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Integrated Data Collection Analysis (IDCA) Program —$KClO_4$/Dodecane Mixture

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ABSTRACT

The Integrated Data Collection Analysis (IDCA) program is conducting a proficiency study for Small-Scale Safety and Thermal (SSST) testing of homemade explosives (HMEs). Described here are the results for impact, friction, electrostatic discharge, and differential scanning calorimetry analysis of a mixture of $KClO_4$ and dodecane—$KClO_4$/dodecane mixture. This material was selected because of the challenge of performing SSST testing of a mixture of solid and liquid materials. The mixture was found to: 1) be less sensitive to impact than RDX, and PETN, 2) less sensitive to friction than RDX and PETN, and 3) less sensitive to spark than RDX and PETN. The thermal analysis showed little or no exothermic features suggesting that the dodecane volatilized at low temperatures. A prominent endothermic feature was observed and assigned to 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 $KClO_4$/dodecane 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 study in order to establish some consistency in test protocols, procedures, and experiments and to understand how to compare results when these testing variables cannot be made consistent.

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

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

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.

Table 1. Materials for IDCA Proficiency study

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.

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.

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

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

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

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, $KClO_4$/dodecane mixture, is the fifth in a series of materials that are in the class of solid oxidizer/fuel mixtures and the second that is a mixture of solid oxidizer and liquid fuel. These materials were chosen for study in the Proficiency Test because of the challenge of testing a fine solid mixed with a low viscosity liquid fuel—adequate mixing on a small scale, representative sampling of a physical mixture, and handling a component that is volatile. The solid was dried as previously described and separated through a 40-mesh sieve. The dodecane was used as received from the manufacture.

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

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

Table 2. Summary of conditions for the analysis of $KClO_4$/dodecane 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[2]
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, (180-grit garnet); LLNL (180-grit garnet, 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[2]
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[2]
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[2]
3. Sample holder—LANL, IHD, and AFRL, pin hole; LLNL, hermetically sealed
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C
6. Sample holder hole size—LANL, IHD, AFRL, 75 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920 and Setaram Sensys; 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, Setaram Sensys DSC, IHD—TA Instruments Model 910, 2910, Q1000, AFRL—TA Instruments Q2000.

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 dodecane was purchased from Alfa-Aesar as *n*-Dodecane (99+%); Lot # L29T050 1 L, CAS # 112-40-3. The $KClO_4$ was dried for 16 h and cooled in a desiccator according to IDCA drying methods[2]; was separated through a 40-mesh (425-μm hole size) sieve. The mixture was prepared by hand, adding the dodecane to the $KClO_4$ while stirring with a spatula in a materials compatible polypropylene container according to IDCA mixing and compatibility procedures[3]. The mixture composition is 88-wt. % $KClO_4$ and 12-wt. % dodecane. The final mixture had the appearance of a wetted solid, with no evidence of free liquid in the vial. Typically, the precursors are mixed at that ratio to give approximately a 1-gram sample. However, AFRL mixed batches that were at the 5- to 10-gram level. This amount is divided up for the various

SSST testing. Three samples were prepared this way and tested separately. The mixing ratio was determined by thermochemical calculations using Cheetah[6] chosen stoichiometric for oxygen balance.

The SSST testing data for the individual participants were obtained from the following reports: Small Scale Safety Test Report for KP/Dodecane (88/12) Mixture (LLNL)[7], 50188 G KP/dodecane (LANL)[8], KP/Dodecane (IHD)[9], and Potassium Perchlorate + Dodecane (AFRL)[10].

3 RESULTS

3.1 $KClO_4$/dodecane mixture

In this proficiency test, all testing 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[11], 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 free-flowing powder with slight mechanical agitation. Particle Size Distribution measurements indicate that the 95% of the sieved KP particles were less than 67 μm. Because the composition of diesel fuel changes regionally and seasonally, dodecane was selected as a surrogate. Although $KClO_4$ and dodecane mixtures can be made at a variety of mixing ratios, the ratio for this study was selected that conforms to the maximum energy output, as determined by thermochemical assessments.

