# Integrated Data Collection Analysis (IDCA) Program —$KClO_3$/Icing Sugar (-100 mesh) 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 scanning calorimetry analysis of a mixture of $KClO_3$ sized through a 100-mesh sieve mixed with icing sugar, also sized through a 100-mesh sieve—$KClO_3$/icing sugar (-100) mixture. This material was selected because of the challenge of performing SSST testing of a mixture of two solid materials. The mixture was found to be: 1) more sensitive to impact than RDX, with sensitivity similar to PETN, 2) the same or more sensitive to friction than PETN, and 3) less sensitive to spark than RDX. The analysis showed that the mixture has thermally stability similar to RDX and is perhaps more energetic upon decomposition but variable results indicate sampling issues.

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 proficiency study is adding SSST testing results for a broad suite of different HMEs to the literature, potentially suggesting new guidelines and methods for HME testing, and possibly establishing SSST testing accuracies needed to develop safe handling practices. 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 $KClO_3$/icing sugar (-100) mixture are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), and Indian Head Division, Naval Surface Warfare Center, (IHD-NSWC). 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 not all protocols are made consistent by necessity.

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

IDCA Explosives Safety Testing of Homemade Explosives
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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 factors—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 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;

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 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. 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, $KClO_3$/icing sugar (-100) mixture, is the first in a series of materials that fall in the class of solid oxidizer/fuel mixtures. 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. Both materials were dried and sized through a 100-mesh sieve before mixing to assist in even distribution, hence the (-100) designation. In subsequent studies, the particle size of the $KClO_3$ will be varied to note the effect of particle size on test results.

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

# 2 EXPERIMENTAL

*General information*. The $KClO_3$ was obtained from Columbus Chemical as a purified powder, Catalog #423000, Lot # 200917615, CAS # 3811-04-9, assay (by manufacturer): $KClO_3$, 99.7%; KCl, 0.05%; $H_2O$, 0.005%. The icing sugar was obtained from a local food market as C & H Icing (powdered) sugar, Lot # 79174-A14. No manufacturer analysis was given on the container, but the C & H sugar web site[2] lists the icing sugar as having 3% cornstarch added as an anti-caking agent. SNL provided elemental composition from combustion analysis and Karl Fischer assay: C, 41.70 ± 0.05%; H, 6.24 ± 0.10%; N, 0.35 ± 0.25%; O, 51.49 ± 0.48%; moisture, 0.29 ± 0.01%; residual 0.21 ± 0.29%[3]. Both precursors were dried for 16 h and cooled in a desiccator according to IDCA drying methods[4]. Both were sieved through a minus 100-mesh (150 μm hole size) sieve. The mixture was prepared by hand, mixing the two solids together in a materials compatible polypropylene container according to IDCA mixing and compatibility procedures[5]. The mixture composition is 74-wt % $KClO_3$ and 26-wt % icing sugar. 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 determined by thermochemical calculations using Cheetah to find the optimum detonation energy output[1]. The ratio chosen matched stoichiometric for oxygen balance.

The SSST testing data for the individual participants was obtained from the following reports: LLNL IDCA Project Report—KC/Sugar (screened) [revised 4.20.11] (LLNL)[6], Potassium Chlorate and Sugar 51088B, revised 4.6.11 (LANL)[7], and KC/Sugar Report [revised 3.30.11] (IHD)[8].

**Table 2. Summary of conditions for the analysis of $KClO_3$/icing sugar (-100) mixture (All = LANL, LLNL, IHD)**

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[4]
3. Sample form—All, loose powder
4. Powder sample configuration—All, conical pile
5. Apparatus—All, Type 12A*
6. Sandpaper—LANL, 150 grit and 180 garnet; IHD, 180 garnet; LLNL, 120 flint S/C paper
7. Sandpaper size—LLNL, 1 inch square; LANL, 1.25 inch diameter disk dimpled; IHD not specified
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL and IHD, 2.5 kg; LANL, 0.8 kg
10. Positive detection—LANL and LLNL, microphones with electronic interpretation as well as observation; IHD, observation
11. Data analysis—All, modified Bruceton and TIL before and above threshold; LANL Neyer also

Friction analysis

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

ESD

1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, dried per IDCA drying methods[4]
3. Sample form—All, powder
4. Tape cover—LANL, scotch tape; LLNL, Mylar; IHD, none
5. Sample configuration—All, cover the bottom of sample holder
6. Apparatus—LANL and IHD, 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[4]
3. Sample holder—All, hermetic with pin hole; LLNL, sealed also
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C
6. Sample holder hole size—LLNL, 50 μm; LANL and IHD, 75 μm
7. Instruments—LANL, TA Instruments Q2000; LLNL, TA Instruments 2920; IHD, TA Instruments Q1000*

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:* 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 $KClO_3$/icing sugar (-100) mixture.

