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Integrated Data Collection Analysis (IDCA) Program - NaClO3/Icing Sugar

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Integrated Data Collection Analysis (IDCA) Program —$NaClO_3$/Icing Sugar

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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 $NaClO_3$ and icing sugar—$NaClO_3$/icing sugar mixture. The mixture was found to: be more sensitive than RDX but less sensitive than PETN in impact testing (180-grit sandpaper); be more sensitive than RDX and about the same sensitivity as PETN in BAM fiction testing; be less sensitive than RDX and PETN except for one participant found the mixture more sensitive than PETN in ABL ESD testing; and to have one to three exothermic features with the lowest temperature event occurring at ~ 160°C always observed in thermal testing. Variations in testing parameters also affected the sensitivity.

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, but the test procedures differ. The results are compared among the laboratories and then compared to historical data from various sources. The testing performers involved 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 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, sodium chlorate, sugar, dodecane, PETN, carbon.

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 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, $NaClO_3$/icing sugar mixture, is the seventh in a series of materials that are in the class of solid oxidizer/fuel mixtures, the fifth that is a mixture of solid oxidizer and a solid fuel, and the third in a series of oxidizers with icing sugar as the 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 $NaClO_3$ was dried as previously described and separated through a 40-mesh sieve. The icing sugar was dried and sized through a 100-mesh sieve before mixing.

The testing performers in this work are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, Indian Head Division, (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 $NaClO_3$ was obtained from Acros Organics as sodium chlorate, 99+%, extra pure white powder, Catalog #22322, Lot # A0265264, CAS # 7775-09-9, assay Certificate of Analysis (by manufacturer): $NaClO_3$, > 99.0%; H_2O, < 0.05%; insoluble matter in H_2O < 0.005%. For the icing sugar, no manufacturer analysis was given on the container, but the C & H sugar web site[4] lists the icing sugar as having 3% cornstarch added as an anti-caking agent. DHS 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%[5]. The $NaClO_3$ was dried for 16 h and cooled in a desiccator according to IDCA drying methods[2]. The $NaClO_3$ was separated through a 40-mesh (425 μm hole size) sieve. The particle size distribution of the $NaClO_3$ was measured on a Horiba LA950 laser particle size analyzer[6]. The mixture was prepared by hand, adding the icing sugar to the $NaClO_3$ while stirring with a spatula in a materials compatible polypropylene container according to IDCA mixing and compatibility procedures[3]. The mixture composition is 79-wt. % $NaClO_3$ and 21-wt. % icing sugar. The final mixture had the appearance of a white powder. Typically, the precursors are mixed at that ratio to give approximately a 1-gram sample. Three samples were prepared this way and tested separately. The mixing ratio was determined by thermochemical calculations using Cheetah[7] and the ratio chosen matched stoichiometric for oxygen balance.

Table 2. Summary of conditions for the analysis of $NaClO_3$/icing sugar 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—All (180-grit garnet); LLNL (120-grit Si/Carbide)
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; LLNL 50 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 $NaClO_3$/icing sugar mixture.

The SSST testing data for the individual participants was obtained from the following reports: Small Scale Safety Test Report for Sodium Chlorate (71%) and Icing Sugar (29%) Mixture [revised 4.1.11] (LLNL)[8], 51088H Sodium Chlorate/Icing Sugar (LANL)[9], Sodium Chlorate/Sugar (IHD)[10], and Sodium Chlorate (SC) + Sugar (AFRL)[11].

3 RESULTS

3.1 $NaClO_3$/icing sugar 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 $NaClO_3$.

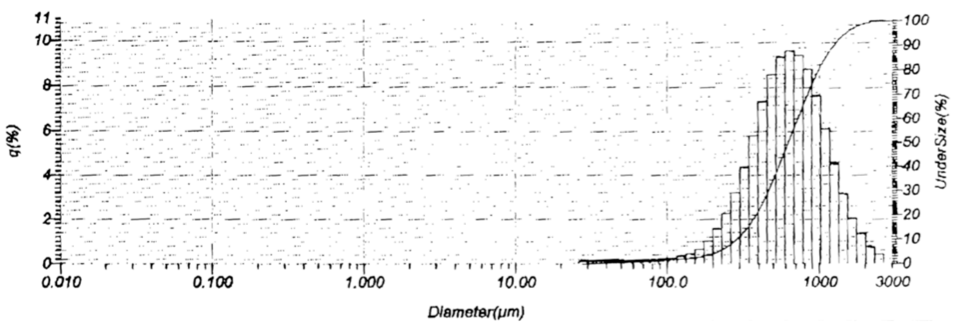

Figure 1. Particle size distribution of $NaClO_3$ by laser diffraction particle size determination method.

