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Challenges of Small-Scale Safety and Thermal Testing of Improvised Explosives: Results from the Integrated Data Collection Analysis (IDCA) Program Proficiency Test

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

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Challenges of Small-Scale Safety and Thermal Testing of Improvised Explosives: Results from the Integrated Data Collection Analysis (IDCA) Program Proficiency Test

José A. Reyes
Applied Research Associates, Inc.
Tyndall Air Force Base, FL USA

Timothy J. Shelley
Air Force Research Laboratory, AFRL-RXQF
Tyndall Air Force Base, FL USA

Mary M. Sandstrom, Geoffrey W. Brown
Los Alamos National Laboratory, LANL
Los Alamos, NM USA

Jason J. Phillips
Sandia National Laboratories, SNL
Albuquerque, NM USA

Kirstin F. Warner, Daniel L. Remmers
Naval Surface Warfare Center, NSWC IHD
Indian Head, MD USA

Peter C. Hsu, John G. Reynolds
Lawrence Livermore National Laboratory, LLNL
Livermore, CA USA

***Abstract*—The IDCA Program has been conducting a Proficiency (Round Robin) Test on the application of Small-Scale Safety and Thermal (SSST) testing to Home Made or Improvised Explosives (HMEs). This Proficiency test has been designed to test the accuracy and relevancy of SSST testing among explosives testing laboratories (3 DOE and 2 DoD), where the testing is performed on the same batches of materials (20 HMEs and 2 Standards), prepared the same way. The results so far have indicated that standard testing methods are not adequate for HMEs, as many conflicting and inconclusive results have been documented.**

- **Impact sensitivity non-predictively affected by testing conditions**
- **Detection of positive reaction (go/no-go) has too much variability**
- **Thermal testing has sampling issues**

As the IDCA continues to compare and evaluate results from the Proficiency Test, many issues are beginning to coalesce about the application of traditional SSST Testing methods to HMEs. Many of the issues show that traditional methods used for military explosives MUST be modified before meaningful results can be obtained for HMEs. The IDCA is finding if traditional methods are not revised, testing can give misleading results that could lead to developing handling practices that are not adequate for working safely.

Keywords: Small-scall safety testing, proficiency test, round-robin test, safety testing protocols, HME

I. Introduction

A critical aspect in developing forensics methods for explosives is understanding the chemical and physical processes that occur when the energetic material reacts, such as in a detonation. Military explosives are well characterized and offer pre- and post-blast signatures for forensics and attribution to some extent. Improvised materials or homemade explosives (HMEs) are less well characterized and little is known of their behavior and even less is known about the required forensics if an "event" occurs. HMEs have few documented pre-blast signatures and essentially no post-blast signatures needed for forensics. The first step in the process of developing signatures is identifying how to handle the improvised or home made explosive (HME) properly so forensic methods can be developed. The IDCA Proficiency Test research [1] addresses many of the issues regarding handling the materials (important information for first responders, EOD techs, three letter agencies, facilities that test performance) and developing the accurate and correct information about the HME.

SSST tests are critical and usually a first step in deciding whether an energetic material is safe to handle [1,2]. These tests were designed for explosives to indicate sensitivity of the material to handling conditions—drop hammer for impact sensitivity; friction for shear force sensitivity; electrostatic discharge for spark or static sensitivity; Differential Scanning Calorimetry (DSC) for thermal stability; many others for specific types of reactivity.

SSST testing is performed when the sensitivity of material is not known, when direct handling is desired, when the performance of an explosive (e.g., release energy and velocity of detonation) is not known (usually very small quantities of less than 1 gram are tested as a first step), when synthesis/formulation is changed, and upon scale-up (showing the effects of preparation equipment). Results determine (depending upon interpretation) whether a material can be

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directly handled, remotely mixed, or requires complete robotic handling.

The IDCA has been conducting testing on a series of HMEs, utilizing standard SSST testing practices as applied to military explosives [3]. The results so far have indicated that standard testing methods are not adequate for HMEs, as many conflicting and inconclusive results have been documented. In this report, several of these issues are described.