# 3 RESULTS

## 3.1 $KClO_3$/icing sugar (-100) 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[9], which compares the different procedures by each testing category. LANL, LLNL, and IHD participated in this part of the SSST testing of the mixture. Screening the $KClO_3$ at -100 mesh was performed primarily so the effect of particle size of the oxidizer could be compared. A subsequent report will be issued where the oxidizer was screened at -40 mesh. Although $KClO_3$ and sugar mixtures can be found at a variety of mixing ratios, the ratio for this study was selected that conforms to stoichiometry[1].

## 3.2 Impact testing results for $KClO_3$/icing sugar (-100) mixture

Table 3 shows the results of impact testing of the $KClO_3$/icing sugar (-100) mixture as performed by LANL, LLNL, and IHD. 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 reaction. LANL used both 150- and 180-grit sandpaper, IHD used 180-grit sandpaper for impact testing, and LLNL used 120-grit S/C flint paper for the impact testing. All participants performed data analysis by normal modified Bruceton method[10,11] and LANL also performed data analysis by the Neyer method[12].

**Table 3. Impact testing results for $KClO_3$/icing sugar (-100) mixture**

| Lab[1] | Test Date | T, °C | RH, %[2] | $DH_{50}$, cm[3] | s, cm[4] | s, log unit[4] |
|---|---|---|---|---|---|---|
| LLNL (120) | 1/22/10 | 23.3 | 21 | 14.5 | 2.18 | 0.065 |
| LLNL (120) | 2/25/10 | 22.8 | 28 | 14.0 | 0.71 | 0.022 |
| LLNL (120) | 2/16/10 | 22.8 | 28 | 16.1 | 0.74 | 0.020 |
| LANL (150) | 2/8/10 | 21.2 | 13.5 | 15.5 | 2.73 | 0.076 |
| LANL (150) | 2/9/10 | 21.1 | 14.2 | 17.7 | 1.80 | 0.044 |
| LANL (150) | 2/10/10 | 21.8 | 13.5 | 18.8 | 1.52 | 0.035 |
| LANL (180) | 4/28/10 | 22.3 | <10 | 10.7 | 1.88 | 0.076 |
| LANL (180) | 4/29/10 | 22.1 | <10 | 11.8 | 4.07 | 0.147 |
| LANL (180) | 5/4/10 | 22.0 | <10 | 9.2 | 1.32 | 0.062 |
| IHD (180) | 1/21/10 | 26 | 40 | 14 | 2.3 | 0.07 |
| IHD (180) | 2/3/10 | 27 | 40 | 15 | 6.4 | 0.18 |
| IHD (180) | 2/3/10 | 27 | 40 | 14 | 4.6 | 0.14 |

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

The test results from the three participating laboratories for impact show a range for $DH_{50}$ from 9.2 to 18.8 cm. The average $DH_{50}$ values, in cm, are LLNL, 14.9 ± 1.1; LANL, 14.0 ± 3.9; IHD, 14.3 ± 0.6. The average $DH_{50}$ values, in cm, based on grit size are 120, 14.9 ± 1.1; 150, 17.3 ± 1.7; 180, 12.5 ± 2.5. The standard deviation, log s, is below the 0.1 log unit range except for IHD, where the value is over 0.1 log units. This appears as a result of IHD using 0.1 log spaced steps while LANL and LLNL use 0.05 log spaced steps. The impact of step spacing will be evaluated in detail in a later report.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method[12]. The $DH_{50}$ values are in the same range as the values analyzed by the Bruceton method, where the average for the Neyer method is 16.9 ± 2.6 cm and 9.3 ± 0.8 cm for the tests that used 150-grit and 180-grit sandpaper, respectively, which compares with 17.3 ± 1.7 cm and 10.6 ± 1.3 cm, respectively.

**Table 4. Impact testing results for $KClO_3$/icing sugar (-100) mixture (Neyer or D-Optimal Method) 150- and 180-grit sandpaper**

| Lab[1] | Test Date | T, °C | RH, %[2] | $DH_{50}$, cm[3] | s, cm[4] | s, log unit[4] |
|---|---|---|---|---|---|---|
| LANL (150) | 2/8/10 | 21.1 | 14.0 | 15.4 | 0.87 | 0.024 |
| LANL (150) | 2/9/10 | 21.1 | 14.2 | 19.9 | 1.91 | 0.042 |
| LANL (150) | 2/10/10 | 21.8 | 13.5 | 15.5 | 0.80 | 0.022 |
| LANL (180) | 4/28/10 | 22.3 | <10 | 10.3 | 0.99 | 0.044 |
| LANL (180) | 4/29/10 | 22.1 | <10 | 8.8 | 1.16 | 0.058 |
| LANL (180) | 5/4/10 | 22.0 | <10 | 8.9 | 1.68 | 0.083 |