3.2 Impact testing results for $KClO_4$/dodecane mixture

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

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm^3	s, log unit[4]	s, cm[4]
LLNL (120)	6/30/10	23.9	21	> 177	NA[5]	NA[5]
LLNL (120)	7/01/10	23.9	23	> 177	NA[5]	NA[5]
LLNL (120)	7/01/10	23.9	22	> 177	NA[5]	NA[5]
LLNL (180)	1/6/12	23.9	18	29.7	0.047	3.4
LLNL (180)	1/6/12	23.9	18	31.2	0.022	1.6
LANL (180)	8/25/10	21.5	52.8	27.0	0.070	4.4
LANL (180)	8/3/10	21.4	62.3	29.4	0.058	3.9
LANL (180)	9/9/10	20.0	57	29.9	0.082	5.7
IHD (180)	1/13/11	26	43	20	0.07	3.2
IHD (180)[6]	1/6/11	25	47	19	0.20	9.1
IHD (180)	2/14/11	24	40	19	0.36	17.6
AFRL (180)	7/28/11	25	60	23.4	0.054	2.9
AFRL (180)	7/28/11	24.4	60	20.2	0.039	1.8
AFRL (180)	12/6/11	22.8	46	17.6	0.093	3.8
AFRL (180)	12/6/11	22.2	47	18.2	0.036	1.5
AFRL (180)	12/6/11	22.8	47	18.8	0.035	1.5

1. Number in parentheses indicates grit size of sandpaper; 2. Relative humidity; 3. DH_{50}, in cm, is from a modified Bruceton method, load for 50% probability of reaction; 4. Standard deviation; 5. Not applicable; 6. This run used only 19-drops because of lack of sample.

Table 3 shows the results of impact testing of the $KClO_4$/dodecane 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 test. In 2010-11 testing, LANL, IHD, and AFRL used 180-grit garnet sandpaper, and LLNL used 120-grit Si/C sandpaper for the impact testing. In subsequent testing by in 2012, LLNL also used 180-grit garnet sandpaper. All participants performed data analysis by normal modified Bruceton method[12,13] and LANL and AFRL also performed data analysis by the Neyer method[14].

For 2010-11 testing, the results from the four participating laboratories for impact show a large range for DH_{50} from 17.6 to >177 cm. The average values are LLNL, >177; LANL, 28.8 ± 1.5; IHD, 19 ± 0.6, and AFRL 19.6 ± 2.3 cm. The average values based on grit size are 120, >177; 180, 22.0 ± 4.6 cm. The s values from the table for the 180-grit sandpaper data are below 10 cm except for one IHD determination (17.6 cm). This appears as a result of IHD using 0.1 log spaced steps while LANL and LLNL use 0.05 log spaced steps. The LLNL data exhibited the positive-negative transition between 141 cm to the limit of the LLNL equipment configuration, 177.4 cm, indicating the DH_{50} is probably in this range. However, the testing did not meet the criteria for applying the Bruceton analysis method, so a complete Bruceton analysis could not be completed. The impact of step spacing will be evaluated in detail in a later report. For 2012 testing, LLNL used 180-grit garnet sandpaper to examine the whether the 120-grit sandpaper was responsible for the 2010 LLNL results. The average value for DH_{50} from this testing by LLNL is 30.4 ± 1.1 cm, very close to the 180-grit garnet sandpaper values obtained by LANL.

Table 4 shows the impact test results from LANL and AFRL using the Neyer or D-Optimal method[10]. The DH_{50} values are in the same range as the values analyzed by the Bruceton method for the 180-grit sandpaper data, where the average DH_{50} for the Neyer method is 24.2 ± 5.0 cm. The standard deviation varies from 4 to 8 cm.

Table 4. Impact testing results for $KClO_4$/dodecane mixture (Neyer or D-Optimal Method) 180-grit sandpaper

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, log unit[4]	s, cm[4]
LANL (180)	8/25/10	20.1	50.3	21.9	0.16	7.92
LANL (180)	8/3/10	21.4	62.3	29.5	0.12	6.90
LANL (180)	9/9/10	20.0	57.6	27.1	0.11	6.74
AFRL (180)	7/27/11	24.4	60	18.4	0.10	4.3

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

3.3 Friction testing results for $KClO_4$/dodecane mixture

Table 5 shows the BAM Friction testing performed by LANL, LLNL and IHD. 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 the threshold initiation level method (TIL)[15], and LANL and LLNL also used a modified Bruceton method[12,13]. The F_{50} friction values show that this material is insensitive to friction. The TIL values also show the same behavior.

Table 6 shows the ABL Friction testing performed by IHD and AFRL on the $KClO_4$/dodecane mixture. LANL did not have the system in routine performance at the time. LLNL does not have ABL Friction. IHD and AFRL performed data analysis using the threshold initiation level method (TIL)[15], and IHD also used a modified Bruceton method[12,13]. The data from IHD show some friction sensitivity. A TIL and one level above are established. In addition, IHD could calculate F_{50} values from their data. The data from AFRL show no friction sensitivity even at the maximum setting for the equipment. These results agree with the BAM friction results in Table 5—this material is not friction sensitive.