II. Small Scale Safety and Thermal Test

SSST testing as applied to the IDCA Proficiency Test has been reviewed elsewhere [3-5]. Briefly, impact sensitivity is measured by drop hammer where the data are analyzed by the Bruceton [6] or Neyer [7] method. Friction sensitivity is measured by BAM or ABL friction systems where the data is analyzed by the Bruceton method or threshold initiation method (TIL) [8]. Spark sensitivity is measured by ABL electrostatic discharge where the data is analyzed by the TIL method [8]. Thermal sensitivity is measured by differential scanning calorimetry (DSC) and the thermal response of the material is analyzed by heat flow into and out of the sample [9]. Note that for a specific material, each laboratory used the material from the same batch, prepared, mixed and handled the same way.

III. Impact Sensitivity Non-Predictively Affected by Testing Conditions

Impact sensitivity is an assessment of how sensitive the material is to being dropped or struck. A sample, ~ 35 mg, is placed on an anvil in the Drop Hammer apparatus. Solid samples are held on sandpaper and a striker rod is placed on the sample. Drop weight (1 to 2.5 kg) is dropped on the striker rod from variable heights until a reaction is detected. The reaction is a pop, flash or smoke (does not necessarily mean a detonation). The drop height is adjusted during a test to map out the reaction region near the 50% reaction level of the material, designated as DH_{50}. The higher the DH_{50} value, the less sensitive the material is to impact.

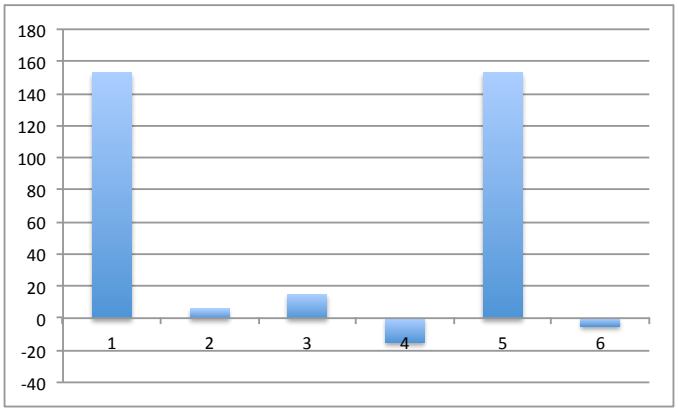

Figure 1. DH_{50} values for selected mixtures relative to RDX standard.

Figure 1 shows impact sensitivity testing of selected HMEs at six different experimental conditions. Material DH_{50} values are set relative to an RDX standard (the DH_{50} of standard is subtracted from the DH_{50} of the material setting the standard to 0). A positive DH_{50} value means the material is more stable than the standard; a negative DH_{50} value means the material is less stable than the standard. The standard is tested under the same conditions at which the sample is tested. 3 mixtures were tested in Drop Hammer at two different conditions. The experiments are:

1. $KClO_4$/Dodecane (120-grit sandpaper) [10];
2. $KClO_4$/Dodecane (180-grit sandpaper) [10];
3. $KClO_3$/Dodecane (120-grit sandpaper) [11];
4. $KClO_3$/Dodecane (180-grit sandpaper) [11];
5. $KClO_4$/Al (120-grit sandpaper) [12];
6. $KClO_4$/Al (180-grit sandpaper) [12].

Figure 1 shows both mixtures **1** and **2** being less sensitive than the standard, but with **1** being much less sensitive than **2**; mixture **3** being more sensitive to than the standard and **4** being less sensitive than the standard; mixture **5** being much less sensitive than the standard and **6** being slightly more sensitive than the standard. Because the only difference in several of these mixture pairs is the use of 120- vs. 180-grit sandpaper to hold the sample, and *the RDX standard changes in a different way than the mixtures*, no relative or absolute sensitivity assessment of the sensitivity is possible.

