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LLNL-TR-523203

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January 17, 2012

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 — $KClO_4$/Aluminum Mixture

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ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a Proficiency Test for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of a mixture of $KClO_4$ and aluminum—$KClO_4$/Al mixture. This material was selected because of the challenge of performing SSST testing of a mixture of two solids. The mixture was found to be: 1) much less sensitive to impact than RDX, (LLNL being the exception) and PETN, 2) more sensitive to friction than RDX and PETN, and 3) extremely sensitive to spark. The thermal analysis showed little or no exothermic character. One prominent endothermic feature was observed in the temperature range studied and identified as a phase transition of $KClO_4$.

This effort, funded by the Department of Homeland Security (DHS), ultimately will put 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 has the potential to 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. Note, however, the test procedures differ among the laboratories. The results are compared among the laboratories and then compared to historical data from various sources. The testing performers involved for the $KClO_4$/Al mixture are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), and Air Force Research Laboratory (AFRL/RXQL). These tests are conducted as a Proficiency Test in order to establish some consistency in test protocols, procedures, and experiments and to understand how to compare results when these things cannot be made consistent.

Keywords: Small-scale safety testing, Proficiency Test, round-robin test, safety testing protocols, HME, RDX, potassium chlorate, potassium perchlorate, sugar, dodecane, aluminum.

IDCA Explosives Safety Testing of Homemade Explosives
Integrated Data Collection Analysis Program

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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[1]. 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.

The IDCA has chosen several formulations 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.

Table 1. Materials for IDCA Proficiency Test

Oxidizer/Explosive	Fuel	Description
Potassium perchlorate	Aluminum	Powder mixture
Potassium perchlorate	Charcoal	Powder mixture
Potassium perchlorate	Dodecane[1]	Wet powder
Potassium chlorate	Dodecane[1]	Wet powder
Potassium chlorate as received	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Potassium chlorate -100 mesh[3]	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Sodium chlorate	Sucrose (icing sugar mixture)[2,3]	Powder mixture
Ammonium nitrate		Powder
Bullseye® smokeless powder[4]		Powder
Ammonium nitrate	Bullseye® smokeless powder[4]	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)

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

Evaluation of the results of SSST testing of explosive materials, such as the HMEs in Table 1, is generally done as a relative process, where a well 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.

The first and basic step in the Proficiency Test is to have representative data on a standard material to allow for basic performance comparisons. Table 1 includes some standard military materials. Class 5 Type II RDX was chosen as the primary standard, and Class 4 PETN was chosen as a secondary material. These materials are being tested in triplicate and RDX will continue to be tested throughout the IDCA Proficiency Test.

The subject of this report, $KClO_4$/Al mixture, is the fourth in a series of materials that fall in the class of solid oxidizer/fuel mixtures, the third that is a mixture of solid oxidizer and solid fuel, and the first having $KClO_4$ as an oxidizer. These materials were chosen for study in the Proficiency Test because of the challenge of testing fine solids mixed with fuels—adequate mixing on a small scale and representative sampling of a physical mixture—as well as an attempt to establish the reactivity of $KClO_4$ mixtures relative to $KClO_3$ mixtures. The $KClO_4$ was dried as previously described and separated through a 40-mesh sieve. The aluminum was a special type, Valimet H-2 spherical, and used as received.

The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), and Air Force Research Laboratory (AFRL/RXQL).

2 EXPERIMENTAL

General information. All samples were prepared according to the IDCA Program report on drying and mixing procedures[2,3]. The $KClO_4$ was obtained from Columbus Chemical as a purified powder, Catalog #441500, Lot # 200917617, CAS # 7778-74-7, assay (by manufacturer): $KClO_4$, > 99.0%; H_2O, < 0.1%; nominal particle size (by Microtrac and Coulter Counter) of 95% < 67 μm[4,5]. The aluminum was Valimet Brand H-2, which is 99.7% Al (Fe content < 0.2%); passes through a 325-mesh (44 μm hole size) sieve with a nominal particle size distribution (by Microtrac) of 90% 6.8 um, 50% 3.2 μm, 10% 1.7 μm[6]. The $KClO_4$ was dried for 16 h and cooled in a desiccator according to IDCA drying methods[3]. The $KClO_4$ was passed through a 40-mesh (425 μm hole size) sieve. The mixture was prepared by hand, adding the aluminum to the $KClO_4$ while stirring with a spatula in a materials compatible polypropylene container according to IDCA mixing and compatibility procedures[2]. The mixture composition is 68-wt. % $KClO_4$ and 32-wt. % aluminum. The final mixture had the appearance of a dry grey solid, with no evidence of free discrete particles of aluminum. Typically, the precursors are mixed at that ratio to give approximately a 1-gram sample. This sample is divided up for the various SSST testing. Three samples were prepared this way and tested separately. The mixing ratio was selected to be stoichiometric for oxygen balance 65.8-wt % $KClO_4$ and 34.2-wt % Al)[7].

The SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for KP/Al (68/32) Mixture [revised 4.1.11 and 8.25.11] (LLNL)[8], Potassium Perchlorate and Aluminum 51088E, revised 4.6.11 (LANL)[9], KP/Aluminum (IHD)[10], and Potassium Perchlorate (KP) + Aluminum (Al), (AFRL)[11].

