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Introducing the LEESA (Low Energy Electrostatic Apparatus)

A Work in Progress

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Energetic Materials Org. (7555)

2021 ET Users Group Meeting
Park City, Utah USA

EXPLOSIVE TECHNOLOGIES 7550 GROUP

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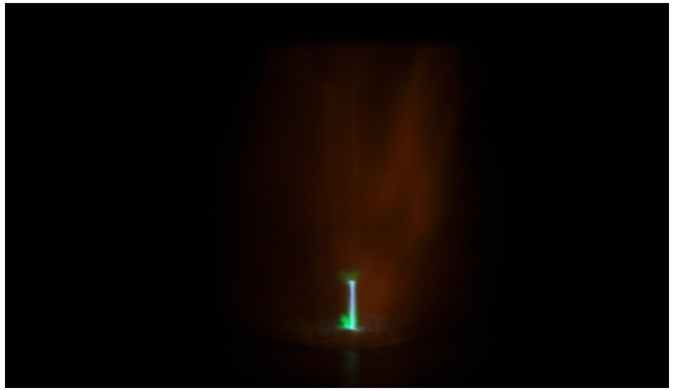
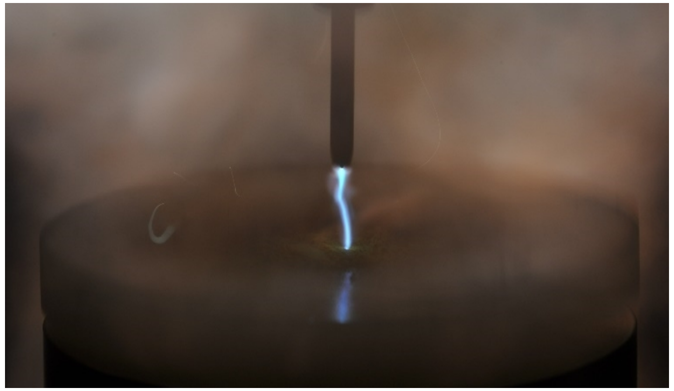
The Problem

- The ABL* tester[2-3] has been in use at SNL, Albuquerque since 2012
- ABL testing can require ~50mg or more per trial and often uses a 0 of 20 TIL†
- Many sensitive materials still initiate at the lowest energy level (TKP, THKP, ZPP, SASN, etc.), preventing a TIL from being performed
- 3 example materials initiating at the lowest energy level (0.0025 Joules):



