease) <sup>3</sup> , dried IPN, 2.5 kg drop weight, 1.0 kg striker
LLNL	159	Bare anvil (no grease) <sup>3</sup> , un-dried IPN, 2.5 kg drop weight, 1.0 kg striker
LLNL	160	120-grit sandpaper, dried <sup>4</sup> IPN, 2.5 kg drop weight, 1.0 kg striker
LLNL	> 177	120-grit sandpaper, un-dried IPN, 2.5 kg drop weight, 1.0 kg striker
LLNL	> 177	180-grit sandpaper, dried <sup>4</sup> IPN, 2.5 kg drop weight, 1.0 kg striker
LLNL	161	180-grit sandpaper, un-dried IPN, 2.5 kg drop weight, 1.0 kg striker
UN	18	Reference 1, (dried?) <sup>5</sup> , 1-kg drop weight, 2-kg striker
UN	14	Rotter Impact, (dried?) <sup>5</sup> , 0-ring seal cup, 5 kg weight (median drop height)

1. DH<sub>50</sub>, in cm, by modified Bruceton method, height for 50% probability of reaction; 2. Reference 27; 3. Grease dissolved in IPN—corning high vacuum grease and Kel F-800; 4. Dried over 12A molecular sieves; 5. Drying status unknown.

*Modification—sample form in drop hammer.* Table 2 shows that the sample form is either pressed or powder—the sample form was never specified in the original methods determined by the IDCA. The pressed form is a standard for LLNL as they produce many materials to that specification. The powder form has advantages because for HMEs, the materials are likely not going to be mechanically pressed.

**Table 8. DH<sub>50</sub> values for RDX pressed and in powder form for the Proficiency Test**

Form <sup>1</sup>	T, °C	RH, %	DH <sub>50</sub> , cm <sup>2</sup>	s, log unit <sup>3</sup>
Pellet	24	18	28.8	0.042
Powder	23	22	24.2	0.015
Powder	23	23	22.0	0.035
Pellet	24	32	34	0.059
Powder	24	18	24.8	0.054

1. Pressed is by mechanical pressing but not to specifications; 2. DH<sub>50</sub>, in cm, by modified Bruceton method, height for 50% probability of reaction; 3. Standard deviation.

Comparing the results in Table 8 show that the pressed RDX is less sensitive to impact than the powder RDX. As a result of these values, the IDCA made a modification to the methodology that the standard form of testing materials for impact will be in the powder form.

*Modification—Striker weight standardization.* Table 9 shows some drop hammer test data from the first draft of the DHS Small-Scale Safety and Thermal Test Guide and some recent Proficiency Test results. LLNL results on liquids were from tests that were performed using a 1.0-kg striker (see Figure 2). LANL tests were performed using a 2.5-kg striker. The data compares the performance of the same or similar materials when the test uses a 2.5-kg striker or a 1.0-kg striker. For standard military materials, such as PETN, the data from the use of the 1.0-kg striker shows a more sensitive material than the corresponding data from the use of the 2.5-kg striker. However, for the data for the H<sub>2</sub>O<sub>2</sub> 90/nitromethane using the two different strikers, the 2.5-kg striker seems to indicate a much more sensitive material compared to the data on the same material obtained using the 1.0 kg-striker.

**Table 9. Drop Hammer (DH<sub>50</sub>, cm) test results at different striker weights**

TMETN (1.0-kg striker LLNL)	14
FEFO (1.0-kg striker LLNL)	32
PETN (1.0-kg striker LLNL)	10
HMX (1.0-kg striker LLNL)	23
PETN (2.5-kg striker LLNL)	15
HMX (2.5-kg striker LLNL)	32
TATP (2.5-kg striker LLNL)	11
HMTD (2.5-kg striker LLNL)	10
PETN (2.5-kg striker LANL)	13
HMX (2.5-kg striker LANL)	28
TMETN (2.5-kg striker LANL)	47
TATP (2.5-kg striker LANL)	6.5
HMTD (2.5-kg striker LANL)	4.2
HP (90)/NM 48.1/51.9 (1.0-kg striker LLNL)	33
HP (90)/NM 48.1/51.9 (1.0-kg striker LLNL)	31
HP (90)/NM 48.1/51.9 (1.0-kg striker LLNL)	27
HP (90)/NM 48.1/51.9 (2.5-kg striker LANL)	8.6
HP (90)/NM 48.1/51.9 (2.5-kg striker LANL)	8.8
HP (90)/NM 48.1/51.9 (2.5-kg striker LANL)	10.5

These results started a detailed discussion on the effects of different striker weights to answer the question “can the data from a 1.0-kg striker be generalized with data from a 2.5-kg striker weight.” A path to eliminate the issue of different striker weights is to make the striker weight uniform. However, LLNL is not sure that this can be done on the LLNL system. LLNL uses a 1-kg striker for liquids because of experimental configuration has not been modified to hold the striker offset with a 2.5-kg striker. With grease holding the liquid sample, the striker is not placed directly on the sample, but is a millimeter or so above (as not to deform the sample until the measurement). The other participants have equipment that has been modified to be able to hold this offset. For example, LANL uses a weak magnet to hold the striker above the liquid sample (see Figure 3). The equipment at LLNL does not have this modification.

