LLNL-TR-641024

LAWRENCE LIVERMORE NATIONAL LABORATORY

Integrated Data Collection Analysis (IDCA) Program - AN and Bullseye Smokeless Powder

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

July 17, 2013

Disclaimer

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 — AN and Bullseye® Smokeless Powder

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], Jason J. Phillips[3], Timothy J. Shelley[4], Jose A. Reyes[5], Peter C. Hsu[6], and John G. Reynolds[6*]

[1]Los Alamos National Laboratory, Los Alamos, NM USA
[2]Indian Head Division, Naval Surface Warfare Center, Indian Head, MD USA
[3]Sandia National Laboratories, Albuquerque, NM USA
[4]Bureau of Alcohol, Tobacco & Firearms, Redstone Arsenal, AL USA
[5]Applied Research Associates, Tyndall Air Force Base, FL USA
[6]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 ammonium nitrate (AN) mixed with Bullseye® smokeless powder (Gunpowder). The participants found the AN/Gunpowder to: 1) have a range of sensitivity to impact, comparable to or less than RDX, 2) be fairly insensitive to friction as measured by BAM and ABL, 3) have a range for ESD, from insensitive to more sensitive than PETN, and 4) have thermal sensitivity about the same as PETN and Gunpowder.

This effort, funded by the Department of Homeland Security (DHS), is putting 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 when developing safe handling practices for HMEs. Each participating testing laboratory uses identical test materials and preparation methods. Note, however, the test procedures differ among the laboratories. The testing performers involved are Lawrence Livermore National Laboratory (LLNL), Los Alamos National Laboratory (LANL), Indian Head Division, Naval Surface Warfare Center, (NSWC IHD), Sandia National Laboratories (SNL), 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, ammonium nitrate, Gunpowder, Bullseye® smokeless powder.

IDCA Explosives Safety Testing of Homemade Explosives

Integrated Data Collection Analysis Program

1 INTRODUCTION

The IDCA Proficiency Test was designed to assist the explosives community in comparing and perhaps standardizing inter-laboratory Small-Scale Safety and Thermal (SSST) testing for improvised explosive materials (homemade explosives or HMEs) and aligning these procedures with comparable testing for typical military explosives[1]. The materials for the Proficiency Test have been selected because their properties invoke challenging experimental issues when 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. Details of the results from the Proficiency Test for the materials examined are documented in IDCA Analysis Reports—RDX first testing[2], RDX second testing[3], RDX testing comparison[4], $KClO_3$/sugar (separated with a 100 mesh sieve)[5], $KClO_3$/sugar (as received)[6], $KClO_3$/Dodecane[7], $KClO_4$/Dodecane[8],

$KClO_4/Al$[9], $KClO_4$/Carbon[10], $NaClO_3$/sugar[11], PETN[12], Methods[13], Ammonium Nitrate[14], and Bullseye® smokeless powder[15].

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 evaluating 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 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 have been tested in triplicate and RDX was tested throughout the IDCA Proficiency Test.

The subject of this report, AN mixed with Gunpowder, is the tenth HME tested in the Proficiency Test and is one of a set of three related tests—AN, Gunpowder and AN/Gunpowder mixture. Gunpowder was selected because it is a solid component that when combined with AN, again demonstrates the challenges of SSST testing of two fine solids mixed together. The Gunpowder chosen is Bullseye® smokeless powder (not a product endorsement), a double-base powder containing nitroglycerin and nitrocellulose. 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, (NSWC IHD).

2 EXPERIMENTAL

General information. All samples were prepared according to IDCA methods on drying and mixing procedures[16,17]. The AN was dried but the Gunpowder was not before testing. The Bullseye® smokeless powder was from Alliant Powder Company. The composition (according to the manufacturer) is NG 40%, NC 58%, Ethyl Centralite (stabilizer) 1%, modifier and graphite 1%. The material was packaged in May of 2003 (the manufacturer suggested the stabilizer level be checked once every 5 years). The

AN was Fisher Brand, Catalog Number A676, Lot #086459. The average particle properties were measured by laser diffraction light scattering method using Microtracs Model FRA9200. The AN and Bullseye® smokeless powder were mixed at a 1 to 1 ratio by weight to form the mixture for this study.

Testing conditions. Table 2 summarizes the SSST testing conditions used by the laboratories that participated in the analyses of the Gunpowder. SSST testing data for the individual participants was obtained from the following IDCA Data Reports: Small Scale Safety Test Report for Ammonium Nitrate/Bullseye® mixture (LLNL)[18], 50188 O AN/Gunpowder (LANL)[19], and AN/Gunpowder (IHD)[20].

Table 2. Summary of conditions for the analysis of RDX (All = LANL, LLNL, IHD)

Impact Testing
1. Sample size—LLNL, IHD, 35 ± 2 mg; LANL, 35 or 40 ± 2 mg
2. Preparation of samples—All, as received/dried by IDCA procedures[16]
3. Sample form—All, loose powder
4. Powder sample configuration—All, conical pile
5. Apparatus—LANL, LLNL, IHD, Type 12*
6. Sandpaper—All, 180-grit garnet dry; LLNL, 120-grit Si/C wet
7. Sandpaper size—LLNL, IHD, 1 inch square; LANL, 1.25 inch diameter disk dimpled;
8. Drop hammer weight—All, 2.5 kg
9. Striker weight—LLNL, IHD, 2.5 kg; LANL, 0.8 kg
10. Positive detection—LANL, LLNL, microphones with electronic interpretation as well as observation; IHD, observation
11. Data analysis—All, modified Bruceton; LANL Neyer also

Friction analysis
1. Sample size—All, ~5 mg, but not weighed
2. Preparation of samples—All, as received/dried by IDCA procedures[16]
3. Sample form—All, powder
4. Sample configuration—All, small circle form
5. Apparatus—LANL, LLNL, IHD, BAM; IHD, ABL*
6. Positive detection—All, by observation
7. Room Lights—LANL on; and LLNL off; IHD, BAM on, ABL off
8. Data analysis—LLNL modified Bruceton (log-scale spacing) and TIL; LANL and IHD, modified Bruceton (linear spacing) and TIL

ESD
1. Sample size—All ~5 mg, but not weighed
2. Preparation of samples—All, as received/dried by IDCA procedures[16]
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, IHD, ABL; LLNL, custom built*
7. Positive detection—All, observation
8. Data analysis methods—All, TIL

Differential Scanning Calorimetry
1. Sample size—All ~ <1 mg
2. Preparation of samples—All, as received/dried by IDCA procedures[16]
3. Sample holder—All, pinhole; LLNL, TA sealed
4. Scan rate—All, 10°C/min
5. Range—All, 40 to 400°C+
6. Sample holder hole size—LANL, IHD, 75 μm; LLNL, 50 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, SNL— MBOM modified for ERL Type 12 Drop Weight; *Friction*: LANL, LLNL, IHD, SNL—BAM Friction Apparatus, LANL, IHD, AFRL—ABL Friction Apparatus; *Spark:* LLNL, LANL, IHD, AFRL, SNL—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.

3 RESULTS

3.1 AN/Gunpowder

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 Analysis Report on method comparisons[13], which compares procedures by each testing category. LANL, LLNL and IHD participated in this testing.

