LLNL-TR-614974

LAWRENCE LIVERMORE NATIONAL LABORATORY

Integrated Data Collection Analysis (IDCA) Program - KClO4/Carbon Mixture

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

January 31, 2013

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 —$KClO_4$/Carbon Mixture

Mary M. Sandstrom[1], Geoffrey W. Brown[1], Daniel N. Preston[1], Colin J. Pollard[1], Kirstin F. Warner[2], Daniel N. Sorensen[2], Daniel L. Remmers[2], Timothy J. Shelley[3], Jose A. Reyes[4], Peter C. Hsu[5], and John G. Reynolds[5*]

[1]Los Alamos National Laboratory, Los Alamos, NM USA
[2]Indian Head Division, Naval Surface Warfare Center, Indian Head, MD USA
[3]Air Force Research Laboratory, Tyndall Air Force Base, FL USA
[4]Applied Research Associates, Tyndall Air Force Base, FL USA
[5]Lawrence Livermore National Laboratory, Livermore, CA USA

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 activated carbon—$KClO_4$/C 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 insensitive to impact, friction, and thermal stimulus, and somewhat sensitive to spark discharge.

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 $KClO_4$/carbon 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, impact-, friction-, spark discharge-, thermal testing, round-robin test, safety testing protocols, HME, RDX, potassium perchlorate, potassium chlorate, sugar, dodecane, PETN, carbon, charcoal.

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

*e-mail: reynolds3@llnl.gov

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 that may be encountered when testing 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, well characterized 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, $KClO_4$/C mixture, is the sixth 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 a solid fuel. These materials were chosen for study in the Proficiency Test because of the challenge of testing fine solids mixed together—adequate mixing on a small scale, representative sampling of a physical mixture, and handling a component that is a very fine powder. The $KClO_4$ was dried as previously described and separated through a 40-mesh sieve. The activated carbon was used as received from the manufacturer.

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 activated carbon was purchased from Sigma-Aldrich as DARCO, particle size -100 mesh, powder, catalog number 24227-6, batch number 11613BH. The mean nominal particle size (by Microtrac) is 48 μm with 87% < 100 μm[6]. The $KClO_4$ was dried at 60°C for 16 h and cooled in a desiccator according to IDCA drying methods[2]. The $KClO_4$ was separated through a 40-mesh (425 μm hole size) sieve to remove clumped material. The mixture was prepared by hand, adding the carbon 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 85-wt. % $KClO_4$ and 15-wt. % carbon. The final mixture had the appearance of a white-grey powder. 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[7] and the ratio was chosen to match stoichiometric for oxygen balance.

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

Impact Testing
1. Sample size—LLNL, AFRL 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 (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 1.0 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 on, AFRL and 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, 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, pin hole and 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.

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

The SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for Potassium Perchlorate (85%) and Charcoal (15%) Mixture [revised 4.1.11] (LLNL)[8], Potassium Perchlorate and Carbon 51088F (LANL)[9], KP/Charcoal (IHD)[10], and Potassium Perchlorate + Charcoal (AFRL)[11].

3 RESULTS

3.1 $KClO_4$/C 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[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 free-flowing powder with slight mechanical agitation. Particle Size Distribution measurements indicate that 95% of the sieved KP particles were less than 67 μm. The particle size distributions of the activated carbon and the $KClO_4$ overlap significantly. Figure 1 shows the particle size distributions using Microtrac laser light scattering method exhibiting this overlap.