The results in Table 9 highlight a panoptic issue—differences in drop hammer results among the participants for the same material. In discussing reasons, the topic of striker weights and the relationship to ringing arose. Ringing comes about due to a mismatch in the drop weight and the striker weight. Many have observed that if the striker weight is smaller than the drop weight, there will be recoil after the initial transfer of the momentum from the drop weight to the striker weight and that this will cause

a second impact of the striker onto the sample. This could cause artificial positive reactions. When an inventory was done of the striker weights, it was found that LANL uses exclusively a 0.8 kg striker, and LLNL uses a 1.0 kg striker on liquids, but both use 2.5-kg drop weights.

To examine whether ringing has an effect and/or whether the striker weight is a critical testing variable, LANL conducted experiments on two HMX formulations of differing sensitivity using 3 different striker weights, but the same size drop weight.

Three strikers of weight 0.8 kg, 1.0 kg, and 2.5 kg were made from a hardened steel rod, 1.25 inches in diameter, the upper round end of 2.50 inch radius. The length varied to accommodate the weight. The HMX standard from the IDCA proficiency study and a formulation of DAAF-GAP-HMX (insensitive) were used as the sample explosives. Fives runs of 15 drops were run for each striker and on both materials for impact sensitivity. The experimental runs were randomized to minimize systematic errors.  $DH_{50}$  and the standard deviation were determined by Neyer D-Optimal test method using Neyer SenTest Software. ANOVA analysis was performed by Minitab Statistical software version 15.

**Table 10. Drop hammer data for HMX and DAAF-GAP-HMX**

HMX/180grit/35mg/d-optimal				DAAF-GAP-HMX/180grit/35mg/d-optimal			
run order	striker, (kg)	H50, (cm)	$\sigma$ , (cm)	run order	striker, (kg)	H50, (cm)	$\sigma$ , (cm)
1	1	19.4	2.3	1	1	64.9	14.2
2	0.8	23.4	1.2	2	2.5	53.7	8.6
3	0.8	23.2	3.2	3	0.8	85.6	43.0
4	2.5	21.4	2.1	4	2.5	52.4	3.6
5	2.5	21.0	3.9	5	0.8	81.1	1.9
6	2.5	19.6	2.7	6	1	61.5	7.1
7	0.8	19.4	0.8	7	0.8	82.3	4.0
8	0.8	20.4	2.0	8	2.5	50.5	4.1
9	1	19.4	1.4	9	1	62.3	4.0
10	1	22.0	2.1	10	0.8	80.7	8.7
11	2.5	19.0	1.4	11	2.5	49.7	3.3
12	1	20.1	1.7	12	2.5	52.0	6.7
13	1	20.4	1.5	13	1	61.1	6.7
14	2.5	22.4	2.7	14	0.8	82.9	11.1
15	0.8	20.7	1.8	15	1	58.6	7.1

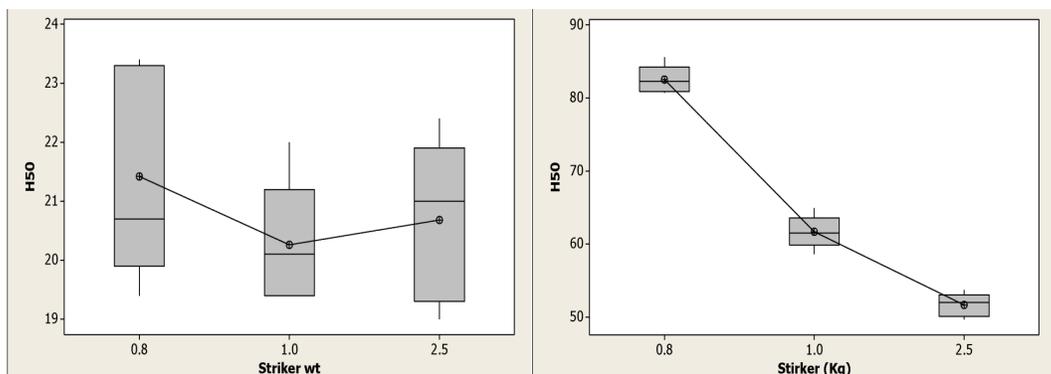
Table 10 shows the results from the experimentation. For the HMX data, the spread in the 50% height is 19.0 to 23.4 cm ( $20.8 \pm 1.4$  cm; 6.7% relative). For the DAAF-GAP-HMX data, the spread in the 50% height is 49.7 to 85.6 cm ( $65 \pm 13.8$  cm; 21.0% relative), and much larger than the HMX case.