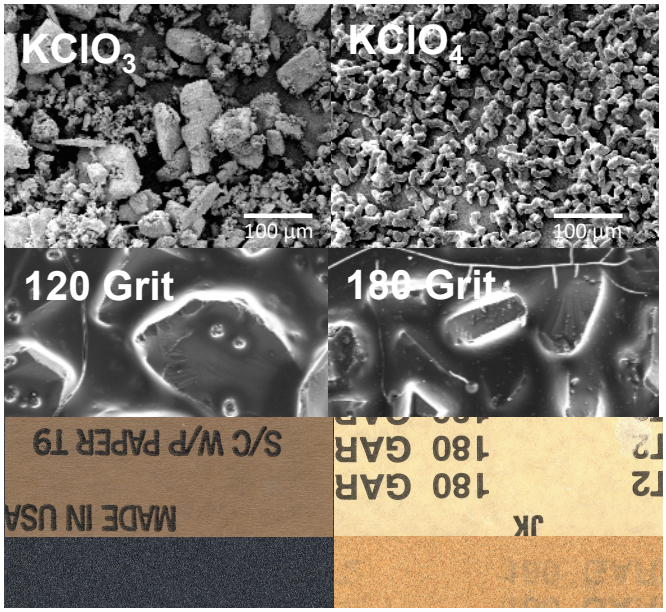

Figure 2. Scanning Electron Micrographs and photographs of 120-grit and 180-grit sandpaper (front and back).

The response difference of a specific mixture to sandpaper as compared to the RDX standard is clear evidence that measurement of the impact sensitivity of the HME by standard methods needs scrutiny. Although it is not clear what property is causing this variability, it probably relates to the details of the sandpapers. Figure 2 shows scanning electron micrographs (SEMs) and photographs of the two sandpapers, showing some of the obvious compositional differences. Some possible relevant properties are:

a mismatch in the relative grain size of the sandpaper as compared to the particle size of the mixture (see below); 120-grit sandpaper is wet/dry type silicon/carbide (Si/C), while the 180-grit is a dry-only garnet; the grit of the 120-grit sandpaper is harder than the grit on the 180-grit sandpaper, (9 to 10 vs. 6.5 to 7.5 on the Mohs hardness scale [13], respectively); the glue on the wet/dry paper is an insoluble resin, while the glue on the 180-grit sandpaper is completely different hide glue; the wet/dry paper is approximately twice as thick as the dry only paper (0.406 vs. 0.229 mm thick, respectively). All of these factors could contribute to the differences seen in the impact data when using 120-grit vs. 180-grit sandpapers.

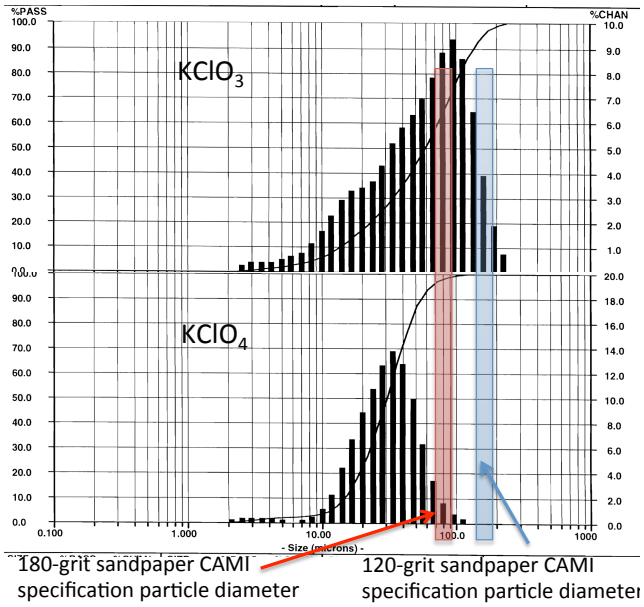

Figure 3. Particle size distribution by laser light scattering of $KClO_3$ and $KClO_4$ with overlay of CAMI specification particle diameters for 120-grit and 180-grit sandpapers.

