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Imaging Indicator for ESD Safety Testing

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Abstract

This report describes the development of a new detection method for electrostatic discharge (ESD) testing of explosives, using a single-lens reflex (SLR) digital camera and a 200-mm macro lens. This method has demonstrated several distinct advantages to other current ESD detection methods, including the creation of a permanent record, an enlarged image for real-time viewing as well as extended periods of review, and ability to combine with most other Go/No-Go sensors. This report includes details of the method, including camera settings and position, and results with well-characterized explosives PETN and RDX, and two ESD-sensitive aluminum powders.

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NOMENCLATURE

ABL	Allegany Ballistics Laboratory
CMOS	complementary metal–oxide–semiconductor
ESD	electrostatic discharge
IDCA	Integrated Data Collection and Analysis program
LANL	Los Alamos National Laboratory
NSWC	Naval Surface Warfare Center
PETN	pentaerythritol tetranitrate
RDX	cyclotrimethylenetrinitramine
SEM	scanning electron microscope
SLR	single-lens reflex
SMS	Safety Management Services, Inc.
SNL	Sandia National Laboratories
TIL	threshold initiation level

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1. INTRODUCTION

Safety testing of explosive materials has become a standard requirement as explosive applications in commercial, mining, and military fields have grown over the past century. Materials are tested to determine the conditions under which the explosive can be detonated, in order to establish standards for safety in handling, storage, and use. Four categories of safety testing have developed:

1) Impact testing, performed by dropping a fixed weight onto a prepared explosive sample and varying the height from which the weight is dropped to determine an initiation level where 50% of the time the samples will react;
2) Friction testing, performed by placing the explosive sample between either a plate and a wheel or a plate and a pin, and moving the plate to create friction at the point of contact to determine a threshold level of reaction;
3) Electrostatic discharge (ESD) testing, performed by discharging a spark of a known energy through the explosive material to a grounded sample holder to determine a threshold level of reaction;
4) Thermal testing, performed either by heating a confined sample until it reacts, or analyzing a material via differential scanning calorimetry (DSC) to determine melting point, phase changes, and decomposition temperature.

All testing methods require the operator to determine whether a reaction has occurred for each test, also referred to as a "go" or "no-go" result. The reaction is typically indicated by audible or visual means, which can include sounds such as a pop or bang and visual indicators including flash, flame, or spark. The presence of organic compounds released into the air during the test can also be determined or measured via infrared analysis for evidence of decomposition.

Determination of a go/no-go result is particularly error prone near the threshold to initiation, as it can suffer from subjective operator judgment. It is common to have several people witness the same test at the same time and half will think it was a go and the other half think it was a no-go. The study described in this report is directed at the development of a new archival method for determination enhancement of a spark initiation during ESD testing of energetic materials.

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2. ABL ESD SENSITIVITY TESTER

The most common ESD tester design is based on the machine designed at the Allegany Ballistics Laboratory (ABL). In this design, the sample is placed on the metal sample holder, which is grounded to a base plate. A capacitor bank is charged to the selected voltage and capacitance. The test is initiated by dropping the discharge needle rapidly towards the sample, which transfers the electrical charge from the needle to the ground, passing through the sample.

The testing apparatus manufactured by Safety Management Services, Inc. (SMS) is shown in Figure 1. A close up view of the sample holder, ESD needle, and sample enclosure is shown in Figure 2. A port on the sample enclosure is connected to an infrared gas detector, which measures the concentration of CO and CO_2 in the reaction/decomposition products. The stainless steel metal sample holder is surrounded by a nylon insulating ring, which can be made thicker to stand up above the metal and form a cup to hold liquids. The standard sample holder has a 0.5-inch diameter area to hold the explosive material; a holder with a 0.25-inch diameter area is also available in order to use less explosive material for each test.

Figure 1. ABL Electrostatic Discharge Apparatus manufactured by SMS.