By the ANOVA analysis, for the HMX, assuming a confidence interval = 95%, the striker (between measurements) mean standard deviation is 1.72, while the error (within measurement), the mean standard deviation is 2.07. These values are almost identical, meaning that the results for the experiment are identical. For the DAAF-GAP-HMX, assuming a confidence level of 95%, the striker (between measurements) mean standard deviation is 1239.2, while the error (within measurement), the mean standard deviation is 3.8. These values are not identical meaning that the results for the experiments are not identical.

Figure 11 shows these results graphically. (Note the differences in y-axis scales.) The left side of the graph shows the HMX data reduced to the mean and the standard deviation for each of the striker weights. The values overlap and are statistically the same. The right side of the graph shows the corre-

sponding DAAF-GAP-HMX data reduced. In this case, there is no overlap. The conclusions are: 1) no significant difference in results due to striker weight at lower drop heights, and 2) large significant difference in results due to striker weight at higher drop heights.

As a result of these studies and comparisons, the IDCA made a modification to the methodology to let each laboratory choose the striker weight for the experiment, but also to make sure the value is reported.



**Figure 11. Graph of the mean and standard deviation for HMX (left side) and DAAF-GAP-HMX (right side).**

### 3.2 ESD Testing

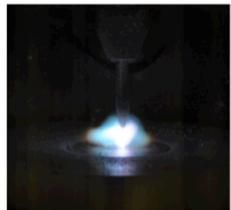
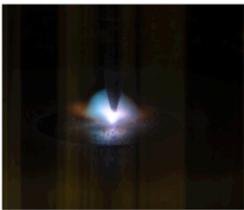
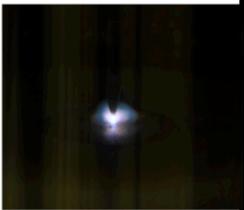
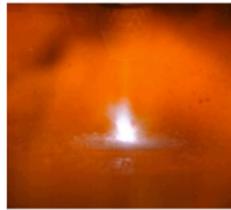
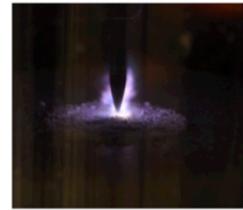
*Modification—Improved detection of positive ESD events.* A difficult part of ESD testing (or any testing for that matter) is proper detection of a positive/negative event (go/no-go). Traditionally, the detection is based on visual observation of some type of reaction over baseline. Baseline for ESD is the action and sound of a spark discharging through a material without causing an energetic reaction. This discharge can be just a spark discharge with a little noise, but also can be a flash or burn. The distinction between the baseline for a specific material and an ESD-driven reaction is difficult and takes much experience to do correctly. It is operator-dependent, and therefore somewhat subjective, casting some doubt on the accuracy of testing results. The field of SSST testing recognizes this problem and is trying to develop measurement equipment that will take the operator subjectivity out of the equation.

The spark in the ESD test, when it interacts with organic-based materials (such as  $\text{KClO}_3/\text{sugar}$ ), produces  $\text{CO}_2$ ,  $\text{CO}$  and sometimes  $\text{NO}_x$ . These are defined, volatile gases that are lightweight and can be detected by various types of meters. If these gases are monitored, detection can be shifted from observational to instrumental, increasing the credibility of the data collection, assuming the instrumentation is used correctly.

Figure 12 illustrates some of the difficulties in ESD detection when testing PETN as an example. Shown are photographs during the spark discharge at different capacitance levels correlated with the gas analysis using the fixed-gas meter monitoring  $\text{CO}_2$  and  $\text{CO}$ . Comparing the blank and test image at 0.1- $\mu\text{F}$  capacitance level indicates the test image clearly shows a reaction, with an intense flash. The gas concentration data before and after testing corroborates that the ESD spark caused a reaction. Both  $\text{CO}_2$  and  $\text{CO}$  increased greatly over baseline. Comparing the blank and the test image at the 0.012- $\mu\text{F}$  capacitance level indicates no difference and visually there would be no reaction assigned, although there is a flash. The gas data supports this also. However, the real advantage of having the gas detec-

tion comes at the transition point between go/no-go. At the 0.02- $\mu\text{F}$  capacitance level, there are two examples. Visually, it would be only the most experienced operator that could tell the difference between the tests and the blank—very little visual difference. However, the gas analysis gives supporting information for the differentiation between go and no-go.

