

DHS small-scale safety and thermal testing of improvised explosives—comparison of testing performance

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Abstract. One of the first steps in establishing safe handling procedures for explosives is small-scale safety and thermal (SSST) testing. To better understand the response of improvised materials or homemade explosives (HMEs) to SSST testing, 16 HME materials were compared to three standard military explosives in a proficiency-type round robin study among five laboratories—two DoD and three DOE—sponsored by DHS. The testing matrix has been designed to address problems encountered with improvised materials—powder mixtures, liquid suspensions, partially wetted solids, immiscible liquids, and reactive materials. More than 30 issues have been identified that indicate standard test methods may require modification when applied to HMEs to derive accurate sensitivity assessments needed for developing safe handling and storage practices. This paper presents a generalized comparison of the results among the testing participants, comparison of friction results from BAM (German Bundesanstalt für Materialprüfung) and ABL (Allegany Ballistics Laboratory) designed testing equipment, and an overview of the statistical results from the RDX (1,3,5-Trinitroperhydro-1,3,5-triazine) standard tested throughout the proficiency test.

1. Introduction

Small-scale safety and thermal (SSST) testing is usually the first step in developing safe handling practices of energetic materials [1]. These tests were designed for explosives to determine sensitivity of the material to handling conditions—drop hammer for impact sensitivity; friction for shear force sensitivity; electrostatic discharge (ESD) 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 or is in question. Results determine (depending upon interpretation) whether a material can be directly handled, remotely mixed, or require complete robotic handling.

The Integrated Data Collection Analysis Program (IDCA) has been conducting SSST testing on a series of homemade (HME) or improvised explosives, utilizing standard SSST testing practices as applied to military explosives [2]. The testing has come about through a round-robin or proficiency test where 19 HMEs and military explosives have been tested by three U.S. Department of Energy and two



U.S. Department of Defense Laboratories—Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Sandia National Laboratories (SNL), Naval Surface Warfare Center, Indian Head (IHD), and Air Force Research Laboratory, Tyndall Air Force Base (AFRL).

The results so far have indicated that standard testing methods are not always adequate for HMEs, as many conflicting and inconclusive results have been documented. However, there are many aspects of the testing results that provide new and critical information to be considered when applying standard SSST testing methods to HMEs. This paper presents a generalized comparison of the results among the participants, comparison of BAM and ABL friction results, and an overview of the preliminary statistical analysis of the RDX standard tested throughout the proficiency test.

2. Small-Scale Safety and Thermal Testing

Test apparatus, *Impact*: LANL, LLNL, IHD—Explosives Research Laboratory (ERL) Type 12 Drop Weight Sensitivity Apparatus, SNL, AFRL—Modified Bureau of Mines (MBOM) modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD, SNL—German Bundesanstalt für Materialprüfung (BAM) Friction Apparatus, LANL, IHD, AFRL—Allegheny Ballistics Laboratory (ABL) Friction Apparatus; *Spark*: LLNL, LANL, IHD, SNL, AFRL—ABL Electrostatic Discharge Apparatus, LLNL—custom-built Electrostatic Discharge Apparatus; *Differential Scanning Calorimetry*: LANL—TA Instruments Q1000, Q2000, LLNL—TA Instruments 2910, 2920, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

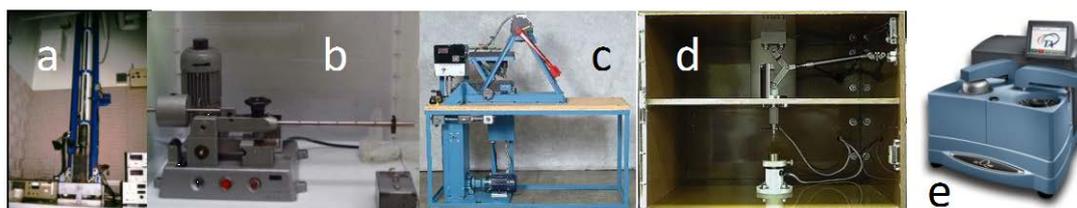


Figure 1. Examples of SSST testing equipment used in Proficiency Test—a. Drop Hammer, b. BAM Friction, c. ABL Friction, d. ABL Electrostatic Discharge, e. Differential Scanning Calorimetry.

