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Challenges of Small-Scale Safety and Thermal Testing of Improvised Explosives

J. G. Reynolds, M. M. Sandstrom, G. W. Brown, K. F. Warner, D. N. Sorensen, D. L. Remmers, T. J. Shelly, J. A. Reyes, L. L. Whinnery, P. C. Hsu

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Challenges of Small-Scale Safety and Thermal Testing of Improvised Explosives

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

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

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

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

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

LeRoy L. Whinnery
Sandia National Laboratories, SNL
Livermore, CA USA

Abstract—**One of the first steps in establishing safe handling procedures for explosives is Small-Scale Safety and Thermal (SSST) testing. Through these tests, an explosive can be evaluated for sensitivity to impact, friction, electrostatic discharge, and thermal instabilities. For military and commercial explosives, there is abundant SSST test data. The opposite is true for the improvised or home made explosives, where little data exists. To better understand the response to SSST testing, several improvised materials are being studied in a proficiency-type round robin study among selected laboratories. 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. This paper discusses experimental difficulties encountered when testing these problematic samples and show inter-laboratory testing results on several improvised materials.**

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

I. INTRODUCTION

Small-Scale Safety and Thermal (SSST) tests are critical and usually a first step in deciding whether an explosive or mixture is safe to handle [1,2]. These tests were designed for explosives to indicate sensitivity of the explosive 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 done when sensitivity of explosive is not known, when direct handling is desired, when the explosive performance (e.g., release energy and velocity of detonation) is not known (usually very small quantities of < 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 directly handled, remotely mixed, or require complete robotic handling.

Recent interest in improvised or Home Made Explosives (HMEs) has shown that there is not much known scientifically about these materials [3]. As a result, explosive performance needs to be experimentally determined, which requires in many cases, as the preliminary step, SSST testing to develop safe handling procedures. However, HMEs tend to have different physical properties than traditional explosives, so some adaptation of standard SSST testing must be considered.

Often, HMEs are formed by mixing oxidizer and fuel precursor materials, and typically, the precursors are combined shortly before use [4]. The challenges to produce a standardized inter-laboratory sample for SSST testing 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.

The Integrated Data Collection Analysis (IDCA) program is conducting a SSST Round Robin Test (Proficiency Test) on selected HMEs. This effort, funded by the Department of Homeland Security (DHS), is to put the issues of safe handling of these materials in perspective with standard military explosives, as well as populate the literature with SSST testing data on HMEs. Each participating testing laboratory uses identical test materials and preparation methods whenever

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possible. The test procedures can differ among the laboratories, so the test results can be compared to understand inter and intra-laboratory testing differences.

Reported here are some examples of the data and the issues that are derived from the Proficiency Test which reflect on the SSST testing of HMEs. These examples are derived from data taken at four testing laboratories [two Department of Energy (DOE) and two Department of Defense], and highlight the nuances of applying standard SSST tests to non-standard materials as well as the different sensitivity of the HME compared to standard military explosives.

The testing participants in the study are Los Alamos National Laboratory (LANL), Naval Surface Warfare Center, Indian Head Division (NSWC IHD), Air Force Research Laboratory-RXQF, Tyndall Air Force Base (AFRL-RXQF), and Lawrence Livermore National Laboratory (LLNL). Sandia National Laboratories (SNL) also contributed through characterization efforts.

II. Small-Scale Safety and Thermal Testing

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

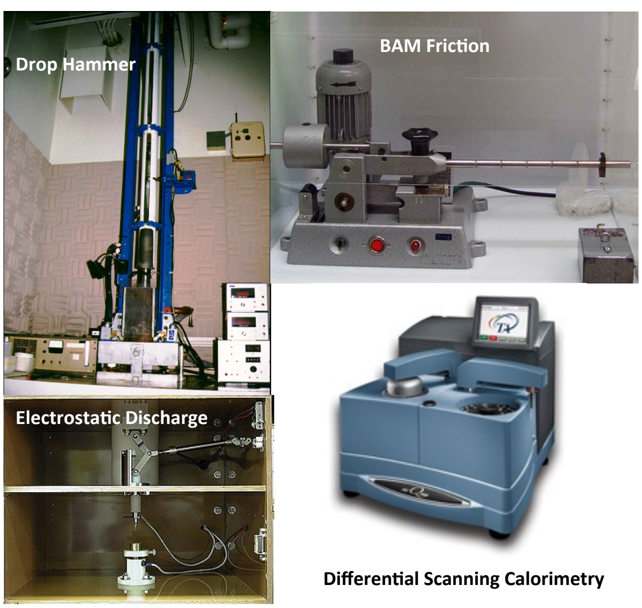

Figure 1. Examples of SSST Testing Equipment.