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

## 3.3 Friction testing results for $KClO_3$/icing sugar (-100) 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)[13], and a modified Bruceton method[10]. The average friction values for $F_{50}$ are: LLNL, 9.9 ± 1.8 kg; LANL, 5.8 kg; IHD, 4.4 ± 0.3 kg. The standard deviation, log s, values range for all $0.03 < s < 0.098$. The threshold values are in the following order IHD < LANL < LLNL. For TIL, LANL and IHD recorded the similar values while LLNL have higher values.

**Table 5. BAM Friction Testing results for $KClO_3$/icing sugar (-100) mixture**

| 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 | 1/21/10 | 23.9 | 21 | 0/10 @ 6.0 | 1/10 @ 6.4 | 7.9 | 0.89 | 0.049 |
| LLNL | 2/19/10 | 22.2 | 31 | 0/10 @ 8.0 | 1/10 @ 8.4 | 10.3 | 0.74 | 0.031 |
| LLNL | 2/16/10 | 22.8 | 21 | 0/10 @ 6.4 | 1/10 @ 6.8 | 11.4 | 1.32 | 0.050 |
| LANL | 2/8/10 | 21.2 | 13.2 | NA[6] | NA[6] | 5.8 | 2.4 | 0.19 |
| LANL | 2/9/10 | 21.3 | 13.4 | NA[6] | NA[6] | <4.3[7] | NA[7] | NA[7] |
| LANL | 2/10/10 | 21.7 | 13.4 | NA[6] | NA[6] | <5.3[7] | NA[7] | NA[7] |
| LANL | 2/19/10 | 22.0 | <10 | 0/10 @ 2.4 | 1/2 @ 4.8 | NA[8] | NA[8] | NA[8] |
| LANL | 2/19/10 | 22.0 | <10 | 0/10 @ 2.4 | 1/2 @ 4.8 | NA[8] | NA[8] | NA[8] |
| LANL | 2/19/10 | 22.0 | <10 | 0/10 @ 2.4 | 1/2 @ 4.8 | NA[8] | NA[8] | NA[8] |
| IHD | 1/25/10 | 27 | 47 | 0/10 @ 1.4 | 1/1 @ 1.6 | NA[8] | NA[8] | NA[8] |
| IHD | 2/12/10 | 26 | 41 | 0/10 @ 2.0 | 1/3 @ 2.5 | NA[8] | NA[8] | NA[8] |
| IHD | 2/12/10 | 27 | 45 | 0/10 @ 2.5 | 1/2 @ 2.9 | NA[8] | NA[8] | NA[8] |
| IHD | 2/26/10 | 26 | 41 | NA[6] | NA[6] | 4.1 | 0.83 | 0.087 |
| IHD | 3/2/10 | 25 | 40 | NA[6] | NA[6] | 4.6 | 1.00 | 0.098 |
| IHD | 2/26/10 | 26 | 41 | NA[6] | NA[6] | 4.4 | 0.86 | 0.084 |

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% Reaction, LLNL and IHD use log spacing; LANL uses linear spacing; 5. Standard Deviation; 6. Not applicable, separate sample used for TIL analysis; 7. The material shows a positive at 2.4 kg, the lowest value available on the LANL BAM friction tester with the 2.4 kg spacing scale (linear spacing). The statistical evaluation is not valid as a result; 8. Not applicable, separate sample used for Bruceton analysis.

Table 6 shows the ABL Friction testing performed by IHD on the $KClO_3$/icing sugar (-100) mixture. IHD was the only participant to report ABL Friction testing results. LANL did not have the system in routine op-

eration at the time. LLNL does not have ABL Friction. The results show the $F_{50}$ is about 40 psig @ 8 fps and the threshold around 30 psig @ 8 fps.

**Table 6. ABL Friction testing results for $KClO_3$/icing sugar (-100) 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, kg[6] | s, log unit[6] |
|---|---|---|---|---|---|---|---|---|
| IHD | 1/25/10 | 27 | 43 | 0/20 @ <30/8 | 1/6 @ 30/8 | 40/8 | 18 | 0.19 |
| IHD | 2/24/10 | 26 | 40 | 0/20 @ <30/8 | 1/2 @ 30/8 | 43/8 | 18 | 0.18 |
| IHD | 2/24/10 | 25 | 41 | 0/20 @ <30/8 | 1/5 @ 30/8 | NA[7] | NA[7] | NA[7] |
| IHD | 2/25/10 | 26 | 43 | NA[8] | NA[8] | 44 | 20 | 0.19 |

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. 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; 4. Next level where positive initiation is detected; 5. $F_{50}$, in psig/fps, modified Bruceton method, load for 50% Reaction; 6. Standard deviation; 7. Not applicable, TIL only was performed; 8. Not applicable, $F_{50}$ measurements only.