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

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4]	s, log unit[5]	s, kg[5]
LLNL	6/30/10	23.9	24	0/10 @ 36.0	1/10 @ > 36.0	> 36.0	NA[6]	NA[6]
LLNL	7/01/10	23.9	21	0/10 @ 36.0	1/10 @ > 36.0	> 36.0	NA[6]	NA[6]
LLNL	7/01/10	23.9	23	0/10 @ 36.0	1/10 @ > 36.0	> 36.0	NA[6]	NA[6]
LANL	8/02/10	21.3	66.7	0/10 @ 36.0	1/10 @ > 36.0	> 36.0	NA[6]	NA[6]
LANL	8/03/10	20.7	57.5	0/10 @ 36.0	1/10 @ > 36.0	> 36.0	NA[6]	NA[6]
LANL	8/04/10	22.0	59.4	0/10 @ 36.0	1/10 @ > 36.0	> 36.0	NA[6]	NA[6]
IHD	2/25/11	25	42	0/10 @ 33.1	1/3 @ 36.7	NA[7]	NA[7]	NA[7]
IHD	2/17/11	24	42	0/10 @ 29.4	1/2 @ 33.1	> 36.0	NA[6]	NA[6]
IHD	2/25/11	27	40	0/10 @ 36.7	0/10 @ 36.7	NA[7]	NA[7]	NA[7]

1. Relative humidity; 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. Next level where positive initiation is detected; 4. F_{50}, in kg, is from a modified Bruceton method, load for 50% probability of reaction, LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. Not applicable, cannot calculate; 7. Not applicable, separate sample used for Bruceton analysis.

Table 6. ABL Friction testing results for $KClO_4$/dodecane 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	3/29/11	23	43	0/20 @315/8	1/12 @ 420/8	ND[7]	ND[7]	ND[7]
IHD	3/29/11	23	43	0/20 @ 420/8	1/6 @ 560/8	ND[7]	ND[7]	ND[7]
IHD	3/29/11	23	44	0/20 @ 315/8	1/5 @ 420/8	ND[7]	ND[7]	ND[7]
IHD	3/29/11	24	43	ND[7]	ND[7]	708/8	147	0.09
IHD	3/29/11	23	43	ND[7]	ND[7]	706/8	232	0.14
IHD	3/29/11	24	41	ND[7]	ND[7]	736/8	136	0.08
AFRL	7/27/11	25	61	0/10 @ 1000/8	ND[7]	ND[7]	ND[7]	ND[7]
AFRL	7/28/11	25	60	0/10 @ 1000/8	ND[7]	ND[7]	ND[7]	ND[7]
AFRL	7/28/11	24.4	60	0/10 @ 1000/8	ND[7]	ND[7]	ND[7]	ND[7]

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. 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; 4. Next level where positive initiation is detected; 5. F_{50}, in psig (at a specific fps), is from a modified Bruceton method, load for 50% probability of reaction; 6. Standard deviation. 7. Not determined.

3.4 Electrostatic discharge testing of $KClO_4$/dodecane mixture

Electrostatic Discharge (ESD) testing of the $KClO_4$/dodecane mixture was performed by LLNL, LANL, IHD and AFRL. All participants performed data analysis using the threshold initiation level method (TIL)[15]. 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. In addition, LLNL uses a custom built ESD system with a 510-Ω resistor in line to simulate a human body, making a direct comparison of the data from LLNL with data generated by the other participants challenging. Recent testing by LLNL with a new ABL spark testing system (2012 data) is also listed.

The testing data from LANL and AFRL show about the same threshold levels for ESD sensitivity for the $KClO_4$/dodecane mixture. The IHD data show a slightly more sensitive material, while the LLNL data from 2010 show a non-sensitive material (expected because of the LLNL experimental configuration). However, the LLNL data from 2012 are in agreement with the other participants. IHD and AFRL were able to measure a level above threshold. This was above the equipment configuration for LANL.

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

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	6/30/10	23.9	24	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	7/01/10	23.9	24	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	7/01/10	23.3	24	0/10 @ 1.0	0/10 @ 1.0
LLNL[5]	1/9/12	23.9	18	0/10 @ 0.250	2/2 @ 0.380
LANL[5]	7/28/10	20.2	68.4	0/20 @ 0.250	1/10 @ > 0.250
LANL[5]	8/03/10	21.3	60.6	0/20 @ 0.250	1/10 @ > 0.250
LANL[5]	8/05/10	21.2	64.1	0/20 @ 0.250	1/10 @ > 0.250
IHD[5]	2/25/11	27	40	0/20 @ 0.165	1/1 @ 0.326
IHD[5]	2/14/11	25	42	0/20 @ 0.095	1/2 @ 0.165
IHD[5]	2/15/11	25	42	0/20 @ 0.095	1/8 @ 0.165
AFRL[5]	7/27/11	24.4	61	0/20 @ 0.25	1/1 @ 0.26
AFRL[5]	7/28/11	25	60	0/20 @ 0.31	1/1 @ 0.38
AFRL[5]	7/28/11	24.4	60	0/20 @ 0.26	1/1 @ 0.28

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-Ω resistor in the discharge unit to mimic the human body. 5. ABL ESD test equipment.