Table 2. Summary of conditions for the analysis of $KClO_4$/Al mixture (All = LANL, LLNL, IHD, AFRL)

Impact Testing

1. Sample size—LLNL and IHD, 35 ± 2 mg; LANL 40 ± 2 mg
2. Preparation of samples—All, dried per IDCA drying methods[3]
3. Sample form—All, loose powder
4. Powder sample configuration—All, conical pile
5. Apparatus—LANL, LLNL, IHD, Type 12; AFRL, MBOM with Type 12 tooling*
6. Sandpaper—LANL, IHD, AFRL, LLNL, 180-grit garnet; LLNL, 120-grit Si/C
7. Sandpaper size—LLNL, IHD, AFRL, 1 inch square; LANL, 1.25 inch diameter disk dimpled;
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL, IHD, AFRL, 2.5 kg; LANL, 0.8 kg
10. Positive detection—LANL and LLNL, microphones with electronic interpretation as well as observation; IHD and AFRL, observation
11. Data analysis—All, modified Bruceton and TIL before and above threshold; LANL and AFRL Neyer also

Friction analysis

1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA procedures[3]
3. Sample form—All, powder
4. Sample configuration—All, small circle form
5. Apparatus—LANL, LLNL, IHD, BAM; IHD, AFRL, ABL
6. Positive detection—All, by observation
7. Room Lights—LANL and AFRL on; LLNL off; IHD, BAM on, ABL off
8. Data analysis—LLNL and IHD, modified Bruceton (log-scale spacing) and TIL; LANL, modified Bruceton (linear spacing) and TIL; AFRL, TIL

ESD

1. Sample size—All ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA drying methods[3]
3. Sample form—All, powder
4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD and AFRL, none
5. Sample configuration—All, cover the bottom of sample holder
6. Apparatus—LANL, IHD, AFRL, ABL; LLNL, ABL and custom built*
7. Positive detection—All, by observation
8. Data analysis methods—All, TIL

Differential Scanning Calorimetry

1. Sample size—All ~ <1 mg
2. Preparation of samples—All, dried per IDCA procedures[3]
3. Sample holder—All, hermetic with pin hole; LLNL also uses sealed pan
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C
6. Pan hole size—LLNL, 50 μm; LANL, IHD, AFRL, 75 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920; IHD, TA Instruments Q1000, AFRL—TA Instruments Q2000*

Footnotes: *Test apparatus, *Impact:* LANL, LLNL, IHD—ERL Type 12 Drop Weight Sensitivity Apparatus, AFRL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark:* LLNL, 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, Seteram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the $KClO_4$/Al mixture.

3 RESULTS

3.1 $KClO_4$/Al mixture

In this Proficiency Test, all participants are required to use materials from the same batch, and mixtures are to be prepared by the same methods. However, the actual testing procedures can be different. These differences are described in the IDCA report on method comparisons[12], which compares the different procedures by each testing category. LANL, LLNL, IHD, and AFRL participated in this part of the SSST testing of the $KClO_4$. Screening the $KClO_4$ at -40 mesh was performed because the material seemed to naturally breakdown to a powder of about this size with slight mechanical agitation. The aluminum was already in a very fine powder form, so it was used as received. Although $KClO_4$ and aluminum mixtures can be made at a variety of mixing ratios, the ratio for this study was chosen to be stoichiometric.

3.2 Impact testing results for $KClO_4$/Al mixture

Table 3 shows the results of impact testing of the $KClO_4$/Al mixture as performed by LANL, LLNL, IHD, and AFRL. Differences in the testing procedures are shown in Table 2, and the notable differences are the sandpaper grit size, amount of sample, and the methods for detection of a positive event. All laboratories used 180-grit sandpaper, and LLNL used 120-grit flint paper, in addition, for the impact testing. All participants performed data analysis by modified Bruceton method[13,14] and LANL and AFRL also performed data analysis by the Neyer method[15].

Table 3. Impact testing results for $KClO_4$/Al mixture

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	5/3/10	23.3	22	> 177	NA[5]	NA[5]
LLNL (120)	5/19/10	22.8	28	> 177	NA[5]	NA[5]
LLNL (120)	5/26/10	23.3	24	> 177	NA[5]	NA[5]
LLNL (180)	10/19/11	23.3	29	17.9	2.9	0.07
LLNL (180)	10/19/11	23.9	30	16.8	2.0	0.04
LLNL (180)	10/20/11	23.3	31	16.1	0.7	0.02
LANL (180)	4/16/10	22.3	24.8	56.7	21.4	0.16
LANL (180)	4/19/10	21.1	26.0	60.0	5.5	0.04
LANL (180)	4/20/10	21.5	24.0	69.7	6.4	0.04
IHD (180)	11/11/10	22	42	43	8.0	0.08
IHD (180)	11/15/10	22	43	39	6.3	0.07
IHD (180)	11/16/10	20	48	42	10.8	0.11
AFRL (180)	9/20/11	23.9	56	42.9	4.9	0.05
AFRL (180)	9/21/11	23.9	54	45.1	8.4	0.08
AFRL (180)	10/19/11	22.8	40	45.4	7.3	0.07
AFRL (180)	10/19/11	23.9	39	36.8	7.7	0.09
AFRL (180)	10/19/11	23.9	38	36.9	2.6	0.03

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. Modified Bruceton method, in cm, load for 50% probability of reaction (DH_{50}); 4. Standard deviation; 5. NA = not applicable.

The test results from the three participating laboratories for impact show a large range for DH_{50} from 16 cm to insensitive. The average values for 180-grit sandpaper are (in cm) LLNL 16.9 ± 0.9; IHD, 41.3 ± 2.1; AFRL 41.4 ± 4.3; LANL, 62.1 ± 6.8. LLNL used two sandpaper sizes, 120-grit and 180-grit. The average values based on grit size are: 120, insensitive (exceeds equipment response): 180, 40.6 ± 15.8 cm (14 determinations). The standard deviation is below the 0.16 log unit range where applicable. The impact of step spacing will be evaluated in detail in a later report.

Table 4. Impact testing results for $KClO_4$/Al mixture (Neyer or D-Optimal Method)

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)	4/16/10	23.4	22.5	78.3	6.73	0.04
LANL (180)	4/19/10	21.2	28.6	53.1	6.96	0.06
LANL (180)	4/20/10	21.5	28.6	67.9	5.21	0.03
AFRL (180)	9/23/11	23.9	56	51.5	25.9	0.21

1. Number in parentheses indicates grit size of sandpaper; 2 Relative humidity; 3. Neyer method, load for 50% reaction (DH_{50}); 4. Standard deviation.