## 3.4 Electrostatic discharge testing of $KClO_3$/icing sugar (-100) mixture

Electrostatic Discharge (ESD) testing of the $KClO_3$/icing sugar (-100) mixture was performed by LANL, IHD and LLNL. 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 series 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. (LLNL has purchased a new ABL ESD tester and is being used for the spark testing on the 3rd RDX calibration run and the remaining IDCA test materials.) All participants performed data analysis using the threshold initiation level method (TIL)[17].

**Table 7. Electrostatic discharge testing $KClO_3$/icing sugar (-100) mixture**

| Lab | Test Date | T, °C | RH, %[1] | TIL, Joule[2] | TIL, Joule[3] |
|---|---|---|---|---|---|
| LLNL[4] | 1/21/10 | 23.9 | 21 | 0/10 @ 1.0[5] | 0/10 @ 1.0[5] |
| LLNL[4] | 2/17/10 | 22.8 | 32 | 0/10 @ 1.0[5] | 0/10 @ 1.0[5] |
| LLNL[4] | 2/22/10 | 22.8 | 23 | 0/10 @ 1.0[5] | 0/10 @ 1.0[5] |
| LANL | 2/8/10 | 21.1 | 13.7 | 0/20 @ 0.0625 | 2/3 @ 0.125 |
| LANL | 2/9/10 | 21.1 | 13.6 | 0/20 @ 0.0625 | 2/13 @ 0.125 |
| LANL | 2/10/10 | 21.6 | 12.7 | 0/20 @ 0.0625 | 2/5 @ 0.125 |
| IHD | 1/21/10 | 27 | 40 | 0/20 @ 0.165 | 1/3 @ 0.326 |
| IHD | 2/2/10 | 27 | 40 | 0/20 @ 0.326 | 1/1 @ 0.853 |
| IHD | 2/2/10 | 26 | 40 | 0/20 @ 0.326 | 1/1 @ 0.853 |

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.

The testing results from LANL indicate the $KClO_3$/icing sugar (-100) mixture is more sensitive than the testing results from the IHD and LLNL. The data from LLNL show a non-sensitive material. This is expected because of the experimental configuration.

## 3.5 Thermal testing (DSC) of $KClO_3$/icing sugar (-100) mixture

Differential Scanning Calorimetry (DSC) was performed on the $KClO_3$/icing sugar (-100) mixture by LLNL, LANL, and IHD. All participating laboratories used different versions of the DSC by TA Instruments.

Table 8 shows the DSC data from each of the participating laboratories. For all three participants there is observed a sharp, low temperature exothermic feature with $T_{max}$ values ranging from 176.7 to 183.7 °C. Some analyses showed a second exothermic feature near 335 °C. For the enthalpy values of the low temperature exothermic feature, when two features are observed, are much lower than the results when one feature is ob-

served—569 to 932 J/g and 2873 to 3095 J/g, respectively. All participants observed instrument limitations that manifest as a positive slope on the cooling part of the feature. This is due to rapid gas loss from the pan and indicates that the reported enthalpy is a lower limit. If the pans could contain the gases, the enthalpies would be higher because no heat would be lost with the mass during the decomposition. The issues with the DSC of this material will be discussed in detail elsewhere, but it should be noted that with additional testing, all three laboratories observed both 1 and 2 exothermic features in at least one sample.

**Table 8. Differential Scanning Calorimetry results for $KClO_3$/icing sugar (-100) mixture, 10°C/min heating rate**

| Lab | Test Date | Exothermic, onset/maximum, °C (ΔH, J/g) | Exothermic, onset/maximum, °C (ΔH, J/g) |
|---|---|---|---|
| LLNL[1] | 1/22/10 | 175.5/182.2 (3095) | NA[2] |
| LLNL[1] | 2/19/10 | 177.2/183.7 (2873) | NA[2] |
| LLNL[1] | 2/04/10 | 176.4/183.2 (3054) | NA[2] |
| LLNL[3] | 2/22/10 | 175.1/180.8 (3689) | NA[2] |
| LLNL[3] | 02/18/10 | 176.0/181.7 (3983) | NA[2] |
| LLNL[3] | 02/24/10 | 176.4/182.3 (3752) | NA[2] |
| LANL[1] | 2/8/10 | 177.3/178.2 (653) | 333.7/334.2 (963) |
| LANL[1] | 2/9/10 | 177.3/177.8 (569) | 334.1/334.5 (724) |
| LANL[1] | 2/10/10 | 175.9/177.0 (637) | 334.4/334.8 (946) |
| IHD[1] | 2/17/10 | 176.0/176.7 (634) | 334.5/334.9 (928) |
| IHD[1] | 11/25/09 | 177.1/178.1 (932) | 334.2/334.5 (660) |
| IHD[1] | 11/25/09 | 176.1/177.4 (848) | 334.7/335.2 (906) |