SASN

$TH_{1.65}KP$



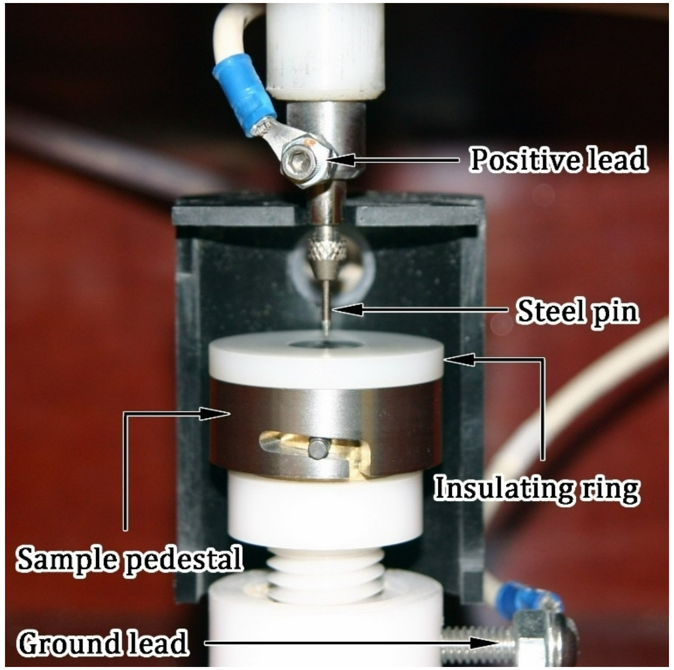
NLS

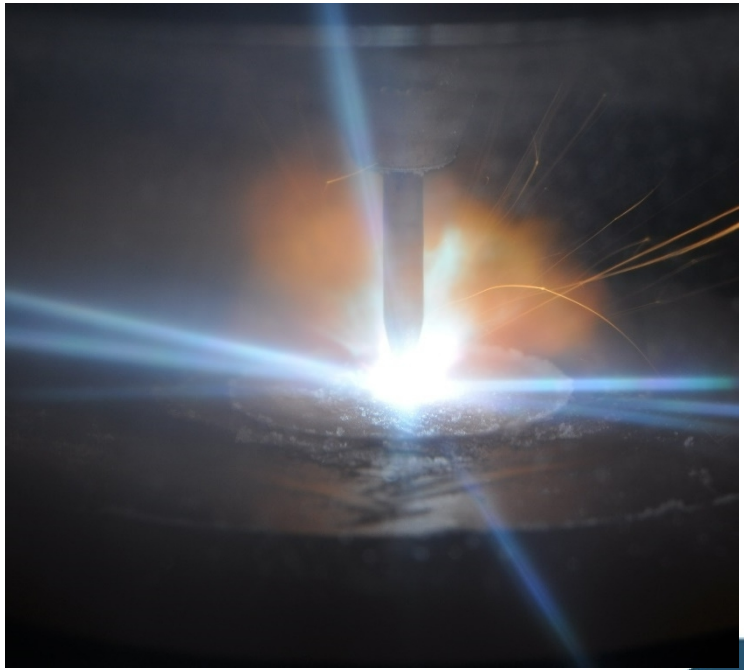
*Allegany Ballistics Laboratory, †Threshold Initiation Level

ABL ESD: Brief Overview

The ABL ESD Tester

- Selectable capacitor bank
  - Additive based on 2-dial selection
  - Minimum energy of 0.0025 Joules (0.0002μF @ 5kV)
  - Maximum energy of 9.375 Joules (0.75μF @ 5kV)
- Commonly uses a 0 of 20 TIL
- System cost: ~$100k





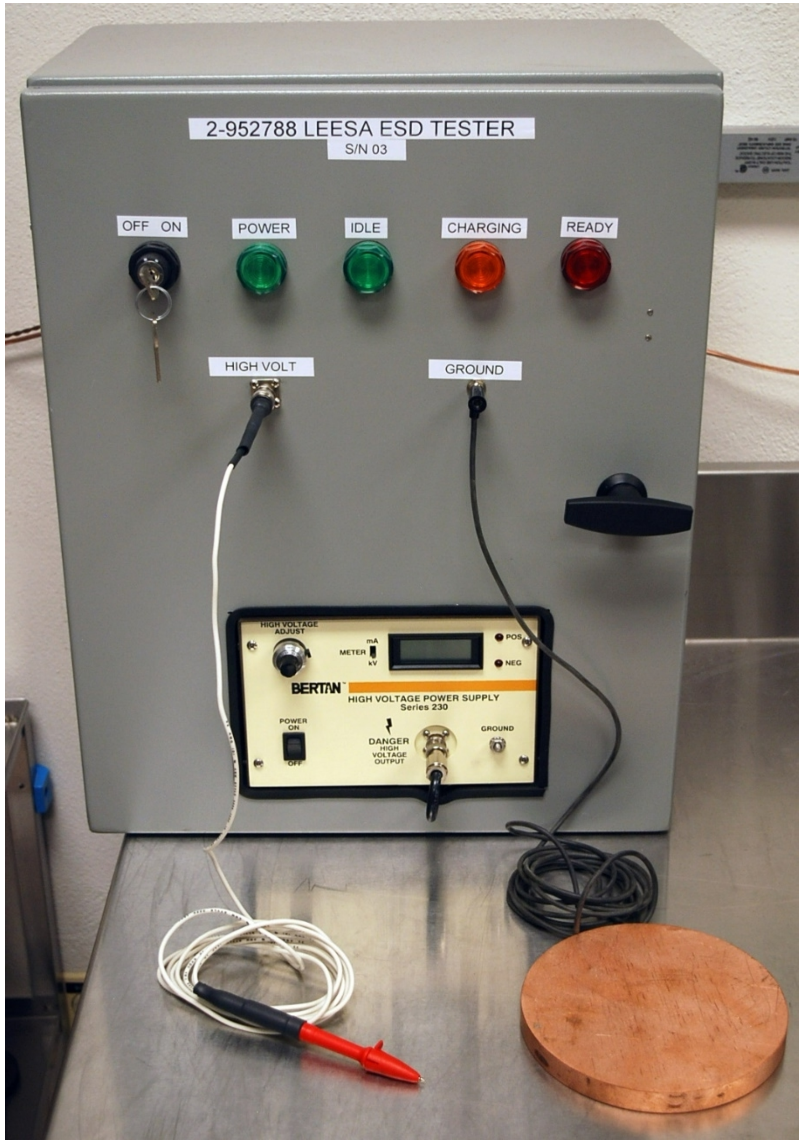
*Allegany Ballistics Laboratory

ABL ESD Energy Levels (Joules)