In Figure 1, experiments 5 and 6 strikingly contrast the effect of the two different sandpapers. Figure 3 illustrates this difference may be caused the mismatch of the relative grain size of the sandpaper to the particle size of the mixtures. The figure displays the particle size distribution (by laser light scattering) for both the $KClO_3$ and $KClO_4$ starting materials. The $KClO_4$ distribution is shifted significantly to small size as compared to the $KClO_3$ distribution. Also shown are the mean diameters of the grit particles of the 120- and 180-grit sandpapers based on the CAMI specification [14]. For the $KClO_3$ mixtures, both the 120- and 180-grit average size fall in the size range of the oxidizer. For the $KClO_4$, only the 180-grit average size falls in the particle size range of the oxidizer. In the mixture cases, the 120-grit and the $KClO_4$/Al mixture are greatly mismatched and the fine powder may fall between the grains of the sandpaper, preventing much contact of the striker. In the 180-grit case, the grit of the sandpaper and the particle size of the $KClO_3$/Al mixture are closer in size (by virtue of the $KClO_3$ size) allowing for better contact. A similar grit size particle size distribution relationship is seen when comparing particle size distributions as measured by Coulter Counter [12].

IV. Detection of Positive Reaction (GO/NO-GO) has too much Variability

The method of detection for a positive reaction in SSST testing is highly dependent upon the testing facility. The general method is by observation, typically done by the operator of the equipment. Use of sound meters, cameras and chemical reaction product analysis are some of the more sophisticated methods. Most positive reactions in impact, friction and spark are marked by a spark, flame, smoke, discoloration and/or sound. These are not trivial to distinguish from background because the testing equipment can make substantial noise, even without samples.

BAM friction is a common method for determining friction sensitivity. The sample (~ 5 mg) is held on a ceramic plate, and a rounded ceramic pin is dragged across the plate, through the sample. Variable force is applied by adding weight to the arm holding the pin, and this weight is varied to cause a reaction and test the material for the 50% reaction level, F_{50}. The reaction is usually a pop, or smoke, or jetting from the sample. The sensitivity is reported either as TIL or F_{50}. TIL is the load (kg) at which zero reactions out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level occurs. F_{50}, in kg, is determined by a modified Bruceton method, load for 50% probability of reaction.

Table 1.BAM friction testing results from selected materials [10-12,15-19].

Material[1]	**LLNL, TIL (kg)**[2]	**LANL, TIL (kg)**[2]	**IHD, TIL (kg)**[2]	**LLNL, F_{50} (kg)**[3]	**LANL, F_{50} (kg)**[3]	**IHD, F_{50} (kg)**[3]
RDX Class 5	19.2	21.6	16.3	25.3	20.8	ND[4]
KC/sugar (100)[5]	6.9	4.8	2.3	9.9	5.8	4.4
KC/sugar (AR)[6]	9.5	2.4	3.2	11.8	4.9	3.6
KC/dodecane	12.3	7.2	16.5	25.5	19.1	26.8
KP/Al	8.7	7.2	12.2	16.7	15.2	14.5
KP/dodecane	36	36	33	>36	>36	>36
KC/C	> 36	> 36	> 36	>36	>36	ND[4]
PETN Class 4	6.4	4.9	4.3	10.4	8.5	6.9

1. KC = $KClO_3$, KP = $KClO_4$; 2. Threshold Initiation Level (TIL) is the load (kg) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 3. F_{50}, in kg, is by a modified Bruceton method, load for 50% Reaction; 4. ND = Not determined; 5. $KClO_3$ separated through a 100-mesh sieve; 6. $KClO_3$ separated through a 40-mesh sieve.

Table 1 shows the BAM testing results for selected materials by LLNL, LANL, and IHD. Shown are both TIL and F_{50} results. In most cases, LLNL testing results indicate a more stable material to friction than the results from the other laboratories, suggesting a systematic issue for at least one of the testing laboratories. However, for the BAM friction testing, all three participants have various versions of the same testing equipment and use observation as the method for detection.