1. sample holder with pin-hole in lid; 2. Not observed in this data set; 3. Sample holder with hermetically sealed lid.

Table 8 also shows the DSC data, by LLNL only, for the $KClO_3$/icing sugar (-100) mixture where the DSC sample holder is closed instead of pinhole vented as used in the other measurements shown in Table 8. The $T_{max}$ values for the exothermic feature are very similar to comparable data from LLNL with the pinhole sample holder; the $T_{max}$ values are slightly lower, but the enthalpy values are around 20% high for the data from the hermetically sealed sample holder. This latter factor is probably due to the closed system having better heat transfer properties retaining the decomposition gases better than the pinhole system does, although, as noted with the other data in Table 8, these sample holders appear to lose mass during decomposition, also.

# 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[14]. The data for PETN was provided by the participating laboratories (when available) from measurements performed outside this Proficiency Test. Table 10 allows the comparison of the average results on $KClO_3$/icing sugar (-100) mixture with standards to obtain relative sensitivities.

## 4.1 Sensitivity of $KClO_3$/icing sugar (-100) mixture compared to standards

*Impact sensitivity*. Although the impact sensitivity varies among the participating laboratories for the $KClO_3$/icing sugar (-100) mixture, the overall trend is that it is more impact sensitive than RDX, roughly similar to PETN.

*Friction sensitivity*. Although LLNL results for BAM friction do not agree with LANL and IHD results, when compared to the RDX standard, the $F_{50}$ friction values for $KClO_3$/icing sugar (-100) mixture are significantly lower than corresponding values for RDX, indicating the mixture is more sensitive to friction. The

only threshold data for PETN comes from LLNL and comparison with the $KClO_3$/icing sugar (-100) mixture threshold data would suggest that it is as sensitive, if not more 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 |
| $KClO_3$/icing sugar (-100)[2] | 14.9[3,4] | 14.0[5] | 14.3[3,6] | ND[7] |
| RDX Class 5 Type II[8] | 24.1[4] | 25.4[9] | 19[6] | 15.3[6] |
| PETN[10] | 15 | 14.7 | ND[5] | ND[4] |
| BAM Friction Testing[11,12] | TIL, kg; $F_{50}$, kg | TIL, kg; $F_{50}$, kg | TIL, kg; $F_{50}$, kg | TIL, kg; $F_{50}$, kg |
| $KClO_3$/icing sugar (-100)[13] | 6.8[14]; 9.9[14] | 2.4[14]; 5.8[14] | 2.0[14]; 4.4[14] | ND[7] |
| RDX Class 5 Type II[8] | 19.2; 25.1 | 19.2; 20.8 | 15.5; ND[7] | ND[7]; ND[7] |
| PETN[10] | 6.4; 10.5 | ND[7]; 9.2 | ND[7]; ND[7] | ND[7]; ND[7] |
| ABL Friction Testing[15-18] | TIL, psig; $F_{50}$, psig | TIL, psig; $F_{50}$, psig | TIL, psig; $F_{50}$, psig | TIL, psig; $F_{50}$, psig |
| $KClO_3$/icing sugar (-100)[19] | ND[7] | ND[7] | < 30[20]; 42[20] | ND[7] |
| RDX Class 5 Type II[8] | ND[7] | ND[7] | 55; 154 | 71; ND[7] |
| PETN[10] | ND[7] | ND[7] | ND[7] | ND[7] |
| Electrostatic Discharge[21] | TIL, Joules | TIL, Joules | TIL, Joules | TIL, Joules |
| $KClO_3$/icing sugar (-100)[22] | 0/10 @ 1.0[23,24] | 0/20 @ 0.0625[24] | 0/20 @ 0.272[24] | ND[7] |
| RDX Class 5 Type II[8] | 0/10 @ 1.0 | 0/20 @ 0.0255 | 0/20 @ 0.0.095 | 0/20 @ 0.044 |
| PETN[10] | 0/10 @ 1.0 | 2/2 @ 0.125 | ND[7] | ND[7] |