Separating the $NaClO_3$ using a 40-mesh sieve was performed because the material as received was inordinately large and would not be a good particle size match for the icing sugar. Previous studies on the metal oxide mixtures have shown the icing sugar used here passes through a 100-mesh sieve (149 um)[13], and some IDCA studies suggest that particle size mismatch affects at least drop hammer testing[14]. Figure 1 shows the particle size distribution of the as-received $NaClO_3$ by laser diffraction after it was dried at 60°C for 16 hours[6]. The mean diameter is 623 um. The material seemed to naturally breakdown to a free-flowing powder with slight mechanical agitation.

There are many commercial sources of icing sugar, most containing about 97% sucrose and the balance anti-caking agents, such as corn starch[15]. The sugar used in this study was selected randomly from a commercial source. This particular batch was used in previous IDCA studies on $KClO_3$/icing sugar mixture using -40 and -100 mesh size $KClO_3$[16,17]. Although $NaClO_3$ and sugar mixtures can be made at a variety of mixing ratios, the ratio for this study was selected that conforms to stoichiometric as determined by thermochemical assessments.

3.2 Impact testing results for $NaClO_3$/icing sugar mixture

Table 3 shows the results of impact testing of the $NaClO_3$/icing sugar 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 performed data analysis by normal modified Bruceton method[18,19]. All participants found the $NaClO_3$/icing sugar mixture to be sensitive to impact testing. Most testing was performed using 180-grit sandpaper to hold the sample. Examining all the values in Table 3 shows wide variation in the DH_{50} values with the average, inclusive of all the testing performed with 180-grit sandpaper, being 15.5 ± 5.5 cm. For the individual performers, the DH_{50} values are, in cm: LLNL 25.7 (one measurement); LANL 18.8 ± 1.4; IHD 15.3 ± 2.6; AFRL 9.1± 1.2. LLNL also performed the testing using 120-grit sandpaper with the average of the DH_{50} values (two measurements) 29.7 ± 2.3 cm.

Table 3. Impact testing results for $NaClO_3$/icing sugar mixture

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	8/02/10	23.2	32	28.1	6.8	0.104
LLNL (120)	8/03/10	23.9	31	31.3	1.6	0.022
LLNL (180)	8/09/10	23.9	31	25.7	5.2	0.088
LANL (180)	8/30/10	20.0	57.1	17.7	2.9	0.071
LANL (180)	8/31/10	20.4	50.4	18.4	2.6	0.062
LANL (180)	9/17/10	22.8	30.5	20.4	1.6	0.035
IHD (180)	3/29/11	24	43	17	6.0	0.15
IHD (180)	4/25/11	29	46	16	3.7	0.10
IHD (180)	4/26/11	28	54	13	2.1	0.07
AFRL (180)	3/21/12	22.2	47	9.7	2.1	0.10
AFRL (180)	3/23/12	22.8	46	9.8	3.6	0.17
AFRL (180)	3/23/12	22.2	50	7.7	1.9	0.11

1. Value in parenthesis is grit size of sandpaper (180 is 180 garnet dry and 120 is 120 Si/Carbide wet/dry); 2 relative humidity; 3. DH_{50}, in cm, is from a modified Bruceton method, height for 50% probability of reaction; 4. Standard deviation.

Table 4 shows the impact test results from LANL and AFRL using the Neyer or D-Optimal method[20]. The LANL average value for DH_{50} is 17.5 ± 3.2 cm, similar to the average value for DH_{50} determined by the Bruceton method. Likewise, the single value determined by AFRL is similar to the average DH_{50} value from determined by the Bruceton method in Table 3.

Table 4. Impact testing results for $NaClO_3$/icing sugar mixture (Neyer or D-Optimal Method) 180-grit sandpaper

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)	8/26/10	19.5	49.5	20.4	2.4	0.05
LANL (180)	8/31/10	20.2	51.2	14.0	3.2	0.10
LANL (180)	9/17/10	21.1	40.1	18.0	6.9	0.17
AFRL (180)	3/27/12	22.8	45	10.2	3.2	0.14

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, height for 50% probability of reaction; 4. Standard deviation.

3.3 Friction testing results for $NaClO_3$/icing sugar 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 conditions for positive detection. All participants performed data analysis using a modified Bruceton method[18,19] and the threshold initiation level method (TIL)[21]. Table 5 shows that data on the sensitivity of the mixture varies by participant. The average values and sensitivity ordering for F_{50}, in kg are: LANL, 5.5 ± 0.6 > LLNL, 8.8 ± 0.3 > IHD, 15.8 ± 2.6. The TIL values for LLNL and LANL follow the same trend.