3.2 Particle Size Distribution of AN/Gunpowder

Figure 1 shows the particle size distribution of the Gunpowder and the AN performed by laser light scattering[21]. For the Gunpowder, the distribution extends from 500 to 1000 μm (10% 530 μm, 95% 970 μm). The average particle size is 753 ± 153 μm. For the AN (dried), the distribution extends from 200 μm to over 1500 μm (10% 364 μm, 95% 1573 μm). The average particle size is 724 ± 401 μm. The figure clearly indicates that the size distributions of the two materials are different but overlap. The AN has a lot more material in the small and large size ranges.

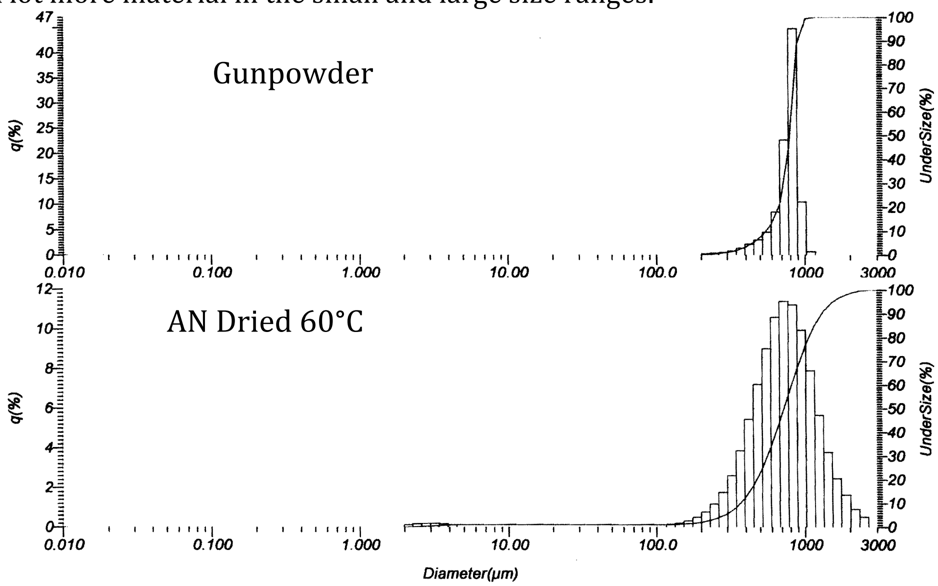

Figure 1. Microtracs laser light scattering particle size distribution for Ammonium Nitrate and Bullseye® Smokeless powder used in this study

3.3 Impact testing results for AN/Gunpowder

Table 3. Impact testing results for AN/Gunpowder

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LLNL (120)	2/04/11	23.3	15	98.6	5.00	0.022
LLNL (120)	2/14/11	23.9	18	73.0	2.35	0.014
LLNL (180)	2/04/11	23.3	15	48.5	2.01	0.018
LLNL (180)	2/07/11	23.3	13	47.1	2.71	0.025
LLNL (180)	4/19/11	23.9	31	44.9	2.28	0.022
LANL (180)	4/04/11	21.0	< 10	28.5	6.82	0.103
LANL (180)	4/06/11	22.0	< 10	29.2	3.91	0.058
LANL (180)	4/06/11	22.6	< 10	29.4	4.82	0.071
IHD (180)	5/20/11	22	46	20	6.1	0.13
IHD (180)	5/20/11	21	44	24	13.3	0.23
IHD (180)	5/23/11	23	46	20	8.5	0.18

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

Table 3 shows the results of impact testing of AN/Gunpowder performed by LANL, LLNL and IHD. Differences in the testing procedures are shown in Table 2, and the notable differences are the amount of sample, and the methods for detection of a positive test. All participants performed data analysis by a modified Bruceton method[22,23]. All participants found AN/Gunpowder to be fairly insensitive to impact testing, but the values span a large range. LLNL used two types of sandpaper—120-grit Si/C wet (LLNL standard) and 180-grit garnet dry (the IDCA standard). The overall average for DH_{50} using 180-grit sandpaper is 32.4 ± 11.4 cm. The large standard deviation comes from the spread in the values based on the participant. Average values for DH_{50}, in cm, are 46.8 ± 1.8, 29.0 ± 0.5, 21.3 ± 2.3 for LLNL, LANL, IHD, respectively. Average value for DH_{50} using 120-grit sandpaper is 85.8 ± 18.1 cm.

Table 4. Impact testing results for AN/Gunpowder (Neyer or D-Optimal Method)

Lab[1]	Test Date	T, °C	RH, %[2]	DH_{50}, cm[3]	s, cm[4]	s, log unit[4]
LANL (180)	4/04/11	21.2	< 10	27.2	0.070	4.4
LANL (180)	4/04/11	22.1	< 10	25.5	0.104	6.0
LANL (180)	4/04/11	22.7	< 10	31.9	0.008	0.6

1. Value in parenthesis is grit size of sandpaper (180 is 180-grit garnet dry); 2. Relative humidity; 3. DH_{50}, in cm, is by the Neyer D-Optimal method, height for 50% probability of reaction; 4. Standard deviation.

Table 4 shows the impact test results from LANL using the Neyer or D-Optimal method[23]. The average value for DH_{50} is 28.2 ± 3.3 cm, similar to the average value by LANL for DH_{50} determined by the Bruceton method.

3.4 Friction testing results for AN/Gunpowder

Table 5. BAM Friction Testing results for AN/Gunpowder

Lab	Test Date	T, °C	RH, %[1]	TIL, kg[2]	TIL, kg[3]	F_{50}, kg[4,5]	s, kg[6]	s, log unit[6]
LLNL	2/04/11	23.3	15	NA[7]	1/10 @ 7.2	> 36	NA[8]	NA[8]
LLNL	2/11/11	22.8	13	0/10 @ 28.8	1/10 @ 32.4	32.4	2.02	0.027
LLNL	2/11/11	23.9	13	0/10 @ 25.2	1/10 @ 28.8	32.9	1.97	0.026
LANL	4/04/11	21.5	< 10	NA[9]	NA[9]	17.4	1.17	0.029
LANL	4/05/11	22.9	< 10	NA[9]	NA[9]	19.7	0.79	0.017
LANL	4/06/11	22.6	< 10	NA[9]	NA[9]	19.8	0.71	0.016
LANL	4/04/11	21.2	< 10	0/10 @ 12.2	1/6 @ 14.7	NA[10]	NA[10]	NA[10]
LANL	4/06/11	22.0	< 10	0/10 @ 14.7	1/7 @ 17.0	NA[10]	NA[10]	NA[10]
LANL	4/06/11	22.2	< 10	0/10 @ 12.2	1/9 @ 14.7	NA[10]	NA[10]	NA[10]
IHD	2/10/12	24	44	NA[9]	NA[9]	13.0	2.4	0.081
IHD	2/10/12	26	42	NA[9]	NA[9]	12.0	3.0	0.011
IHD	2/10/12	26	43	NA[9]	NA[9]	13.2	2.7	0.090
IHD	12/22/11	28	43	0/10 @ 12.2	1/1 @ 14.7	NA[10]	NA[10]	NA[10]
IHD	12/22/11	28	43	0/10 @ 12.2	1/1 @ 14.7	NA[10]	NA[10]	NA[10]
IHD	12/22/11	28	43	0/10 @ 12.2	1/3 @ 14.7	NA[10]	NA[10]	NA[10]

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. LLNL uses log spacing and LANL and IHD use liner spacing for the Bruceton up and down method experimentation and data analysis 6. Standard deviation; 7. Not applicable, TIL was not established; 8. Not applicable, outside of the range of the BAM friction apparatus; 9. Not applicable, separate measurements performed for TIL analysis; 10. Not applicable, separate measurements performed for modified Bruceton analysis.