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 (22.8–23.3; 21–28), LANL (21.1–22.3; <10–14.2), IHD (26–27; 40); 3. Average of three measurements from Table 3; 4. 120-grit sandpaper data only; 5. Average of six measurements (150- and 180-grit sandpaper) from Table 3; 6. 180-grit sandpaper; 7. ND = Not determined; 8. RDX average values from reference 14; 9. 150-grit sandpaper; 10. PETN values from testing outside of the Proficiency test (sand paper grit size not specified); 11. 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; 12. $F_{50}$, in kg, is by a modified Bruceton method, load for 50% Reaction; 13. Temperature and humidity values varied during the sets of measurements ($T_{range}$, °C; $RH_{range}$, %)—LLNL (22.2–23.9; 21–31), LANL (21.2–22.0; <10–13.4), IHD (25–27; 40–47); 14. Average of three measurements from Table 5; 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% Reaction; 18. Measurements performed at 8 fps; 19. Temperature and humidity values varied during the sets of measurements ($T_{range}$, °C; $RH_{range}$, %)—IHD (25–27; 40–43); 20. Average of three measurements from Table 6; 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 (22.8–23.9; 21–32), LANL (21.1–21.6; 12.7–13.7), IHD (26–27; 40); 23. LLNL has 510-Ω series resistor in circuit; 24. Average of three measurements from Table 7.

For the current set of ABL friction data, IHD is the only participant that provided any data that can be compared to standards. When examining the RDX standard data, $KClO_3$/icing sugar (-100) mixture appears to be much more sensitive, consistent with the BAM friction results. Currently, there is no ABL friction data for PETN.

*Spark sensitivity*. Comparing the $KClO_3$/icing sugar (-100) mixture spark sensitivity values to the corresponding RDX values, the mixture is less spark sensitive than RDX. There are limited values on PETN, but the comparison shows the mixture to have about the same sensitivity as PETN.

*Thermal sensitivity*. The $KClO_3$/icing sugar (-100) mixture shows two exothermic features—one with a $T_{max}$ around 177°C and one around 334°C. The occurrence of these features in a specific sample depends upon factors such as sampling. When comparing this result to RDX (exothermic feature around 250°C with 2200

ΔH in J/g), the $KClO_3$/icing sugar (-100) mixture has an on-set of thermal reactivity slightly below RDX, and is evaluated to be about as sensitive. In additional samples, when only one exothermic feature is observed (this occurs near 177°C), the enthalpy is similar to or more than that observed for RDX decomposition. The behavior of the $KClO_3$/icing sugar (-100) mixture in DSC will be discussed in more detail elsewhere—particularly the impact of sample preparation as well as mixing. However, for SSST evaluation of this material, the low temperature exothermic feature is the most important feature by which to evaluate the material, because it indicates a low-temperature thermal sensitivity, and is observed in all samples.

## 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, all participants show about the same sensitivity for the $KClO_3$/icing sugar (-100) mixture. However, the LANL results are based on using two different grit sizes of sand paper—180 and 150. The 150-grit set of results indicates a more stable material than the 180-grit set of results. This could be fortuitous, because looking at the trend based on grit size and not participant shows no simple relationship. However, to address this issue, the IDCA participants have agreed to use 180-grit sand paper all from the same batch for future measurements. The average results from RDX exhibited a participant-to-participant variation as well.

For BAM Friction, LLNL average values for both TIL and $F_{50}$ indicate a less sensitive material than the comparable values from LANL and IHD. For the RDX average values for $F_{50}$, also show that LLNL finds the material less friction sensitive than the other participants. For ESD, LLNL consistently shows a much more stable material, highlighting the large design difference between the LLNL spark testing system and the others participants. In addition, the ESD averages from IHD indicate a more stable material than the averages from LANL, paralleling the averages for the RDX data from the respective laboratories.

## 4.3 Comparisons of DSC among participants

As noted in the Results section, the DSC behavior of the $KClO_3$/icing sugar (-100) mixtures is highly sample dependent, where one or two exothermic features are observed. Figure 1 shows examples of the DSC profiles from each of the participants. For these specific profiles, LANL and IHD show two exothermic features, while the profile from LLNL shows only one exothermic feature. However, all three participants generated profiles showing both one and two exothermic features when studying additional samples.

The low temperature exothermic event seen in Figure 1 is due to a combination of the sugar melting (endothermic features) and rapid decomposition of the mixture (exothermic features) where $C_{12}H_{22}O_{11} + 8\ KClO_3 \rightarrow 8\ KCl + 12\ CO_2 + 11\ H_2O$. This has been documented in previous DTA and TGA experiments on $KClO_3$ and $KClO_4$ + sugar mixtures[15,16].