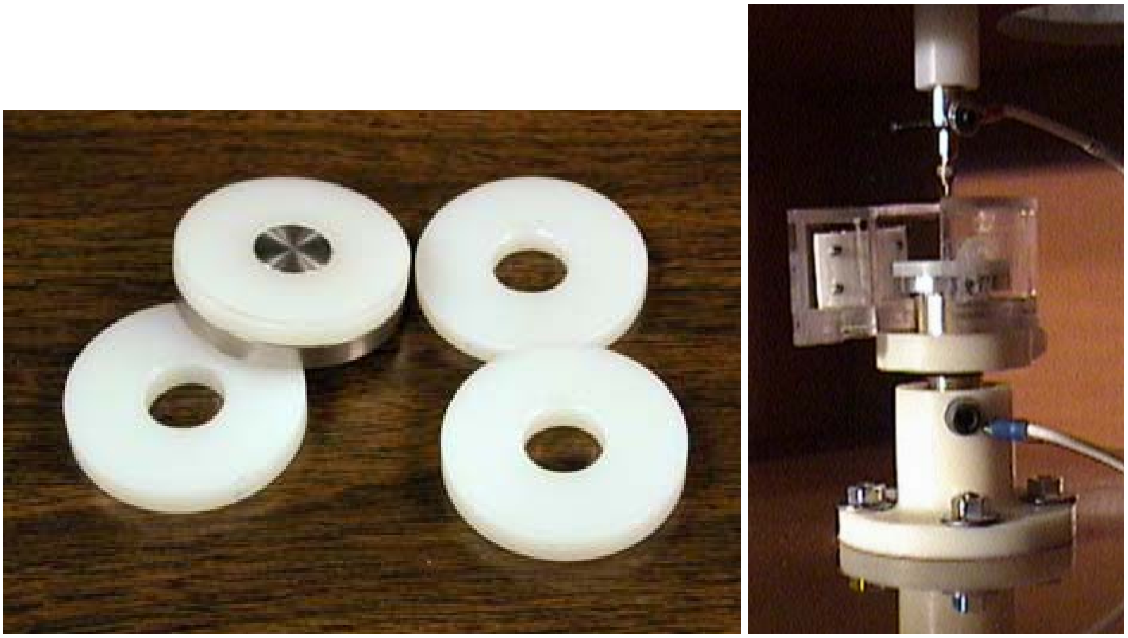

Figure 2. Sample holders (left) and sample holder, enclosure, and ESD needle (right) for the ABL ESD Apparatus manufactured by SMS.

Methods of Observation for ESD Reactions

There are several methods of detection of a positive event, including visual, auditory, and olfactory observations in real time, physical evidence, gas analysis of reaction products, tape analysis, and high-speed video. Signs of a "go" reaction are described below, with their respective positive and negative attributes.

Visual observations: made in real-time, can be extremely quick and difficult to see at low energy levels, but quite obvious at higher energy levels. Visible signs of reaction can be produced by discharging onto a blank sample holder.

- Color change in flame
- Flash or flame
- Flame trace
- Spark
- Propagating fire
- Small and faint ejecta ("flyers")
- Smoke

Auditory observations: made in real-time, can be difficult to distinguish from the loud "clunk" sound made by the lowering of the needle and transfer of the electrical charge.

- Pop/snap
- Bang
- Loud report

Olfactory observations: made in real-time, not always present, shielded by the sample enclosure and needle compartment door. There are also personnel exposure concerns with this method.

- Odor (burned/smoke)

Physical evidence: Not always present, but can be examined post-test and recorded by photo.

- Sample consumed by propagating fire
- Hardware damage
- Tape changes

Gas Analysis: A port from the sample enclosure connects to a NDIR Series 600 Gas Analyzer manufactured by California Analytical Instruments. The analyzer constantly samples the air from the sample enclosure and measures the concentration of various organic molecules present in the air. A change of 40 ppm from the baseline immediately following a test indicates a detonation. This method is not applicable to materials without organic content (perchlorate/aluminum, for example).

- CO, CO_2
- NO_x

Tape Analysis: This method is not typically used at SNL/CA, but is commonly used by other laboratories. A piece of transparent pressure-sensitive tape, such as 3M Scotch Magic Tape, is placed over the sample. The electrical discharge is transferred through the tape and sample to the grounded sample holder. The tape can show physical evidence of a reaction, and the tape can be preserved as a record of the test. Unfortunately, the tape can often appear torn or even singed while just running a blank sample. After a successful reaction, the tape can appear:

- Burned/singed
- Soot deposition
- Torn

High-Speed Video: Provides a detailed record of the test, and allows a good view of the discharge and/or any possible reactions. High-speed video is extremely expensive to implement, on the order of $50,000. A commercial system high-speed video system has been developed by SMS to use with their ESD tester. Without automated image analysis software, this can also be very time consuming.

Overall, the current detection options, with the exception of high-speed video, are quite subjective, requiring hands-on experience to become practiced at observing the visual evidence of a reaction, especially at lower energy levels. The tape analysis method is a variant that has not been widely used, thus limiting its comparison to previous results.

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3. NEW DETECTION METHOD: SLR CAMERA

Camera Specifications and Settings

A method of reaction detection during ESD sensitivity testing of explosives has been developed using a single-lens reflex (SLR) digital camera and a 200-mm macro lens. The Nikon D90 camera body, with a 12.3 megapixel CMOS imaging sensor, and the Micro-Nikkor 200-mm Lens combine to make the basis of the detection equipment, for a cost of approximately $2000. The end of the lens is positioned approximately 18 inches from the sample holder. A tripod is required to hold the camera, and a television connected to the A/V Output signal of the camera is helpful for immediate review of the image in a magnified format. Figure 3 shows an example of this detection setup with the ABL ESD tester. It should be noted that less expensive options might be commercially available; this image capture method was first demonstrated with a small digital camera that had a manual focus (although very coarse) and allowed the shutter speed and aperture to also be set manually.