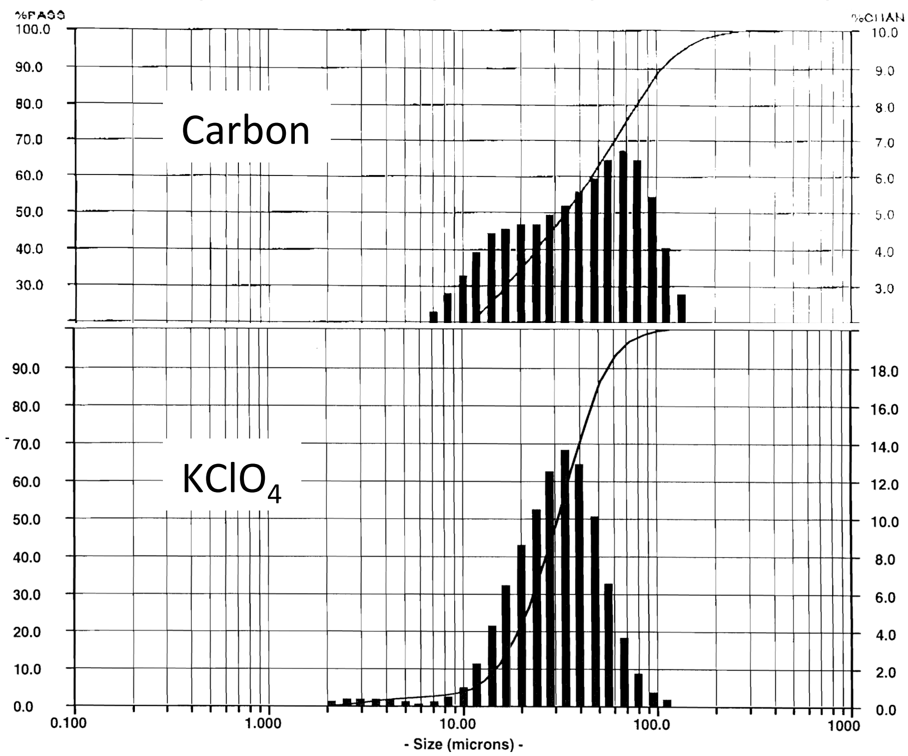

Figure 1. Particle Size Distribution of $KClO_4$ and activated carbon by the Microtrac method.

There are multiple sources and types of activated carbon because of the use in the commercial purification community. The carbon for this study was selected because of local availability at the time. These types of carbon have high adsorptive capacities for specific colored compounds, and are used generally in organic synthesis to remove off-color contaminants[13]. Although $KClO_4$ and carbon mixtures can be made at a variety of mixing ratios, the ratio for this study was selected that conforms to stoichiometric.

3.2 Impact testing results for $KClO_4$/C mixture

Table 3 shows the results of impact testing of the $KClO_4$/C 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. All participants found the $KClO_4$/C to be insensitive to impact testing within the limits of the testing equipment. AFRL observed positives for some testing at the upper height limits of the drop weight, but not enough to obtain valid statistical data. All participants performed data analysis by normal modified Bruceton method[14,15].

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

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	7/8/10	22.8	33	> 177	NA[5]	NA[5]
LLNL (120)	7/09/10	24.4	30	> 177	NA[5]	NA[5]
LLNL (120)	7/12/10	22.8	36	> 177	NA[5]	NA[5]
LANL (180)	5/26/10	22.3	36.3	> 320	NA[5]	NA[5]
LANL (180)	5/28/10	23.3	50.1	> 320	NA[5]	NA[5]
LANL (180)	5/28/10	23.7	44.7	> 320	NA[5]	NA[5]
IHD (180)	12/2/10	20	42	> 320	NA[5]	NA[5]
IHD (180)	12/9/10	23	41	> 320	NA[5]	NA[5]
IHD (180)	12/9/10	28	40	> 320	NA[5]	NA[5]
AFRL (180)	6/28/11	25	62	> 116[6]	NA[5,]	NA[5]
AFRL (180)	7/5/11	25	59	> 116[6]	NA[5]	NA[5]
AFRL (180)	7/6/11	25	59	> 116[6]	NA[5]	NA[5]

1. Value in parenthesis is grit size of sandpaper (180 is 180 garnet dry and 120 is 120 Si/C wet/dry); 2 relative humidity; 3. DH_{50}, in cm, is from a modified Bruceton method, and is the load for 50% probability of reaction; 4. Standard deviation; 5. NA = not applicable; 6. AFRL observed positives for some testing at the upper height limits of the drop weight, but not enough to obtain valid statistical data.

Table 4 shows the impact test results from LANL and AFRL using the Neyer or D-Optimal method[16]. The DH_{50} from the Neyer method show the same behavior as the DH_{50} by the Bruceton method—$KClO_4$/C is very insensitive material to impact testing. One LANL test exhibited sensitivity, but the DH_{50} value indicates a very insensitive material just the same.