The company that manufactures the ABL ESD equipment, SMS, now provides the gas meter as a standard component. This feature is used only by the SNL and the new LLNL system. Even though the other participants have this feature, they did not use it. However, the IDCA modified to the methodology to allow the determination of the positive event in ESD by confirming with the gas meter.

Capacitance Level ( $\mu\text{F}$ )	0.1		0.02				0.012	
Blank Image								
Test Image								
Gas	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO
Starting Conc. (ppm)	384	90.5	344	81.5	344	86.0	364	91.0
Ending Conc. (ppm)	1100	157	351	89	403	99.3	371	94.9
$\Delta$ Conc. (ppm)	716	66.5	7	7.5	59	13.3	7	3.9
Result	GO		NO-GO		GO		NO-GO	

**Figure 12. ESD testing of PETN monitoring visible emissions and gas evolution**

*Modification—LLNL addition of ABL ESD testing equipment.* Table 11 compares the results of the ESD testing using the LLNL custom-built system to the ABL systems used by the various participants. The LLNL custom-built system indicates most of the material to be insensitive to ESD. Only the KClO<sub>4</sub>/Al mixture exhibits sensitivity to ESD. However, for the ABL systems, all laboratories reported measurable sensitivity of these same materials. This is to be expected because the LLNL system has a 510- $\Omega$  resistor in series.

Prior to enrolling the ABL ESD system purchased by LLNL into the Proficiency Test, comparison of ESD results of the other participants with results from LLNL was difficult. By engaging the new ESD in the

program, the LLNL results are now generated on a similar platform and therefore are now reasonably comparable to those of the other participants. After the initial results on the new system, LLNL retested several of the materials that had already been tested with the custom built system. As a result of the initial comparison data, the IDCA made a modification to the methodology to allow incorporation of LLNL generated ESD data from both the custom built system and the new ABL system.

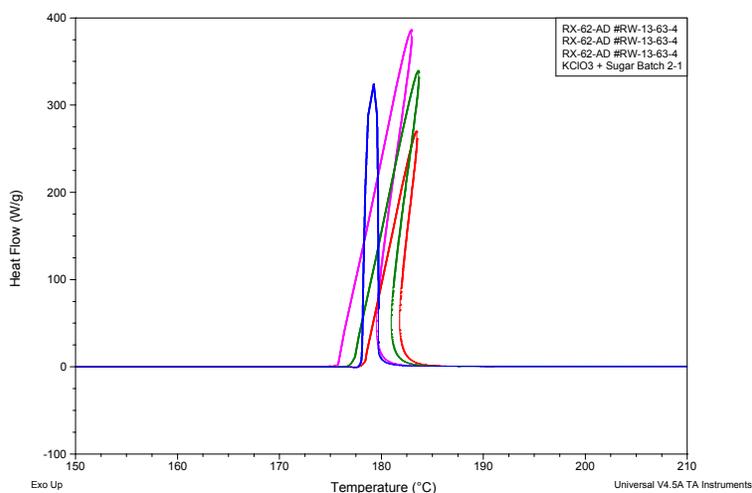
**Table 11. Comparison of ESD TIL levels of the LLNL custom-built system and the ABL ESD systems**

Sample	Custom 510-Ω, TIL	ABL 0-Ω, TIL	ABL, 0-Ω Above TIL	Lab
RDX	0/10 @ 1.0 J	0/10 @ 0.038 J 0/20 @ 0.025 J 0/20 @ 0.095 J 0/20 @ 0.028 J	1/3 @ 0.063 J 1/3 @ 0.063 J 1/7 @ 0.165 J 1/3 @ 0.063 J	LLNL LANL IHD AFRL
HMX	0/10 @ 1.0 J	0/10 @ 0.065 J 0/20 @ 0.025 J	1/8 @ 0.075 J 1/5 @ 0.063 J	LLNL LANL
PETN	0/10 @ 1.0 J	0/10 @ 0.031 J 0/20 @ 0.025 J	2/5 @ 0.038 J 1/4 @ 0.063 J	LLNL LANL
UN/Al	0/10 @ 1.0 J	0/10 @ 0.038 J 0/20 @ 0.125 J	1/10 @ 0.063 J 1/6 @ 0.25 J	LLNL LANL
KClO <sub>4</sub> /Al	0/10 @ 0.25 J	0/10 @ 0.088 J N/A 0/20 @ 0.015 J	2/3 @ 0.013 J 3/8 @ 0.063 J 1/4 @ 0.023 J	LLNL LANL IHD
KClO <sub>3</sub> /sugar	0/10 @ 1.0 J	NA 0/20 @ 0.063 J 0/20 @ 0.165 J	NA 2/3 @ 0.125 J 1/3 @ 0.326 J	LLNL LANL IHD