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

Differential Scanning Calorimetry (DSC) was performed on the $KClO_4$/dodecane mixture by LLNL, LANL, IHD, and AFRL. All participating laboratories used different versions of the DSC by TA Instruments. In addition, LLNL used the Setaram Sensyn system.

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

Lab	Test Date	Endothermic, onset/minimum, °C (ΔH, J/g)	Exothermic, onset/maximum, °C (ΔH, J/g)	Endothermic, onset/minimum, °C (ΔH, J/g)
LLNL[1]	6/30/10		206.9/218.5 (46)	302.9/305.1 (91)
LLNL[1]	6/30/10		226.7/244.7 (87)	302.8/304.3 (95)
LLNL[1]	6/30/10			302.5/304.5 (92)[3]
LANL[2]	8/16/10			303.2/305.3 (103)
LANL[2]	8/16/10			303.1/304.8 (102)
LANL[2]	8/16/10			303.7/304.8 (101)
IHD[2]	3/2/10	148.2/185.3 (33)		301.4/306.2 (72)
IHD[2]	3/2/10	132.8/164.2 (23)		303.1/305.2 (69)
IHD[2]	3/2/10	137.2/161.1 (15)		302.8/305.4 (74)
AFRL[2]	7/26/11			303.7/305.0 (127)
AFRL[2]	7/27/11			303.7/305.0 (95)
AFRL[2]	7/28/11			303.6/306.4 (91)

1. LLNL—hermetically sealed sample holder; 2. LANL, IHD and AFRL—open pinhole sample holder; 3. high temperature exothermic feature 475/531.9, by Setaram closed system 481/508 (4308)

Table 8 shows the DSC data differs somewhat 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 to 306 °C. This is assigned to the phase transition in $KClO_4$ from previous work on the thermal behavior of $KClO_4$/fuel mixes by TGA, DTA, and DSC[16-18]. The LLNL data shows a small broad exothermic feature in the low 200°C range, not obvious in the data from the other participants. LLNL used a sealed sample holder instead of a closed sample holder. This behavior is discussed below. IHD also observes a very small endothermic feature in the 100 to 200°C range. This is also discussed below.

Table 9. Average Comparison values

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
$KClO_4$/dodecane[2]	>177[3,4]	28.8[4,5]	19[4,5]	19.6[5,6]
$KClO_3$/dodecane[7]	9.3[5]	8.1[5]	10[5]	ND[8]
RDX Class 5 Type II[9]	23.8[10]	25.4[11]	19[5]	15.3[5]
PETN[12]	15	14.7	ND[8]	ND[8]
BAM Friction Testing[13,14]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
$KClO_4$/dodecane[15]	36[16]; >36[16]	36[16]; >36[16]	33[16]; >36[17]	ND[8]; ND[8]
$KClO_3$/dodecane[7]	12.3; 25.5	7.2; 19.1	16.5; 26.8	ND[8]; ND[8]
RDX Class 5 Type II[9]	19.2; 25.1	19.2; 20.8	15.5; ND[8]	ND[8]; ND[8]
PETN[12]	6.4; 10.5	ND[8]; 9.2	ND[8]; ND[8]	ND[8]; ND[8]
ABL Friction Testing[18-21]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
$KClO_4$/dodecane[22]	ND[8]; ND[8]	ND[8]; ND[8]	350[23]; 717[23]	1000[23]; ND[8]
$KClO_3$/dodecane[7]	ND[8]; ND[8]	ND[8]; ND[8]	135; 498	ND[8]; ND[8]
RDX Class 5 Type II[9]	ND[8]; ND[8]	ND[8]; ND[8]	74; 154	93; ND[8]
PETN[12]	ND[8]; ND[8]	ND[8]; ND[8]	ND[8]	ND[8]
Electrostatic Discharge[24]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
$KClO_4$/dodecane[25]	0/10 @ 1.0[26,27]	0/20 @ 0.250[27]	0/20 @ 0.118[27]	0/20 @ 0.273[27]
$KClO_3$/dodecane[7]	0/10 @ 1.0	0/20 @ 0.125	0/20 @ 0.140	ND[8]
RDX Class 5 Type II[9]	0/10 @ 1.0	0/20 @ 0.0250	0/20 @ 0.095	0/20 @ 0.044
PETN[12]	0/10 @ 1.0	0/20 @ 0.0625	ND[8]	ND[8]