Figure 3. SLR Camera detection method for ESD testing, including the SLR camera body, 200-mm lens, tripod, and television, displaying the results of a test.

A series of tests were conducted to determine the best camera settings for this method, using a blank sample holder. In particular, the reflections caused by the sample enclosure should be minimized. The exposure time, aperture (f-stop), and ISO sensitivity were varied, and Figures 4 and 5 show examples of the effects of those variations. Based on these experiments and a series of tests on various materials, the standard settings have been set at aperture f/20, exposure time 1 second, and ISO sensitivity 200.

Figure 4. Effect of exposure time on image capture of spark (0.05 μF-5000 V) on blank sample holder, aperture f/20, for 0.5 seconds (left), 1 second (center), and 2 seconds (right).

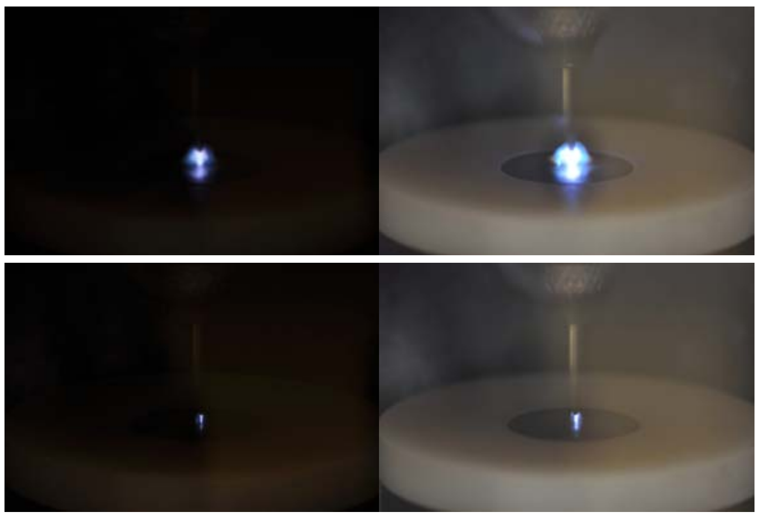


Figure 5. Effect of aperture on image capture of spark. Top: 0.01 μF-5000 V spark, 2 seconds exposure, at f/40 (left) and f/10 (right). Bottom: 0.0002 μF-5000 V spark, 2 seconds exposure, at f/29 (left) and f/10 (right).

Data Organization: A Single Spreadsheet Approach

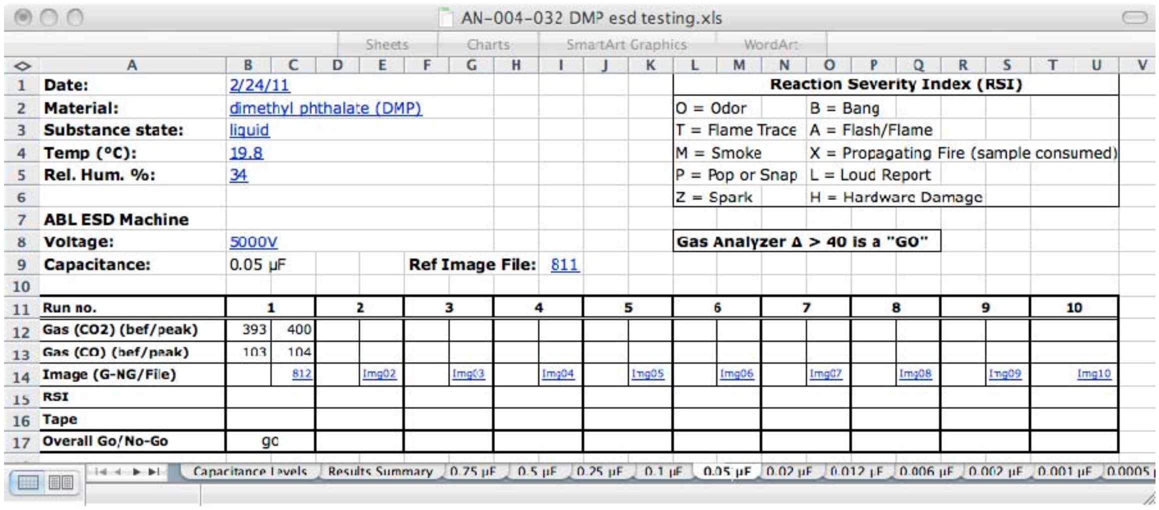
Several pieces of data must be collected for each test:

- a blank sample holder image at each energy level, with the reference number of the image recorded (for post-test retrieval)
- an image for each test (with the reference number of the image recorded)
- the ESD tester settings (capacitance, voltage)
- any notes on the reaction (particular sound, odor, or visual observation)
- starting and ending CO and CO_2 values from the gas analyzer (optional, not required for the image capture method)

To facilitate the collection and review of this data, a set of Excel spreadsheets has been created. Figure 6 shows examples of the Excel spreadsheets. The general design is based on a data worksheet supplied by SMS for use with the ABL ESD tester. A worksheet is created for each capacitance level as specified by the original SMS worksheet, with spaces to collect information about the sample, lab conditions, and data for each test, including gas analyzer levels and any observations. Additional worksheets can be added for different capacitance levels as necessary.