Table 5. BAM Friction Testing results for $NaClO_3$/icing sugar mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4]	s, kg[5]	s, log unit[5]
LLNL	8/02/10	23.9	31	0/10 @ 6.4	1/10 @ 7.2	8.8	0.99	0.049
LLNL	8/03/10	23.3	32	0/10 @ 6.0	1/10 @ 6.4	9.0	1.79	0.086
LLNL	8/04/10	23.9	31	0/10 @ 6.0	1/10 @ 6.4	8.5	2.42	0.122
LANL	8/25/10	20.9	49.0	0/10 @ 2.4	1/2 @ 4.8	5.0	1.1	0.093
LANL	9/01/10	20.3	45.2	0/10 @ 2.4	1/1 @ 4.8	5.2	1.6	0.138
LANL	9/16/10	23.0	18.6	0/10 @ 2.4	1/2 @ 4.8	6.2	1.9	0.138
IHD	5/9/11	24	41	NA[6]	NA[6]	18.8	9.8	0.251
IHD	5/9/11	23	40	NA[6]	NA[6]	14.4	4.5	0.140
IHD	5/11/11	23	42	NA[6]	NA[6]	14.1	8.4	0.298
IHD	5/11/11	23	43	0/10 @ 6.1	1/3 @ 7.3	NA[7]	NA[7]	NA[7]
IHD	5/12/11	23	43	0/10 @ 4.2	1/5 @ 4.9	NA[7]	NA[7]	NA[7]
IHD	5/18/11	23	42	0/10 @ 2.9	1/7 @ 3.3	NA[7]	NA[7]	NA[7]

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the weight (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 weight level; 3. Next level where positive initiation is detected; 4. F_{50}, in kg, is by a modified Bruceton method, weight for 50% probability of reaction; 5. Standard deviation; 6. Not applicable, separate measurement performed for TIL; 7. Not applicable, separate measurements performed for modified Bruceton analysis.

Table 6 shows the ABL Friction testing performed by IHD and AFRL on the $NaClO_3$/icing sugar mixture. LANL did not have the system in routine performance at the time. LLNL does not have ABL Friction. IHD performed data analysis using a modified Bruceton method[18,19], and both IHD and AFRL performed data analysis using the threshold initiation level method (TIL)[21]. The data from IHD show some friction sensitivity. A TIL and one level above are established. In addition, IHD calculated F_{50} values from their data. The data from AFRL show friction sensitivity also, but at a much more sensitive level. A TIL could not be established, as well, the modified Bruceton analysis was not performed.

Table 6. ABL Friction testing results for $NaClO_3$/icing sugar mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[4]	F_{50}, psig/fps[5]	s, cm[6]	s, log unit[6]
IHD	6/6/11	24	41	0/20 @ 315/8	1/3 @ 420/8	NA[7]	NA[7]	NA[7]
IHD	7/7/11	24	42	0/20 @ 180/8	1/3 @ 235/8	NA[7]	NA[7]	NA[7]
IHD	7/7/11	23	42	0/20 @ 180/8	1/12 @ 235/8	NA[7]	NA[7]	NA[7]
IHD	7/7/11	23	44	NA[8]	NA[8]	464/8	118	0.11
IHD	7/7/11	23	44	NA[8]	NA[8]	492/8	80	0.07
IHD	7/7/11	23	44	NA[8]	NA[8]	476/8	111	0.10
AFRL	3/26/12	22.2	44	NA[9]	4/10 @ 5/8	NA[10]	NA[10]	NA[10]
AFRL	3/26/12	22.2	43	NA[9]	2/10 @ 5/8	NA[10]	NA[10]	NA[10]
AFRL	3/26/12	22.8	41	NA[9]	1/10 @ 5/8	NA[10]	NA[10]	NA[10]

1. Relative humidity; 2. psig/fps = pressure in psig at test velocity in feet per sec; 3. Threshold Initiation Level (TIL) is the force (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 force level; 4. Next level where positive initiation is detected; 5. F_{50}, in psig/fps, is by a modified Bruceton method, force for 50% probability of reaction; 6. Standard deviation; 7. Not applicable, separate measurements done for modified Bruceton analysis; 8. Not applicable, separate measurements performed for TIL analysis; 9. AFRL did not measure a TIL; 10. AFRL did not determine a modified Bruceton analysis.

3.4 Electrostatic discharge testing of $NaClO_3$/icing sugar mixture

Electrostatic Discharge (ESD) testing of the $NaClO_3$/icing sugar 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 that 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. All participants performed data analysis using the threshold initiation level method (TIL)[21].