Capacitor (µF)	0.25	0.1	0.05	0.02	0.01	0.005	0.002	0.001	0.0005	0.0002	0.0001	0
0.5	9.3750	7.5000	6.8750	6.5000	6.3750	6.3125	6.2750	6.2625	6.2563	6.2525	6.2513	6.2500
0.25	-	4.3750	3.7500	3.3750	3.2500	3.1875	3.1500	3.1375	3.1313	3.1275	3.1263	3.1250
0.1	-	-	1.8750	1.5000	1.3750	1.3125	1.2750	1.2625	1.2563	1.2525	1.2513	1.2500
0.05	-	-	-	0.8750	0.7500	0.6875	0.6500	0.6375	0.6313	0.6275	0.6263	0.6250
0.02	-	-	-	-	0.3750	0.3125	0.2750	0.2625	0.2563	0.2525	0.2513	0.2500
0.01	-	-	-	-	-	0.1875	0.1500	0.1375	0.1313	0.1275	0.1263	0.1250
0.005	-	-	-	-	-	-	0.0875	0.0750	0.0688	0.0650	0.0638	0.0625
0.002	-	-	-	-	-	-	-	0.0375	0.0313	0.0275	0.0263	0.0250
0.001	-	-	-	-	-	-	-	-	0.0188	0.0150	0.0138	0.0125
0.0005	-	-	-	-	-	-	-	-	-	0.0088	0.0075	0.0063
0.0002	-	-	-	-	-	-	-	-	-	-	0.0038	0.0025
0.0001	-	-	-	-	-	-	-	-	-	-	-	0.0013

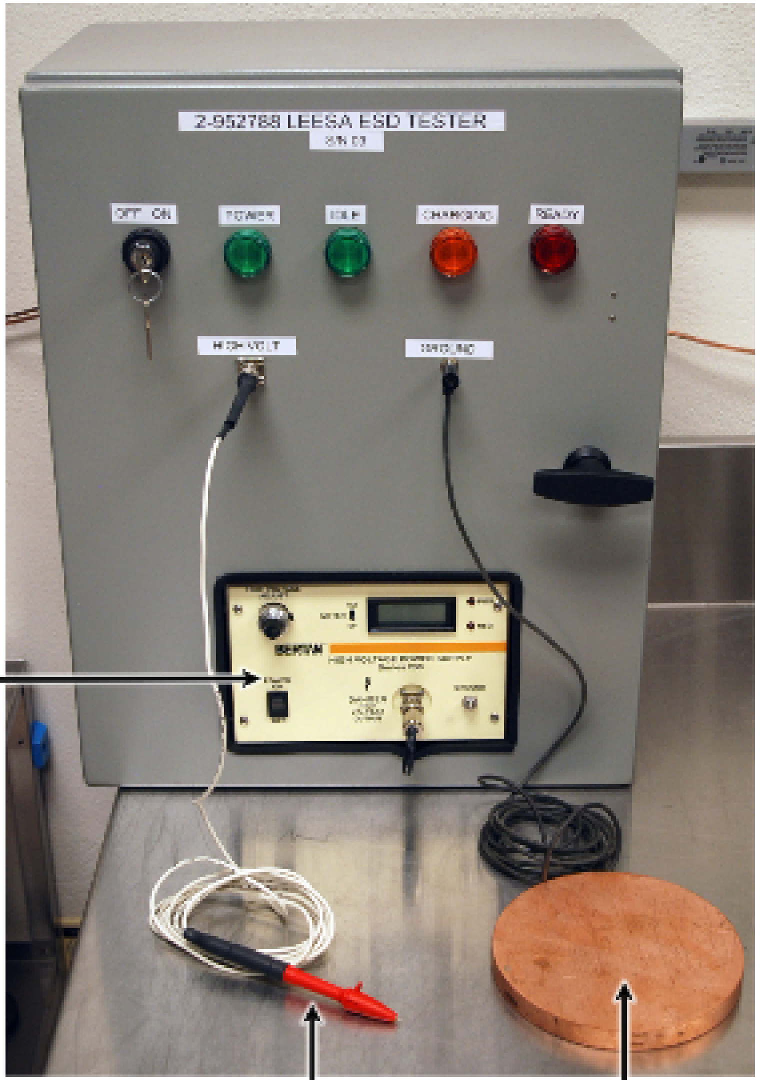
LEESA:
Brief Overview

LEESA (Low Energy Electrostatic Apparatus)