Figure 1 shows representative examples of the SSST testing equipment used in this program [3]. Each laboratory has purchased and built equipment over decades. The versions and configurations are similar but not identical. However, for each test, the equipment generally functions by the same principal, so most of the results can be compared among the participants. The exceptions to this are BAM and ABL friction equipment at IHD and AFRL and the custom-built spark equipment at LLNL.

Four basic tests are reported—impact, friction, spark, and thermal. The impact test is to evaluate the material for sensitivity to being dropped or having something dropped on it. During this test, a sample is placed on an anvil and a weight, which can be adjusted for both size and height, is dropped on it. The friction test (both BAM and ABL) evaluates the sensitivity of the material to shear forces, such as scraping or pinching. During BAM friction testing, the material is placed on a flat surface and a stylus set at different forces (using weights) is dragged through the material. During ABL friction testing, the sample is placed on a stage that is moved under a grooved surface set at different forces (using pressure). The spark test evaluates the response of the material to an electric discharge. During ESD testing, the material is placed on a grounded surface and a spark is sent through it. The thermal test evaluates if energy will be released upon heating, indicating thermal stability. During DSC testing, the sample is in a holder and the temperature is increased at a constant heating rate, as the heat flow in and out of the sample is monitored.

Positive results (indication of stimulus level where the material exhibits sensitive) for the first three tests are usually a pop, a flash, or the evolution of smoke, or any combination thereof. The way a positive or negative result is assessed varies among laboratories. Personnel do the monitoring in most tests, but electronic equipment is used in some tests. Positive results for a thermal test are usually indicated as positive heat flow as a function of time, but are usually displayed as a function of temperature.

3. Comparison of results among testing participants

In this proficiency test, at least two participants, but usually three to four participants tested each of the 19 materials. Except in rare cases, the testing included drop hammer, BAM and ABL friction, ESD, and DSC. The graphs in this section compare the testing results from each of the participants to the average of all the participants for each of the materials. The axes are the value of the sensitivity parameter. The x-axis is for the average data value for a specific material, and the y-axis is for the corresponding data from the individual participant for the specific material. The red line connects the average of all the data from the participants for the specific materials and the markers are the sensitivity value from the individual participants for each of the materials [4].

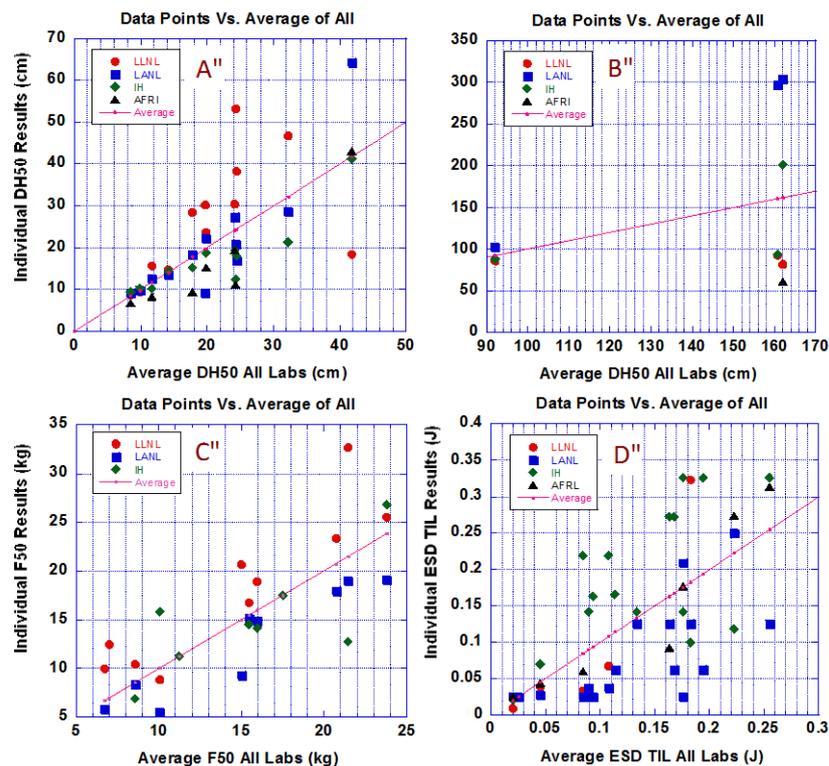


Figure 2. Testing results compared for all the participants—A and B, Impact; C, Friction; D, ESD.