The higher temperature exothermic feature seen in the data appears to have sample size dependence and possibly can be attributed to the reaction of the potassium chlorate with residual carbon from an incomplete oxidation that occurred around the 180°C temperature. $KClO_3$ alone starts to melt near 300°C[15] but does not decompose until over 400°C.

Physical effects of mixing and reactant contact on the local level can explain the appearance of one vs. two exothermic features. Consider different sample sizes in the DSC sample holders. First, smaller samples will likely have isolated islands of material in the DSC sample holder that do not mix well, even when the sugar melts, because of their physical separation. Some of these will have more sugar than stoichiometric, leaving

carbon residue that can react with the oxidizer rich areas at higher temperatures later, when the $KClO_3$ melts. Larger sample sizes, however, allow for a more continuous/connected mixture in the DSC sample holder as the sugar melts so the solid oxidizer has a better chance to react without isolated pockets of each material occurring on the inner sample holder surface. This will produce less residual carbon to contribute to the second reaction at higher temperatures and often will result in only one or none reactant being in excess at the end so that the second exothermic feature is not observed. Obviously there will not be a mass threshold where this effect occurs since it will vary somewhat with the uncontrollable distribution of the sample in the holder–larger samples will have a better chance of mixing.

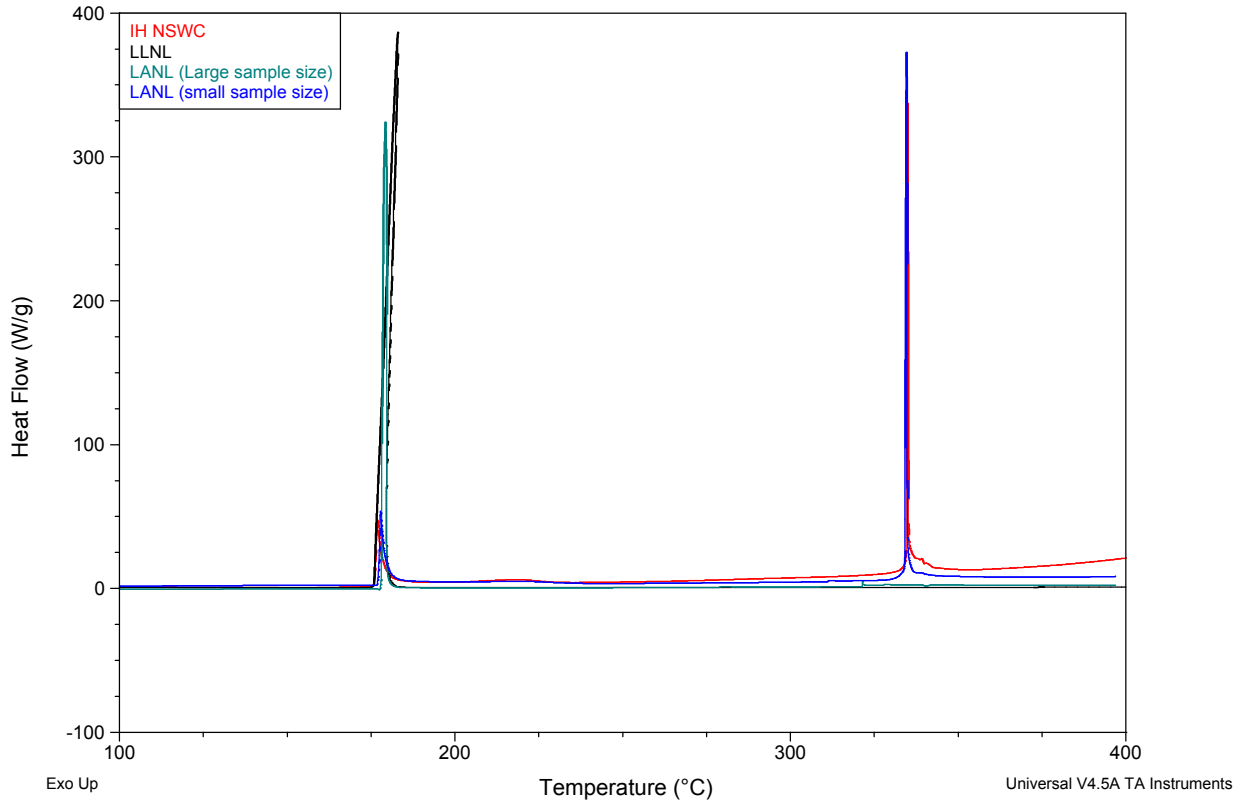


**Figure 1. DSC profiles of $KClO_3$/icing sugar (-100) mixture from LANL, IHD, and LLNL at 10°C/min heating rate.**

Regardless of the mechanism, these results indicate that the low temperature exothermic event is more appropriate for characterizing bulk samples of $KClO_3$/icing sugar mixtures since bulk samples will always be well-connected mixtures and not exhibit isolated pockets of materials. This indicates that $KClO_3$/icing sugar (-100) mixture is then as thermally stable as RDX and perhaps more energetic upon decomposition.