The section for each test includes a link to a second worksheet where the images from each test are placed; the image reference number can be noted during testing, and the images can be added at a later time. The images worksheet also includes the reference image of the blank sample holder, to allow easy comparison of each test image to the reference. The testing results are summarized in a table, representing each test as a green box for a positive reaction ("go") or a red box for no reaction ("no-go"). For the test shown here, the Threshold Initiation Level (TIL) is established as the energy level below which 10 tests in a row failed to react. A normal TIL requires 20 no-go's. As shown in Figure 6, a TIL of 0.002 μF or 25 mJ was determined for the material being tested.

While data and images can be organized through many different approaches, this set of Excel spreadsheets has served well and is included here as an example of one method of organization.



AN-004-032 DMP esd testing.xls

Date:	2/24/11
Material:	dimethyl phthalate (DMP)
Substance state:	liquid
Temp (°C):	19.8
Rel. Hum. %:	34
ABL ESD Machine	
Voltage:	5000V
Capacitance:	0.05 µF
Ref Image File:	811

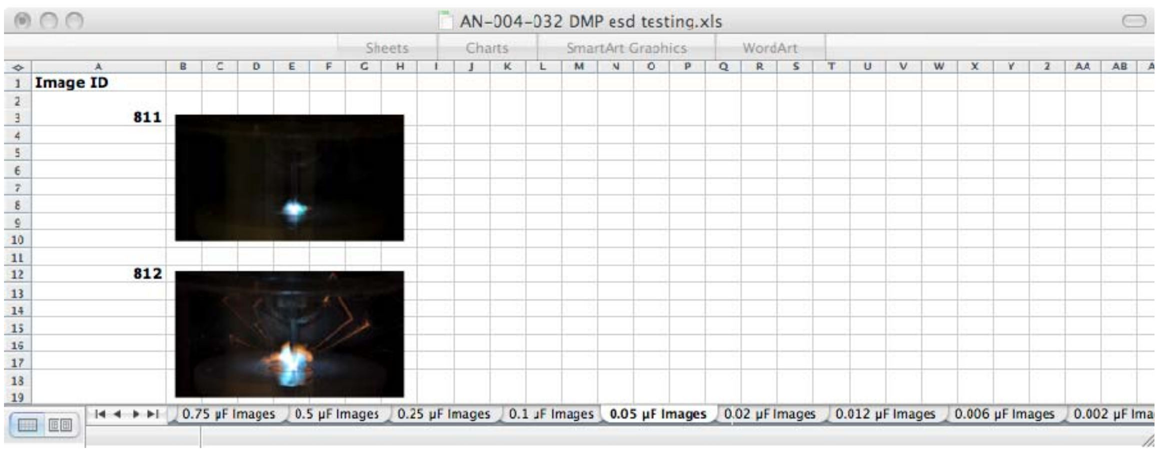
Reaction Severity Index (RSI)

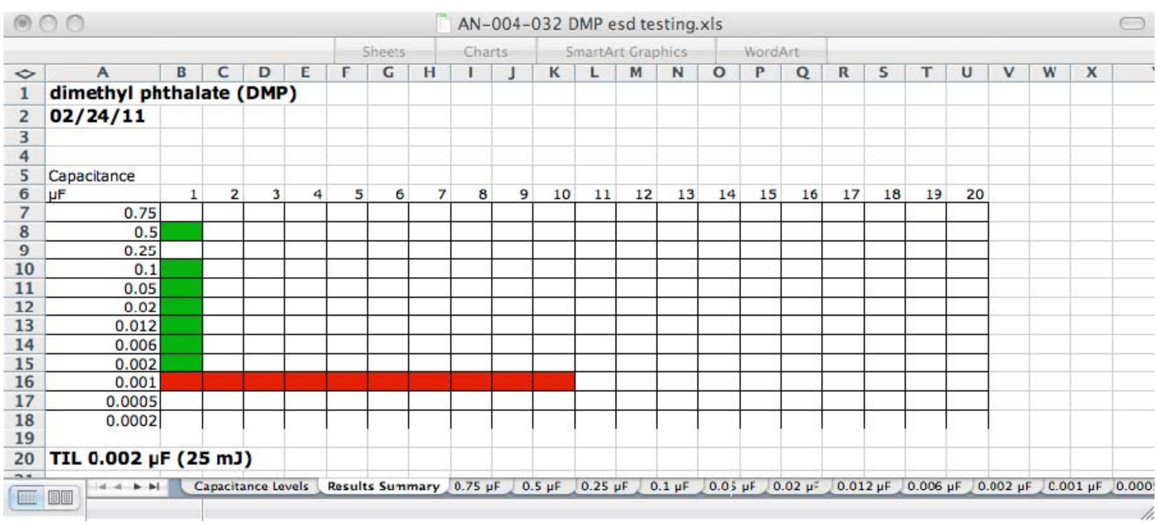
O = Odor	B = Bang
T = Flame Trace	A = Flash/Flame
M = Smoke	X = Propagating Fire (sample consumed)
P = Pop or Snap	L = Loud Report
Z = Spark	H = Hardware Damage