3.1. Impact testing comparisons

Figures 2A and 2B compare the impact data as reported by DH_{50} determined by the Bruceton method. The individual laboratory data values above the line indicate a material determined to be less sensitive than the corresponding average, and values below the line indicate a material determined to be more sensitive than the corresponding average. Figure 2A shows materials that are very to moderately sensitive to impact and figure 2B shows materials that are reasonably insensitive to impact. Another set of data could not be included because the results were beyond the measuring limits of the equipment (insensitive materials).

Figures 2A and 2B show the following general behavior for the materials: LLNL values (circles) are above the average for DH_{50} values below 40 cm: LLNL values (circles) are below the average for DH_{50} values above 90 cm; LANL values (squares) are usually the same as the average for DH_{50} values below 40 cm; LANL values (squares) are above the average for DH_{50} values of 90 cm or more; AFRL DH_{50} values (triangles) usually are the lowest of the group for a specific material; IHD values (diamonds) usually track corresponding LANL DH_{50} values, but are lower in value.

Although the individual participants derive different values for a specific material, the trends above show some consistency and have a speculated basis in experimental parameters. One strong possibil-

ity is linked to the method of detection of a positive event. Both LLNL and LANL use microphones as well as observation. However, the microphones are different types with different response factors and different placement. LANL and IHD have almost identical equipment, but differ in the detection method. AFRL has equipment that is MBOM with type 12 tooling, which is different than the rest of the participants. This configuration could produce a different type of acoustic and reaction environment so observation could be categorically different.

3.2. BAM Friction testing comparisons

Figure 2C shows the comparison of the F_{50} values determined by a modified Bruceton method. As before, the red line is the average value line and the data points are from each participant. Only data generated by LLNL, LANL, and IHD are including because AFRL does not have BAM friction equipment.

In general, the LLNL values (circles) are above the average line, indicating LLNL finds the materials less sensitive to friction than the other participants. The corresponding F_{50} values for LANL and IHD track each other in many cases. These behaviors can be explained by differences in equipment configurations. The LLNL apparatus is inside a sealed glove box with a HEPA filter and driving ventilation fan to contain volatiles, while the other participants have just ventilation hoses. The result is that the LLNL equipment is acoustically more isolated and that the operator cannot hear a positive reaction as well as in the other cases while LANL and IHD have similar configurations and therefore obtain similar results.

3.3. ESD testing comparisons

Figure 2D shows the comparison of the Threshold Initiation Level (TIL) values. This graph includes mostly LANL and IHD data with a few measurements by LLNL and AFRL. The red line is the average value line.

In general, the IHD values are above the average values, and LANL values are below the average values, although there are exceptions to this. Possibly this difference can be explained by humidity effects, which has been documented to effect static discharge. IHD has typically 50% relative humidity all year; LANL has <10% relative humidity except for one or two months a year.

4. BAM Friction testing results compared to ABL Friction testing results

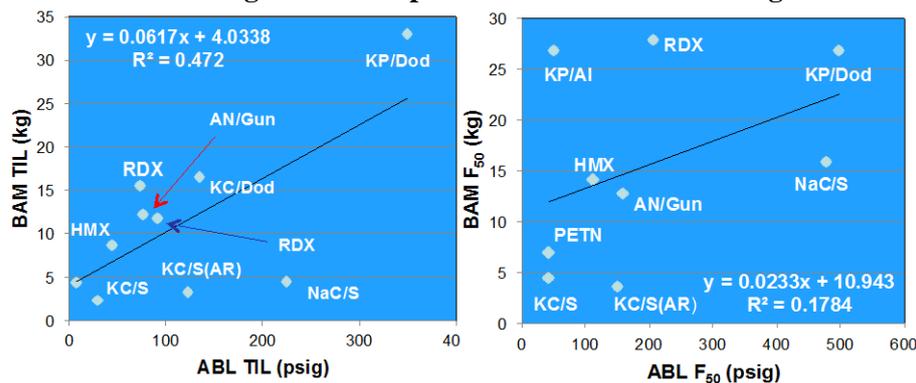


Figure 3. BAM friction data compared to ABL friction data.