Table 7. Electrostatic discharge testing $NaClO_3$/icing sugar mixture

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	8/02/10	23.9	31	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	8/03/10	23.3	32	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	8/04/10	23.9	31	0/10 @ 1.0	0/10 @ 1.0
LANL	8/26/10	20.8	48.8	0/13 @ 0.250	1/10 @ > 0.250
LANL	9/01/10	20.3	45.1	0/13 @ 0.250	1/10 @ > 0.250
LANL	9/16/10	23.0	18.4	0/20 @ 0.125	1/1 @ 0.250
IHD	3/30/11	24	44	0/20 @ 0.095	1/2 @ 0.165
IHD	4/13/11	21	48	0/20 @ 0.165	1/1 @ 0.326
IHD	4/14/11	22	40	0/20 @ 0.165	1/7 @ 0.326
AFRL	3/21/12	22.8	45	0/20 @ 0.15	1/7 @ 0.19
AFRL	3/22/12	22.8	48	0/20 @ 0.19	1/2 @ 0.25
AFRL	3/23/12	22.8	47	0/20 @ 0.19	1/1 @ 0.25

1. Relative humidity; 2. Threshold Initiation Level (TIL) is the energy (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 energy level; 3. Next level where positive initiation is detected; 4. LLNL used a custom built ESD with a 510-ohm resistor in the discharge unit to mimic the human body.

For TIL, LANL, IHD and AFRL results are about the same—zero events around 0.1 joules. The LLNL values using the custom built system show a material with no sensitivity.

3.5 Thermal testing (DSC) of $NaClO_3$/icing sugar mixture

Differential Scanning Calorimetry (DSC) was performed on the $NaClO_3$/icing sugar 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 taken in a pin-hole or a hermetically seal sample holder. For the LLNL data, there is only one exothermic event with a T_{max} around 166°C. There also appears little difference in the pin-hole sample holder data and the hermetically sealed sample holder data. For the LANL, IHD and AFRL data, three exothermic events are apparent with T_{max} values at ~ 160°C, ~ 216°C, and ~ 260°C. These three exothermic features vary in intensity from each participant reflected in the enthalpy values varying. For example, the middle temperature exothermic feature in the LANL data is so prominent and broad that it significantly overlaps with the other exothermic features such that an onset temperature could not be assigned automatically.

The assignment of these exothermic features is discussed below. AFRL also observed an endothermic feature with a T_{min} at ~ 258°C. This feature is in a region of overlap with the high temperature exothermic feature observed by LANL, IHD and AFRL.

Table 8. Differential Scanning Calorimetry results for $NaClO_3$/icing sugar mixture, 10°C/min heating rate

Lab	Test Date	Exothermic, on-set/maximum, °C (ΔH, J/g)	Exothermic, on-set/maximum, °C (ΔH, J/g)	Endothermic, on-set/minimum, °C (ΔH, J/g)	Exothermic, on-set/maximum, °C (ΔH, J/g)
LLNL[1]	7/26/10	156.5/164.6 (3706)	ND[4]	ND[4]	ND[4]
LLNL[1]	7/26/10	155.9/165.7 (4391)	ND[4]	ND[4]	ND[4]
LLNL[1]	7/26/10	155.5/165.8 (4503)	ND[4]	ND[4]	ND[4]
LLNL[2]	7/26/10	157.2/167.2 (3348)	ND[4]	ND[4]	ND[4]
LLNL[2]	6/29/10	157.0/167.1 (3345)	ND[4]	ND[4]	ND[4]
LLNL[2]	6/29/10	157.0/165.9 (3333)	ND[4]	ND[4]	ND[4]
LANL[1]	8/26/10	157.0/160.6 (2588)	214.1	ND[4]	257.6/260.0 (558)
LANL[1]	9/2/10	157.4/160.8 (2416)	207.5	ND[4]	256.4/258.6 (516)
LANL[1]	9/20/10	155.2/160.0 (2124)	214.1	ND[4]	256.0/262.1 (421)
IHD[1]	4/4/11	156.1/157.7 (2453)	200.2/213.8 (1630)	ND[4]	256.9/261.5 (496)
IHD[1]	4/4/11	156.1/158.8 (2003)	200.4/214.2 (1401)	ND[4]	257.2/259.1 (828)
IHD[1]	4/4/11	146.9/153.8 (660)	200.7/216.6 (2273)	ND[4]	260.0/262.8 (830)
AFRL[1, 3]	3/21/12	156.4/158.6 (716)	193.2/216.0 (667)	255.3/257.0 (12)	258.3/259 (117)
AFRL[1, 3]	3/22/12	157.5/158.8 (739)	193.2/216.6 (705)	253.9/260.6 (24)	258.7/260.6 (44)
AFRL[1, 3]	3/23/12	156.5/158.0 (619)	193.4/216.6 (540)	254.0/257.3 (84)	258.9/260.5 (49)

1. pin-hole sample holder; 2. Hermetically sealed sample holder; 3. AFRL also observed an endothermic event correlating to the melting of $NaClO_3$; 4. ND = not detected.