Gas Analyzer Δ > 40 is a "GO"

Run no.	1		2	3	4	5	6	7	8	9	10
Gas (CO2) (bef/peak)	393	400									
Gas (CO) (bef/peak)	103	104									
Image (G-NG/File)		812	Img02	Img03	Img04	Img05	Img06	Img07	Img08	Img09	Img10
RSI											
Tape											
Overall Go/No-Go	go										

Capacitance Levels | Results Summary | 0.75 µF | 0.5 µF | 0.25 µF | 0.1 µF | 0.05 µF | 0.02 µF | 0.012 µF | 0.006 µF | 0.002 µF | 0.001 µF | 0.0005





AN-004-032 DMP esd testing.xls

dimethyl phthalate (DMP)
02/24/11

Capacitance µF	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
0.75																				
0.5																				
0.25																				
0.1																				
0.05																				
0.02																				
0.012																				
0.006																				
0.002																				
0.001																				
0.0005																				
0.0002																				

TIL 0.002 µF (25 mJ)

Capacitance Levels | Results Summary | 0.75 µF | 0.5 µF | 0.25 µF | 0.1 µF | 0.05 µF | 0.02 µF | 0.012 µF | 0.006 µF | 0.002 µF | 0.001 µF | 0.000

Figure 6. Excel spreadsheet for ESD sensitivity testing, showing the data, images, and TIL table (top to bottom) collected during an ESD sensitivity test.

4. TESTING OF ENERGETIC SAMPLES

Record Blanks for Comparison

To effectively characterize the display created by a reaction in the ESD tester, the images created by a blank sample holder should be reviewed first. A blank spark discharge creates a significant blue-white flash accompanied by a loud "chunk" sound. Figures 4, 5, and 7 show examples of the flash at various energy levels. The flash diminishes in size and intensity with decreasing energy level, becoming a single spark as seen in the bottom images in Figure 5. An orange color can be visible on the outer edges of larger flashes as well, which can be mistaken for flames created by a positive reaction.