Only IHD used both BAM and ABL friction equipment in the proficiency test. This provided a plethora of data by both methods at the same laboratory [5]. The IDCA examined this data to see if there were any numerical relationships between the corresponding data from the two test methods. Figure 3 shows the TIL (left side) and F_{50} (right side) values for each of the materials. The x-axis is for the ABL data values, and the y-axis is for the corresponding BAM data values. Clearly there is no correlation of the data between the two testing methods. Dividing the data into subgroups does not provide

any correlations (military standards, TIL $R^2 = 0.6818$, $F_{50} R^2 = 0.9332$; HMEs, TIL $R^2 = 0.5372$, $F_{50} R^2 = 0.16708$).

5. Preliminary statistical analysis of the RDX standard

The RDX Standard was tested four times throughout the proficiency test and each test was at least in triplicate. This provided a substantial amount of data for possible statistical analysis and the results could be used as the basis for the statistical evaluation of the other materials in the proficiency test [6].

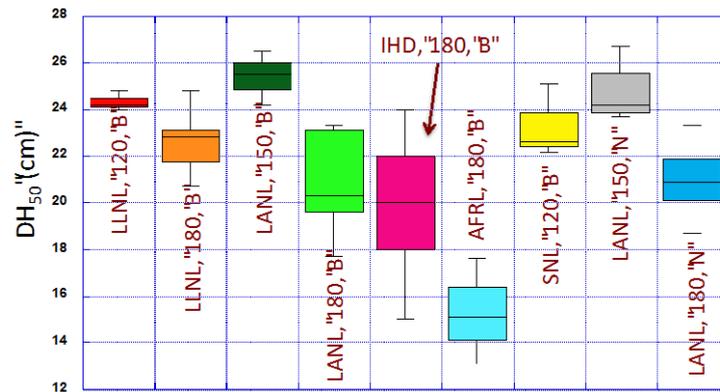


Figure 4. DH_{50} values grouped by parameters and participants—participant is indicated; sandpaper is designated by grit size; DH_{50} values derived by B = Bruceton method; N = Neyer (D-optimal) method.

The RDX was from the same source, prepared, and stored in the same manner for all participants. There were variations in the testing conditions—sandpaper type, striker weight, temperature, humidity, and detection method, as well as minor equipment variables, such as surface conditions, age, and calibration. Figure 4 shows the impact data grouped according to the participant, sandpaper (identified by grit-size), and data method. The boxes are 50% of the data, the median is the horizontal line, the mean is the middle of the box and extremes indicated by vertical bars. Clearly, the groups are spread apart, but some extreme values indicate overlap. ANOVA analysis indicates that at least one set is statistically different. Subgrouping, based on specific parameters such as sandpaper type, tightens up the distribution. Application of further statistical analyses, such as Tukey and Fisher, show that the AFRL, 180, B stands out as different than the rest. The results of these analyses will be discussed in more detail elsewhere [7].

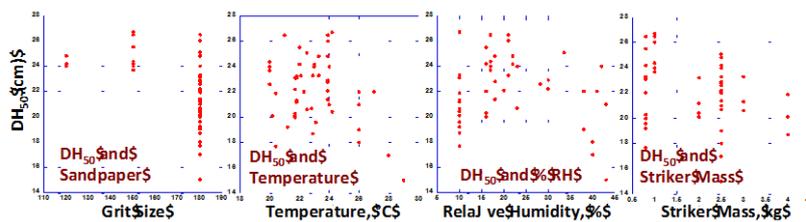


Figure 5. Overall DH_{50} values compared as a function of impact testing parameters.

Figure 5 shows the DH_{50} values as a function of some of the testing parameters discussed above. The only parameter that shows any systematic effect is the sandpaper type. Temperature, humidity, and striker weight exhibit essentially random patterns.

6. Summary and conclusions

Differences in testing results from the proficiency test indicate that equipment configuration and detection modalities are the principal causes for the differences in results. These differences are sufficient to establish statistical differences in results among the testing laboratories for a specific material. These differences appear to be more systematic than random.

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