- Originally designed at Mound Laboratory in 1990[1]
  - Newly manufactured by Pacific Scientific Energetic Materials Company LLC
- Designed to test ESD-sensitive pyrotechnic powders
- Energy levels overlap with ABL machine, but go far lower
  - Minimum energy of 0.00005 Joules (10pF @ 3kV)
  - Maximum energy of 0.00743 Joules (1650pF @ 3kV)
- System cost: ~$45k

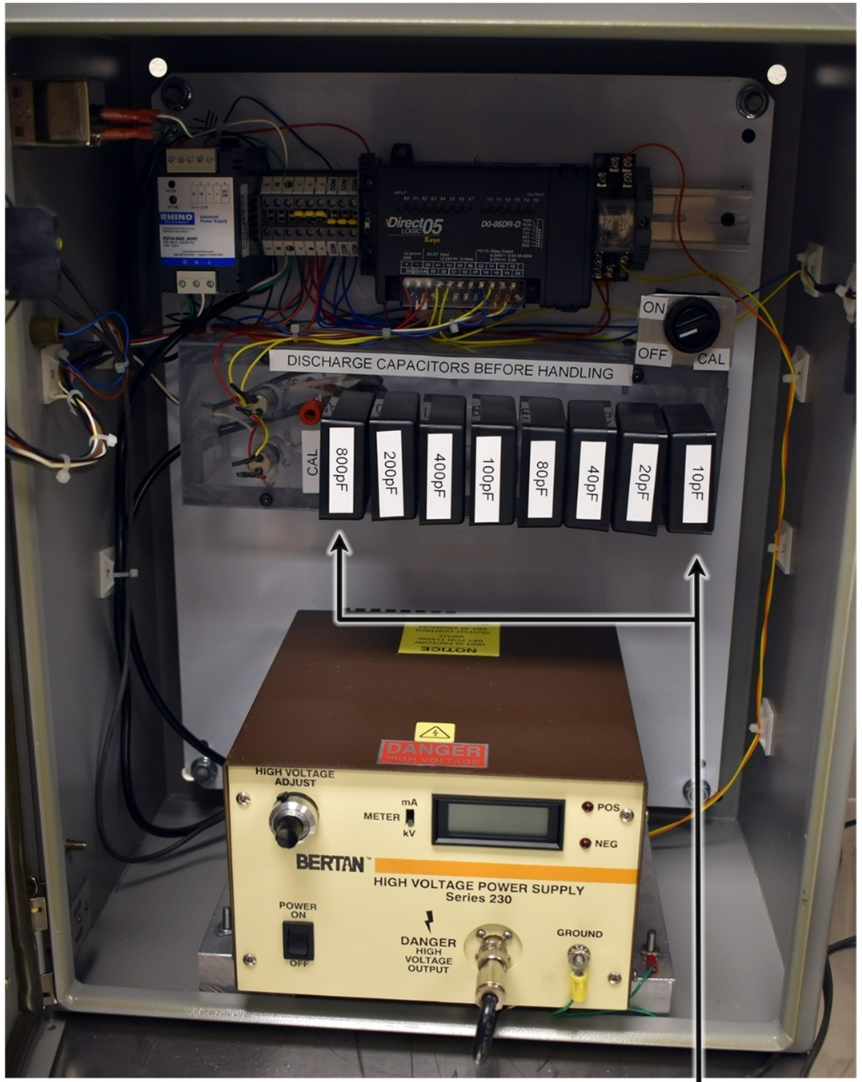
LEESA (Low Energy Electrostatic Apparatus)