1. DH_{50}, in cm, is by a modified Bruceton method, load for 50% reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.2; 21-23), LANL (20.0-21.5; 52.8-62.3), IHD (24-26; 40-47), AFRL (22.2-25.0; 46-60); 3. 120-grit sandpaper data only; 4. Average of three measurements from Table 3; 5. 180-grit sandpaper; 6. Average of five measurements from Table 3; 7. From reference 20; 8. ND = Not determined; 9. From reference 19; 10. 120-grit Si/C wet/dry sandpaper; 11. 150-grit garnet sandpaper; 12. From data taken outside of the Proficiency Test; 13. 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; 14. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 15. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 21-24), LANL (20.7-22.0; 57.5-66.7), IHD (24-27; 40-42); 16. Average of three measurements from Table 5; 17. One value only from Table 5; 18. LLNL and LANL did not perform measurements; 19. 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; 20. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% Reaction; 21. Measurements performed at 8 fps; 22. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (23; 43-44), AFRL (24.4-25.0; 60-61); 23. Average of three measurements from Table 6; 24. 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; 25. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3–23.9; 24), LANL (20.2-21.3; 60.6-68.4), IHD (25-27; 40-42), AFRL (24.4-25.0; 60-61); 26. LLNL has 510-Ω resistor in circuit; 27. Average of three measurements from Table 7.

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[19]. 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 $KClO_4$/dodecane mixture with standards to obtain relative sensitivities.

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

Impact sensitivity. Table 3 shows the impact data where the testing was done using two different sandpapers—120-grit silicon carbide and 180-grit garnet. As a result, the impact data shows a large spread in values. For the LLNL data, the DH_{50} values for RDX and PETN in Table 9 are averages of data taken with the same type of 120-grit sandpaper used for the LLNL data from 2010 for $KClO_4$/dodecane mixture, so the direct comparison is relevant—the $KClO_4$/dodecane mixture is much less sensitive to impact than the RDX and PETN. For the other participants, the DH_{50} values in Table 9 are averages of data sets taken with the same type of 180-grit sandpaper. Note: the LANL RDX data is an average of data taken with 150-grit sandpaper. LANL, IHD, and AFRL find the $KClO_4$/dodecane mixture to be about on the same sensitivity level as RDX, based on the comparison at each laboratory with their corresponding RDX standard data. In the LANL case, the $KClO_4$/dodecane mixture is less sensitive than PETN.

Friction sensitivity. For BAM friction, LLNL, LANL and IHD performed this testing and found the $KClO_4$/dodecane mixture insensitive, usually challenging the limits of the settings of the equipment. The IHD data shows some response at the highest applied weights, but these values, as with the other laboratories show the material is insensitive. For ABL friction, IHD and AFRL performed this testing and also found the $KClO_4$/dodecane mixture to be very insensitive.

Spark sensitivity. Comparing the $KClO_4$/dodecane mixture spark sensitivity values to the corresponding standards, LANL, IHD, and AFRL found the mixture to be less sensitive to spark stimulation than the RDX and PETN standards. LLNL found the material to be insensitive (LLNL ESD equipment is custom built). However, LLNL, when using the ABL ESD, found the material to be about the same sensitivity as found by the other participants

Thermal sensitivity. The thermal sensitivity of $KClO_4$/dodecane compared to the RDX standard is difficult to assess examining the data in Table 8, because of the lack of exothermic features when using standard DSC sample holders (pin-hole vented). On the surface, this would indicate that the mixture is not as thermally sensitive as RDX. However, the data, when using a sealed sample holder, suggest that there may be more chemistry occurring, some causing exothermic heat flow. This is discussed below. However, at this time, the thermal sensitivity of $KClO_4$/dodecane cannot be reliably assessed by this technique in the standard configuration, so it is not clear that is more or less sensitive 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 methods. 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, LANL, IHD and AFRL show about the same sensitivity for the $KClO_4$/dodecane mixture. LANL data suggests a material that is apparently less sensitive than suggested by the IHD and AFRL data, but when the data is compared to the corresponding RDX data, all three laboratories afford the same assessment. This is interesting because the testing by LANL for RDX is with 150-grit garnet sandpaper, while the $KClO_4$/dodecane is with 180-grit garnet sandpaper. In addition, LLNL data from 2012 was taken with 180-grit garnet sandpaper and essentially matches the LANL data obtained in 2010.

For BAM and ABL Friction, all participants find this material is insensitive. For the most part, TIL and F_{50} values could not be assessed due to the insensitivity of the mixture to friction.

For ESD testing, LANL, IHD, and AFRL show about the same sensitivity for the $KClO_4$/dodecane mixture. Experimental configuration accounts for LANL not finding a level above TIL. LLNL results indicate even a less sensitivity material, not registering any spark sensitivity. This also can be accounted for by the experiment configuration. However, with the new ABL friction testing system, LLNL obtained results that are in excellent agreement with the other participants.