Table 5 shows the BAM Friction testing of AN/Gunpowder performed by LLNL, LANL, 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)[25] and a modified Bruceton method[22,23]. Table 5 shows that data on the sensitivity of the mixture varies depending upon on the participant. The average values for F_{50}, in kg, are: LLNL 32.7 ± 0.4, LANL 19.0 ± 1.4 and IHD 12.7 ± 0.6. The averages TIL values follow the same trend. The order and average TIL values, in kg, are: LLNL 27.0 > LANL 13.0 > IHD 12.2.

Table 6 shows the ABL Friction testing of AN/Gunpowder performed by IHD. LANL did not have the system in routine performance at the time. LLNL does not have ABL Friction testing equipment. IHD performed data analysis using the TIL method[25] and a modified Bruceton analysis[22,23]. The F_{50} data show that the AN/Gunpowder exhibits friction sensitivity, with an average of 159 ± 5 psig/8 fps. The TIL values also are consistent with the average F_{50} value, with an average of 77 psig/8 fps.

Table 6. ABL Friction testing results for AN/Gunpowder

Lab	Test Date	T, °C	RH, %[1]	TIL, psig/fps[2,3]	TIL, psig/fps[4]	F_{50}, psig/fps[5]	s, psig[6]	s, log unit[6]
IHD	9/23/11	24	42	0/20 @ 55/8	1/3 @ 75/8	NA[7]	NA[7]	NA[7]
IHD	9/23/11	24	42	0/20 @ 75/8	1/4 @ 100/8	NA[7]	NA[7]	NA[7]
IHD	12/7/11	29	40	0/20 @ 100/8	1/5 @ 135/8	NA[7]	NA[7]	NA[7]
IHD	9/23/11	27	42	NA[8]	NA[8]	158	37	0.10
IHD	12/7/11	29	40	NA[8]	NA[8]	165	46	0.12
IHD	12/22/11	28	43	NA[8]	NA[8]	155	47	0.13

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; 7. Not applicable, separate measurements performed for Bruceton analysis; 8. Not applicable, separate measurements performed for TIL analysis.

3.5 Electrostatic discharge testing of AN/Gunpowder

Electrostatic Discharge (ESD) testing of AN/Gunpowder was performed by LLNL, LANL and IHD. Table 7 shows the results. Differences in the testing procedures are shown in Table 2, and the notable differences are the use of tape covering the sample. In addition, LLNL uses a custom built ESD system with a

Table 7. Electrostatic discharge testing results for AN/Gunpowder

Lab	Test Date	T, °C	RH, %[1]	TIL, Joule[2]	TIL, Joule[3]
LLNL[4]	2/04/11	23.3	20	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	2/04/11	22.9	16	0/10 @ 1.0	0/10 @ 1.0
LLNL[4]	2/07/11	22.9	16	0/10 @ 1.0	0/10 @ 1.0
LANL[5]	4/06/11	22.0	< 10	0/20 @ 0.0625	1/2 @ 0.125
LANL[5]	4/05/11	23.0	< 10	0/20 @ 0.0625	1/1 @ 0.125
LANL[5]	4/06/11	22.6	< 10	0/20 @ 0.0625	1/1 @ 0.125
IHD[5]	5/24/11	24	44	0/20 @ 0.165	1/3 @ 0.326
IHD[5]	5/23/11	24	44	0/20 @ 0.165	1/13 @ 0.326
IHD[5]	5/25/11	24	43	0/20 @ 0.165	1/6 @ 0.326

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. ABL ESD equipment.

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)[25].

For TIL, IHD found the material to be the least sensitive, while LANL found it to be the most sensitive. The LLNL values using the custom built system show a material with no sensitivity.

3.6 Thermal testing (DSC) results for AN/Gunpowder

Differential Scanning Calorimetry (DSC) was performed on AN/Gunpowder by LLNL, LANL and IHD. All participating laboratories used different versions of the DSC by TA Instruments. Table 8 shows the data obtained at a 10°C/min heating rate.

Table 8. Differential Scanning Calorimetry results for AN/Gunpowder, 10°C/min heating rate[1,2]

Lab	Test Date	Transition T^1, onset/T_{min}, °C (ΔH, J/g)	Transition T^2, onset/T_{min}, °C (ΔH, J/g)	Transition T^3, onset/T_{min}, °C (ΔH, J/g)	Transition T^4, onset/T_{min}, °C (ΔH, J/g)	Transition T^5, T range, °C (ΔH, J/g)
LLNL[3]	1/26/11	51.7/53.0 (-14)	93.8/94.3 (-6)	126.2/127.2 (-42)	168.9/169.6 (-55)	176-226 (+466)/226-276 (-749)
LLNL[3]	1/27/11	51.7/52.8 (-10)	92.1/93.7 (-3)	126.1/127.0 (-26)	169.0/169.5 (-31)	174-223 (+642)/188-224 (-103)
LLNL[3]	1/27/11	51.8/52.9 (-11)	~ 94[4]	126.1/127.0 (-29)	168.3/169.3 (-33)	175-240 (+1015)/240-260 (-76)
LLNL[5]	1/26/11	51.6/52.9 (-13)	90.7/92.4 (-4)	126.2/127.3 (-36)	168.8/169.6 (-36)	175-230 (+723)/230-270 (-350)
LLNL[5]	1/27/11	51.6/52.9 (-16)	92.7/93.3 (-5)	126.1/127.2 (-45)	169.0/169.6 (-61)	175-220 (+519)/220-280 (-854)
LLNL[5]	1/27/11	51.6/53.1 (-13)	93.9	126.1/127.3 (-36)	169.0/169.6 (-43)	175-240 (+592)/240-258 (-224)
LANL[5]	3/21/11	52.7/53.6 (-6)	92.0/92.5 (-6)	126.8/128.3 (-18)	164.0/167.1 (-15)	160-220 (+1892)[6]
LANL[5]	4/5/11	53.2/55.0 (-14)	91.1/92.5 (-10)	127.1/130.3 (-32)	166.5/169.2 (-31)	170-300 (+1771)
LANL[5]	4/12/11	53.2/53.9 (-9)	92.0/92.3 (-4)	127.0/128.7 (-19)	164.9/167.1 (-17)	165-220 (+1881)
IHD[5]	6/24/12	53.5/54.3 (-7)	~90[4]	127.0/127.7 (-12)	164.1/166.0 (-9)	180-240 (+1906)
IHD[5]	6/24/12	53.5/54.1 (-13)		127.0/128.1 (-25)	165.7/167.6 (-24)	167-215 (+2028)
IHD[5]	6/24/12	53.1/54.5 (-15)	~90[4]	126.9/127.8 (-30)	166.4/167.6 (-32)	167-228 (+2204)

1. Exothermic = ΔH positive, endothermic = ΔH negative; 2. Minimum temperature of transition, T_{min}; 3. Hermetically sealed sample holder; 4. Temperature estimated on hard copy; 5. pinhole sample holder; 6. Unexplained extremely sharp transition at 190°C.