HV Power Supply

HV Probe

Grounded plate



Capacitors

Foot pedal

LEESA Energy Levels*

Available capacitors (pF): 10 20 40 80 100 200 400 800

0.00743	0.00738	0.00734	0.00729	0.00725	0.00720	0.00716	0.00711	0.00707	0.00702	0.00698
0.00675	0.00671	0.00666	0.00662	0.00657	0.00653	0.00648	0.00644	0.00639	0.00635	0.00630
0.00608	0.00603	0.00599	0.00594	0.00590	0.00585	0.00581	0.00576	0.00572	0.00567	0.00563
0.00540	0.00536	0.00531	0.00527	0.00522	0.00518	0.00513	0.00509	0.00504	0.00500	0.00495
0.00473	0.00468	0.00464	0.00459	0.00455	0.00450	0.00446	0.00441	0.00437	0.00432	0.00428
0.00405	0.00401	0.00396	0.00392	0.00387	0.00383	0.00378	0.00374	0.00369	0.00365	0.00360
0.00338	0.00333	0.00329	0.00324	0.00320	0.00315	0.00311	0.00306	0.00302	0.00297	0.00293
0.00270	0.00266	0.00261	0.00257	0.00252	0.00248	0.00243	0.00239	0.00234	0.00230	0.00225
0.00203	0.00198	0.00194	0.00189	0.00185	0.00180	0.00176	0.00171	0.00167	0.00162	0.00158
0.00135	0.00131	0.00126	0.00122	0.00117	0.00113	0.00108	0.00104	0.00099	0.00095	0.00090
0.00068	0.00063	0.00059	0.00054	0.00050	0.00045	0.00041	0.00036	0.00032	0.00027	0.00023
0.00743	0.00738	0.00734	0.00729	0.00725	0.00720	0.00716	0.00711	0.00707	0.00702	0.00698
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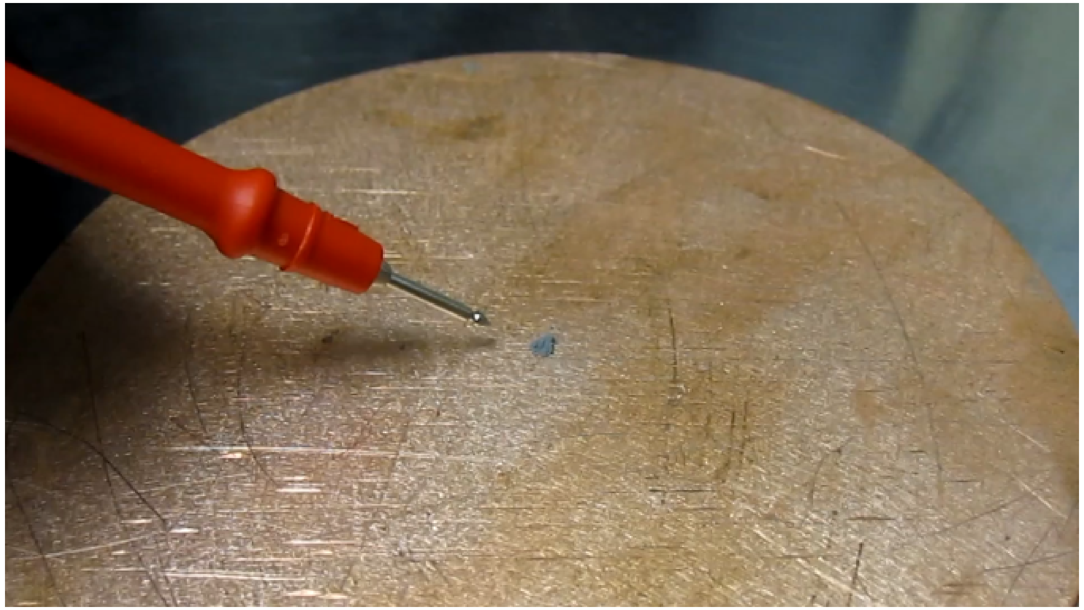
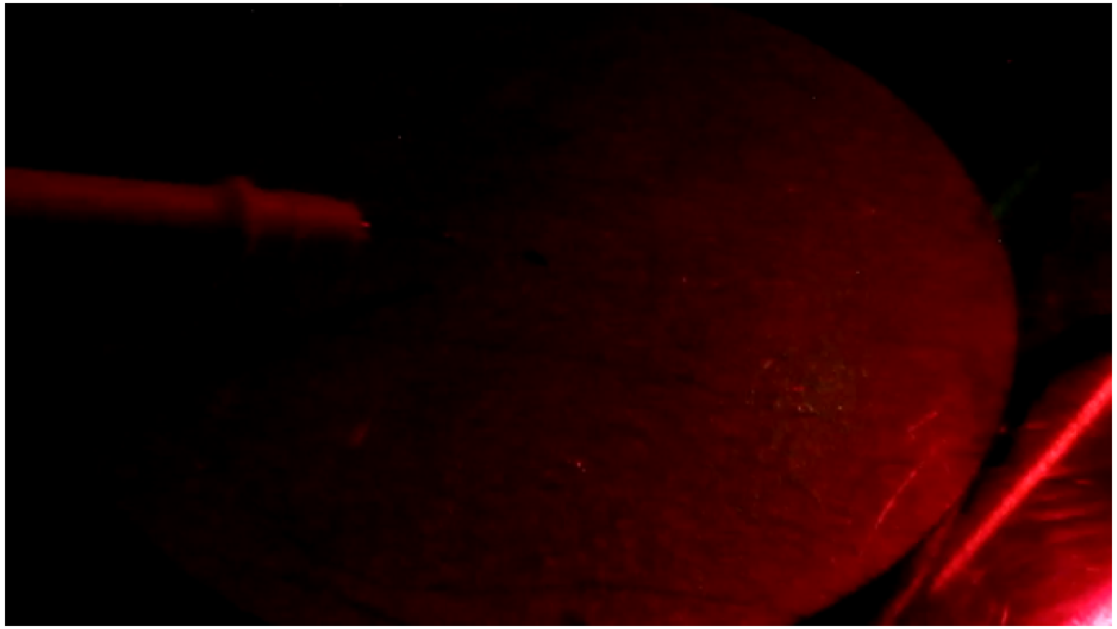
0.00540	0.00536	0.00531	0.00527	0.00522	0.00518	0.00513	0.00509	0.00504	0.00500	0.00495