4 DISCUSSION

Table 9 shows the average values for the data from each participant and compares it to corresponding data for standards, RDX and PETN. The data for RDX comes from the IDCA first iterative study of RDX as part of this Proficiency Test[22], and the data for PETN comes from the examination of PETN Class 4 as part of this Proficiency Test[23]. Table 9 allows the comparison of the average results on $NaClO_3$/icing sugar mixture with standards to obtain relative sensitivities.

4.1 Sensitivity of $NaClO_3$/icing sugar mixture compared to standards

Impact sensitivity. LANL, IHD, and AFRL found the $NaClO_3$/icing sugar mixture to be more sensitive to impact than the RDX standard used in this IDCA study. LLNL found it to be less sensitive to impact than the RDX standard. All participants found the $NaClO_3$/icing sugar mixture to be less sensitive than PETN.

Friction sensitivity. For BAM friction, LLNL, LANL and IHD found the $NaClO_3$/icing sugar mixture to be more sensitive than the RDX standard and on the order of sensitivity of the PETN standard. For ABL friction, IHD found the mixture to be significantly less sensitive than both the RDX and PETN standards. AFRL, however, could not find a TIL level, indicating an extremely sensitive material.

Spark sensitivity. LANL and AFRL found the $NaClO_3$/icing sugar mixture to be less sensitive than both the RDX and PETN standards. IHD found the mixture to be less sensitive than the RDX standard but more sensitive than the PETN standard. LLNL found the material to be insensitive (LLNL ESD equipment is custom built).

Thermal sensitivity. All participants found the $NaClO_3$/icing sugar mixture to have a fairly low thermal on-set temperature (exothermic feature) that is close to the melting/decomposition point of the icing sugar. LANL, IHD, and AFRL also found evidence of higher temperature exothermic features.

Table 9. Average Comparison values

	LLNL	LANL	IHD	AFRL
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
$NaClO_3$/icing sugar[2-4]	29.7[5,6]	18.8[7,8]	15.3[7,8]	9.1[7,8]
$KClO_3$/icing sugar (AR)[4,9,10]	15.6[5]	10.4[7]	10.3[7]	8.3[7]
$KClO_3$/icing sugar (-100)[4,11,12]	14.8[5]	10.6[7]	14.3[7]	ND[13]
RDX Class 5 Type II[14]	24.3[5]	20.9[7,15]	19[7]	15.3[7]
PETN[16]	10.9[5]	8.0[7]	9.3[7]	6.8[7]
BAM Friction Testing[17,18]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
$NaClO_3$/icing sugar[3,4,19]	6.1[20]; 8.8[20]	2.4[20]; 5.5[20]	4.4[20]; 15.8[20]	ND[13]; ND[13]
$KClO_3$/icing sugar (AR) [4,9,10]	9.5; 11.8	2.4; 4.9	3.2; 3.6	ND[13]; ND[13]
$KClO_3$/icing sugar (-100)[4,11,12]	6.9; 9.9	4.8; 5.8	2.3; 4.4	ND[13]; ND[13]
RDX Class 5 Type II[14]	19.2; 25.1	19.2; 20.8	15.5; ND[13]	ND[13]; ND[13]
PETN[16]	6.4; 10.5	4.9, 8.5	4.3, 6.9	ND[13]; ND[13]
ABL Friction Testing[21-24]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
$NaClO_3$/icing sugar[3,4,25]	ND[13]; ND[13]	ND[13]; ND[13]	225[26]; 477[26]	<1[13]; ND[13]
$KClO_3$/icing sugar (AR) [4,9,10]	ND[13]; ND[13]	ND[13]; ND[13]	123; 150	43; ND[13]
$KClO_3$/icing sugar (-100)[4,11,12]	ND[13]; ND[13]	ND[13]; ND[13]	30; 42	ND[13]; ND[13]
RDX Class 5 Type II[14]	ND[13]; ND[13]	ND[13]; ND[13]	74; 154	93; ND[13]
PETN[16]	ND[13]; ND[13]	ND[13]; ND[13]	7.7, 42	ND[13]; ND[13]
Electrostatic Discharge[27]	TIL, Joules	TIL, Joules	TIL, Joules	TIL, Joules
$NaClO_3$/icing sugar[3,4,28]	0/10 @ 1.0[29,30]	0/20 @ 0.208[30]	0/20 @ 0.141[30]	0/20 @ 0.176[30]
$KClO_3$/icing sugar (AR) [4,9,10]	0/10 @ 1.0[29]	0/20 @ 0.125	0/20 @ 0.272	0/20 @ 0.091
$KClO_3$/icing sugar (-100) [4,11,12]	0/10 @ 1.0[29]	0/20 @ 0.0625	0/20 @ 0.272	ND[13]
RDX Class 5 Type II[14]	0/10 @ 1.0[29]	0/20 @ 0.0250	0/20 @ 0.095	0/20 @ 0.044
PETN[16]	0/10 @ 0.033[31]	0/20 @ 0.025	0/20 @ 0.219	0/20 @ 0.076