Table 8 shows five transitions that were observed by all the participants. Transitions T^1 through T^4 are primarily endothermic features from the AN. Transition T^2 is a very weak transition and is observed is the sample is rigorously dried at 60°C. The values with the approximate sign reflect that the transition is observed in the DSC profile, but is too weak to assign T_{min} and ΔH. Transition T^5 is in the region of overlap of a high temperature transition from AN and Gunpowder. Overlapping endothermic and exothermic behavior further complicates T^5. Temperature ranges are indicated because of the multiple features that overlap. The differences in the values in Table 8 compared to those of the pure materials—AN and Gunpowder—are discussed below, as well as more details about the Transition T^5.

4 DISCUSSION

Table 9 shows the average values for the data for AN/Gunpowder from each participant and compares it to corresponding data for standards, RDX Type II Class 5 and PETN Class 4 done previously and Gunpowder[14] and AN done previously[15]. The data for RDX comes from the evaluation of all of the RDX examinations as part of this Proficiency Test[4], and the data for PETN comes from the examination of PETN Class 4 as part of this Proficiency Test[12].

Table 9. Average Comparison values

	LLNL	LANL	IHD
Impact Testing[1]	DH_{50}, cm	DH_{50}, cm	DH_{50}, cm
AN/Gunpowder[2-4]	46.8	29.0	21.3
Gunpowder[5]	54.2	20.7	12.3
AN[6]	82	> 320	201
RDX Type II Class 5[3,7]	22.6	20.9	19.7
PETN[3,8]	8.3	8.0	9.3
BAM Friction Testing[9,10]	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg	TIL, kg; F_{50}, kg
AN/Gunpowder[11,12]	27.0[13]; 32.7	13.0; 19.0	12.2; 12.7
Gunpowder[5]	16.4; 20.7	5.6; 9.3	13.8; NA[14]
AN[6]	> 36; > 36	> 36.7; > 36.7	> 36.7; > 36.7
RDX Type II Class 5[7]	19.2; 25.1	19.2; 20.8	15.5; ND[15]
PETN[8]	6.4; 10.5	4.9; 8.5	4.3; 6.9
ABL Friction Testing[16-19]	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig	TIL, psig; F_{50}, psig
AN/Gunpowder[20,21]	ND[15]; ND[15]	ND[15]; ND[15]	77; 159
Gunpowder[5]	ND[15]; ND[15]	ND[15]; ND[15]	ND[15]; 317
AN[6]	ND[15]; ND[15]	ND[15]; ND[15]	385; 388
RDX Type II Class 5[7]	ND[15]; ND[15]	ND[15]; ND[15]	74; 154
PETN[8]	ND[15]; ND[15]	ND[15]; ND[15]	7.7; 42
Electrostatic Discharge[22]	TIL, Joules	TIL, Joules	TIL, Joules
AN/Gunpowder[23,24]	0/10 @ 1.0[25]	0/20 @ 0.0625[26]	0/20 @ 0.165[26]
Gunpowder[5]	0/10 @ 1.0[25]	0/20 @ 0.0250[26]	0/20 @ 0.1625[26]
AN[6]	0/10 @ 1.0[25]	0/20 @ 0.125[26]	0/20 @ 0.326[26]
RDX Type II Class 5[7]	0/10 @ 1.0[25]	0/20 @ 0.0250[26]	0/20 @ 0.095[26]
PETN[8]	0/10 @ 0.033[26]	0/20 @ 0.025[26]	0/20 @ 0.219[26]

1. DH_{50}, in cm, is by a modified Bruceton method, load for 50% probability of reaction; 2. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (23.3-23.9; 15-31), LANL (21.0-22.6; <10), IHD (21-23; 44-46); 3. 180-grit sandpaper; 4. Average of three measurements from Table 3; 5. Gunpowder reference 15; 6. AN reference; 14; 7. From reference 4; 8. From reference 12; 9. 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; 10. F_{50}, in kg, is by a modified Bruceton method, load for 50% probability of reaction; 11. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.8-23.9; 13-15), LANL (21.2-22.9; <10), IHD (24-28; 42-44); 12. Average of three measurements from Table 5; 13. Average of 2 values from Table 5; 14. Outside the range of the Bruceton analysis; 15. ND = Not determined; 16. LLNL and LANL did not perform measurements; 17. Threshold Initiation Level (TIL) is the load (psig) at test velocity (fps) at which zero reaction out of twenty or fewer trials with at least one reaction out of twenty or fewer trials at the next higher load level; 18. F_{50}, in psig/fps, is by a modified Bruceton method, load for 50% probability of reaction; 19. Measurements performed at 8 fps; 20. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—IHD (24-29; 40-43); 21. Average of three measurements from Table 6; 22. 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; 23. Temperature and humidity values varied during the sets of measurements (T_{range}, °C; RH_{range}, %)—LLNL (22.9-23.3; 16-20), LANL (22.0-23.0; <10), IHD (24; 43-44); 24. Average of three measurements from Table 7; 25. LLNL has 510-Ω resistor in circuit; 26. ABL ESD apparatus.

4.1 Comparison of participating laboratory testing results of AN/Gunpowder mixture

Impact sensitivity. All the drop hammer data in Table 9 for the AN/Gunpowder mixture was taken using 180-grit garnet sandpaper. All three participants show the AN/Gunpowder mixture has distinctively different sensitivities. LLNL found the lowest sensitivity and IHD found the highest. LANL analysis by the Neyer method yielded about the same sensitivity as the LANL values using Bruceton analysis.

Friction sensitivity. For BAM Friction, all three participants found a different sensitivity of the AN/Gunpowder mixture. For TIL, the order is IHD > LANL > LLNL. For F_{50}, the order is the same. IHD was the only participant to test friction sensitivity by ABL Friction and found moderate sensitivity for the AN/Gunpowder mixture. LLNL recorded the least sensitivity of the participants. This has been seen before in the friction testing of other materials by LLNL, and has been attributed to safety shielding of the LLNL equipment[26].

ESD. All three participants found different levels of sensitivity for AN/Gunpowder mixture. The differences were quite large. The order is LANL > IHD > LLNL. The results by LLNL indicating the AN/Gunpowder mixture is completely insensitive can be explained by LLNL using a custom built system that has a 510-Ω resistor in the circuit. This system is completely different than the ABL ESD systems of LANL and IHD.

Thermal sensitivity. All participants found the thermal behavior of the AN/Gunpowder mixture to be approximately the same. Roughly five transitions are observed. The first four features, Transitions T^1 to T^4, are endothermic and have about the same T_{min} and ΔH_{endo} for corresponding values as measured by each participant. The biggest difference among the participants is the thermal behavior measured in Transition T^5. Table 8 shows a broad range of values with complicated overlapping features. The range is roughly the same—160 to 250°C, but the T_{max} and ΔH values are much more convoluted. These differences will be discussed below with respect to comparison with the behavior of the pure components.

4.2 Comparison of average values for AN/Gunpowder mixture with standards

Table 9 shows the comparison of the impact, friction and ESD sensitivity of the AN/Gunpowder mixture with the standards RDX Type II Class 5 and PETN Class 4.

Impact sensitivity. Even though there is a wide range of sensitivity values reported, the participants found the AN/Gunpowder mixture to be less sensitive than the RDX standard and PETN standards. IHD found the AN/Gunpowder mixture to be very close in sensitivity to, but still less sensitive than RDX.

Friction sensitivity. LANL and IHD found the AN/Gunpowder mixture to be more sensitive than the RDX standard, but less sensitive than PETN standard. LLNL found the AN/Gunpowder mixture to be less sensitive than both standards.