### 3.3 Thermal Testing Modifications

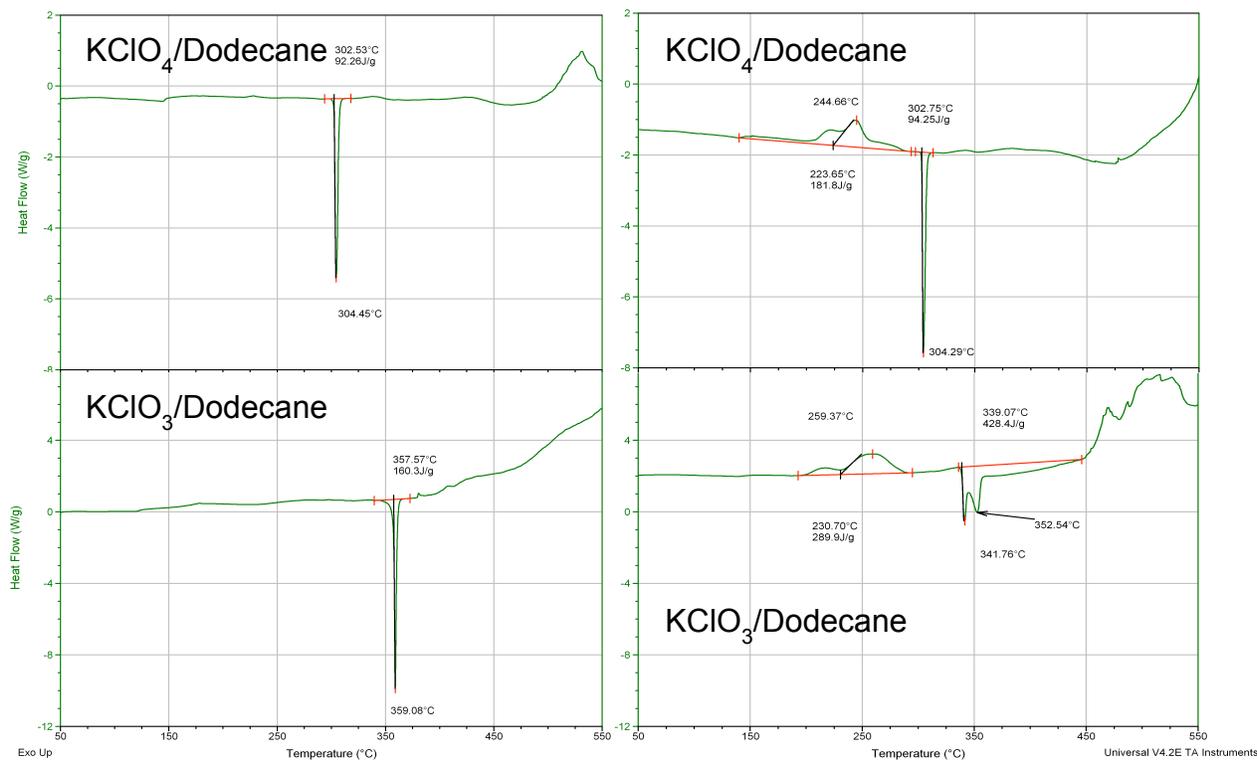
In the Proficiency Test, the standard DSC method is: constant heating rate of 10°C/min, open pinhole lid on the sample holder, and < 3 mg sample size. However, results show on certain HME mixtures, the application of these methods, which are used on conventional explosives, does not always give clear results when applied to HMEs.

*Modification—Energetic material overdriving DSC performance.* One of the most illustrative examples demonstrating the inadequacy of standard DSC test method used by the IDCA is the evaluation of the thermal properties of the KClO<sub>3</sub>/sugar mixture. Figure 13 shows DSC profiles of this mixture under standard operating conditions taken of various samples sizes (and different laboratories). The exothermic feature has a maximum of around 180°C. It also has an abnormal shape—narrow but slanted. This is equipment artifacts due to too much energy release over a very short period of time—the sample is over driving the heating, so the DSC heating shuts down for a short time. The solution to this is to use a much smaller sample. Any negative slope on the front of the exothermic feature or positive slope on the backside of the exothermic feature indicates the sample size is too large. The correct sample size may only be determined by previous results or by trial and error. As a result of these issues, the IDCA made a modification to the methodology to let sample size be determined on a case-by-case basis for each material.



**Figure 13. KClO<sub>3</sub>/sugar DSC at 10°C/min heating rate, large sample size**

*Modification—Standard pinhole DSC sample holder not adequate for volatile species.* Figure 14 shows a comparison of DSC profiles for KClO<sub>4</sub>/dodecane and KClO<sub>3</sub>/dodecane mixtures using a standard sample holder and a sealed sample holder. The standard sample holder has a 75 um laser drilled hole in the center of the sample holder lid (see Figure 10), which allows gases to escape if produced during heating. This prevents the sample holder from violently venting and potentially destroying the equipment.