*All possible sums (no repeated values) via $E = \frac{1}{2}CV^2$ at 3kV; nominal values are reported, actual capacitances will slightly vary

LEESA Testing: TKP





ABL vs. LEESA Comparison

Energy Level Comparison (Joules): ABL vs. LEESA

☐ Overlapping ABL energy levels

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0.00675	0.00671	0.00666	0.00662	0.00657	0.00653	0.00648	0.00644	0.00639	0.00635	0.00630
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0.00473	0.00468	0.00464	0.00459	0.00455	0.00450	0.00446	0.00441	0.00437	0.00432	0.00428
0.00405	0.00401	0.00396	0.00392	0.00387	0.00383	0.00378	0.00374	0.00369	0.00365	0.00360
0.00338	0.00333	0.00329	0.00324	0.00320	0.00315	0.00311	0.00306	0.00302	0.00297	0.00293
0.00270	0.00266	0.00261	0.00257	0.00252	0.00248	0.00243	0.00239	0.00234	0.00230	0.00225
0.00203	0.00198	0.00194	0.00189	0.00185	0.00180	0.00176	0.00171	0.00167	0.00162	0.00158
0.00135	0.00131	0.00126	0.00122	0.00117	0.00113	0.00108	0.00104	0.00099	0.00095	0.00090
0.00068	0.00063	0.00059	0.00054	0.00050	0.00045	0.00041	0.00036	0.00032	0.00027	0.00023
0.00743	0.00738	0.00734	0.00729	0.00725	0.00720	0.00716	0.00711	0.00707	0.00702	0.00698
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0.00540	0.00536	0.00531	0.00527	0.00522	0.00518	0.00513	0.00509	0.00504	0.00500	0.00495