Table 4 shows the impact test results from LANL and AFRL using the Neyer or D-Optimal method[12]. The DH_{50} values are higher than the corresponding values analyzed by the Bruceton method. The average values are, in cm (Neyer, Bruceton): LANL—(66 ± 12.7, 62 ± 6.8), AFRL—(52, 41).

3.3 Friction testing results for $KClO_4$/Al mixture

Table 5 shows the BAM Friction testing performed by LANL, LLNL and IHD (AFRL does not have BAM friction testing). The difference in testing procedures by the three laboratories is shown in Table 2, and the notable differences are in the methods for positive detection. All participants performed data analysis using a modified Bruceton method[13,14] and the threshold initiation level method (TIL)[16]. The average friction values for F_{50} are, in kg: LLNL, 17.1 ± 2.6; LANL, 15.2 ± 0.9; IHD, 14.5 ± 1.0. The standard deviation values range for all $0.042 < s < 0.349$. The threshold values using averages of determinations by each laboratory, 0/10 @ (X) kg, are in the following order LANL (7.2) < LLNL (9.6) < IHD (12.2). For 1 or more positive events, 1/Y @ (Z) kg, the order is the same LANL (9.6) < LLNL (10.3) < IHD (14).

Table 5. BAM Friction Testing results for $KClO_4$/Al mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4]	s, kg[5]	s, log unit[5]
LLNL	4/30/10	23.3	18	0/10 @ 9.6	1/10 @ 10.2	14.1	1.37	0.04
LLNL	5/19/10	22.8	24	0/10 @ 10.8	2/10 @ 11.2	18.9	1.31	0.03
LLNL	5/24/10	23.3	21	0/10 @ 8.4	1/10 @ 9.6	18.3	7.34	0.17
LANL	4/16/10	22.1	26.5	NA[6]	NA[6]	14.1	9.42	0.35
LANL	4/19/10	21.8	21.5	NA[6]	NA[6]	15.6	4.00	0.11
LANL	4/20/10	21.2	27.4	NA[6]	NA[6]	15.8	3.65	0.10
LANL	4/16/10	23.5	22.6	0/10 @ 7.2	1/3 @ 9.6	NA[7]	NA[7]	NA[7]
LANL	4/19/10	21.7	21.4	0/10 @ 7.2	1/3 @ 9.6	NA[7]	NA[7]	NA[7]
LANL	4/20/10	21.5	25.8	0/10 @ 7.2	1/4 @ 9.6	NA[7]	NA[7]	NA[7]
IHD	11/11/10	23	44	NA[6]	NA[6]	15.4	1.71	0.05
IHD	11/11/10	24	44	NA[6]	NA[6]	14.8	1.62	0.05
IHD	11/11/10	24	45	NA[6]	NA[6]	13.4	1.85	0.06
IHD	11/4/10	25	44	0/10 @ 9.8	1/2 @ 11.0	NA[7]	NA[7]	NA[7]
IHD	11/9/10	23	44	0/10 @ 14.7	1/1 @ 16.3	NA[7]	NA[7]	NA[7]
IHD	11/9/10	23	45	0/10 @ 12.2	1/7 @ 14.7	NA[7]	NA[7]	NA[7]

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load, in 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. Next level where positive initiation is detected; 4. Modified Bruceton method, load, in kg, for 50% probability of reaction (F_{50}), LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. Not applicable, separate sample used for TIL analysis; 7. Not applicable, separate sample used for Bruceton analysis.

Table 6 shows the ABL Friction testing performed by IHD and AFRL on the $KClO_4$/Al mixture. LLNL does not have and LANL did not perform ABL friction testing. The results from IHD show the F_{50} is around 51 psig @ 8 fps and the TIL value was too low to measure. AFRL did only TIL measurements and found the material was sensitive to the lowest level of stimulation so a TIL was not attainable.

Table 6. ABL Friction testing results for $KClO_4$/Al mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[2,4]	F_{50}, psig/fps[2,5]	s, psig[6]	s, log unit[6]
IHD	11/5/10	26	43	NA[6]	1/4 @ 30/8	NA[6]	NA[6]	NA[6]
IHD	11/5/10	25	42	NA[6]	1/5 @ 30/8	NA[6]	NA[6]	NA[6]
IHD	11/5/10	24	41	NA[6]	1/2 @ 30/8	NA[6]	NA[6]	NA[6]
IHD	11/5/10	24	42	NA[6]	NA[6]	53/8	26	0.21
IHD	11/5/10	22	43	NA[6]	NA[6]	48/8	7	0.14
IHD	11/5/10	22	44	NA[6]	NA[6]	52/8	13	0.11
AFRL	9/20/11	22.8	49	NA[6]	6/10 @ 25/8	NA[6]	NA[6]	NA[6]
AFRL	9/20/11	23.9	52	NA[6]	10/13 @ 25/8	NA[6]	NA[6]	NA[6]
AFRL	9/21/11	23.9	52	NA[6]	9/12 @ 25/8	NA[6]	NA[6]	NA[6]

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the load, in psig, 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; 4. Next level where positive initiation is detected; 5. Modified Bruceton method, load, in psig, for 50% probability of reaction (F_{50}); 6. Standard deviation; Not applicable.

3.4 Electrostatic discharge testing of $KClO_4$/Al mixture

Electrostatic Discharge (ESD) testing of the $KClO_4$/Al mixture was performed by LANL, IHD, LLNL, and AFRL. Table 7 shows the results. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape and what covers the sample. All participants performed data analysis using the threshold initiation level method (TIL)[16]. In addition, LLNL also uses a custom built ESD system with a 510-Ω series resistor to simulate a human body, in addition to the new ABL system.