For thermal sensitivity, the major event observed by all participants is the phase transition of $KClO_4$ near 300°C. This transition is an endothermic event and the only prominent feature in the DSC. IHD observed some lower temperature endothermic features in the 100 to 200°C range which are probably due to degassing or evaporating (dodecane and or water). LLNL observed broad exothermic features above 200°C which have been assigned in the $KClO_3$/dodecane study[20] as possible thermal reaction of the mixture. This will be discussed in more detail below. Note that LLNL used a hermitically sealed sample holder and the other participants use pin-hole vented sample holders.

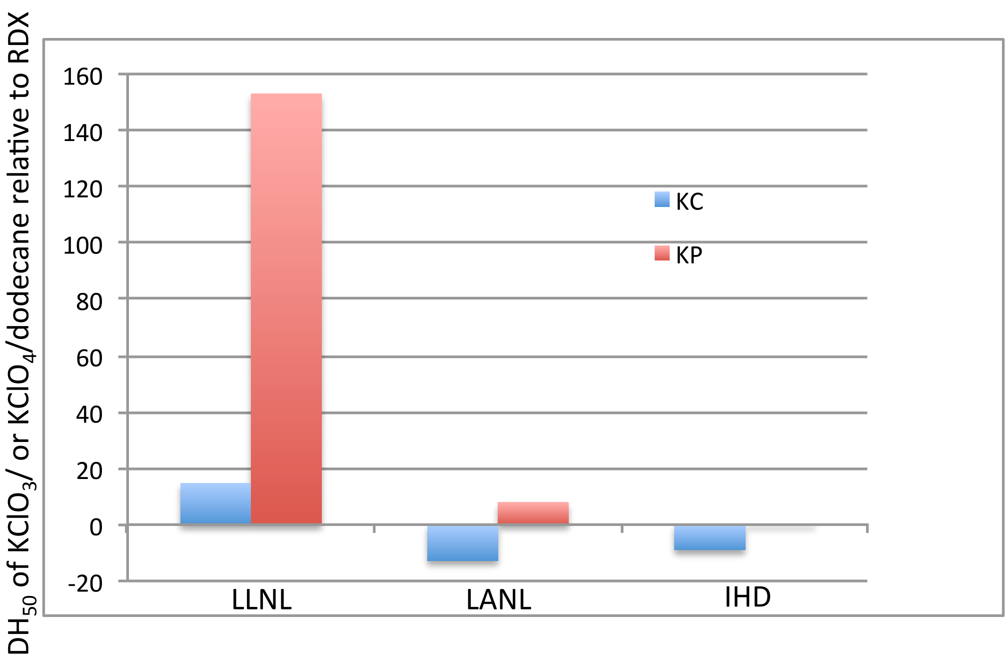

Figure 1. Comparison of average impact data from LLNL, LANL, and IHD for $KClO_4$ (KP)/dodecane and $KClO_3$ (KC)/dodecane mixtures (LLNL data, 120-grit sandpaper; LANL and IHD data, 180-grit sandpaper) relative to RDX standard (LLNL data, 120-grit sandpaper; LANL and IHD data, 180-grit sandpaper).

4.3 T Comparison of $KClO_3$/dodecane and $KClO_4$/dodecane mixtures

$KClO_3$/dodecane mixture at 89/11-mixture ratio has been studied in the Proficiency Test previously[20]. Combining these results with those of the current study gives an opportunity to realize the differences of two oxidizers, $KClO_3$ and $KClO_4$, when combined with dodecane. Table 9 compares the SSST testing average values of both mixtures. LANL, LLNL, and IHD studied both mixtures completely.

Table 9 shows that LLNL, LANL, and IHD found the $KClO_4$/dodecane mixture is less impact sensitive than the $KClO_3$/dodecane mixture. The table also shows the $KClO_4$/dodecane mixture to be less sensitive than RDX and the $KClO_3$/dodecane mixture to be more impact sensitive than RDX for all participants that measured impact sensitivity on both. However, the comparison is Table 9 does not account for the grit size of the sandpaper in the impact test. Figure 1 shows the comparison of impact data relative to RDX (average DH_{50} value of mixture minus average DH_{50} value of RDX) when the same grit size of sandpaper is used for standard and mixtures. LLNL used 120-grit Si/C wet/dry sandpaper, LANL and IHD used the 180-grit garnet sandpaper. In this comparison, LLNL found both mixtures to be less sensitive than RDX, while LANL and IHD found the $KClO_4$/dodecane mixture to be equal or less sensitive and the $KClO_3$/dodecane mixture to be more sensitive than RDX. Note, the LLNL data came from the $KClO_3$/dodecane study[20], and the LANL RDX data came form the 2nd RDX study in the Proficiency Test[21].