1. DH_{50}, in cm, is by a modified Bruceton method, height for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.2-23.9; 31-32), LANL (20.0-22.8; 30.5-57.1), IHD (24-29; 43-54), AFRL (22.2-22.8; 46-50); 3. $NaClO_3$ separated through 40-mesh sieve; 4. Icing sugar separated through 100-mesh sieve; 5. 120-grit sandpaper data only; 6. Average of two values from Table 3; 7. 180-grit sandpaper; 8. Average of three measurements from Table 3; 9. $KClO_3$ used as received from manufacturer, not separated through sieve; 10. From reference 16; 11. $KClO_3$ separated through a 100-mesh sieve; 12. From reference 17; 13. ND = Not determined; 14. From reference 22; 15. From reference 24; 16 From reference 23; 17. Threshold Initiation Level (TIL) is the weight (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 weight level; 18. F_{50}, in kg, is by a modified Bruceton method, weight for 50% probability of reaction; 19. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3-23.9; 31-32), LANL (20.3-23.0; 18.6-49.0), IHD (23-24; 40-42); 20. Average of three measurements from Table 5; 21. LLNL and LANL did not perform measurements; 22. Threshold Initiation Level (TIL) is the force (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 force level; 23. F_{50}, in psig/fps, is by a modified Bruceton method, force for 50% probability of reaction; 24. Measurements performed at 8 fps; 25. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (23-24; 41-44), AFRL (22.2-22.8; 41-44); 26. Average of three measurements from Table 6; 27. Threshold Initiation Level (TIL) is the energy (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 energy level; 28. 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); 29. LLNL has 510-Ω resistor in circuit; 30. Average of three measurements from Table 7. 31. ABL ESD apparatus.

4.2 Comparison of results based on participants

Table 9 allows absolute and relative comparison of testing data among participants. Note that for impact testing, the standards have been examined with the same grit size sandpaper as done by each participant in this study (LLNL all materials 120-grit Si/Carbide wet/dry sandpaper; LANL, IHD, and AFRL all materials 180-grit garnet dry sandpaper). This is different than what has been presented in comparison tables in previous IDCA Program Analysis reports. In future reports, all the standard measurements will be with 180-grit garnet dry sandpaper.

For impact testing, the sensitivity of the $NaClO_3$/icing sugar mixture depends upon the sandpaper used in the drop hammer apparatus. The LLNL DH_{50} values in Table 9 are determined with 120-grit sandpaper. The data indicates a material that is less sensitive than the standards examined using the same type of Si/Carbide sandpaper (120 grit sandpaper for standards and mixture). The DH_{50} values from LANL, IHD and AFRL indicate a mixture than is more sensitive than the RDX standard. In these cases, 180-grit garnet sandpaper was used for both the mixture and standards. The effect of the sandpaper on the DH_{50} results have been documented in previous IDCA studies of solid oxidizer and fuel mixtures[14,25,26], where DH_{50} measurements with 120-grit paper consistently show a more stable mixture to impact than measurements with the 180-grit garnet paper. For the $KClO_4$/Al and $KClO_4$/Dodecane mixtures, this difference based on sandpaper type is dramatic[27].

For BAM friction testing, LLNL determined the mixture to be less the sensitive than that determined by LANL and IHD. This has been seen before in several of the mixtures and has been attributed to the facility-required extra containment of the LLNL BAM system raising the threshold of a positive event. For ABL friction testing, IHD determined the mixture to be more stable than even RDX. AFRL on the other hand, found the mixture to be so sensitive that it could not establish a TIL.

For ESD testing, LANL, IHD and AFRL determined the mixture to have about the same sensitivity. Interestingly, the large variations in the absolute sensitivities for the PETN standard match the relative sensitivities for $NaClO_3$/icing sugar determined by these participants. This variation is not so pronounced for the RDX standard. LLNL determined the mixture to be non-sensitive. However, the experimental configuration of the LLNL system is different than the ABL ESD system. The results form LLNL are in keeping with previous IDCA determinations.

Figure 2 shows the DSC profiles of $NaClO_3$/icing sugar mixture measured by AFRL. There are four events possible and they are assigned as the following:

1. Exothermic at ~ 160°C (melting/decomposition of sugar[28-30] causes flow and then reaction with of $NaClO_3$)
2. Exothermic at ~ 220°C (carbonization of sugar)[29-31]
3. Endothermic slightly below 260°C (melting of $NaClO_3$)[32]
4. Exothermic slightly above 260°C (melted $NaClO_3$ reacting with residual carbon of the sugar).

Different participants derive different results—AFRL data show all four events; LANL and IHD data show three events (the melting of $NaClO_3$ appears masked by the large exothermic feature at ~ 260°C); LLNL data show only the low temperature exothermic feature (probably due to overdriving heat flow due to too much sample used in the DSC sample holder).