Table 7. Electrostatic discharge testing $KClO_4$/Al mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	4/30/10	23.3	18	ND[5]	1/10 @ 0.49
LLNL[4]	5/19/10	22.2	23	0/10 @ 0.25	2/3 @ 0.64
LLNL[4]	5/24/10	22.2	23	0/10 @ 0.25	2/6 @ 0.64
LLNL	11/30/11	23.9	13	0/10 @ 0.0088	2/3 @ 0.013
LLNL	12/1/11	23.9	13	0/10 @ 0.0088	2/4 @ 0.013
LANL	4/16/10	22.6	23.5	< 0.0625	< 0.0625
LANL	4/19/10	21.3	26.0	< 0.0625	< 0.0625
LANL	4/20/10	21.6	28.7	< 0.0625	< 0.0625
LANL	10/18/11	22.2	12.2	0/20 @ 0.0125	2/5 @ 0.025
IHD	11/5/10	22	44	0/20 @ 0.023	1/3 @ 0.037
IHD	11/5/10	22	44	0/20 @ 0.015	1/4 @ 0.023
IHD	11/5/10	22	45	0/20 @ 0.023	1/3 @ 0.037
AFRL	9/20/11	22.8	47	0/20 @ 0.025	3/21 @ 0.026
AFRL	9/20/11	22.8	51	0/20 @ 0.019	1/1 @ 0.025
AFRL	9/20/11	23.9	50	0/20 @ 0.031	1/4 @ 0.038

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the load, in 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 simulate the human body. 5. No TIL could be measured.

The testing results from all four participants indicate the $KClO_4$/Al mixture is a very spark sensitive material. The LLNL data from the custom built system are substantially higher than the rest of the group because of the experimental configuration but the LLNL data from the ABL system is comparable to the other participants. All participants were able to measure TIL values.

3.5 Thermal testing (DSC) of $KClO_4$/Al mixture

Differential Scanning Calorimetry (DSC) was performed on the $KClO_4$/Al mixture by all participating laboratories using different versions of the DSC by TA Instruments.

Table 8. Differential Scanning Calorimetry results for $KClO_4$/Al mixture, 10°C/min heating rate

Lab	Test Date	Endothermic, onset/minimum °C (ΔH, J/g)
LLNL[1]	5/4/10	302.3/304.3 (71)
LLNL[1]	5/4/10	302.3/304.3 (70)
LLNL[1]	5/4/10	302.4/304.3 (68)
LLNL[2]	5/4/0	302.5/304/4 (75)
LLNL[2]	5/5/10	302.5/304.5 (67)
LLNL[2]	5/5/10	302.4/304.3 (59)
LANL[1]	4/19/10	303.1/304.9 (67)
LANL[1]	5/04/10	303.3/305.5 (66)
LANL[1]	5/04/10	303.2/305.2 (66)
IHD[1]	3/2/11	302.9/304.7 (69)
IHD[1]	3/2/11	302.0/304.6 (68)
IHD[1]	3/2/11	302.7/304.6 (70)
AFRL[1]	9/19/11	303.9/306.2 (69)
AFRL[1]	9/20/11	303.8/306.1 (73)
AFRL[1]	9/21/11	303.9/306.7 (70)

1. pin-hole vented sample holder lid; 2. Sealed sample holder lid.

Table 8 shows the DSC data from each of the participating laboratories. For all participants there is observed a sharp, high temperature endothermic feature with T_{min} values ranging from 304.2 to 306.7 °C. This is assigned as the rhombic-cubic transition of $KClO_4$ from previous work on the thermal behavior of $KClO_4$/fuel mixes by TGA, DTA, and DSC[17-20].

Table 8 shows the DSC data, by LLNL, for the $KClO_4$/Al mixture where the DSC sample holder is closed instead of pinhole vented as used in the other measurements shown in Table 8. The behavior of the profiles is identical to the pinhole vented samples. In previous studies, where the fuel was volatile, the difference between the pinhole and sealed sample holders indicated that the sealed pans were needed to observe thermal instability. In this case, the fuel is completely non-volatile and does not even melt at these temperatures (Al melts at 660°C[21]), so the thermal profile is only due to the $KClO_4$ rhombic to cubic transition[20].

4 DISCUSSION

Table 9 shows the average values for the data from each participant and compares it to corresponding data for standards, RDX and PETN. The data for RDX comes from the IDCA first iterative study of RDX as part of this Proficiency Test[22]. The data for PETN was provided by the participating laboratories (when available) from measurements performed outside this Proficiency Test. Table 9 allows the comparison of the average results on $KClO_4$/Al mixture with standards to obtain relative sensitivities.

4.1 Sensitivity of $KClO_4$/Al mixture compared to standards

Impact sensitivity. Compared to the standards RDX and PETN, the average values for impact sensitivity shown in Table 9 indicate $KClO_4$/Al is relatively insensitive to impact when using 180-grit sandpaper to hold the sample. LLNL found the material to be more sensitive than RDX, but less sensitive than PETN. LANL, IHD and AFRL found it considerably less sensitive than both RDX and PETN.

Friction sensitivity. All the participants that performed BAM friction agree, for the most part, on the sensitivity. The trends show that for both TIL and F_{50}, the $KClO_4$/Al mixture is more sensitive than RDX, but less sensitive than PETN.