Figure 4 shows the configuration of the IHD and LLNL BAM Friction testing system. The configuration of the IHD system shows a vent hose that removes gases formed during testing. The configuration of the LLNL system shows

complete enclosure of the system also to vent gases formed during testing. The enclosure was designed to control the atmosphere in the testing room, but it also dampens noise from a positive reaction. This effects determination of positives events that generate sound (pop or crackle), and to a lesser extent, flashes. LANL has a system that is similar to IHD and the results reflect that.

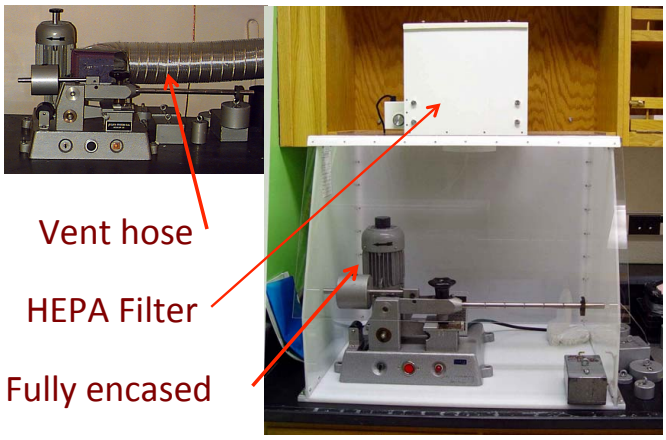

Figure 4. BAM friction apparatus configurations at IHD and LLNL.

The core aspect of this issue is the detection method for positive reaction relies on the senses of the operator of the equipment. Because that person must make a decision based on hearing or seeing the event, the detection becomes somewhat subjective depending upon how acute these senses are in the individual. There is certainly variation from operator-to-operator that adds to the variability of the determination of a positive reaction. There are also no standards for testing the ability of operators to hear or see positive reactions. Additionally, the secondary containment is optional to the standardized BAM friction system, and is custom designed. This only adds more variability in the detection. It appears that the LLNL system inhibits the ability to determine positive events above background, and therefore the material seems less sensitive than they might truly be. As a result of issues such as these, efforts are on going to make the decision more equipment based [20].

V. THERMAL TESTING HAS SAMPLING ISSUES

Thermal stability in SSST testing is commonly determined by differential scanning calorimetry. This technique has advantages as it uses a very small sample size (< 1 mg), can be a fast survey and can be automated. The sample is placed in a sample holder (open or sealed) and is heated, usually at a constant heating rate (10°C/min). The heat flow into and out of the sample is measured. Heat flow into the sample indicates an endothermic response and is generally not hazardous. Heat flow out of sample indicates an exothermic response and suggests an energetic material if the response is large.

For standard military type explosives, this technique is reliable. However, for HMEs, the application to mixtures, both solid-solid and solid-liquid, has shown issues in reproducibility. Figure 5 shows the DSC of $KClO_3$/sugar mixture heated at 10°C/min [16]. Three exothermic features are visible which have been assigned previously [16]—Ex_1, with T_{max} at ~ 180°C is the $KClO_3$/Sugar mixture reacting (sugar melts and then mixes); Ex_2, with T_{max} at ~ 220°C is the sugar carbonizing (sugar that did not react); and Ex_3, with T_{max} at ~ 340°C is the $KClO_3$ melting and reacting with residual carbon. In Figure 5, IHD observed only Ex_1, LANL observed Ex_1 and Ex_3, AFRL and LLNL observed Ex_1, Ex_2, and Ex_3. An important point to note is in other cases, all participants have observed all combinations. The issues are therefore due to sampling.

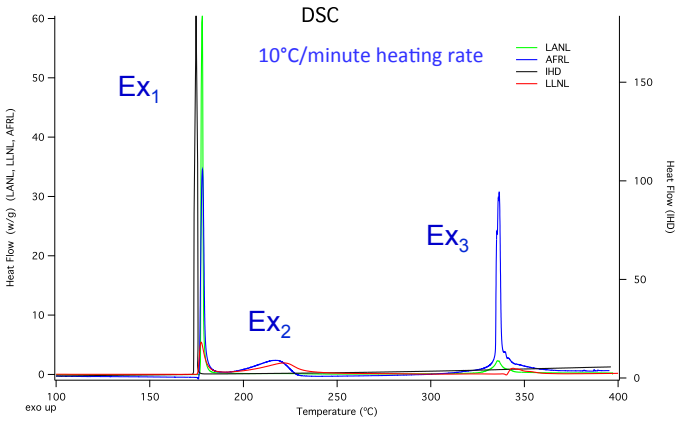

Figure 5. DSC of $KClO_3$/sugar mixture by LANL, AFRL, IHD, and LLNL.