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

Lab	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)	5/26/10	23.3	33.1	> 320	NA[5]	NA[5]
LANL (180)	5/28/10	23.3	50.1	> 320	NA[5]	NA[5]
LANL (180)	5/28/10	23.8	44.8	306.4	7.2	0.01
AFRL (180)	7/1/11	25	58	> 116[6]	NA[5]	NA[5]

1. Value in parenthesis is grit size of sandpaper (180 is 180 garnet dry); 2 relative humidity; 3. DH_{50}, in cm, is from the Neyer method, and is the load for 50% probability of reaction; 4. Standard deviation; 5. NA = not applicable; 6. AFRL did get some positive reactions during Neyer testing, but was at the upper limit of the force range, so a full Neyer analysis was not valid.

3.3 Friction testing results for $KClO_4$/C 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 a modified Bruceton method[14,15] and the threshold initiation level method (TIL)[17]. The friction values for F_{50} and TIL values show that this material is insensitive to friction.

Table 5. BAM Friction Testing results for $KClO_4$/C

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4]	s, cm[5]	s, log unit[5]
LLNL	7/7/10	23.9	32	0/10 @ 36	1/10 @ > 36	> 36	NA[6]	NA[6]
LLNL	7/07/10	24.4	30	0/10 @ 36	1/10 @ > 36	> 36	NA[6]	NA[6]
LLNL	3/22/10	24.4	30	0/10 @ 36	1/10 @ > 36	> 36	NA[6]	NA[6]
LANL	5/26/10	22.7	31.4	0/13 @ 36	1/10 @ > 36	> 36	NA[6]	NA[6]
LANL	5/28/10	22.8	36.5	0/13 @ 36	1/10 @ > 36	> 36	NA[6]	NA[6]
LANL	5/28/10	23.6	44.7	0/13 @ 36	1/10 @ > 36	> 36	NA[6]	NA[6]
IHD	12/2/10	23	42	0/10 @ 36	1/10 @ > 36	NA[7]	NA[6]	NA[6]
IHD	12/2/10	23	42	0/10 @ 36	1/10 @ > 36	NA[7]	NA[6]	NA[6]
IHD	12/2/10	23	42	0/10 @ 36	1/10 @ > 36	NA[7]	NA[6]	NA[6]

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 by a modified Bruceton method, load for 50% probability of reaction; 5. Standard deviation; 6. Not applicable, could not determine; 7. Because no TIL could be found to the limit of the equipment, Bruceton analysis was not valid.

Table 6 shows the ABL Friction testing performed by IHD and AFRL on the $KClO_4$/C mixture. LANL did not have the ABL system in routine use at the time. LLNL does not have ABL Friction. Both IHD and AFRL performed data analysis using a modified Bruceton method[14,15], and the threshold initiation level method (TIL)[17]. 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 also show friction sensitivity, but at much higher force values indicating that $KClO_4$/C is not very friction sensitive.

Table 6. ABL Friction testing results for $KClO_4$/C 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	11/19/10	23	48	0/20 @ 135/8	1/8 @ 180/8	NA[6]	NA[6]	NA[6]
IHD	11/19/10	24	43	0/20 @ 100/8	1/4 @ 135/8	NA[6]	NA[6]	NA[6]
IHD	11/19/10	23	44	0/20 @ 100/8	1/9 @ 135/8	NA[6]	NA[6]	NA[6]
IHD	12/10/10	24	44	NA[6]	NA[6]	246/8	86	0.15
IHD	12/10/10	24	44	NA[6]	NA[6]	293/8	132	0.19
IHD	12/10/10	24	44	NA[6]	NA[6]	304/8	107	0.15
AFRL	6/29/11	25	62	0/10 @ 500/8	1/3 @ 630/8	NA[6,7]	NA[6]	0.00
AFRL	7/7/11	25	58	0/10 @ 500/8	1/5 @ 630/8	NA[6,7]	NA[6]	0.04
AFRL	7/8/11	25	60	0/10 @ 630/8	1/5 @ 795/8	NA[6,7]	NA[6]	0.03
AFRL	6/29/11	25	62	NA[6]	NA[6]	891/8		0.00
AFRL	7/7/11	25	58	NA[6]	NA[6]	813/8		0.04
AFRL	7/8/11	25	60	NA[6]	NA[6]	851/8		0.03

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/fps, is by a modified Bruceton method, load for 50% probability of reaction; 6. Standard deviation; Not applicable; 7. AFRL did get some positive reactions during Bruceton testing, but was at the upper limit of the force range, so a full Bruceton analysis could not be performed.