Table 9 compares the BAM friction results from LLNL, LANL, and IHD for $KClO_4$/dodecane and $KClO_3$/dodecane. Essentially, $KClO_4$/dodecane is not friction sensitive while $KClO_3$/dodecane shows sensitivity similar to RDX. IHD is the only participant that performed ABL friction testing on both mixtures, and found that the $KClO_4$/dodecane mixture is much less sensitive than the $KClO_3$/dodecane mixture and RDX.

Table 9 also shows the electrostatic discharge results on both mixtures from LLNL, LANL, and IHD. LLNL found both mixtures to be insensitive. The LLNL data was taken using a custom built system that has a 510-Ω resistor in the circuit, so comparison with the other data is problematic. The LANL and IHD data show sensitivity about the same for the mixtures and both less ESD sensitive than RDX.

Figure 2 shows the DSC profiles from LLNL, comparing $KClO_4$/dodecane thermal behavior to $KClO_3$/dodecane thermal behavior. These sets of profiles were chosen because they more clearly exhibit multiple features. The profiles on the left side of the figure are similar to the profiles from IHD, LANL and AFRL (not shown) and were taken using a pinhole sample holder. The LLNL data were taken with a hermetically sealed sample holder.

Examining the profiles on the left side of the figure shows prominent endothermic features for both the $KClO_4$ and $KClO_3$ mixtures. This corresponds to a phase transition and a melting of the oxidizer, respectively. These endothermic features are also seen in the corresponding DSC profiles from IHD, LANL and AFRL. For the IHD profiles, less prominent endothermic features are also observed in the 100 to 200 °C range. Because these are endothermic, they are likely due to the volatilization of the dodecane. These low temperature endothermic features are not prominent in the LANL, AFRL and LLNL DSC profiles. However, closer inspection of these profiles reveals the low temperature endotherms are present in all the $KClO_4$/dodecane mixture <u>and</u> the $KClO_3$/dodecane mixtures DSC profiles.

Examining the profiles on the right side of the figure, shows a much more complicated thermal behavior. A series of very broad exothermic features are observed in the 200 to 300°C temperature range. The origin of these exothermic features has not been resolved, but has been discussed in the $KClO_3$/dodecane mixture study[20]. The occurrence of the low temperature exothermic features is possibly due to the hermetically sealed sample holder preventing total evaporation of the dodecane and therefore providing some contact with the oxidizer at higher temperatures. Dodecane has a boiling point of 218°C[22] so much of it is vaporized in the above temperature range, but if the system is closed, some vapor (not much) is still available for reaction. This same argument could be extended to the $KClO_4$/dodecane mixture. However, that these reactions are occurring in both samples also suggests that the chemistry occurring is due to the dodecane on a solid surface and may be independent of the solid.