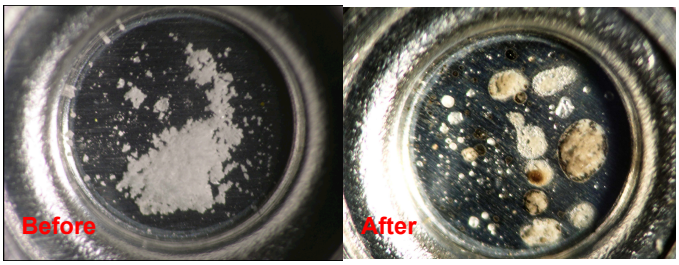

Figure 6. Photographs of a 0.15 mg DSC sample in sample holder before and after thermal scan at 10°C/min.

Figure 6 [21] shows LANL photographs of a 0.15 mg sample in the DSC sample holder before and after exposure to a 10°C/min thermal heating ramp from room temperature to 400°C. The close-up taken before testing shows that the sample does not fully cover the surface of the sample holder. (Note that very small sample sizes must be used for energetic materials to avoid bursting the sample holder and potentially damaging the DSC equipment.) Because this is a mixture of two different solids, these areas could be areas where the ratios of the two solids are not uniform. This is substantiated in the close-up of the sample after thermal testing where there are regions of different color reflecting possible different reaction products. For example, some of the areas are still white while some show brown and even black. It is postulated that the white regions are oxidizer rich because the starting components are white, while the brown and black regions are fuel rich because the thermal reaction products show carbonization of the fuel. The DSC profile of this sample suggests that these correspond with exothermic responses Ex_1, Ex_2, and Ex_3.

In this particular case, Ex_1 is the exothermic feature of most importance because it is the shows the lowest temperature for instability of the mixture. In the experimental series referred to above, as the sample size increased, Ex_1 became the dominant exothermic feature, although other experimental complications were pushing the reliability of the measurement. This suggests that if a perfectly mixed sample could be obtained, there would be only Ex_1. However, this is not the case because of the experimental considerations for this material. $KClO_3$/dodecane [10] and $KClO_4$/dodecane [11] mixtures also exhibit sampling issues that produce variable DSC results, indicating that the standard conditions for DSC screening applied to military materials may be misleading for HMEs.

VI. Summary

SSST testing techniques have been used for military explosives to develop safe handling practices. These techniques have also been applied to HMEs, using the same protocols for testing. The results have shown that for HMEs, the testing techniques are not as reliable in establishing safe handling conditions because the results are, in many cases, ambiguous or confusing. The physical and chemical properties of the HME are the source of the issues. These issues can produce misleading results for both absolute and relative stability. The problem caused in thermal testing by DSC is an example of misleading results when looking for absolute stability. The problem caused by sandpaper grit size is and example of misleading results when looking for relative stability.

The above issues are not well appreciated by the energetic materials community probably because of the lack of experience with HMEs. However, the results reported here are just some of the many examples that have been observed in the IDCA testing of HMEs, and the community needs to be aware that the SSST testing methods applied to military explosives may yield misleading results when applied to HMEs. Standardization is the solution for the testing community—develop testing methods, materials standards and equipment calibrations for HMEs.

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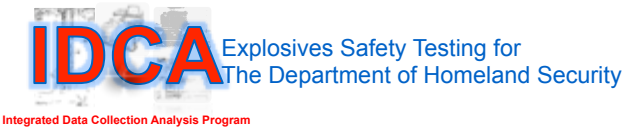

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