3.4 Electrostatic discharge testing of $KClO_4$/C mixture

Electrostatic Discharge (ESD) testing of the $KClO_4$/C mixture was performed by LLNL, LANL, IHD 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. 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. All participants performed data analysis using the threshold initiation level method (TIL)[17].

Table 7. Electrostatic discharge testing $KClO_4$/C

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	7/07/10	23.9	31	0/10 @ 1.0[5]	> 1.0[5]
LLNL[4]	7/09/10	23.9	31	0/10 @ 1.0[5]	> 1.0[5]
LLNL[4]	7/09/10	23.9	30	0/10 @ 1.0[5]	> 1.0[5]
LLNL[5]	1/26/12	23.9	30	0/10 @ 1.3	1/3 @ 1.5
LLNL[5]	1/27/12	23.9	28	0/10 @ 0.88	1/4 @ 1.3
LANL	5/26/10	23.0	37.3	0/20 @ 0.25	> 0.25
LANL	5/28/10	23.2	43.5	0/20 @ 0.125	1/2 @ 0.25
LANL	5/28/10	23.4	44.0	0/20 @ 0.25	> 0.25
IHD[6]	2/2/12	28	44	0/20 @ 0.326	1/6 @ 0.853
IHD[6]	2/6/12	26	42	0/20 @ 0.326	1/1@ 0.853
IHD[6]	2/8/12	26	42	0/20 @ 0.165	1/9 @ 0.326
AFRL	3/13/12	25	48	0/20 @ 0.13	1/1 @ 0.14
AFRL	3/13/12	26.1	47	0/20 @ 0.13	1/3 @ 0.14
AFRL	3/13/12	27.8	45	0/20 @ 0.13	1/1 @ 0.14

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 used a custom built ESD with a 510-Ω resistor in the discharge unit to mimic the human body; 5. LLNL used ABL friction tester; 6. Retesting by IHD—original results were not complete tests.

For TIL, LANL, IHD and AFRL results are about the same—zero events 0.13 to 0.326 joules. In addition, AFRL was able to establish a very narrow transition from TIL to 1 level above TIL of 0.01 joules. The LLNL values using the custom built system show a material with no sensitivity, while LLNL testing with the ABL system show an equally insensitive material.

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

Differential Scanning Calorimetry (DSC) was performed on the $KClO_4$/C mixture by LLNL, LANL, IHD, and AFRL. All participating laboratories used different versions of the DSC by TA Instruments. Results were obtained at a 10°C/min heating rate.

Table 8 shows in the DSC data 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[18-21]. The LLNL data also shows a high temperature exothermic feature in the low 500°C range.

Table 8 also shows the DSC data, by LLNL, for the $KClO_4$/C 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 the same as the pinhole vented samples for LLNL. In previous studies, where the fuel was volatile, the difference between the pinhole and sealed pans 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 (C melts at 3500°C[22]), so the thermal profile is only due to the $KClO_4$ rhombic to cubic transition[18].

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

Lab	Test Date	Endothermic, onset/minimum °C (ΔH, J/g)	Exothermic, onset/maximum °C (ΔH, J/g)
LLNL[1]	6/29/10	303.0/304.7 (87)	508.4/512.6 (3625)
LLNL[1]	6/29/10	303.0/304.5 (86)	509.3/514.0 (4020)
LLNL[1]	6/29/10	303.0/304.7 (87)	508.8/513.1 (3702)
LLNL[2]	6/29/10	302.9/304.5 (88)	509.1/512.9 (3150)
LLNL[2]	6/29/10	302.9/304.6 (89)	508.2/511.9 (2993)
LLNL[2]	6/29/10	302.9/304.6 (85)	508.4/512.6 (3058)
LANL[1]	6/01/10	303.4/305.2 (84)	
LANL[1]	6/03/10	303.5/305.2 (84)	
LANL[1]	6/11/10	303.3/305.1 (87)	
IHD[1]	3/18/11	303.2/304.6 (76)	
IHD[1]	3/18/11	302.9/304.4 (79)	
IHD[1]	3/18/11	302.9/304.2 (77)	
AFRL[1]	6/29/11	304.0/305.6 (100)	
AFRL[1]	6/29/11	304.0/305.7 (85)	
AFRL[1]	7/1/11	304.0/305.6 (94)	

1. Pinhole sample holder lid; 2. Hermetically sealed sample holder.

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[23], and the data for PETN comes from the examination of PETN Class 4 as part of this Proficiency test[24]. Table 9 allows the comparison of the average results on $KClO_4$/C mixture with standards to obtain relative sensitivities.