## 4.4 Comparison with other data

There is some SSST testing data on $KClO_3$/sugar from other sources[17-20]. Because the materials, methods and equipment configurations were not always delineated, the comparison of the data in these sources can only be qualitative. In addition, the $KClO_3$ in some cases was from different manufacturers and sized differently. The sugar was from several batches, some from C&H Brand and some from Dixie Crystal Brand; some were icing sugar and some cane sugar. The data come from primarily two laboratories—LLNL and LANL, with

the earliest data from 2003. Data were selected only if a complete set was available (impact, friction and ESD), and extreme outliers were excluded (one data set reported a $DH_{50}$ of 69 cm).

Comparing the 74/26 weight ratio formulations, only, the impact values range from roughly 12 to 38. The IDCA values are in the middle of this range. The TIL friction data ranges from 7.2 to 11.2, and the IDCA values are reasonably close to this range, overlapping on the low end. Most of the ESD data is from the LLNL system, which has a 500-Ω resistor in the discharge line, and shows no sensitivity seen for this formulation ratio. However, previous work at LANL shows $KClO_3$/sugar has an ESD sensitivity that is on the order of PETN, slightly more than what is seen with the IDCA testing results.

# 5 CONCLUSIONS

$KClO_3$/icing sugar (-100) mixture was found through SSST testing to be a moderately sensitive mixture toward impact, friction, spark and thermal handling conditions—generally more sensitive than RDX, and on the order of sensitivity of PETN.

For RDX and the $KClO_3$/icing sugar (-100 mesh) mixture studied with the current equipment configurations and experimental methods used in this Proficiency study, the impact results from LANL tend to show materials to have more stability than the results from LLNL or IHD. For friction and ESD, results from LLNL tend to show the $KClO_3$/icing sugar (-100 mesh) mixture to be less sensitive than the corresponding results from IHD and LANL. For thermal results, unlike in the case for RDX, where all the participants had results that were virtually identical, sampling issues are causing inconsistent results.

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# ACRONYMS AND INITIALISMS

| | |
|---|---|
| -100 | solids sized through a 100-mesh sieve |
| $\Delta H$ | Change in enthalpy |
| ABL | Allegany Ballistics Laboratory |
| AFRL | Air Force Research Laboratory, RXQF |
| AN | ammonium nitrate |
| BAM | German Bundesanstalt für Materialprüfung Friction Apparatus |
| C | Carbon |
| $C_{12}H_{22}O_4$ | Sugar |
| CAS# | chemical abstract services log number |
| $CO_2$ | Carbon Dioxide |
| $DH_{50}$ | 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 |
| $F_{50}$ | For BAM Friction, $F_{50}$, in kg, is by a modified Bruceton method, load for 50% probability of reaction; For ABL Friction, F50 in psig/fps, is by modified Bruceton Method, load for 50% probability of reaction |
| fps | Feet per second |
| g | grams |
| H | Hydrogen |
| $H_2O$ | Water |
| HME | homemade explosives or improvised explosives |
| HMX | cyclotetramethylene-tetranitramine |
| HP/F | hydrogen peroxide/fuel |
| IDCA | Integrated Data Collection Analysis |
| IHD | Indian Head Division, Navel Surface Warfare Center |

| | |
|---|---|
| j | joules |
| KCl | Potassium chloride |
| $KClO_3$ | Potassium Chlorate |
| $KClO_4$ | Potassium perchlorate |
| LANL | Los Alamos National Laboratory |
| LLNL | Lawrence Livermore National Laboratory |
| MEKP | methyl ethyl ketone peroxide |
| N | nitrogen |
| O | oxygen |
| PETN | Pentaerythritol tetranitrate |
| psig | pounds per square inch, gauge reading |
| RDX | Research Department Explosive, 1,3,5-Trinitroperhydro-1,3,5-triazine |
| s | Standard Deviation |
| S/C | Silicon carbide |
| SNL | Sandia National Laboratories |
| SO/F | solid oxidizer/fuel |
| SSST | small-scale safety and thermal |
| TGA | Thermogravimetric Analysis |
| TIL | Threshold Initiation Level (TIL) is the load (in kg for BAM friction, in psig/fps for ABL friction, in joules for ESD) 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; |
| $T_{max}$ | Temperature maximum of peak |
| UN | urea nitrate |

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