Spark sensitivity. LANL found the AN/Gunpowder mixture to be less sensitive than either standard. IHD found the AN/Gunpowder mixture to be less sensitive than the RDX standard but more sensitive than the PETN standard. LLNL measured no sensitivity on the custom built system with a 510-Ω resistor in the circuit.

Thermal sensitivity. The lower temperature transitions T^1 through T^4 are all endothermic and do not indicate thermal sensitivity for the AN/Gunpowder mixture. However, Transition T^5, exhibits exothermic behavior, in some cases, in the regions around 200°C, which is close to the sensitivity of the PETN standard. The T_{max} and ΔH_{exo} for RDX[4] and PETN[12], respectively are: ~ 240°C, ~ 2200 J/g; ~ 205 °C, ~ 1100 J/g. However, the magnitude of the enthalpy is much lower than the standards. Caution needs to be taken in the interpretation of these results because this low value for AN/Gunpowder is likely an artifact of the experimental method. This is discussed in detail below.

4.3 Comparison of average values for AN/Gunpowder mixture with components

For this study, pure AN and pure Bullseye® Smokeless Powder were mixed at a 1 to 1 ratio by weight to give the AN/Gunpowder mixture. Both of the components of the mixture have been studied by the IDCA previously[14,15]. This provides the opportunity to assess the effects of mixing the two energetic materials has on the SSST testing properties.

Impact sensitivity. All the participants found the AN component insensitive in impact testing. LLNL found Gunpowder to be essentially insensitive also, but LANL and IHD found the Gunpowder to be as or more sensitive than RDX, respectively. Interestingly, all participants found the AN/Gunpowder mixture to have similar sensitivity as the Gunpowder. For LANL and IHD, this sensitivity was decreased from the corresponding sensitivity of the pure Gunpowder, but for LLNL, the mixing produced a more sensitive material.

Friction sensitivity. All participants found the AN to be completely insensitive in BAM friction testing. On the other hand, all participants found Gunpowder more sensitive than RDX. LANL found the AN/Gunpowder mixture to be less sensitive than pure Gunpowder, but still more sensitive than RDX. IHD found the mixture to be slightly more sensitive than Gunpowder, and more sensitive than what LANL found. LLNL found the AN/Gunpowder mixture to be less sensitive than the pure Gunpowder and less sensitive than RDX. This has been seen repeatedly in the LLNL results when compared to the other participants due to the amount of safety shielding around the BAM friction equipment[26].

IHD is the only participant to study all three materials by ABL Friction. IHD found the AN to be the least sensitive, the AN/Gunpowder mixture to be the most sensitive, and the Gunpowder in between these two. This parallels the IHD finding by BAM friction except in the ABL study, all materials were found less sensitive than RDX.

ESD sensitivity. LANL and IHD found the AN to be the least sensitive, the Gunpowder to be the most sensitive and the AN/Gunpowder mixture to be somewhere in between these two values. LLNL found all the materials insensitive, like due to the design of the custom built system.

Thermal sensitivity. All participants found the DSC essentially a composite of the two component materials. The first four transitions shown in Table 8 are due to the AN. Table 10 compares the averages, deviations and ranges for the DSC data taken for AN and the AN/Gunpowder mixture. The AN values come from the previous IDCA report on AN[14]. The nature of Transitions T^1-T^4 are discuss in that report also. A breakdown of these averages for each participant and condition are listed in the Appendix.

Table 10. Summary DSC Transition T^1-T^4 Data for AN and AN/Gunpowder

Parameters	Onset T, Range (°C)	T_{min}, Range (°C)	Enthalpy, Range (J/g)
All T^1 AN	52.3 ± 1.9, 46.4 to 55.8	53.6 ± 2.2, 46.6 to 57.5	-19 ± 5, -3 to -25
All T^1 AN/Gunpowder	52.4 ± 0.8, 51.6 to 53.5	53.6 ± 0.8, 52.8 to 55.0	-12 ± 3, -16 to -6
All T^2 AN	90.7 ± 1.6, 85.5 to 92.6	92.7 ± 2.3, 86.6 to 96.2	-14 ± 13, -57 to -3
All T^2 AN/Gunpowder	92.3 ± 1.1, 90.7 to 93.9	92.5 ± 1.5, 90.0 to 94.3	-5 ± 2, -10 to -3
All T^3 AN	126.5 ± 0.5, 125.9 to 128.1	128.3 ± 1.0, 127.0 to 131.1	-53 ± 6, -60 to -38
All T^3 AN/Gunpowder	126.6 ± 0.4, 126.1 to 127.1	127.8 ± 0.9, 127.0 to 130.0	-29 ± 10, -45 to -12
All T^4 AN	168.5 ± 1.1, 164.8 to 169.4	169.5 ± 0.6, 167.9 to 170.7	-71 ± 9, -82 to -32
All T^4 AN/Gunpowder	167.1 ± 2.0, 164.0 to 169.0	168.4 ± 1.3, 166.0 to 169.6	-32 ± 15, -61 to -9

For T_{min}, the maximum difference of the corresponding average values is 1.1 °C. The average enthalpies show bigger differences between the AN and the corresponding AN/Gunpowder mixture. In almost every case for the individual laboratories, the enthalpies measured for the AN were of higher magnitude than the corresponding enthalpies for the AN/Gunpowder mixture. This is to be expected because the enthalpies are measured on a per gram basis, and the AN/Gunpowder mixture is 1/1 where the AN is 100 % pure.

For Transition T^5, the DSC behavior is much more complicated. Figure 2 shows the all DSC data for the T^5 region for each of the participants. LANL and IHD each performed 3 separate DSC measurements on the AN/Gunpowder. LLNL performed 6—3 with the pinhole sample holder and 3 with the TA sealed sample holder. This region is complicated because this is in the range for: 1) the Gunpowder to show an exothermic transition (average T_{max} = 200.6 ± 1.6 °C; ΔH_{exo} = 2044 ± 129 J/g)[15], 2) the AN (average (vented sample holders only) T_{min} = 297.8 ± 18.5 °C; ΔH_{endo} = -894 ± 575 J/g)[14] to show an endothermic transition if the measurements are made in a vented sample holder, and 3) for a synergistic transition to appear between the Gunpowder and the AN.