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

Impact sensitivity. All participants found the $KClO_4$/C mixture to be insensitive compared to both RDX and PETN. Within the equipment limits, no participant could find a drop height that showed sensitivity.

Friction sensitivity. For BAM friction, LLNL, LANL and IHD found the $KClO_4$/C mixture to be insensitive. Within the equipment limits, no participant could find a force that showed sensitivity. For ABL friction, IHD and AFRL performed this testing and also found the $KClO_4$/C mixture to be very insensitive, although both found pressures where the mixture would respond.

Spark sensitivity. LANL, IHD, and AFRL found the $KClO_4$/C mixture to respond to high-energy stimulation. However, the mixture is less sensitive to spark stimulation than the RDX and PETN standards. LLNL found the material to be insensitive (LLNL ESD equipment is custom built).

Thermal sensitivity. The $KClO_4$/C mixture exhibited no exothermic events at normal DSC evaluation temperatures, rendering the mixture much more thermally stable than the RDX and PETN standards.

Table 9. Average Comparison values

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
$KClO_4$/C[2]	> 177[3,4]	> 320[4,5]	> 320[4,5]	> 116[4,5]
$KClO_4$/Al[6]	17[5]	62[5]	41[5]	41[5]
RDX Class 5 Type II[7]	24.1[3]	25.4[8]	19[5]	15.3[5]
PETN[9]	8.3[5]	8.0[5]	9.3[5]	6.8[5]
BAM Friction Testing[10,11]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
$KClO_4$/C[12]	> 36[13]; >36[13]	> 36[13]; >36[13]	> 36[13]; ND[14]	ND[14]; ND[14]
$KClO_4$/Al[6]	12.3; 25.5	7.2; 19.1	16.5; 26.8	ND[14]; ND[14]
RDX Class 5 Type II[7]	19.2; 25.1	19.2; 20.8	15.5; ND[14]	ND[14]; ND[14]
PETN[9]	6.4; 10.5	4.9, 8.5	4.3, 6.9	ND[14]; ND[14]
ABL Friction Testing[15-17]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
$KClO_4$/C[18]	ND[14]; ND[14]	ND[14]; ND[14]	112[19,20]; 281[19,20]	543[19,20]; 851[19,20]
$KClO_4$/Al[6]	ND[14]; ND[14]	ND[14]; ND[14]	< 30[20]; 51[20]	28[20]; ND[14]
RDX Class 5 Type II[7]	ND[14]; ND[14]	ND[14]; ND[14]	74[20]; 154[20]	93[20]; ND[14]
PETN[9]	ND[14]; ND[14]	ND[14]; ND[14]	7.7[20], 42[20]	ND[14]; ND[14]
Electrostatic Discharge[21]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
$KClO_4$/C[22]	0/10 @ 1.0[23,24]	0/20 @ 0.250[24]	0/20 @ 0.118[24]	0/20 @ 0.113[24]
$KClO_4$/Al[6]	0/10 @ 1.0	0/20 @ 0.125	0/20 @ 0.140	ND[14]
RDX Class 5 Type II[7]	0/10 @ 1.0	0/20 @ 0.0250	0/20 @ 0.095	0/20 @ 0.044
PETN[9]	0/10 @ 0.033[25]	0/20 @ 0.025	0/20 @ 0.219	0/20 @ 0.076

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 (22.8-24.4; 30-36), LANL (22.3-23.7; 36.3-50.1), IHD (20-28; 40-42), AFRL (25.0; 59-62); 3. 120-grit sandpaper data only; 4. Average of three measurements from Table 3; 5. 180-grit sandpaper; 6. From reference 25; 7. From reference 23; 8. 150-grit garnet sandpaper; 9. From reference 24; 10. 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; 11. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 12. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9-24.4; 30-32), LANL (22.7-23.6; 31.4-44.7), IHD (23; 42); 13. Average of three measurements from Table 5; 14. ND = Not determined; 15. LLNL and LANL did not perform measurements; 16. 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; 17. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 18. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (23-24; 43-48), AFRL (25.0; 58-62); 19. Average of three measurements from Table 6; 20. At 8 fps; 21. 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; 22. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.9; 30-31), LANL (23.0-23.4; 37.3-44.0), IHD (26-28; 42-44), AFRL (25.0-27.8; 45-48); 23. LLNL has 510-Ω resistor in circuit; 24. Average of three measurements from Table 7. 25. ABL ESD apparatus.