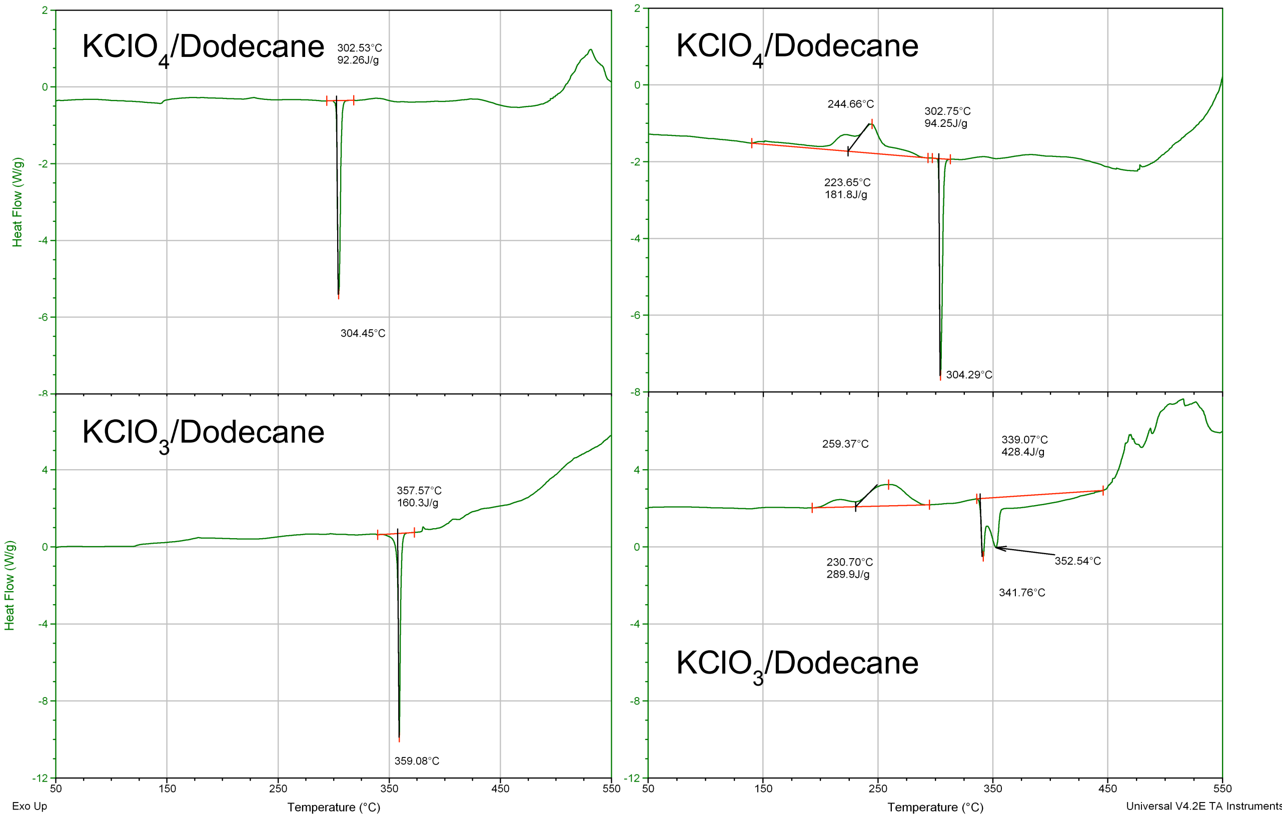

Figure 2. DSC profiles of $KClO_4$/dodecane mixture and $KClO_3$/dodecane from LLNL; note: scales of corresponding axes are not the same in some cases.

The other exothermic features are not particularly intense in both cases and are fairly complicated so full analysis of these is beyond the scope of this report, and will be discussed elsewhere. However, it is important to note that even though the lack of features in data from the pin-hole vented sample holder implies no thermal reactivity, the appearance of exothermic features in the sealed pan, even though weak, implies something is happening in the sample. The application of the standard DSC is just not adequate for evaluating the thermal sensitivity of this sample.

5 CONCLUSIONS

$KClO_4$/dodecane mixture was found through SSST testing to be a low sensitive mixture toward impact, friction, and spark handling conditions—generally less sensitive than RDX, and PETN. Standard thermal testing by DSC probably does not adequately describe the system.

The proficiency study shows that for $KClO_4$/dodecane mixture examined by current equipment configurations and experimental methods, the impact sensitivity greatly depended upon the type of sandpaper used in the drop hammer test, something that has been seen with previously studied HMEs. For example, with 120-grit Si/C wet/dry paper, the $KClO_4$/dodecane mixture appears very insensitive to impact, while with 180-grit garnet sandpaper, the mixture is on the order of sensitivity of RDX. This is important because the impact sensitivity of the RDX standard does not show this strong dependency on grit size. Most testing laboratories use relative sensitivity to an established standard as the metric for evaluating sensitivity of a new material. This finding casts doubt on using the relative method for HME evaluation. For friction, all participants found the $KClO_4$/dodecane mixture to be much less sensitive than RDX or PETN, by both BAM and ABL methods. For ESD, LLNL found the material to be insensitive, and LANL, IHD, and AFRL found the material to have less sensitivity than both RDX and PETN. For thermal results, unlike in the case for RDX, where all the participants had results that were virtually identical, unlike the case of $KClO_3$/icing sugar mixtures[18,23], where sampling issues have complicated the interpretation of the results, and like the $KClO_3$/dodecane[20] mixture, no prominent exothermic features were seen for the $KClO_4$/dodecane mixture when using the standard DSC configuration. Sealed sample holder data suggests that there are exothermic events occurring in the 200 to 300°C range.

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ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH_{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
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine
IDCA	Integrated Data Collection Analysis
IHD	Indian Head Division, Naval Surface Warfare Center
j	joules

$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
kg	kilograms
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
N	Chemical symbol for nitrogen
$NaClO_3$	Sodium Chlorate
NSWC	Naval Surface Warfare Center
O	Chemical symbol for oxygen
PETN	Pentaerythritol tetranitrate
psig	pounds per square inch, gauge reading
RDX	Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine
RH	Relative humidity
RT	Room Temperature
RXQL	The Laboratory branch of the Airbase Sciences Division of the Materials & Manufacturing Directorate of AFRL
s	Standard Deviation
SEM	Scanning Electron Micrograph
Si	silicon
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

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