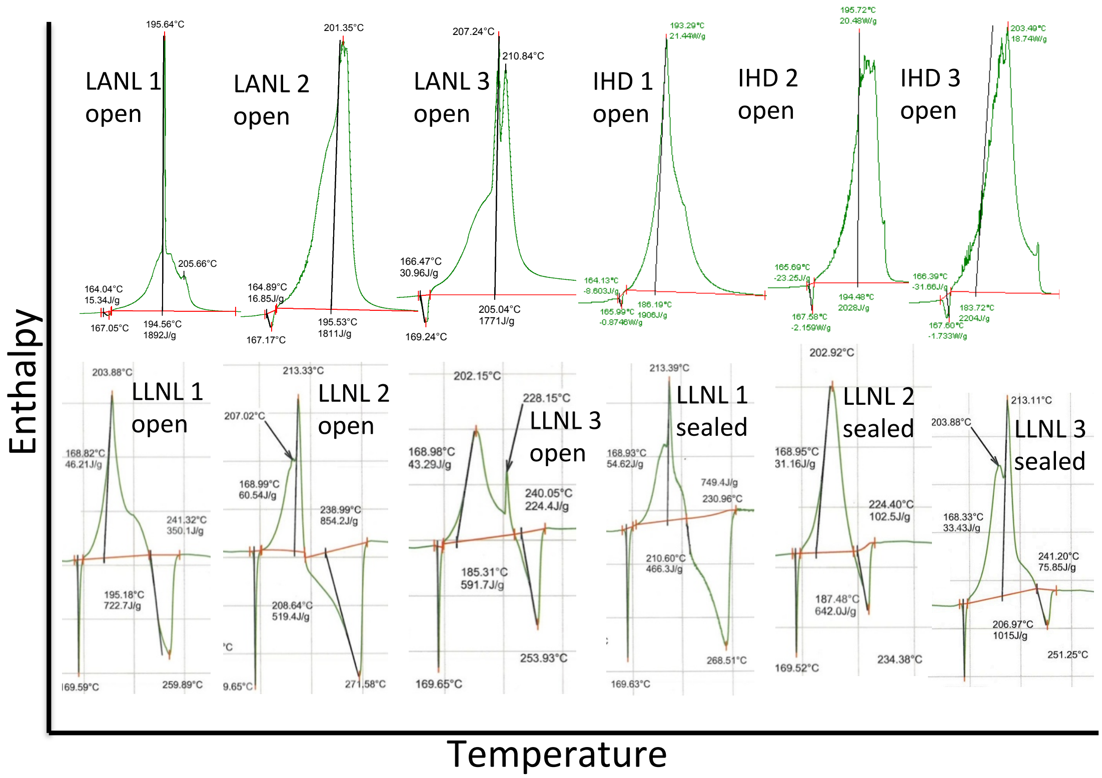

Figure 2. DSC of AN/Gunpowder Transition T^5 Region from LANL, IHD and LLNL; data labeled open is from pinhole sample holders, and data labeled sealed is from sealed TA sample holders

LANL data shows a large variation because the LANL 1 DSC data has a narrow unidentified feature at 195 °C that is not seen in any of the other profiles. This feature overlaps the T_{max} of a very broad exothermic feature. The T_{max} value is near that of the T_{max} for gunpowder. The other LANL profiles are also complicated, exhibiting a shoulder on the prominent feature and a sharper, but still broad, maximum. The consistent feature of the LANL profiles is the ΔH_{exo} = 1825 ± 62 J/g.

The IHD 1 profile exhibits a shoulder on the high temperature side of the broad maximum. The other IHD profiles appear as complex combinations of exothermic features, although the maximum temperatures are near that of the Gunpowder. The ΔH_{exo} = 2046 ± 150 J/g.

LLNL profiles exhibit both the exothermic features that could be due to the Gunpowder, but also the endothermic features of the AN. However, a broad exothermic feature appears underneath the sharper exothermic feature. This broad feature is on the high temperature side in LLNL 1 open and the low temperature side for LLNL 2 open and LLNL 1 sealed. LLNL 1 sealed and LLNL 3 sealed appear to have two of these broader shoulders. Note that the open and sealed profiles are similar because the sealed sample holder is thought to rupture during the measurements.

These complex features seen in the T^5 range do not preclude the possibility of a synergy between the AN and the Gunpowder. The broad underlying features that often appear as shoulders in many of the profiles could be due to a thermal sensitivity that could be assigned to a thermally sensitive complex made by the AN and the Gunpowder. Likewise, the sharper dominant feature could be such a complex also. The region needs further examination to adequately explain the details of the features.

4.4 Speculation on Differences in Testing Results from Participants

In the AN, Gunpowder, and AN/Gunpowder series, there have been a wide range of SSST testing values for each of the materials, differing based on the participant. Table 9 shows the average values that reflect these differences. The IDCA has been addressing these issues to some extent throughout the Proficiency Test. The following is speculation about some of the reasons for these differences:

1. For AN, the average DH_{50} value determined by LLNL is lower (more sensitive) than the average DH_{50} values determined by LANL and IHD. *Possible reason*: microphone placement—LLNL uses a microphone that is close in compared to LANL; for low sensitivity materials, the drop hammer background effects the perception of a positive reaction. *Possible reason*: difficulty in determining a positive reaction for AN—IDCA Analysis Report 025[14] discusses the difficulty with impact testing of AN sometimes showing visual signs of a reaction, but not necessarily showing physical evidence of a reaction.
2. For Gunpowder, the average DH_{50} value determined by LLNL is twice that (less sensitive) of the DH_{50} values determined by LANL and IHD. *Possible reason*: type of microphone—even though LLNL has a microphone that is several feet closer to the anvil, the type of microphone is different, so the response is different. This makes the detection system less sensitive than the detection system of LANL or the observation method of IHD.
3. For AN/Gunpowder, the DH_{50} value determined by LLNL is not in between the DH_{50} values of the two components while the corresponding DH_{50} values determined by LANL and IHD are in between the two components. *Possible reason*: sampling—Figure 1 shows the AN and Gunpowder have very different particle size distributions, where the Gunpowder distribution is much narrower. Although there is overlap, AN has a lot more particles of small size. When sampling small quantities, this can lead to a different ratio of the AN to Gunpowder on the local level.
4. For BAM friction testing, LLNL shows Gunpowder and the AN/Gunpowder mixture to be more stable than the other participants. (In the case of AN, no one found any sensitivity.) *Possible reason*: safety shielding on the LLNL equipment—in IDCA Analysis Report 009[15], the comparison of shielding of the BAM friction equipment shows that LLNL has a system that is acoustically much more shielded than the other participants. This leads to TIL and F_{50} values from LLNL that indicate a much less sensitive material.
5. For BAM friction testing, the average TIL value determined by IHD is not between the two components, while the average TIL values determined by LANL and LLNL are between the two

components. *Possible reason*: sampling—Figure 1 shows the AN and Gunpowder have very different particle size distributions, where the Gunpowder is much narrower. Although there is overlap, AN has a lot more particles of small size. This can lead to a different ratio of the AN to Gunpowder on the local level.

6. For ESD, average TIL values determined by LLNL show no sensitivity for any of the materials. *Possible reason*: custom built ESD equipment—LLNL has custom built equipment with a 510-Ω resistor in the circuit. The other participants have ABL ESD equipment.
7. For ESD, the average TIL values determined by LANL show a more sensitive material than the corresponding average TIL values determined by IHD. *Possible reason*: humidity—consistently, LANL has < 10% relative humidity and IHD has 40+% relative humidity. Humidity affects spark sensitivity.
8. For DSC, LLNL resolves Transition T^5 into exothermic and endothermic components, while LANL and IHD record only evidence of an exothermic transition. *Possible reason*: sampling issue—LLNL samples are ~ 0.3 mg and LANL and IHD are over 1.0 mg. At a sample size of 0.3 mg, obtaining a representative sample is very difficult (seen before for $KClO_3$/sugar mixtures[5,6], for examples), especially with the mismatch of the particle sizes of the Gunpowder and the AN. It appears that the LLNL samples may be richer in AN compared to the Gunpowder. This is verified by examining the averages of the T^1 through T^4, shown in the Appendix. As indicated above, the enthalpies are categorically less in magnitude for T^1 through T^4 for the AN/Gunpowder mixture compared to AN. This is because the formulation of the mixture is 1/1 AN to Gunpowder, so the enthalpy values for AN/Gunpowder should be about ½ the enthalpy values of pure AN. However, the enthalpy values measured by LLNL are more than the rest of the participants, indicating a different composition, richer in AN than Gunpowder. As a result, the endothermic behavior of AN more dominates the DSC features for LLNL profiles.