4.2 Comparison of results based on participants

Essentially all the participants derived the same conclusions about the $KClO_4$/C mixture by impact, friction, and ESD testing—the mixture is very insensitive and sensitivity levels could not be established because of the limitations of the equipment. The only exception to this is the ABL results which both IHD and AFRL were able to find a response level. However, this was at a very high pressure and so much above the pressures for the standards that the conclusion is the mixture is highly insensitive.

For thermal sensitivity, the major event observed by all participants is the phase transition of $KClO_4$ near 300°C. Figure 2 shows the DSC profiles from LLNL. This transition is an endothermic event and the only prominent feature in the DSC. LLNL observed strong exothermic features around 500°C, associated with the carbon assisted decomposition of the $KClO_4$. However, this feature is substantially higher in temperature than the standards to claim any appreciable thermal sensitivity.

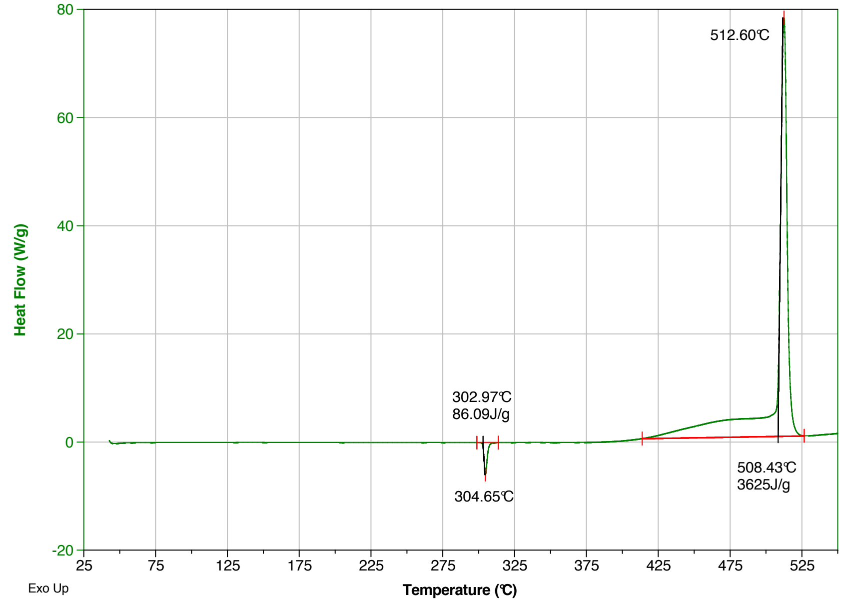

Figure 2. DSC of $KClO_4$/C from LLNL at 10°C/min heating rate.

The higher temperate exothermic feature is interesting, as it has been assigned to the decomposition of the $KClO_4$. Pure $KClO_4$ thermally decomposes around 600°C (10°/min heating rate)[18] according to equation **1**.

$KClO_4 \rightarrow KCl + 2O_2$ Equation **1**

Carbonaceous compounds have been cited in a number of cases of causing a lower decomposition temperature for this oxidizer—sucrose, 410°C[18]; carbon black, 525°C[26]; charcoal, 550°C[26].