These features have been seen in other oxidizer/fuel mixtures[16,17,29,33,34] and specifically for $KClO_3$/icing sugar mixtures[16,17]. In the previous reports on $KClO_3$/icing sugar, up to three exothermic events were observed and assigned to parallel thermal chemistry. The differing effects were primarily attributed to sample size issues[27]. In addition, previous LLNL data[29], show three exothermic features at the same positions (but differing relative intensities) for $NaClO_3$ mixed with different sugar fuels as well as $NaClO_3$ melting at 261°C. $NaClO_3$ + sorbitol shows exothermic release at the melting point of $NaClO_3$ but no low temperature exothermic release because sorbitol melts/decomposes at 332°C, above the temperature that $NaClO_3$ melts.

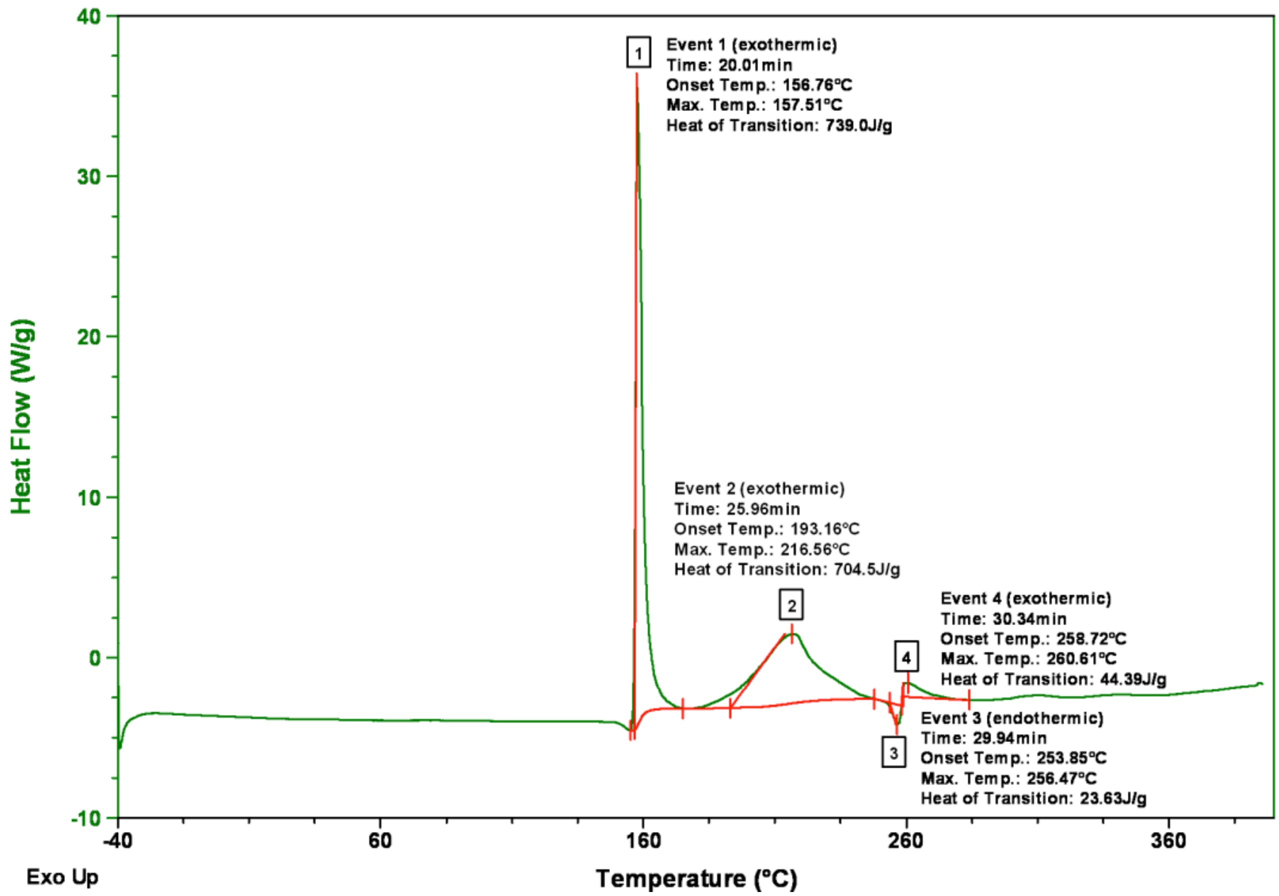

Figure 2. DSC of $NaClO_3$/icing sugar from AFRL at 10°C/min heating rate.