**Figure 14. DSC profiles of KClO<sub>4</sub>/dodecane and KClO<sub>3</sub>/dodecane mixtures with a vented or a sealed sample holder; note: scales of corresponding axes are not the same in some cases.**

Examining the profiles on the left side of the figure shows prominent endothermic features for both the  $\text{KClO}_4$  and  $\text{KClO}_3$  mixtures. This corresponds to a phase transition and a melting of the oxidizer, respectively. Examining the profiles on the right side of the figure, shows a much more complicated thermal behavior. For the  $\text{KClO}_4$ /dodecane mixture, a series of very broad exothermic features are observed in the 200 to 300°C temperature range. The occurrence of these features is possibly due to the sealed sample holder preventing total evaporation of the dodecane and therefore providing some contact with the oxidizer at reaction temperatures. Dodecane has a boiling point of 218°C<sup>28</sup> so much of it is vaporized in the above temperature range, but if the system is closed, some vapor (not much) is still available for reaction. This same argument can be extended to the  $\text{KClO}_3$ /dodecane mixture.

The exothermic features are not particularly intense in both cases so the profiles are probably not totally representative of the thermal behavior. However, it is important to note that even though the lack of features in data from the pinhole vented sample holder implies no thermal reactivity, the appearance of exothermic features in the sealed sample holder, even though weak, implies something is happening that is different in the sample holder that is vented.

The participants also had varied results for the thermal decomposition of AN. Temperature ranges of endothermic features and enthalpy values were different (LANL and IHD enthalpy values were about 1/3 of the LLNL values). There was also a disagreement between DSC observation and intuition because the region where the oxidizer decomposes was exhibiting endothermic decomposition where exothermic decomposition is expected. As well, the literature shows this same disagreement—Gunawan and Zhang<sup>29</sup> vs. Oxley et al.<sup>30</sup>.

Figure 15 exhibits the DSC profiles of AN in the literature. The left profile is from Gunawan and Zhang and the right profile is from Oxley et al. The profiles are similar except for exact minimum temperature of the endothermic features and the high temperature transition is an endothermic feature in Gunawan and Zhang and is an exothermic feature in Oxley et al. The former issue can be explained by the different heating rates. An exothermic feature is expected for the latter issue because the feature is due to an energetic material decomposing.

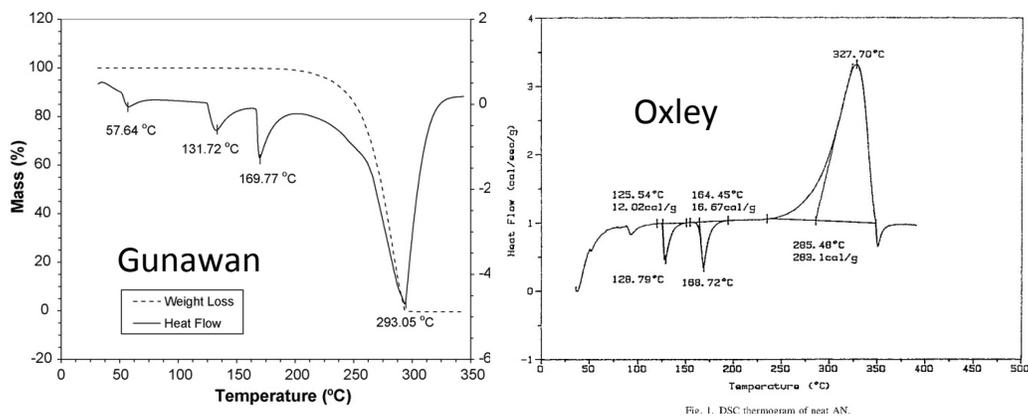
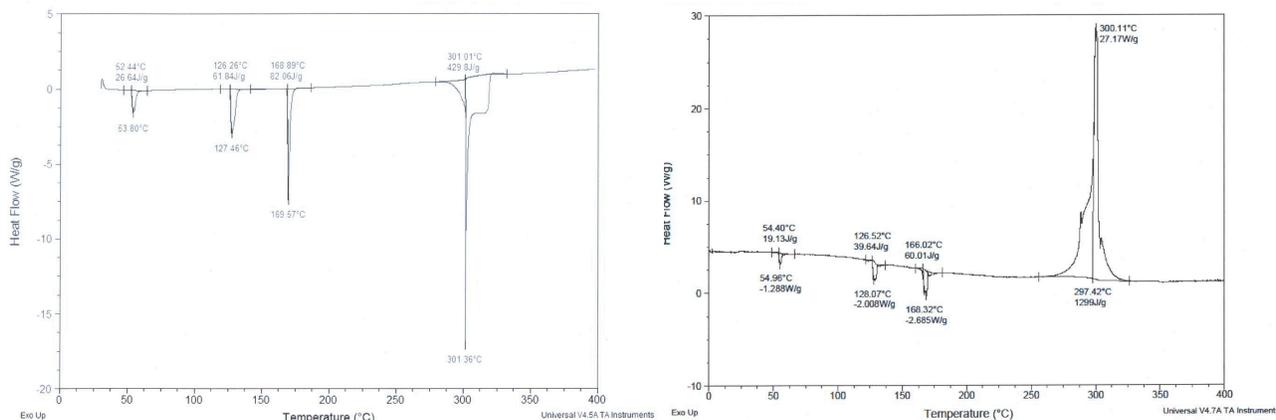