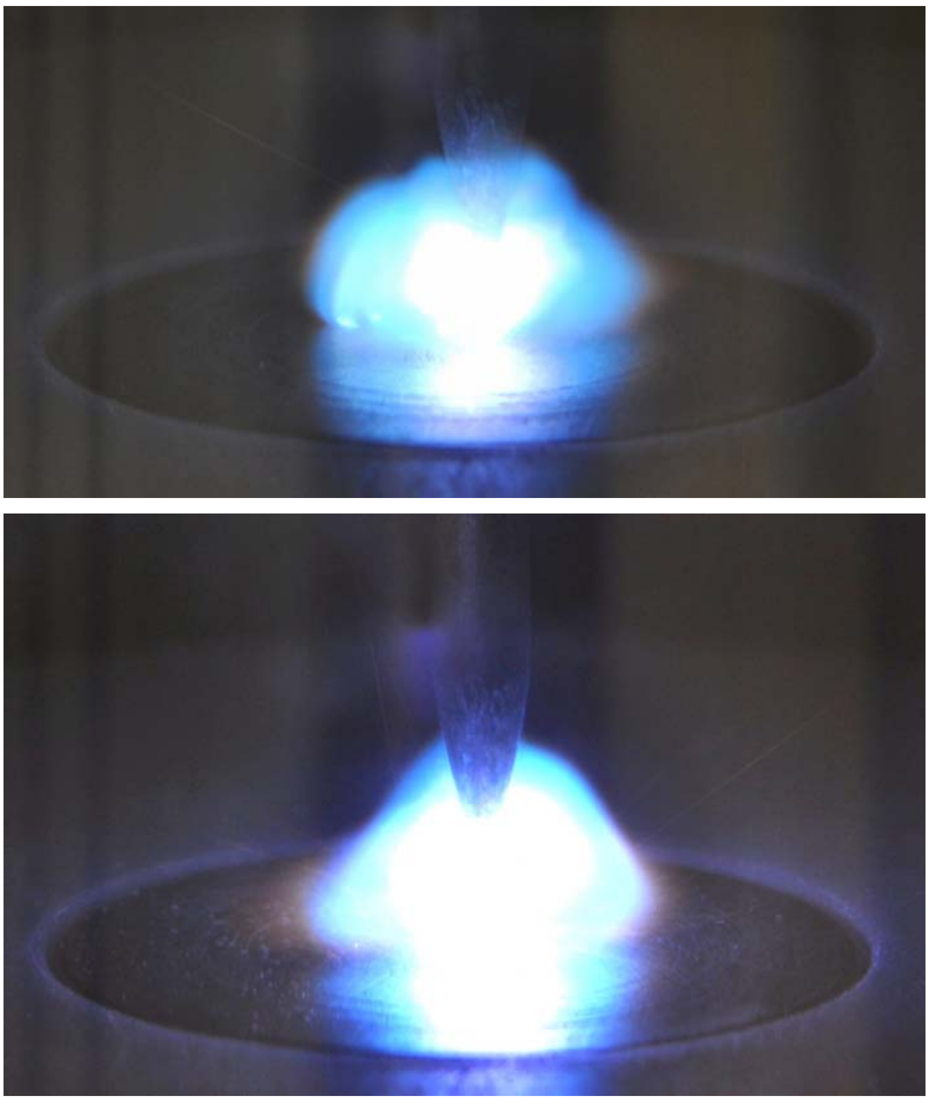


Figure 7. Blank sample holder with a 0.05 μF (0.625 J) discharge. Small "flyer" sparks can be seen in the upper left quadrant of top image, and in several directions away from the discharge in the bottom image.

In addition to the flash, tests on a blank sample holder can also create "flyers", very small and faint ejecta from the baseplate that only become visible when recorded by a camera system and reviewed under magnification. Figure 7 shows two such tests, with ejecta visible as faint orange lines radiating from the flash. These ejecta are more common at the highest energy levels, and rarely seen at lower levels. The camera method, however, allows the results of all tests to be scrutinized, and these ejecta may be visible on a test of energetic material and cause a "false positive" result. The net result of this "false positive" would be a lower TIL, causing the material to be handled with more caution than may be necessary. However, it is advised to take these ejecta into account when evaluating test images, and consider rating those with one or two "flyers" to be non-reactions.

PETN, A Well-Characterized Explosive

The camera detection system was used on some familiar and widely characterized materials to compare results with published literature values. PETN (pentaerythritol tetranitrate) is used in many different explosive mixtures and is typically used as a "standard" for safety testing. Selected data and images are summarized in Table 1, and the TIL table is presented as Table 2.

Table 1. Images and data from ESD sensitivity testing of PETN powder

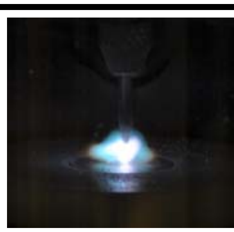
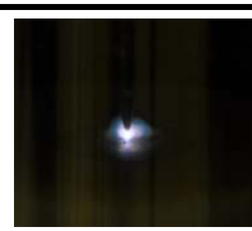
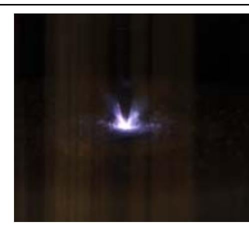
Capacitance Level (μF)	0.1		0.02				0.012	
Blank Image								
Test Image								
Gas	CO_2	CO	CO_2	CO	CO_2	CO	CO_2	CO
Starting Conc. (ppm)	384	90.5	344	81.5	344	86.0	364	91.0
Ending Conc. (ppm)	1100	157	351	89	403	99.3	371	94.9
Δ Conc. (ppm)	**716**	66.5	7	7.5	**59**	13.3	7	3.9
Result	**GO**		**NO-GO**		**GO**		**NO-GO**	

Table 2. Threshold Initiation Level (TIL) Summary for PETN Powder

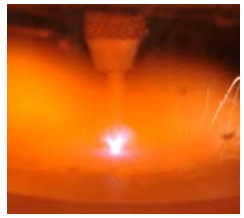
Capacitance Level (µF)																				
0.1																				
0.05																				
0.02																				
0.012																				
0.006																				
0.002																				
0.001																				

The final TIL of 0.02 µF at 5000 V corresponds to an energy level of 0.25 J. This is comparable to various reported values for PETN: 0.19 J from industry reports, 0.062 J from testing at Los Alamos National Laboratory (LANL), and 0.325 J from the FBI's Database of Range Evaluated Improvised Explosives (DBREIE).

RDX, A Reference Explosive

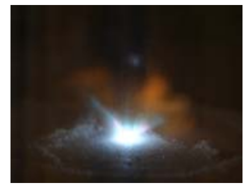
The camera system was also used to characterize RDX (cyclotrimethylenetrinitramine), one of the most powerful and brisant of the high explosives used by the military, and used in many explosive mixtures. RDX is typically used as the reference material to calibrate the drop tower for impact safety testing, as well as a standard for the IDCA round robin testing. The material used in this study was rated as Class 5 and received from Kirstin Warner at NSWC, Indian Head Division. Selected data and images are summarized in Table 3.

Table 3. Images and data from ESD sensitivity testing of RDX powder.