Figure 1 shows representative examples of the SSST testing equipment used in this program. 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 tester 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. The actual test involves a sample on an anvil where a weight that can be varied in amount, is dropped on it. The friction test (both BAM and ABL) evaluates the sensitivity of the material to sheer forces such as scraping or pinching. The actual test involves the material placed on a flat surface and a stylus set at different forces (using weights or pressures) is dragged through the material. The spark test evaluates if the material will respond to an electric discharge. The actual test involves the material 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. The actual test utilizes a DSC where the sample is in a holder and the temperature is increased at a constant heating rate. The heat flow in and out of the sample is monitored.

Positive results (indication of where the material becomes sensitive) for the first three tests are usually a pop, a flash, or the evolution of smoke, or any combination. The way a positive or negative is assessed varies among laboratories. Most tests use personnel to do the monitoring, but some tests use electronic equipment. Positive results for a thermal test are usually indicated on a graph as positive heat flow as a function of time, but are usually displayed as a function of temperature.

III. Results

Through the Proficiency Test, several comparisons have come to the forefront when comparing SSST data among the participants. These can be grouped into two categories—differences due to testing techniques and differences in results. Below are some, but not all, examples of these issues.

A. *Impact Testing—Is Sample Form a Factor?*

TABLE I. Impact Testing 1,3,5-Trinitroperhydro-1,3,5-triazine (RDX)

Lab	Form[a]	T, °C	RH, %[b]	DH_{50}, cm[c]	s, log unit[d]
LLNL	Pellet	24	18	28.8	0.042
LLNL	Pellet	23.9	32	34.0	0.059
LLNL	Powder	23	22	24.2	0.015
LLNL	Powder	23	23	24.0	0.035

a. Form of sample. b. Relative humidity. c. Modified Bruceton method load for 50% reaction (DH_{50}). d. Standard deviation.

Table 1 shows the form of the sample is very important in determining the correct impact sensitivity. For this test, the sample size is ~35 mg. In the pressed form, the sample is mechanically pressed to about 90% of Theoretical Maximum Density. In the powder form, the sample is a conical pile formed by pouring the sample into place. In this case, with RDX standard, the pressed sample shows more stability to impact than the powder sample. Many military materials are used pressed, but because many of the HME materials are used as powders, the appropriate sample configuration is powder.

B. *Impact Testing—Is Sandpaper Grit Size a Factor?*

Table 2 shows properties of the sandpaper used in holding the sample in place in the impact test can directly influence the indication of sensitivity. In the impact test, solid samples are typically held in place on the testing anvil using sandpaper. The type of sandpaper is only suggested in the specifications of the Type 12 Impact test [5]. As a result, each testing laboratory usually chooses sandpaper and uses that paper for routine testing, comparing the results with standards that are run using the same grit-size sandpaper. The drop hammer test was performed on a $KClO_3$/Dodecane mixture using different grit size sandpapers. All three participants did some testing with the 180-grit size and the DH_{50} values are roughly the same. LANL did testing with 150-grit size sandpaper also. Those values, on average, are slightly higher (indicating the material is more stable) than the corresponding data from the 180-grit sandpaper. LLNL also tested 120-grit sandpaper and those results are significantly higher (indicating a much more stable material) than the corresponding data from the 180-grit sandpaper.

TABLE II. $KClO_3$/DODECANE IMPACT DATA

Lab	Grit[a]	T, °C	RH, %[b]	DH_{50}, cm[c]	s, log unit[d]
LLNL	120	23.9	23	38.2	0.041
LLNL	120	23.9	22	40.5	0.020
LLNL	120	22.2	16	36.7	0.095
LLNL	180	23.3	20	9.0	0.054
LLNL	180	23.3	18	9.6	0.048
LANL	150	24.0	<10	12.6	0.048
LANL	150	23.3	<10	9.0	0.068
LANL	150	24.0	<10	12.1	0.040
LANL	180	22.7	<10	6.4	0.061
LANL	180	21.3	<10	7.6	0.027
LANL	180	21.6	<10	10.2	0.080
IHD	180	20	42	9	0.10
IHD	180	20	45	12	0.07
IHD	180	20	46	10	0.13

a. grit size of sandpaper. b. Relative humidity. c. Modified Bruceton method, load for 50% reaction (DH_{50}). d. Standard deviation.