Table 9. Average Comparison values

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
$KClO_4$/Al[2,3]	17[4]	62[5]	41[5]	41[6]
RDX Class 5 Type II[7]	24.1[8]	25.4[9]	19[3]	15.3[3]
PETN[10]	15	14.7	ND[11]	ND[11]
BAM Friction Testing[12,13]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
$KClO_4$/Al[14]	8.7[15]; 16.7[15]	7.2[15]; 15.2[15]	12.2[15]; 14.5[15]	ND[11]; ND[11]
RDX Class 5 Type II[7]	19.2; 25.1	19.2; 20.8	15.5; ND[11]	ND[11]; ND[11]
PETN[10]	6.4; 10.5	ND[11]; 9.2	ND[11]; ND[11]	ND[11]; ND[11]
ABL Friction Testing[16-19]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL[20], psig; F_{50}, psig	TIL[20], psig; F_{50}, psig
$KClO_4$/Al[21]	ND[11]; ND[11]	ND[11]; ND[11]	> 30, 8[22]; 51, 8[22]	25, 8[22]; ND[11]
RDX Class 5 Type II[7]	ND[11]; ND[11]	ND[11]; ND[11]	74, 8; 154, 8	93, 8; ND[11]
PETN[10]	ND[11]; ND[11]	ND[11]; ND[11]	ND[11]; ND[11]	ND[11]; ND[11]
Electrostatic Discharge[23]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
$KClO_4$/Al[24]	0/10 @ 0.0088[25,26]	0/20 @ 0.0125[27]	0/20 @ 0.0203[28]	0/20 @ 0.025[28]
RDX Class 5 Type II[7]	0/10 @ 1.0[29]	0/20 @ 0.0250	0/20 @ 0.095	0/20 @ 0.044
PETN[10]	0/10 @ 1.0[29]	0/20 @ 0.0625	ND[8]	ND[8]

1. DH_{50}, in cm, is by modified Bruceton method, load for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 24), LANL (21.1–22.3; 24.0–26.0), IHD (20–22; 42–48), AFRL (22.8-23.8; 38-56); 3. 180-grit sandpaper; 4. Average of three determinations at 180-grit sandpaper (DH_{50} > 177 cm for 120-grit sandpaper) from Table 3; 5. Average of three measurements from Table 3; 6. Five determinations from Table 3; 7. From reference 22; 8. 120-grit silicon carbide sandpaper; 9. 150-grit garnet sandpaper; 10. From data taken outside of the Proficiency Test; 11. ND = Not determined; 12. 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; 13. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 14. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.8–23.3; 18–24), LANL (21.2–23.5; 21.4–27.4), IHD (23–25; 44–45); 15. Average of three measurements from Table 5; 16. LLNL and LANL did not perform measurements; 17. Threshold Initiation Level (TIL) is the load (psig) at test velocity (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; 18. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 19. Measurements performed at 8 fps; 20. Values reported are not threshold level, but the next level above threshold. RDX standard also reported as level above threshold; 21. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (22–26; 41–44), AFRL (22.8-23.9; 49-52); 22. Average of three measurements from Table 6; 23. 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; 24. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.2–23.3; 18–23), LANL (21.3–22.6; 23.5–28.7), IHD (22; 44–45). AFRL (22.8-23.9; 47-51); 25. LLNL data with 510-Ω series resistor in circuit is 0/10 @ 0.25 joules; 26. Average of two measurements from Table 7; 27. One measurement from Table 7. 28. Average of three measurements in Table 7; 29. LLNL data from custom built system with 510-Ω series resistor in circuit.

For the current set of ABL friction data, IHD and AFRL are the only participants that provided any data that can be compared to standards. Note: values reported in Table 9 are not threshold level values, but the next level above threshold, because $KClO_4$/Al mixture was too sensitive to determine threshold levels. RDX standard is also reported as level above threshold[22]. When comparing with the RDX standard data, $KClO_4$/Al mixture is more sensitive, consistent with the BAM friction results. Currently, there is no ABL friction data for PETN.

Spark sensitivity. All participants find the $KClO_4$/Al mixture to be very spark sensitive, much more sensitive than PETN. LLNL actually registered spark sensitivity using their custom-built spark tester (510-Ω resister in testing circuit to simulate the human body). LLNL also retested the $KClO_4$ mixture with a recently acquired ABL spark tester, and found an extremely low TIL values, consistent with the values derived by the other laboratories. There are limited values on PETN, but the comparison shows the mixture to be more sensitive than PETN.

Thermal sensitivity. The thermal sensitivity of $KClO_4$/Al by DSC is reflective of high melting solid oxidizer mixed with high melting solid fuel—no reaction is observed in DSC in the temperature range selected. The only feature is the endothermic event associated with the phase change of the $KClO_4$. Even when this occurs, the Al is far from melting, so the likelihood of thermal instability is very slight. By this measure, the $KClO_4$/Al mixture, showing only endothermic heat flow between RT and 400°C is much more thermally stable than RDX.

4.2 Comparison of results based on participants

There are differences in methodologies and equipment configurations among the participating laboratories, so comparison of results for the same test is useful to highlight any differences in SSST testing techniques. Using the average values shown in Table 9, although not statistically precise, at least allows for a qualitative comparison of any trends that may be seen among the participants.

For impact testing, when using 180-grit sandpaper, all participants show the $KClO_4$/Al mixture to be relatively insensitive. LLNL found it more sensitive than the LLNL-determined sensitivity of RDX while the other participants found the $KClO_4$/Al mixture value less sensitive than their determined sensitivity of RDX. This is further complicated by the LLNL determined sensitivity of the $KClO_4$/Al mixture using 120-grit sandpaper. LLNL could not measure a positive event in the drop hammer testing range, which has a high limit of > 177 cm.

There are two issues presented by these results: 1) a substantial difference in DH_{50} values when different grit size sandpapers are used, and 2) a difference in DH_{50} values when the same grit size sandpaper is used. Sandpaper grit size has been determined a factor in previous materials in the Proficiency Test. The sensitivity of the RDX standard, as measured by LLNL using 120-grit sandpaper, LANL using 150-grit sandpaper, and IHD and AFRL using 180-grit sandpaper, has been in the range of 15 to 25 cm. Over the course of the Proficiency Test, when all the participants use 180-grit sandpaper, the average DH_{50} value for RDX is 19.8 ± 2.8 cm (19 determinations), or a 14% relative deviation. A much bigger variation was observed in the average DH_{50} value for the $KClO_3$/dodecane mixture[23] when the participants used the different grit size sandpapers shown above—15.9 ± 12.3 cm, or a relative deviation of 77%. Much less variation was observed in the average DH_{50} value for the $KClO_3$/dodecane mixture when only 180-grit sandpaper was used—9.2 ± 1.7 cm, or a relative deviation of 18%.