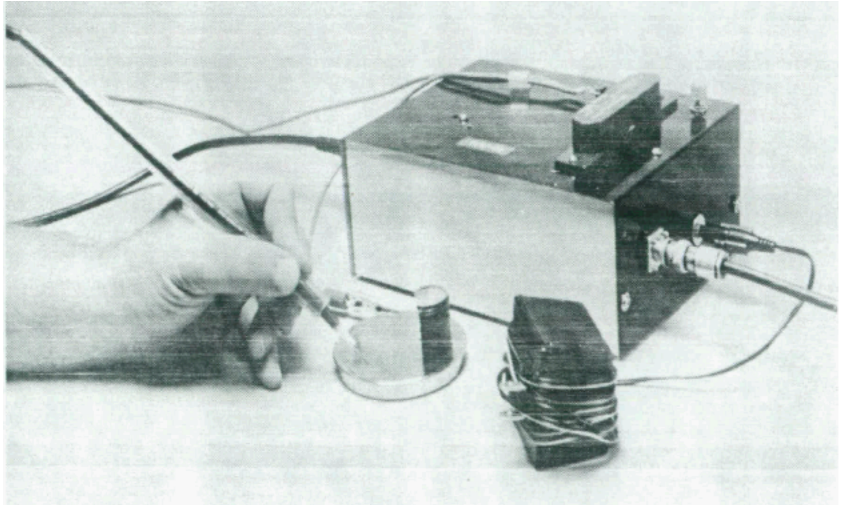
Summary: ABL ESD vs. LEESA

- ABL ESD
  - Significantly larger sample sizes (~20-60mg), volume based and highly dependent upon sample density
  - Fix gap or pneumatic approaching needle
  - No resistor in firing circuit
  - Higher energy levels (9.4 - 0.0025 Joules)
  - Integrated reaction detection options (gas analyzer, high-speed camera)
  - 5kV, typical charging voltage

- LEESA
  - Significantly smaller sample sizes (<3mg)
  - Hand-held needle probe
  - 500Ω resistor in firing circuit for "simulating body resistance"[1]
  - Lower and more numerous energy levels (0.00743 – 0.00005 Joules)
  - Operator-based detection, though others could be added
  - 3kV charging voltage

Test Method Development

Experimental Methods at Mound Laboratory[1]



- Voltage varied at a pre-selected capacitance, followed by subsequent tests at other capacitance levels
- Approx. 100-200 trials per voltage level
- % fire (ignition probability) calculated and plotted for that capacitance
- Categorized materials based on 10% ignition probably level:
  - Extremely sensitive $< 10^{-5}$ J Ex: ZPP
  - Very sensitive < 1mJ Ex: TKP
  - Sensitive < 0.1 J Ex: BCTK
  - Less Sensitive > 0.1 J Ex: $TH_{1.65}KP$
  - Relatively insensitive: B/CuO, FFFF black powder, Al/CuO, 1µm Al (spherical), $Al/KClO_4$
- Determined that plotting Probability of Ignition vs. Energy was preferred over voltage plots

Experimental Methods at PacSci[4]

- Small pile(s) placed into the sample holder (popsicle stick or Velostat) on the copper plate
- Repeated shocks with pile reconsolidated every ~5 trials if no reactions noted
- The pile is replaced after a reaction (Go)
- Signs of a Go include: Propagating reaction or total consumption (not flyers)
- Typically, ~100 trials per energy level, followed by an All Fire/No Fire analysis

Developmental Methods at SNL

- Samples loaded into templates on the copper plate
- Voltage fixed at 3kV, capacitors changed to vary energy level
- Signs of a Go include: Propagating reaction or total consumption (not flyers)
- Piles replaced if any reaction is observed
- A range of energy levels has been selected for initial testing:

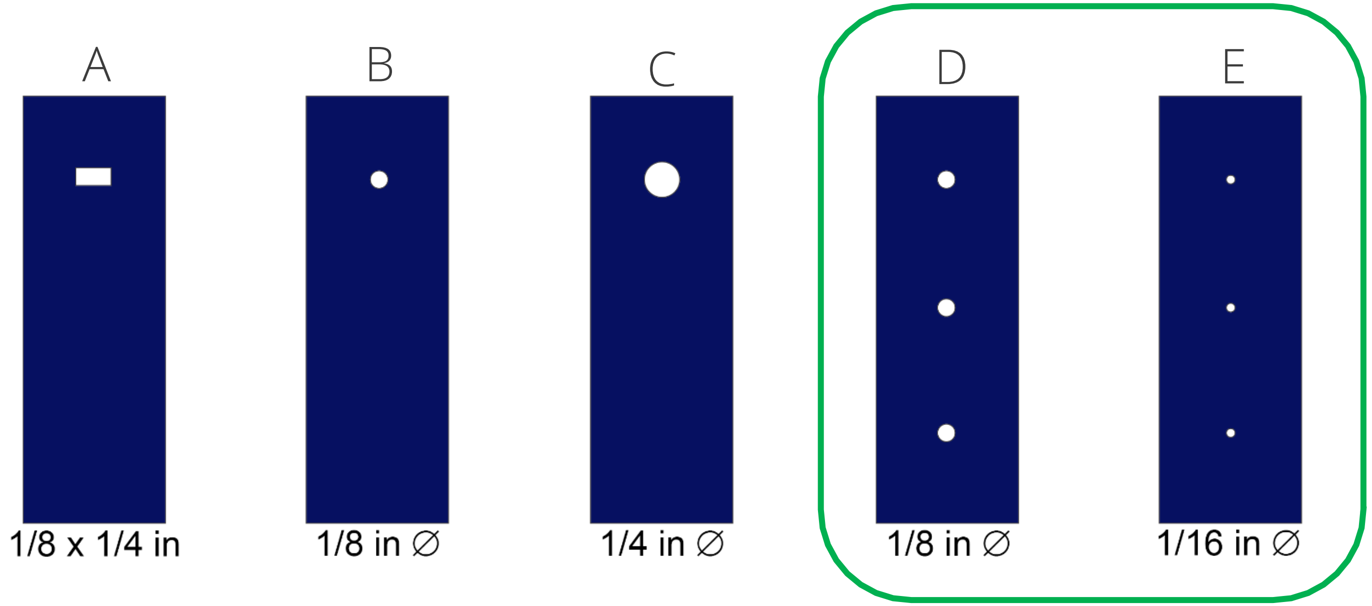
Capacitance (pF)	Required Capacitors (pF)	Ln Steps	Energy at 3000V (mJ)
1650	10 + 20 + 40 + 80 + 100 + 200 + 400 + 800	7.4	7.425
1400	200 + 400 + 800	7.2	6.300
1100	100 + 200 + 800	7.0	4.950
650	10 + 40 + 200 + 400	6.5	2.925
400	400	6.0	1.800
150	10 + 40 + 100	5.0	0.675
60	20 + 40	4.1	0.270
20	20	3.0	0.090