Fig. 1. DSC thermogram of neat AN.

**Figure 15. DSC profiles of AN by Gunawan and Zhang (2009)<sup>29</sup> and by Oxley et al. (2002)<sup>30</sup>.**

The differences in this high temperature features are simply explained by the type of DSC sample cell that is used for the measurement. The pinhole vented sample holders (standard type used by the IDCA, see Figure 10) allow for the gases to escape causing evaporative cooling, an endothermic event, which overrides any positive heat flow from decomposition, an exothermic event. When the gases are not al-

lowed to escape, an exothermic feature is observed instead because of net exothermic heat flow. Figure 16 exhibits this behavior from the IDCA participants comparing the DSC of AN measured under standard and non-standard conditions.



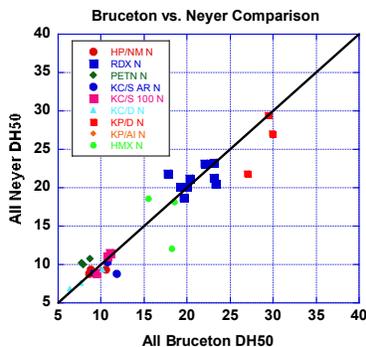
**Figure 16. DSC of AN using a pinhole sample holder (left) and gold sealed holder (right) at 10°C/min heating rate.**

The left side of Figure 16 shows the AN sample heated in the standard Proficiency Test DSC sample holder with a pinhole sample lid. The right side of Figure 16 shows the AN sample heated in a gold sealed sample holder (Gold High Pressure pans, SWISSI crucibles sold in US by Fauske<sup>31</sup>). The high temperature exothermic feature is clearly seen.

As a result of these major discrepancies found in standard test methods, the IDCA made a modification to the methodology to allow incorporation of DSC data taken with sealed sample holders and to recognize that the application of the standard DSC is not always adequate for evaluating the thermal sensitivity of samples.

### 3.4 Data Reduction

*Modification—Bruceton vs. Neyer Data reduction methods.* The standard method for drop hammer data reduction for the participating laboratories has been a modified Bruceton method. However, the Neyer D-Optimal method is becoming popular and, as stated above, has some advantages. Because of the cost of the Neyer software program, the IDCA decided to use the modified Bruceton method as the standard method. However, LANL agreed to perform the analysis with both methods for comparison.



**Figure 17. DH<sub>50</sub> data comparing the Neyer D-Optimal method to the modified Bruceton method**

Figure 17 shows the results of the comparisons of the  $DH_{50}$  values of the two techniques using LANL testing data only. The agreement of the two methods is very good except for PETN.

## 4 SUMMARY

Table 12 lists the changes in methodologies that the IDCA made during the Proficiency Test.