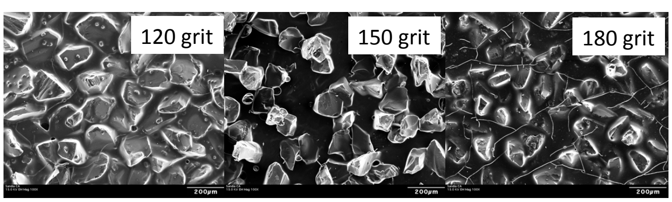

Figure 2. SEM images of sandpaper in the impact tests.

Figure 2 shows Scanning Electron Microscope images of the sandpapers used in the impact testing. Although the density of particles appears to remain fairly constant among the images, the particle size of the grit decreases with increasing grit size, consistent with the CAMI specifications for the sandpaper [6].

C. *Impact Testing—Is Particle Size a Factor?*

Table 3 shows, for this limited data set, the particle size of the sample may not have importance in the impact sensitivity of a $KClO_3$/sugar mixture. For this test, mixtures at two different particle sizes were prepared where the $KClO_3$ was sized through a 100-mesh sieve and used As Received (AR—fit through a 40-mesh sieve). The mixtures were then tested for impact sensitivity using 180-grit sandpaper in the impact test. There is a spread in values for DH_{50}, but it is difficult to definitely assign it to particle size, instead more aligned with inter-laboratory differences. For example, the data from IHD shows some differences between the -100 and the AR values, but the data from LANL does not.

TABLE III. IMPACT TESTING RESULTS FOR $KClO_3$/ICING SUGAR MIXTURES

Lab	Sample[a]	T, °C	RH, %[b]	DH_{50}, cm[c]	s, log unit[d]
LANL	-100	22.3	<10	10.7	0.076
LANL	-100	22.1	<10	11.8	0.147
LANL	-100	22.0	<10	9.2	0.062
LANL	AR	22.5	<10	11.0	0.139
LANL	AR	21.5	<10	10.7	0.105
LANL	AR	21.0	<10	9.5	0.043
IHD	-100	26	40	14	0.07
IHD	-100	27	40	15	0.18
IHD	-100	27	40	14	0.14
IHD	AR	20	45	9	0.120
IHD	AR	20	50	11	0.090
IHD	AR	20	50	11	0.070
AFRL	AR	26.7	54	10.2	0.146
AFRL	AR	26.1	57	6.9	0.279
AFRL	AR	25.6	57	7.9	0.447

a. Particle size of $KClO_3$, -100—sized through a 100-mesh sieve, AR—sized through a 40-mesh sieve. b. relative humidity. c. Modified Bruceton method load for 50% reaction (DH_{50}). d. Standard deviation.

D. *Impact Testing—Is the Analysis Method a Factor?*

Table 4 shows the effect of using different analysis methods, Bruceton vs. Neyer, for the impact testing. The Bruceton method [7] is a manual assessment method that is the traditional way to determine test parameters for impact sensitivity. The Neyer method [8] is more recently developed and utilizes computer assistance in determining impact test parameters.

TABLE IV. IMPACT DATA BRUCETON VS. NEYER $KClO_3$/DODECANE MIXTURE

Lab[a]	Type[b]	T, °C	RH, %[c]	DH_{50}, cm[d]	s, log unit[e]
LANL (150)	B	24.0	<10	12.6	0.048
LANL (150)	B	23.3	<10	9.0	0.068
LANL (150)	B	24.0	<10	12.1	0.040
LANL (150)	N	24.0	<10	12.3	0.082
LANL (150)	N	24.0	<10	10.2	0.175
LANL (150)	N	23.0	<10	13.6	0.111
LANL (180)	B	22.7	<10	6.4	0.061
LANL (180)	B	21.3	<10	7.6	0.027
LANL (180)	B	21.6	<10	10.2	0.080
LANL (180)	N	22.0	<10	6.9	0.038
LANL (180)	N	22.0	15.4	7.6	0.029
LANL (180)	N	21.3	<10	9.3	0.071

a. grit size of sandpaper. b. analysis method, B = Bruceton, N = Neyer. c. relative humidity. d. Bruceton or Neyer method, load for 50% reaction (DH_{50}). e. standard deviation.

Both methods of analysis were used for the impact analysis of a $KClO_3$/Dodecane mixture. As well, two sets of data were collected 180- and 150-grit sandpaper. The DH_{50} values are similar for both methods where 180-grit sandpaper was used. Likewise, the same holds true for the 150-grit sandpaper results.