Capacitance Level (µF)	0.05		0.02				0.01	
Blank Image								
Test Image								
Gas	CO_2	CO	CO_2	CO	CO_2	CO	CO_2	CO
Starting Conc. (ppm)	357	73.1	357	73.4	352	72.7	355	72.5
Ending Conc. (ppm)	911	80.4	362	72.7	610	77.2	355	72.6
Δ Conc. (ppm)	**554**	7.3	5	-0.7	**258**	4.5	0	0.1
Result	**GO**		**NO-GO**		**GO**		**NO-GO**	

Of the twenty no-go results recorded at a capacitance level of 0.01 µF, five images revealed an orange color in the spark. While the appearance of color such as this might indicate a go, the gas analysis clearly indicates that no reaction took place, with similar results across all samples at 0.01 µF regardless of spark color. Examples of results at 0.01 µF with and without the orange color are summarized in Table 4.

Table 4. Images and Data from "no-go" results at 0.01 µF for RDX powder.

Capacitance Level (µF)	0.01							
Blank Image								
Test Image								
Gas	CO_2	CO	CO_2	CO	CO_2	CO	CO_2	CO
Starting Conc. (ppm)	356	71.8	341	71.6	356	71.6	339	71.9
Ending Conc. (ppm)	352	71.7	342	72	356	71.7	339	72.0
Δ Conc. (ppm)	**-4**	**-0.1**	**1**	**0.4**	**0**	**0.1**	**0**	**0.1**
Result	**NO-GO**		**NO-GO**		**NO-GO**		**NO-GO**	

The final TIL of 0.01 µF at 5000 V corresponds to an energy level of 0.125 J. This is slightly higher, but comparable to reported results from other laboratories: 0.025 J from LANL, 0.095 J from Indian Head Division, and 0.06 J from the Air Force Research Laboratory. The full TIL summary of the testing of RDX for this report can be found in Table 5.

Table 5. Threshold Initiation Level (TIL) Summary for RDX Powder

Capacitance Level (µF)																				
0.05																				
0.02																				
0.01																				

Aluminum Powders, A Common Ingredient in Explosives, Propellants, and Pyrotechnics

As an example of another ESD-sensitive ingredient to various explosive mixtures, two aluminum powders were tested. Spherical powder, grade H2 from Valimet, has a size range with 90% < 6.8 µm, and German Blackhead flake powder, similar to Eckart 5413 H-Super, has particles generally < 10 µm. Figure 8 shows SEM images of both powders to compare size and shape of the particles. Table 6 summarizes example images from ESD testing. As the aluminum powders do not contain an organic component, gas analysis was not conducted.

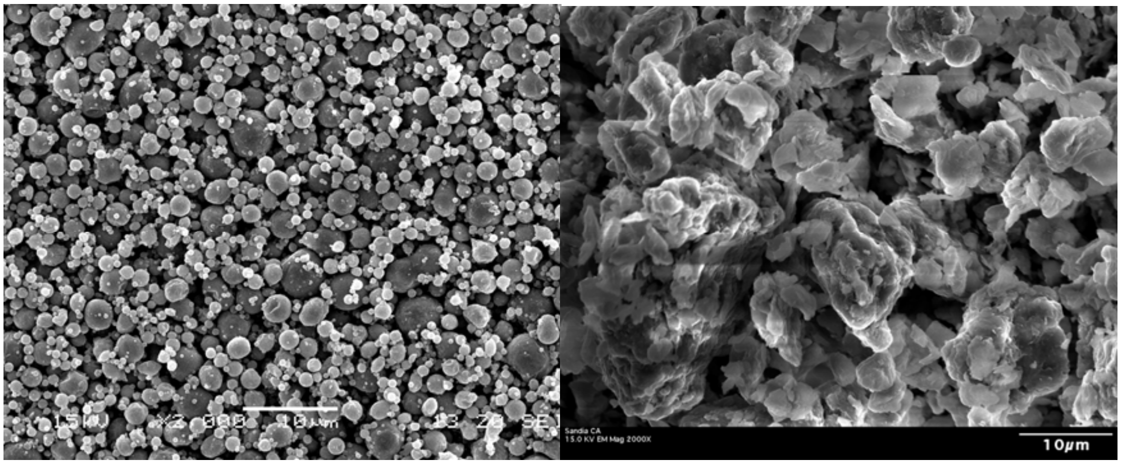
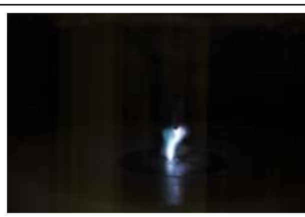
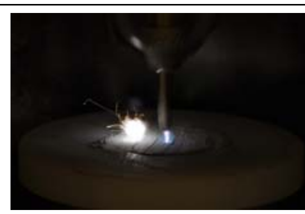
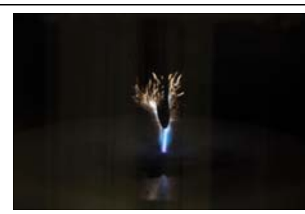
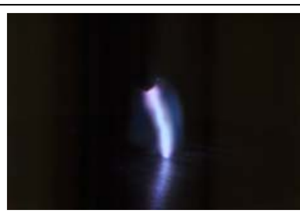
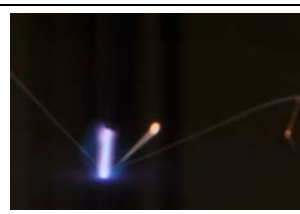
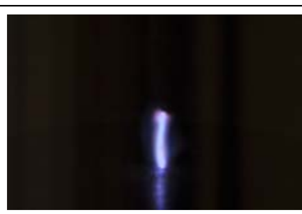
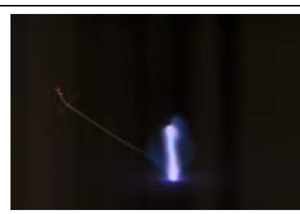
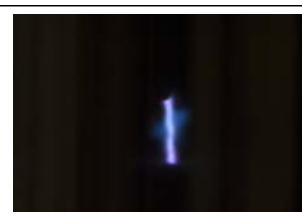