In this case for the $KClO_4$/Al, use of the standardization of the sandpaper did not bring the DH_{50} values in agreement, with LLNL showing the material more sensitive than the other participants, even when the same adjustment in method worked well for RDX and $KClO_3$/dodecane. This would suggest that a different parameter is affecting the comparison. At this time, the cause is unknown.

The radical difference in sensitivity found using 120-grit sandpaper compared to 180-grit sandpaper can be explained a number of ways. The sandpaper in the impact experiment provides two functions—to hold the sample in place and to provide sites for reactions to occur. Sandpapers are generally made of very hard solid materials to withstand erosion during use. In the drop hammer application, erosion is not the primary affect on structure of the sandpaper, fracturing is. The particles that make up the grit are subjected to impacting shock in the drop hammer as opposed to friction wear by a constant rubbing action in normal use. These particles make the sites for reaction during the drop hammer experiment, and if the materials are reactive, the amount of these sites can determine whether the reaction will be detected during the experiment.

Table 3 shows different sensitivity of the $KClO_4$/Al mixture based on the grit size of the sandpaper. The reason or reasons are not clear at this time. However, there are several potential reasons for differences between 120-grit sandpaper DH_{50} values vs. the 180-grit sandpaper DH_{50} values. These potential sources are being examined with further experimentation.

- Particle size of mixture vs. grit size of sandpaper—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 $KClO_4$/Al mixture are closer in size allowing for better contact.

  Figure 1 illustrates the size differences. Shown are the Scanning Electron Micrograph (SEM) images of $KClO_3$ (top left), $KClO_4$ (top right), 120-grit sandpaper (bottom left) and 180-grit (bottom right). The $KClO_4$ image shows a very fine material. The Al used in these tests is even finer (image not shown). Mixed together, they make an extremely fine material. The 120-grit sandpaper has very large grain size compared to this mixture. If the $KClO_4$/Al mixture is put on the 120-grit sandpaper, the $KClO_4$/Al mixture could possibly get lost in the grit matrix. Then the striker of the Drop Hammer does not really have much contact with the $KClO_4$/Al mixture because the grit of the 120-grit sandpaper physically prevents much contact and therefore does not provide enough sites for the reactions to start. With the 180-grit sandpaper, however, the grit size is small, the density of grains is about the same as the 120-grit sandpaper[23], and so the striker has better contact and more sites for reactions to start. For $KClO_3$, this mismatch of mixture particle size and sandpaper grit size is not as extreme, so there are more sites for reactions for both sandpapers. As a result, our particular $KClO_3$/fuel mixtures show less sandpaper grit size dependency than the $KClO_4$/Al mixture.

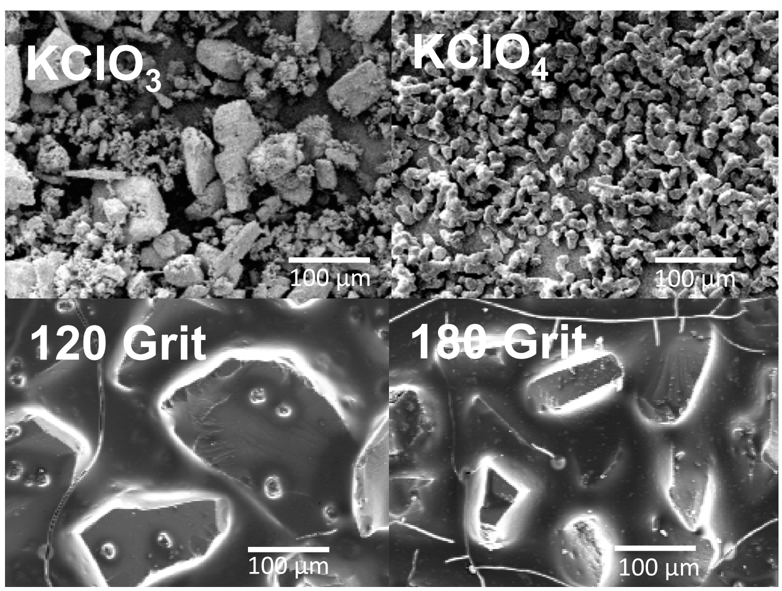

Figure 1. Scanning Electron Micrographs of $KClO_3$, $KClO_4$, 120-grit sandpaper, and 180-grit sandpaper at the same magnification.

The mismatch of the particle size of the $KClO_4$ and the 120-grit Si/C sandpaper is further illustrated in Figure 2 that 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 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[24]. For the $KClO_3$, 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 fall in the particle size range. The 120-grit average size does not overlap at all with the $KClO_4$ particle size range further supporting the argument above. A similar grit size particle size distribution relationship is seen when comparing particle size distributions as measured by Coulter Counter[4,5].