E. Friction Testing—Is the Testing Equipment a Factor?

TABLE V. BAM FRICTION AND ABL FRICTION $KClO_3$/ICING SUGAR MIXTURE

Lab	Method[a]	T, °C	RH, %[b]	TIL[c]
IHD	BAM	29	39	0/10 @ 2.5 kg
IHD	BAM	29	40	0/10 @ 2.9 kg
IHD	BAM	29	38	0/10 @ 2.9 kg
IHD	ABL	27	45	0/20 @ 100/8 psig/fps
IHD	ABL	27	42	0/20 @ 75/8 psig/fps
IHD	ABL	27	43	0/20 @ 100/8 psig/fps

a. Analysis equipment. b. relative humidity. c. 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.

Table 5 shows the non-orthogonal nature of two different types of friction testing equipment. Friction testing is commonly done with BAM, and less commonly with ABL friction testing equipment. BAM friction testing uses a weighted stylus to vary the loading on the sample. ABL uses pressure to do the same action. Although these methods seem similar, there are big issues in inter-converting results, and the table shows the information is not obviously inter-convertible. However, this Proficiency Test is testing the same material using both types of equipment, providing some basis for analysis equivalence.

F. Electrostatic Discharge Testing—Very Sensitive Material

Table 6 shows $KClO_4$ and aluminum mixture is highly spark sensitive. Spark sensitivity is analyzed to prevent issues from discharge of static charge during handling. Although the testing apparatus are different for each participant, this material shows that it is very spark sensitive by all participants.

TABLE VI. ELECTROSTATIC DISCHARGE TESTING $KClO_4$/AL MIXTURE

Lab	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	23.3	18	nd[5]	1/10 @ 0.49[5]
LLNL[4]	22.2	23	0/10 @ 0.25[5]	2/3 @ 0.64[5]
LLNL[4]	22.2	23	0/10 @ 0.25[5]	2/6 @ 0.64[5]
LANL	22.6	23.5	< 0.0625	< 0.0625
LANL	21.3	26.0	< 0.0625	< 0.0625
LANL	21.6	28.7	< 0.0625	< 0.0625
IHD	22	44	0/20 @ 0.023	1/3 @ 0.037
IHD	22	44	0/20 @ 0.015	1/4 @ 0.023
IHD	22	45	0/20 @ 0.023	1/3 @ 0.037

1. Relative humidity. 2. Threshold Initiation Level (TIL) is the load (joules) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level. 3. Next level where positive initiation is detected. 4. LLNL uses a 510-ohm resistor in the discharge unit to mimic the human body.

G. Thermal Testing—Is Sampling an Issue?

Figure 3 shows that sampling can affect the thermal behavior of some HME materials. Thermal sensitivity is typically done using DSC. In this test, a very small sample is heated at a constant heating rate while the energy flow from the sample is measured. If there is exothermic energy flow, the material is considered energetic and, depending upon the temperature of this discharge, the material can be considered highly thermally sensitive. This analysis is routine in many laboratories, and for military and commercial explosives, is fairly reliable. However, with the HMEs, this analysis method is showing to be problematic.

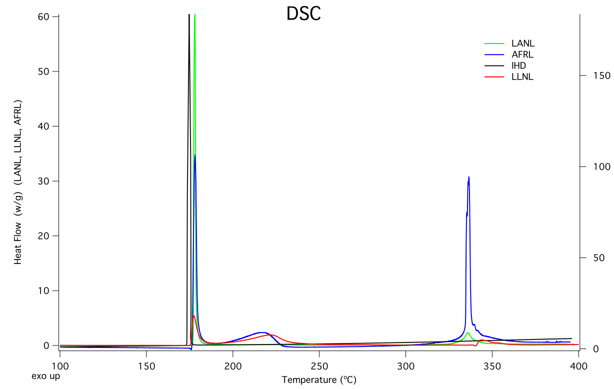

Figure 3. DSC of $KClO_3$/Sugar mixtures at 10°C/min heating rate.

The figure shows DSC profiles for the same $KClO_3$/sugar mixture examined at four different laboratories. In this figure, only the positive heat flow is seen indicating exothermic events (no endothermic events are shown in these profiles). Depending upon the laboratory, there are 1 to 3 exothermic events seen. Because all laboratories observed any combination of these features with at least one sample, this effect is not due to laboratory expertise, but appears to be an issue with sampling. The important point to glean from this behavior is that the low temperature exothermic feature must be used to indicate thermal stability even though the overall behavior is yet to be clearly understood—why the number of exothermic features varies base on sample.

H. Thermal Testing—Is the Sample Container an Issue?

Figure 3 shows that sample containment can be an issue for DSC. Some HME materials have a liquid component that is volatile. This can be problematic for any of the SSST testing because the component could evaporate before the test is completed. To circumvent this issue for impact, friction, and spark testing, because these tests are generally performed at room temperature, the sample is freshly made and tested quickly before the volatile component can escape.