4.3 Comparison of $NaClO_3$/icing sugar and $KClO_3$/icing sugar mixtures

Table 9 shows the comparison of SSST testing results for $NaClO_3$/icing sugar mixture and compares the results to the testing results for $KClO_3$/icing sugar mixtures where the $KClO_3$ has been separated through a 40-mesh (AR) or a 100-mesh sieve (-100). This comparison gives the opportunity to compare two different oxidizers with the same fuel.

For impact testing by all the participants, the $NaClO_3$/icing sugar mixture was found to be less sensitive to impact than the two $KClO_3$/icing sugar mixtures. LLNL used 120-grit sandpaper for all mixtures and LANL, IHD, and AFRL used 180-grit sandpaper.

For the BAM friction testing, the comparisons are more complicated, and depend on the method of evaluation. Table 10 shows a relative comparison of the sensitivities. AFRL was not included because they did not test $KClO_3$ (-100)/icing sugar.

Table 10. Order of sensitivity by BAM Friction of oxidizer mixed with icing sugar[1,2]

	Sensitivity by TIL[3]	Sensitivity by F_{50}[4]
LLNL (BAM)[5]	$NaClO_3$ > $KClO_3$ (AR) > $KClO_3$ (-100)	$NaClO_3$ > $KClO_3$ (AR) > $KClO_3$ (-100)
LANL (BAM)[5]	$NaClO_3$ = $KClO_3$ (AR) > $KClO_3$ (-100)	$KClO_3$ (AR) > $NaClO_3$ > $KClO_3$ (-100)
IHD (BAM)[5]	$KClO_3$ (-100) > $KClO_3$ (AR) > $NaClO_3$	$KClO_3$ (AR) > $KClO_3$ (-100) > $NaClO_3$
IHD (ABL)[6]	$KClO_3$ (-100) > $KClO_3$ (AR) > $NaClO_3$	$KClO_3$ (-100) > $KClO_3$ (AR) > $NaClO_3$

1. Oxidizer only listed, all are mixtures with icing sugar as listed in Table 9; 2. AR = as received by manufacturer through a 40-mesh sieve, -100 = separated with a 100-mesh sieve; 3. Threshold Initiation Level (TIL) is the force (kg for BAM or psig/fps for ABL) 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 force level; 4. F_{50} (in kg for BAM or psig/fps for ABL), is by a modified Bruceton method, force for 50% probability of reaction; 5. By BAM Friction method based on average values from Table 9; 6. By ABL Friction method based on average values from Table 9.

For LLNL, results from both methods (TIL and F_{50}) show that the $NaClO_3$/icing sugar mixture is more sensitive than the $KClO_3$/icing sugar mixtures. For LANL, the results from the TIL values indicate that the $NaClO_3$/icing sugar mixture is the same sensitivity as the $KClO_3$ (AR)/icing sugar mixture, but is more sensitive than the $KClO_3$ (-100)/icing sugar mixture. The F_{50} values do not have the same relative order. For IHD, all but the F_{50} values determined by BAM exhibit the same relative order—BAM TIL, ABL TIL, and ABL F_{50} agree.

For the ESD testing, LANL and AFRL found the $NaClO_3$/icing sugar mixture to be less sensitive while IHD found the $NaClO_3$/icing sugar mixture to be more sensitive than the $KClO_3$/icing sugar mixtures. LLNL found all the mixtures insensitive.

The thermal testing exhibited essentially parallel properties among the three mixtures—three exothermic features depending upon icing sugar melting, sugar carbonization, and the oxidizer melting. Each participant more or less observed all three exothermic features.

5 CONCLUSIONS

$NaClO_3$/icing sugar mixture exhibited the following sensitivities in small-scale safety and thermal testing:

1. Impact testing
    a. Using 180-grit sandpaper—more sensitive than RDX but less sensitive than PETN
    b. Using 120-grit sandpaper—less sensitive than RDX and PETN
2. Friction testing
    a. Using BAM friction—more sensitive than RDX and about the same sensitivity as PETN
    b. Using ABL friction—less sensitive than RDX for one participant, but extra sensitive by the other (no TIL could be determined)
3. ESD testing
    a. Using ABL friction—less sensitive than RDX and PETN except for one participant found the mixture more sensitive than PETN
    b. Using a custom built ESD with a 510-Ω resistor—insensitive material
4. Thermal testing at 10°C/min heating rate

a. Sharp low temperature exothermic feature observed that corresponds to the sugar melting/decomposing and reacting with oxidizer (most likely scenario for large scale mixtures)
b. Broad mid temperature exothermic feature observed that correlates to carbonization of the sugar
c. A high temperature exothermic feature observed corresponds to the melting of the $NaClO_3$ which then can react with residual fuel
d. The thermal features parallel other solid oxidizer fuel mixtures when the nature of the oxidizer is accounted for.

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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	Naval Surface Warfare Center, Indian Head Division
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
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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