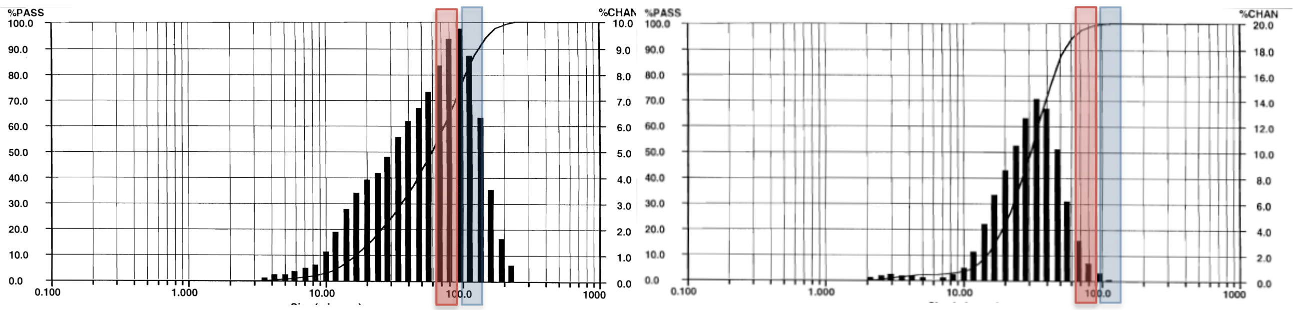

Figure 2. Particle size distribution of $KClO_3$ (left side) and $KClO_4$ (right side) and 180-grit sandpaper (red overlay) and 120-grit sandpaper (blue overlay) from CAMI specifications.

- Grit composition of the sandpaper—the 180-grit sandpaper is garnet while the 120-grit composition is silicon carbide (previously mislabeled as flint). The latter material has different crystal morphology than the garnet paper, and, as well has different hardness and fracture properties than garnet (garnet—6.5 to 7.5, silicon carbide—9 to 10, on Mohs' hardness scale)[25]. This could greatly affect the interaction of the sandpaper with the $KClO_4$/Al, and therefore the number of sites for reaction. The action of the striker on the more friable garnet paper could generate more sites for reaction compared to the Si/C paper.
- Thickness of the sandpaper backing—the 120-grit paper is a "wet" paper indicating that it can be used in wet or dry applications. The backing appears almost like a woven fabric (consistent with wet/dry papers). The 180-grit garnet paper is visibly much thinner and appears more paper like. Simple measurements of the thickness of the unused intact paper are: 180-grit garnet—0.229 mm (0.009 in); 120-grit Si/C—0.406 mm (0.016 in). Because the drop hammer is a shock experiment, the thicker paper could absorb more of the impact and diffuse the amount of energy transferred from the striker to the $KClO_4$/Al.
- Bonding agent on the sandpaper—the 120-grit paper is a "wet" paper indicating that it can be used in wet or dry applications, while the 180-grit sandpaper is for dry use only. Figure 3 shows photographs of the front and back of both sandpapers. The color and coding show the differences in the two types of paper. The adhesive to keep the grit in place is likely to be different[26]. The standard garnet generally has an adhesive, such as hide glue (animal connective tissue). The wet type sandpaper is likely to have a water insoluble resin. The effect on the DH_{50} is unknown. However, NSWC-IHD has seen effects of adhesive on impact testing of ammonium perchlorate (NH_4ClO_4) mixtures. These effects are large enough that IHD does not use sandpaper when testing mixtures containing NH_4ClO_4.

Experimentation is under way to clarify these issues.

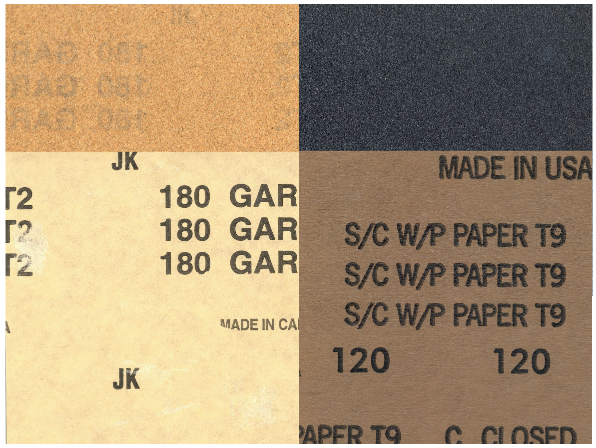

Figure 3. Photographs of the front and back of sandpapers used in drop hammer testing—180-grit garnet paper on the left side, 120-grit Si/C paper on the right side.

For BAM Friction, the TIL values roughly agree, among the participants, although values measured by IHD are slightly higher than the values measured by LANL and LLNL. The F_{50} values also roughly agree among the participants. LANL average values for both TIL and F_{50} indicate a more sensitive material than the comparable values from LLNL and IHD. This is not the same as seen for RDX, where average values for F_{50}, show that IHD finds RDX more friction sensitive than the other participants. This is also unlike the behavior seen previously where LLNL found $KClO_3$/sugar mixtures to be much more stable to friction than the other participants[19,27].

For ABL friction testing, neither IHD nor AFRL could find a threshold level because the material was found too sensitive. Both participants measured similar sensitivity for the lowest level that could be measured—IHD, approximately 1/3 positives @ 30 psig (8 ft./sec); AFRL, approximately 8/11 @ 25 psig (8 ft./sec). Because there is no known transfer function between the BAM and ABL friction methods, it is not clear why BAM exhibited TIL levels while the ABL did not. Perhaps the design differences of the sample contact mechanisms may account for some of the differences in sensitivity between the two methods and, as a result, the ABL method not finding a TIL. The ABL method uses a fixed steel wheel sliding on a steel plate that rotates in between testing to provide a fresh surface, to test for sensitivity, while the BAM method uses a ceramic plate that moves side-to-side in contact with a rounded ceramic pin to test for sensitivity[21].

For ESD, for all the Proficiency Test materials so far, LLNL consistently shows a much more stable material, highlighting the large design difference between the LLNL spark testing system and the others. This is the first time LLNL data shows a material with spark sensitivity. In addition, with the recently purchased ABL tester, LLNL obtained test values consistent with the other laboratories.

Initially, LANL did not find a TIL in their testing (below the energy level set when conducting the tests). However, LANL performed additional experiments to highlight the sensitivity of the mixture, and then found a very low TIL value as reported. In one experiment, the reaction of the $KClO_4$/Al mixture during ESD testing was monitored using a camera. Figure 4 shows the photos during (left hand side) and after the reaction (right hand side). Full propagation was observed and all the material was consumed during the reaction. The figure shows the reaction was very bright. As well, a loud sound was heard. The reaction was extremely hot as the witness tape was uncharacteristically burned and melted rather than torn after the reaction.