This volatility is also a particular issue for thermal testing because in DSC the temperature is increased as a function of time and is quickly elevated. Figure 4 demonstrates this issue. In this set of data, a $KClO_3$/dodecane mixture was examined using a vented sample (open) holder, where any gases formed during the analysis can escape, and using a hermetically sealed sample holder where gases forming cannot escape unless there is a catastrophic failure of the holder. In the top profile, the endothermic feature (negative heat flow) is due to the $KClO_3$ melting [9]. The high temperature broad exothermic features are due to $KClO_3$ decomposing [10]. There is no evidence of a thermally unstable species formed in this case. In the bottom profile (sealed sample holder), several exothermic features are observed, indicating that the sample does have low temperature thermal instability. The most likely explanation is that the configuration of the sample pan affects the ability of the Dodecane to contact the $KClO_3$.

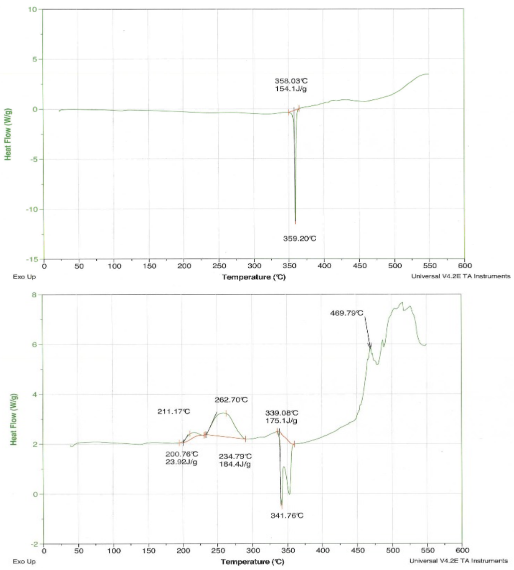

Figure 4. DSC profiles of $KClO_3$/Dodecane mixture with open (top) and sealed (bottom) sample holders.

I. Comparison of Results—Interlaboratory Variations?

TABLE VII. COMPARISON OF AVERAGE VALUES

	LLNL	LANL	IHD	AFRL
Impact Testing, DH_{50}, cm				
$KClO_3$/dodecane	9.3	8.1	10	ND
$KClO_3$/icing sugar (AR)	15.6	12.7	10.3	8.3
$KClO_3$/icing sugar (-100)	14.8	14.0	14.3	ND
RDX Class 5 Type II	23.8	25.4	19	15.3
PETN	15	14.7	ND	ND
BAM Friction Testing, TIL, kg				
$KClO_3$/dodecane	12.3	7.2	16.5	ND
$KClO_3$/icing sugar (AR)	9.5	2.4	3.2	ND
$KClO_3$/icing sugar (-100)	6.9	4.8	2.3	ND
RDX Class 5 Type II	19.2	19.2	15.5	ND
PETN	6.4	ND	ND	ND
BAM Friction Testing, F_{50}, kg				
$KClO_3$/dodecane	25.5	19.1	26.8	ND
$KClO_3$/icing sugar (AR)	11.8	4.9	3.6	ND
$KClO_3$/icing sugar (-100)	9.9	5.8	4.4	ND
RDX Class 5 Type II	25.1	20.8	ND	ND
PETN	10.5	9.2	ND	ND
ESD, TIL, J 0/20 @				
$KClO_3$/dodecane	1.0	0.125	0.140	ND
$KClO_3$/icing sugar (AR)	1.0	0.125	0.272	0.092
$KClO_3$/icing sugar (-100)	1.0	0.0625	0.272	ND
RDX Class 5 Type II	1.0	0.0250	0.095	0.044
PETN	1.0	0.0625	ND	ND

Notes: Temperature and humidity varied. Pentaerythritol tetranitrate (PETN) values from data taken outside of the Proficiency Test. 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. For impact, modified Bruceton method, load for 50% reaction (DH_{50}). For friction, modified Bruceton method, load for 50% Reaction (F_{50}). ND = not determined by a specific laboratory. LLNL has 500-ohm series resistor in circuit.

Table 7 shows comparison values from each laboratory for specific materials. The values were compiled by taking the average value of three of the same test (usually) from each laboratory. In some cases, not all laboratories participated in the analysis of each of the materials.

IV. SUMMARY

Several HMEs are undergoing SSST testing in a Proficiency (round robin) test sponsored by DHS. Four laboratories are involved in the SSST test measurements. The materials were chosen to represent HMEs in general. Specific materials were chosen to challenge analysis of unusual materials, such as solid-solid and solid-liquid mixtures. Some oddities were found in the results on almost every material.

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