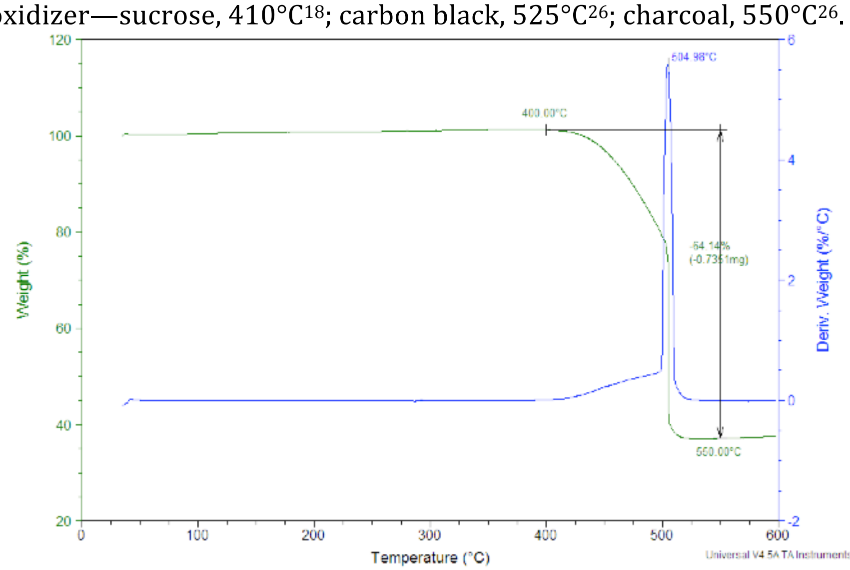

Figure 3. TGA of $KClO_4$/C mixture at 10°C/min heating rate and 50 mL/min N_2 flow.

Figure 3 shows the TGA for the $KClO_4$/C mixture for this study. The decomposition of the $KClO_4$ is clearly seen occurring around 500°C, due to the reaction of the C with the $KClO_4$ by equation 2.

$KClO_4 + 2C \rightarrow KCl + 2CO_2$ Equation **2**

4.3 Comparison of $KClO_4$/C and $KClO_4$/Al mixtures

Table 9 shows the comparison of SSST testing results for $KClO_4$/C and compares the results to $KClO_4$/Al to highlight the differences in using elemental Al and elemental C. The $KClO_4$/Al mixture has the same or higher sensitivity for all SSST testing properties than the $KClO_4$/C. In some cases, the Al mixture has similar sensitivity as PETN.

5 CONCLUSIONS

$KClO_4$/C mixture was found through SSST testing to be an insensitive mixture toward impact, friction, and spark handling conditions—generally less sensitive than RDX, and PETN. Standard thermal testing by DSC does not show any thermal exothermic events below 475°C.

REFERENCES

1. Integrated Data Collection Analysis (IDCA) Program—Proficiency Study for Small Scale Safety Testing of Homemade Explosives, B. D. Olinger, M. M. Sandstrom, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. S. Moran, T. J. Shelley, L. L. Whinnery, P. C. Hsu, R. E. Whipple, M. Kashgarian, and J. G. Reynolds, *IDCA Program Analysis Report* **001**, LLNL-TR-416101, December 3, 2009.
2. Integrated Data Collection Analysis (IDCA) Program—Drying Procedures, B. D. Olinger, M. M. Sandstrom, G. W. Brown, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. S. Moran, T. J. Shelley, L. L. Whinnery, P. C. Hsu, R. E. Whipple, and J. G. Reynolds, *IDCA Program Analysis Report* **004**, LLNL-TR-465872, April 27, 2010.
3. Integrated Data Collection Analysis (IDCA) Program—Mixing Procedures and Materials Compatibility, B. D. Olinger, M. M. Sandstrom, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. S. Moran, T. J. Shelley, L. L. Whinnery, P. C. Hsu, R. E. Whipple, M. Kashgarian, and J. G. Reynolds, *IDCA Program Analysis Report* **002**, LLNL-TR-422028, December 27, 2009.
4. $KClO_3$ and $KClO_4$ particle size distribution by Microtrac, K. B. Proctor and K. F. Warner, *IDCA Program Data Report* **075**, December 12, 2011.
5. Particle size analysis of $KClO_3$ and $KClO_4$ by Coulter Counter, E. L. Hartline and M. M. Sandstrom, *IDCA Program Data Report* **076**, January 4, 2012.
6. Activated Carbon, Kimberly B. Proctor, *IDCA Program Data Report* **072**, December 8, 2011.
7. Exp6-polar Thermodynamics of dense supercritical water, S. Bastea and L. E. Fried, *Journal of Chemical Physics*, 128, 174502 (2008).
8. Small Scale Safety Test Report for Potassium Perchlorate (85%) and Charcoal (15%) Mixture [revised 4.1.11], P. C. Hsu and J. G. Reynolds, *IDCA Program Data Report* **045**, LLNL-TR-450511, August 20, 2010.
9. Potassium Perchlorate and Carbon 51088F, M. M. Sandstrom, *IDCA Program Data Report* **018**, September 9, 2010.
10. KP/Charcoal, D. L. Remmers, D. N. Sorensen, K. F. Warner, *IDCA Program Data Report* **006**, April 13, 2011.
11. Potassium Perchlorate + Charcoal, J. A. Reyes and T. J. Shelley, *IDCA Program Data Report* 073, December 9, 2011.
12. Integrated Data Collection Analysis (IDCA) Program—SSST Testing Methods, B. D. Olinger, M. M. Sandstrom, G. W. Brown, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. S. Moran, T. J. Shelley, L. L. Whinnery, P. C. Hsu, R. E. Whipple, and J. G. Reynolds, *IDCA Program Analysis Report* in preparation.
13. Sigma-Aldrich Technical Bulletin, AL-143, Mineral Absorbents, Filter Agents and drying Agents, Activated Carbon, http://www.sigmaaldrich.com/chemistry/chemical-synthesis/learning-center/technical-bulletins/al-1430/activated-carbon.printerview.html.
14. A Method for Obtaining and Analyzing Sensitivity Data, W. J. Dixon and A.M. Mood, *J. Am. Stat. Assoc.,* **43**, 109-126, 1948.
15. The Bruceton method also assumes that testing begins in the vicinity of the mean. Often this is not true and the initial testing to home in on the mean can skew the statistics. In practice, a "Modified" Bruceton method is used in which initial tests are used to bracket the mean before starting to count Go's and No-Go's. This is used by LANL in this work.
16. A. D-Optimality-Based Sensitivity Test, B. T. Neyer, *Technometrics*, **36**, 48-60 1994.
17. Department of Defense Ammunition and Explosives Hazard Classification Procedures, TB 700-2 NAVSEAINST 8020.8B TO 11A-1-47 DLAR 8220.1, January 5, 1998.