Figure 4. Photographs of decomposing the $KClO_4$/Al mixture during electrostatic discharge testing using the ABL Spark tester at 0.0625 J—left, reaction during testing; right witness tape after reaction

All participants found this mixture to be extremely spark sensitive. This is not a surprising finding because oxidizers and metals have been used as flash powders for years[28] and are known to be spark initiated, in some cases, with energies as little as 0.1 to 10 millijoules[29].

Figure 5 shows the DSC profile for the $KClO_4$/Al mixture obtained by LANL, LLNL, IHD, and AFRL. Other than the noticeable slope in baseline IHD data, the thermal behavior was found by all the participants to be essentially identical—a sharp large endothermic feature that is assigned to the phase transition of $KClO_4$[17-20].

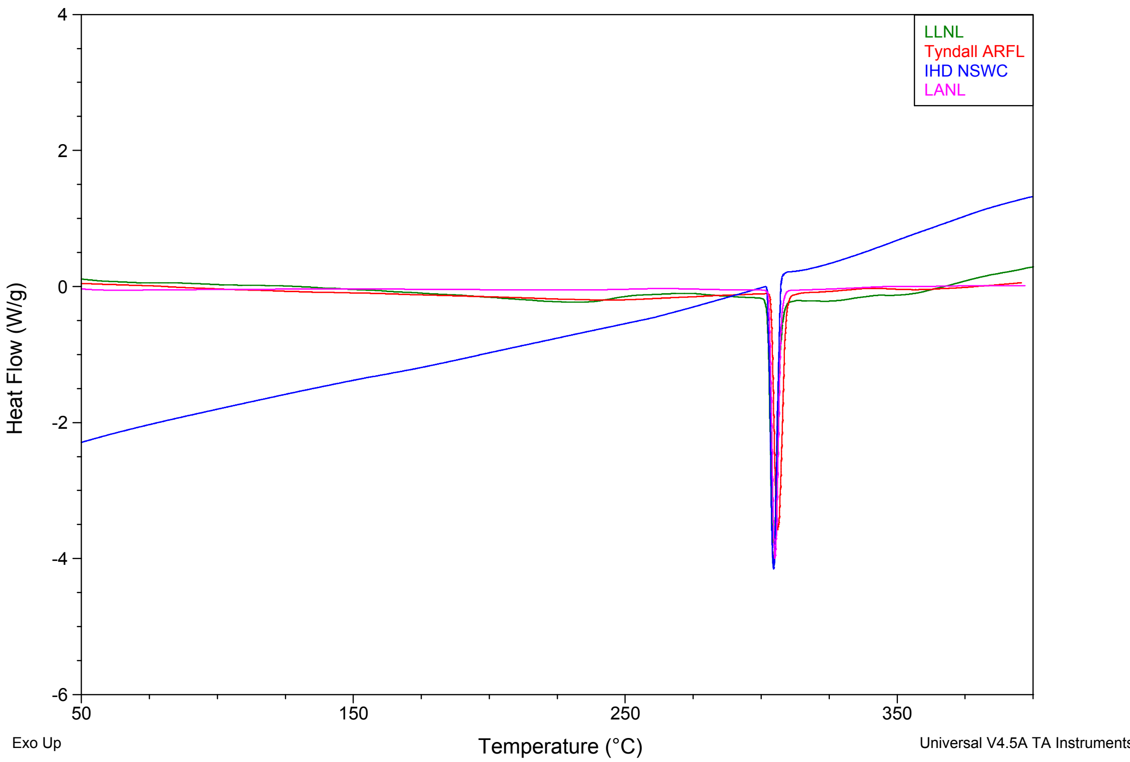

Figure 5. DSC profiles of $KClO_4$/Al mixture from pinhole sample holder at 10°C/min heating rate.

5 CONCLUSIONS

The following conclusions were found through SSST testing of the sensitivity of the $KClO_4$/Al mixture.

- Relatively insensitive toward impact
  - LLNL found it to be more sensitive than RDX
  - LANL, IHD and AFRL found it less than RDX
  - Where the data is available, the mixture was found less sensitive than PETN
- Relatively more sensitive than RDX for friction
  - For BAM friction, the mixture was found to be more sensitive than RDX
  - For BAM friction, the mixture was found to be less sensitive than PETN
  - For ABL friction, the mixture was found to be much more sensitive than RDX (no data on PETN)
  - For the ABL method, no level was found that did not cause reaction (no TIL).
- Very sensitive to spark
  - All participants found the mixture extremely sensitive
  - LLNL found sensitivity even with a 500 Ω resistor in the circuit
- Thermally stable
  - All participants found the mixture to exhibit only an endothermic phase transition for $KClO_4$ in the temperature range of RT to 400°C

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ABREVIATIONS, ACRONYMS AND INITIALISMS

ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AN	ammonium nitrate
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus

DH_{50}	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_{50}	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
Fe	Iron
H_2O	Water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
HP/F	hydrogen peroxide/fuel
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
MEKP	methyl ethyl ketone peroxide
NSWC	Naval Surface Warfare Center
PETN	Pentaerythritol tetranitrate
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
SEM	Scanning Electron Microscopy
SNL	Sandia National Laboratories
SO/F	solid oxidizer/fuel
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event
UN	urea nitrate

ACKNOWLEDGMENTS

The IDCA thanks Heidi Turner (LLNL), Fowsia Zaka (LLNL), Gary Hust (LLNL), Ernest L. Hartline (LANL), and Kimberly B. Proctor (IHD) for supporting data in this document.

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 multiprogram 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/RXQF and NSWC IHD is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division.

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