5 CONCLUSIONS

Conclusions from this study of AN/Gunpowder are:

1. Impact testing
    a. The DH_{50} values for the mixture varied among participants
    b. IHD found the mixture to be the most sensitive
    c. LLNL and LANL found the mixture less sensitive than the RDX standard
    d. IHD found the mixture to be about the same sensitivity as the RDX standard
    e. All participants found the mixture to be less sensitive than the PETN standard
    f. LANL and IHD found the mixture to have sensitivity between the two component materials
    g. LLNL found the mixture to be more sensitive than the two component materials
2. Friction testing
    a. IHD found, by BAM Friction, the mixture to be the most sensitive
    b. LLNL found, by BAM Friction, the mixture to be moderately sensitive
    c. LANL and IHD found, by BAM Friction, the mixture to be more sensitive than the RDX standard
    d. LANL and IHD found, by BAM Friction, the mixture to be more sensitive than what LLNL found
    e. IHD found, by ABL Friction, similar sensitivity of the mixture with the RDX standard
    f. LLNL, LANL, and IHD found the mixture to be less sensitive than the PETN standard
    g. LLNL and IHD found the mixture to have sensitivity between the two component materials

    h. LANL found the mixture to have sensitivity greater than the two component materials

3. Spark testing
    a. Of all the participants, LANL found the mixture to be the most sensitive
    b. LANL and IHD found the sensitivity of the mixture to be between the sensitivity of the two components
    c. LANL and IHD found the mixture to be less sensitive than the RDX standard
    d. LLNL found the mixture to be insensitive
4. Thermal testing
    a. All participants found the mixture to exhibit thermal behavior reminiscent of the two component materials added together
    b. All participants found the mixture to have about the same thermal sensitivity as PETN and the RDX standards.

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—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.
3. Integrated Data Collection Analysis (IDCA) Program—RDX Standard Data Set 2, M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. J. Phillips, T. J. Shelley, J. A. Reyes, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **020**, LLNL-TR-619637 (725352), February 20, 2013.
4. Integrated Data Collection Analysis (IDCA) Program—RDX Standard Data Sets, M. M. Sandstrom, G. W. Brown, D. N. Preston, C. J. Pollard, K. F. Warner, D. N. Sorensen, D. L. Remmers, J. J. Phillips, T. J. Shelley, J. A. Reyes, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **024**, LLNL-TR-624214 (733312), March 4, 2013.
5. 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** (2011), LLNL-TR-482149, May 10. 2011.
6. 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.
7. Integrated Data Collection Analysis (IDCA) Program—$KClO_3$/Dodecane Mixture, 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, L. L. Whinnery, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **012**, LLNL-TR-484788, June 21, 2011.
8. Integrated Data Collection Analysis (IDCA) Program—$KClO_4$/Dodecane 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, L. L. Whinnery, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **015**, LLNL-TR-522941, May 11, 2012.
9. 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**, LLNL-TR-518531, December 5, 2011.
10. Integrated Data Collection Analysis (IDCA) Program—$KClO_4$/Carbon, 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* **018**, LLNL-TR-614974 (717752), January 31, 2013.
11. Integrated Data Collection Analysis (IDCA) Program—$NaClO_3$/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* **019**, LLNL-TR-617403 (721773), February 11, 2013.
12. 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, 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.
13. Integrated Data Collection Analysis (IDCA) Program—SSST Testing Methods, M. M. Sandstrom, G. W. Brown, K. F. Warner, D. N. Sorensen, D. L. Remmers, L. L. Whinnery, J. J. Phillips, T. J. Shelley, J. A. Reyes, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **009**, LLNL-TR-630173 (742792), March 25, 2013.

14. Integrated Data Collection Analysis (IDCA) Program—Ammonium Nitrate, M. M. Sandstrom, G. W. Brown, K. F. Warner, D. N. Sorensen, D. L. Remmers, L. L. Whinnery, J. J. Phillips, T. J. Shelley, J. A. Reyes, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **025**, LLNL-TR-636915 (755705), May 17, 2013.
15. Integrated Data Collection Analysis (IDCA) Program—Bullseye® Smokeless Powder, M. M. Sandstrom, G. W. Brown, K. F. Warner, D. N. Sorensen, D. L. Remmers, L. L. Whinnery, J. J. Phillips, T. J. Shelley, J. A. Reyes, P. C. Hsu, and J. G. Reynolds, *IDCA Program Analysis Report* **026**, LLNL-TR-637818 (756807), May 30, 2013.
16. 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.
17. 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.
18. Small Scale Safety Test Report for Ammonium Nitrate/Bullseye® mixture, P. C. Hsu and J. G. Reynolds, *IDCA Program Data Report* **050**, LLNL-TR-481096, April 19, 2011.
19. 50188 O AN/Gunpowder, M. M. Sandstrom and G. W. Brown, *IDCA Program Data Report* **034**, April 13, 2011.
20. AN/Gunpowder, D. L. Remmers, D. N. Sorensen and K. F. Warner, *IDCA Program Data Report* **110**, June 15, 2012.
21. Particle Size Analysis—Gunpowder, AN Dried @ 60°C (AR), AN Dried @ 60°C (100), K. B. Proctor, *IDCA Program Data Report* **113**, August 13, 2012.
22. A Method for Obtaining and Analyzing Sensitivity Data, W. J. Dixon and A.M. Mood, *J. Am. Stat. Assoc.,* **43**, 109-126, 1948.
23. 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.
24. D-Optimality-Based Sensitivity Test, B. T. Neyer, *Technometrics*, **36**, 48-60, 1994.
25. 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.
26. Challenges of Small-Scale Safety and Thermal Testing of Home Made Explosives—Results from the Integrated Data Collection Analysis (IDCA) Program Proficiency Test, J. G. Reynolds, M. M. Sandstrom, G. W. Brown, K. F. Warner, T. J. Shelley, P. C. Hsu, *IDCA Program Presentation* **009**, LLNL-PRES-547780, May 2, 2012.

ABREVIATIONS, ACRONYMS AND INITIALISMS

-100	Solid separated through a 100-mesh sieve
ABL	Allegany Ballistics Laboratory
AFRL	Air Force Research Laboratory, RXQL
Al	Aluminum
AR	As received (separated through a 40-mesh sieve)
ARA	Applied Research Associates
BAM	German Bundesanstalt für Materialprüfung Friction Apparatus
C	Chemical symbol for carbon
CAS	Chemical Abstract Services registry number for chemicals
cm	centimeters
DH_{50}	The height the weight is dropped in Drop Hammer that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
DHS	Department of Homeland Security
DSC	Differential Scanning Calorimetry
DTA	Differential Thermal Analysis
ESD	Electrostatic Discharge
F_{50}	The weight or pressure used in friction test that cause the sample to react 50% of the time, calculated by the Bruceton or Neyer methods
fps	feet per second
H	Chemical symbol for hydrogen
H_2O	Chemical formulation for water
HME	homemade explosives or improvised explosives
HMX	Her Majesty's Explosive, cyclotetramethylene-tetranitramine

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

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.