18. Thermal decomposition of pyrotechnic mixtures containing sucrose with either potassium chlorate or potassium perchlorate, S. G. Hosseini, S. M. Pourmortazavi, and S. S, Hajimirsadeghi, *Combustion and Flame* **141**, 322-326, 2005.
19. Thermal Analysis of Pyrotechnic Compositions Containing Potassium Chlorates and Lactose, F. S. Scanes, *Combustion and Flame*, **23** (3), 363-371 (1974).
20. Integrated Data Collection Analysis (IDCA) Program—$KClO_3$ (as received)/Icing Sugar**,** M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, T. J. Shelley, P. C. Hsu, R. E. Whipple, and J. G. Reynolds, *IDCA Program Analysis Report*, **011**, LLNL-TR-484715, May 26, 2011.
21. Integrated Data Collection Analysis (IDCA) Program—$KClO_3$/Icing Sugar (-100) mixture**,** M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, T. J. Shelley, P. C. Hsu, R. E. Whipple, and J. G. Reynolds, IDCA Program Analysis Report, **007** LLNL-TR-482149 May 10. 2011.
22. Periodic Table: Carbon, http://www.chemicalelements.com/elements/c.html
23. Integrated Data Collection Analysis (IDCA) Program—RDX Standard, Data Set 1, M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. S. Moran, T. J. Shelley, P. C. Hsu, R. E. Whipple, and J. G. Reynolds, *IDCA Program Analysis Report* **006**, LLNL-TR-479891, April 19, 2011.
24. Integrated Data Collection Analysis (IDCA) Program—PETN Class 4 Standard, M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. S. Moran, T. J. Shelley, P. C. Hsu, R. E. Whipple, and J. G. Reynolds, *IDCA Program Analysis Report* 017, LLNL-TR-568299 (634352), August 1, 2012.
25. Integrated Data Collection Analysis (IDCA) Program—$KClO_4$/Aluminum Mixture, M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, T. J. Shelley, P. C. Hsu, and J. G. Reynolds, IDCA Program Analysis Report **013**, TR-518531, December 5, 2011.
26. Effect of additives on the decomposition of $KClO_4$, G. Hussain and G. J. Rees, *Fuel*, **70**(2), 1399-1401, 1991.

ABREVIATIONS, ACRONYMS AND INITIALISMS

ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Carbon (in this report, activated C)
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
H_2O	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
$KClO_3$	Potassium Chlorate
$KClO_4$	Potassium Perchlorate
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
MBOM	Modified Bureau of Mines
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
SNL	Sandia National Laboratories
SSST	small-scale safety and thermal
TGA	Thermogravimetric Analysis
TIL	Threshold level—level before positive event

ACKNOWLEDGMENTS

The IDCA thanks Heidi Turner (LLNL) and Gary Hust (LLNL) 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/RXQL and NSWC IHD is under sponsorship of the US Department of Homeland Security, Office of Science and Technology, Energetics Division.