Figure 8. SEM images of Valimet grade H2 aluminum powder (left) and German Blackhead aluminum powder (right).

Table 6. Images from ESD testing of aluminum powders. Images at 0.001 and 0.0005 μF capacitance levels are enlarged 2x.

Capacitance Level (μF)	Blank Image	Valimet H2 Spherical Al powder	German Blackhead flake Al powder
0.006			
0.002			
0.001 (2x zoom)			
0.0005 (2x zoom)			

As can be seen from the results, test images for aluminum powder result in “flyers” similar to the ejecta seen from discharge to a blank sample holder, as seen in Figure 7. However, the single sparks created from the aluminum sample occurred at very low energy levels, while higher energy levels are required to create ejecta from a blank holder. The ejecta phenomenon was taken into account when making a final ignition determination, and test images with a single flyer, such as that for the Valimet Al powder at 0.0005 µF, were labeled as a “no-go”.

The final TIL of 0.0005 µF at 5000 V for both Al powders corresponds to an energy level of 6.25 mJ. This falls within 0.1-10 mJ range of values found in the literature. The full TIL summary of the testing of Valimet H-2 Spherical Al powder for this report can be found in Table 7, and the summary for German Blackhead flake powder can be found in Table 8.

Table 7. Threshold Initiation Level (TIL) Summary for Valimet H-2 Aluminum Powder

Capacitance Level (µF)																				
0.5																				
0.25																				
0.1																				
0.05																				
0.02																				
0.01																				
0.007																				
0.006																				
0.005																				
0.004																				
0.003																				
0.002																				
0.001																				
0.0005																				
0.0002																				
0.0001																				

Table 8. Threshold Initiation Level (TIL) Summary for German Blackhead Aluminum Powder

Capacitance Level (µF)																				
0.006																				
0.002																				
0.001																				
0.0005																				

5. DISCUSSION AND CONCLUSIONS

The still camera method for ESD testing interpretation has proven to be an extremely useful tool for determining the electrostatic sensitivity of various explosive mixtures and other materials. Compared to other methods of detection, almost all of which require close visual observation of a very rapid test with very little resulting physical evidence, the still camera method has demonstrated several distinct advantages, including:

- A permanent record, allowing for interpretation post-test.
- Inexpensive and not difficult to implement.
- An enlarged image can be viewed in real time on a television.
- Resulting images can be reviewed for an extended period of time.
- Camera method can be combined with most other Go/No-Go sensors, including gas analysis.

The method has been used to characterize several materials, from standard explosives such as PETN and RDX, to ESD sensitive materials including aluminum powders, and experimental formulas in liquid and powder form. For each material, testing was completed in an efficient manner, and the resulting images were analyzed both in real time and post-testing, together with CO/CO_2 gas analysis data when available, to quickly determine a Go/No-Go result and an overall TIL for each material.

However, there are a few disadvantages to this method as well, which should be kept in mind while conducting ESD testing:

- Images, especially when enlarged, pick up subtleties missed by other methods, making this method possibly “too sensitive” and creating a lower TIL. This does err on the side of caution, however.
- The tape method is not compatible with the still camera method.
- Glare from the plastic sample enclosure surrounding the sample holder can distract from the spark.
- Ejecta from the base of the sample holder can create a false positive.
- An orange color in the spark has been observed on blanks as well as samples.

These effects can be minimized through refinement of the still camera method, including camera placement to reduce glare, and Go/No-Go criteria established to account for flyers and other “false positive” indicators. Overall, the still camera-imaging indicator for electrostatic discharge testing of explosive materials can be an excellent addition to an ABL testing system. In addition, a still camera may be a useful tool during impact and friction small scale safety testing.

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