Table A.1. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^1 for AN and AN/Gunpowder

Participant and Parameters	Onset T^1, °C	T^1_{min}, °C	Enthalpy T^1, J/g
All AN dried	52.3 ± 1.9 46.4 to 55.8	53.6 ± 2.2 46.6 to 57.5	-19 ± 5 -25 to -3
All AN dried/Gunpowder	52.4 ± 0.8 51.6 to 53.5	53.6 ± 0.8 52.8 to 55.0	-12 ± 3 -16 to -6
LLNL pinhole AN dried	51.6 ± 0.1 51.6 to 51.7	53.0 ± 0.2 52.8 to 53.1	-20 ± 1 -21 to -20
LLNL pinhole AN dried/Gunpowder	51.7 ± 0.1 51.7 to 51.8	52.9 ± 0.2 52.8 to 53.0	-12 ± 2 -14 to -10
LLNL sealed AN dried	51.7 ± 0.1 51.6 to 51.7	53.0 ± 0.2 52.8 to 53.1	-20 ± 1 -21 to -19
LLNL sealed AN dried/Gunpowder	51.6 ± 0.0 51.6 to 51.6	53.0 ± 0.1 52.9 to 53.1	-14 ± 2 -16 to -13
LANL pinhole AN dried	52.8 ± 0.2 52.7 to 53.0	54.3 ± 0.2 54.1 to 54.5	-19 ± 2 -21 to -18
LANL pinhole AN dried/Gunpowder	53.0 ± 0.3 52.7 to 53.2	54.2 ± 0.7 53.6 to 55.0	-10 ± 4 -14 to -6
IHD pinhole AN dried	52.2 ± 0.1 52.1 to 52.3	53.0 ± 0.1 52.1 to 52.3	-5 ± 2 -7 to -3
IHD pinhole AN dried/Gunpowder	53.4 ± 0.2 53.1 to 53.5	54.3 ± 0.2 54.1 to 54.5	-12 ± 4 -15 to -7

Table A.2. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^2 for AN and AN/Gunpowder

Participant and Parameters	Onset T^2, °C	T^2_{min}, °C	Enthalpy T^2, J/g
All AN dried	90.7 ± 1.6 85.5 to 92.6	92.7 ± 2.3 86.6 to 96.2	-14 ± 13 -57 to -3
All AN dried/Gunpowder	92.3 ± 1.1 90.7 to 93.9	92.5 ± 1.5 90.0 to 94.3	-5 ± 2 -10 to -3
LLNL pinhole AN dried	91.2 ± 0.7 90.5 to 91.8	93.2 ± 1.1 92.0 to 94.2	-5 ± 2 -6 to -3
LLNL pinhole AN dried/Gunpowder	93.0 ± 1.2 92.1 to 93.8	94.0 ± 0.3 93.7 to 94.3	-5 ± 2 -6 to -3
LLNL sealed AN dried	91.4 ± 0.4 91.1 to 91.8	93.7 ± 0.3 93.4 to 94.0	-5 ± 2 -7 to -3
LLNL sealed AN dried/Gunpowder	91.7 ± 1.4 90.7 to 92.7	92.9 ± 0.6 92.4 to 93.3	-5 ± 1 -5 to -4
LANL pinhole AN dried	90.9 ± 0.5 90.2 to 91.4	92.7 ± 0.8 92.0 to 93.5	-17 ± 1 -18 to -17
LANL pinhole AN dried/Gunpowder	91.7 ± 0.5 91.1 to 92.0	92.4 ± 0.1 92.3 to 92.5	-7 ± 3 -10 to -4

Table A.3. Temperature and Enthalpy Averages and Ranges of DSC data Transition T3 for AN and AN/Gunpowder

Participants and Parameters	Onset T^3, °C	T^3_{min}, °C	Enthalpy T^3, J/g
All AN dried	126.5 ± 0.5 125.9 to 128.1	128.3 ± 1.0 127.0 to 131.1	-53 ± 6 -60 to -38
All AN dried/Gunpowder	126.6 ± 0.4 126.1 to 127.1	127.8 ± 0.9 127.0 to 130.0	-29 ± 10 -45 to -12
LLNL pinhole AN dried	126.2 ± 0.1 126.1 to 126.2	127.6 ± 0.2 127.3 to 127.7	-55 ± 1 -55 to -54
LLNL pinhole AN dried/Gunpowder	126.1 ± 0.1 126.1 to 126.2	127.1 ± 0.1 127.0 to 127.2	-32 ± 9 -42 to -26
LLNL sealed AN dried	126.2 ± 0.0 126.2 to 126.2	127.6 ± 0.1 127.5 to 127.7	-55 ± 1 -56 to -55
LLNL sealed AN dried/Gunpowder	126.1 ± 0.1 126.1 to 126.2	127.2 ± 0.1 127.2 to 127.3	-39 ± 5 -45 to -36
LANL pinhole AN dried	126.7 ± 0.2 126.6 to 127.0	129.1 ± 0.1 129.0 to 129.2	-55 ± 2 -57 to -54
LANL pinhole AN dried/Gunpowder	127.0 ± 0.1 126.8 to 127.1	129.1 ± 1.1 128.7 to 130.3	-23 ± 8 -32 to -18
IHD pinhole AN dried	126.1 ± 0.1 126.0 to 126.1	127.3 ± 0.2 127.2 to 127.5	-58 ± 2 -60 to -56
IHD pinhole AN dried/Gunpowder	127.0 ± 0.1 126.9 to 127.0	127.9 ± 0.2 127.7 to 128.1	-22 ± 9 -30 to -12

Table A.4. Temperature and Enthalpy Averages and Ranges of DSC data Transition T^4 for AN and AN/Gunpowder

Participant and Parameter	Onset T^4, °C	T^4_{min}, °C	Enthalpy T^4, J/g
All AN dried	168.5 ± 1.1 164.8 to 169.4	169.5 ± 0.6 167.9 to 170.7	-71 ± 9 -82 to -32
All AN dried/Gunpowder	167.1 ± 2.0 164.0 to 169.0	168.4 ± 1.3 166.0 to 169.6	-32 ± 15 -61 to -9
LLNL pinhole AN dried	168.8 ± 0.6 168.1 to 169.9	169.6 ± 0.4 169.2 to 169.9	-74 ± 0 -74 to -74
LLNL pinhole AN dried/Gunpowder	168.7 ± 0.4 168.3 to 169.0	169.5 ± 0.2 169.3 to 169.6	-40 ± 13 -55 to -31
LLNL sealed AN dried	169.1 ± 0.1 169 to 169.2	169.8 ± 0.1 169.7 to 169.9	-75 ± 1 -76 to -74
LLNL sealed AN dried/Gunpowder	168.9 ± 0.3 168.8 to 169.0	169.6 ± 0.0 169.6 to 169.6	-47 ± 13 -61 to -43
LANL pinhole AN dried	169.3 ± 0.2 169.0 to 169.4	170.2 ± 0.5 169.8 to 170.7	-76 ± 3 -79 to -74
LANL pinhole AN dried/Gunpowder	165.1 ± 1.3 164.0 to 166.5	167.8 ± 1.2 167.1 to 169.2	-21 ± 9 -31 to -15
IHD pinhole AN dried	168.7 ± 0.1 168.6 to 168.8	169.1 ± 0.1 169.1 to 169.2	-78 ± 4 -82 to -74
IHD pinhole AN dried/Gunpowder	165 .4 ± 1.2 164.1 to 166.4	167.1 ± 0.9 166.0 to 167.6	-22 ± 12 -32 to -9

Disclaimer

Lawrence Livermore National Laboratory is operated by Lawrence Livermore National Security, LLC, for the U.S. Department of Energy, National Nuclear Security Administration under Contract DE-AC52-07NA27344.