**Table 12. Changes in methods during the IDCA Proficiency Test (modified from testing needs)**

Method Issue	Situation Before	Results After
Sandpaper	<ul style="list-style-type: none"> <li>- LLNL tested using 120-grit silicon carbide paper</li> <li>- LANL tested using 150-grit garnet paper</li> <li>-IHD and AFRL testing using 180-grit garnet paper</li> </ul>	<ul style="list-style-type: none"> <li>- All laboratories test using 180-grit garnet paper</li> </ul>
Sample Preparation	<ul style="list-style-type: none"> <li>- All laboratories tested using loose powder samples</li> <li>- LLNL also tested using pressed powder samples</li> </ul>	<ul style="list-style-type: none"> <li>- All laboratories test using loose powder samples</li> </ul>
Liquid Testing— Drop Hammer	<ul style="list-style-type: none"> <li>- All laboratories tried IPN as a new liquid standard</li> <li>- Methods at specific laboratories varied: bare anvil, sandpaper, cavity, grease</li> <li>- Tried to standardize striker weight</li> </ul>	<ul style="list-style-type: none"> <li>- Multiple methods for impact testing of liquids are allowed</li> </ul>
Liquid Testing— volatile components	<ul style="list-style-type: none"> <li>- All laboratories tested using open pin-hole sample holder lids (loss of components)</li> </ul>	<ul style="list-style-type: none"> <li>- Testing using sealed sample holders for thermal testing is an option</li> <li>- Standard DSC methods may be inadequate</li> </ul>
Data reduction methods	<ul style="list-style-type: none"> <li>- All participants proposed to test using both modified Bruceton and Neyer methods</li> <li>- Report results as 50% probability of reaction</li> <li>- Neyer may be better for standard deviation</li> </ul>	<ul style="list-style-type: none"> <li>- LANL does both and is used for the comparison platform</li> </ul>
Testing Equipment	<ul style="list-style-type: none"> <li>- LANL, IHD and AFRL use ABL ESD</li> <li>- LLNL uses custom equipment</li> <li>- Results very difficult to compare</li> </ul>	<ul style="list-style-type: none"> <li>- LLNL purchased a new ABL ESD apparatus on-line</li> <li>- SNL purchased a new ABL ESD and incorporated it into the new testing facility</li> <li>- Results comparable from all laboratories</li> </ul>
Testing Roles	<ul style="list-style-type: none"> <li>- SNL only performed support characterization work</li> </ul>	<ul style="list-style-type: none"> <li>- SNL brought an entire SSST system on-line</li> </ul>

## 5 CONCLUSIONS AND RECOMMENDATIONS

Table 12 shows many modifications to the standard tests methods were made by the IDCA during the Proficiency Test. Many of these modifications addressed issues that were not realized before testing commenced. Also, many of these modifications are not the ultimate solutions to the problems encountered.

tered when testing HMEs using standard methods, but they were the best solutions at the time considering the scope of the Proficiency Test. Without extensive experimentation to test whether these modifications are the *best* solution to the problems encountered, the modifications listed in Table 12 should be considered when testing HMEs. The following are IDCA recommendations derived from the experience of the Proficiency Test:

1. Standardize the sandpaper and consider using more than one type of sandpaper in the drop hammer experiment. The relationship of the particle size of solid HMEs to the grit-size of the sandpaper (and other sandpaper properties) is important. From the results of the Proficiency Test, it appears that the choice of sandpaper is a much more critical issue for HMEs than for military type-materials. To adequately compare results, for both standard materials and HMEs, the same sandpaper from a common distributed batch needs to be used. To adequately understand the reactivity of the material, multiple sandpapers should be used.
2. Develop standard methods of sample preparation and adhere to these methods strictly. This includes pretreatment, mixing, storage and handling conditions. There is enough variance in the testing that elimination of as many variables as possible is highly recommended.
3. Find a liquid test standard that can be used by all liquid test methods and is readily available. This should be done before the testing of HMEs commences. The liquid test standard should reflect the liquid properties of the HMEs to be tested.
4. Develop methods for pretreatment, mixing, storage, handling and testing of materials that have volatile liquid components. Failure to account for the volatile component can lead to the volatile component disappearing from the sample before testing commences. This would lead to a misleading representation of the reactivity of the HMEs.
5. Consider employing multiple data reduction methods. Having more than one analysis method for the data helps better compare the results and develop an appreciation for the variance of results.
6. Standardize test equipment as much as possible. Although hard to do in SSST testing, try to use the same test platforms as much as possible to minimize testing variables. Comparing the same type of test result taken on different testing platforms may prove to be impossible.
7. Utilize unique testing resources that each contributor may have. Even though these are standard tests, to understand working with new materials, such as HMEs, may require additional characterization of samples to interpret test results. Each contributor brings additional resources that probably will be needed due to the unusual chemical and physical nature of HMEs compared to military type materials.

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## ABBREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH <sub>50</sub>	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
ESD	Electrostatic Discharge
F <sub>50</sub>	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
H	Chemical symbol for hydrogen
H <sub>2</sub> O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
j	joules
KClO <sub>3</sub>	Potassium Chlorate
KClO <sub>4</sub>	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
NaClO <sub>3</sub>	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon

SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

## ACKNOWLEDGMENTS

This work was performed by the Integrated Data Collection Analysis (IDCA) Program, a five-lab effort supported by Los Alamos National Laboratory, Lawrence Livermore National Laboratory, Sandia National Laboratories, the Air Force Research Laboratory, and Indian Head Division, Naval Surface Warfare under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division. Los Alamos National Laboratory is operated by Los Alamos National Security, LLC, for the United States Department of Energy under Contract DE-AC52-06NA25396. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. This work was performed under the auspices of the U. S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. The Air Force Research Laboratory (AFRL/RXQF) and Indian Head Division, Naval Surface Warfare (NSWC IHD) also performed work in support of this effort. The work performed by AFRL/RXQL and NSWC IHD is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division.

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