- Ignition Probability at each energy level is calculated
  - Probability of Total Consumption is also calculated

Sample Template Development

- Small, loose piles tend to disperse after multiple sparks, making testing increasingly difficult
- Laserable acrylic sheets were used to create templates as an alternative to wood (cleaning issues) or Velostat® (availability issues)
  - Initially, templates A,B,C were tested. B utilized the smallest sample size and did not appear to compromise the test, so D was created for subsequent tests.
  - After a trial with a primary explosive cracked template D, template E was created and used for all subsequent tests with primaries

Template Dimensions:
1 x 3 x 0.025 inches



Note: Only one hole (D,E) was filled flush for a trial. Once this hole was sufficiently damaged by reactions, a new hole was used.

What is a "Go"?

- While this has always been an issue with any sensitivity test (impact, friction, ESD), the especially small sample sizes with the LEESA exacerbate this

  - **Flyers** – Not counted by PacSci. These are commonly seen with pyrotechnics containing fine metal powders and do not always lead to a propagating reaction.

  - **Smoke** – Difficult to quantify/detect, especially if local ventilation is used.

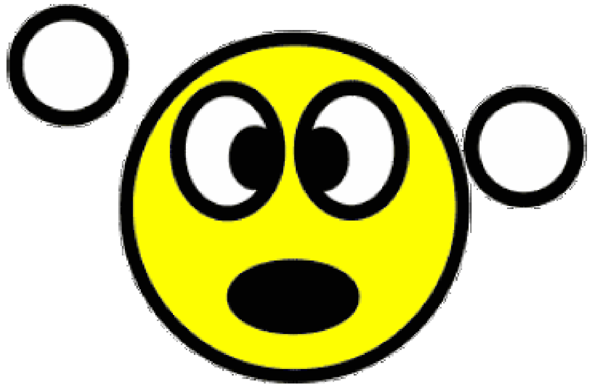
  - **Report** – Difficult to quantify/detect since sparks create reports. This is not an issue when testing primary explosives.

  - ➢ **Propagating reaction** – Results in partial sample consumption in the sample template.

  - ➢ **Total sample consumption** – The sample template is empty or only contains reaction residue.

Questions Raised

- Selection of an energy level subset for routine testing?
  - 165 possible energy levels available for the LEESA
- Criteria of a Go?
  - Same question as always
- Lights?
  - On or off? Material or energy level specific?
- How to analyze and present data?
  - % Ignition plot vs. Bruceton, SEQ, PROBIT, TIL, Neyer, etc.
- How to compare data to other ESD tests? (if at all possible)
  - ABL has no resistor in the firing circuit
  - ABL is at 5kV instead of 3kV



References

1. MLM-3652. Carlson, R., & Wood, R. (1990). Development and Application of LEESA (Low Energy Electrostatic Sensitivity Apparatus). Mound Laboratory: Miamisburg, OH USA.
2. MIL-STD-1751A (2001). Safety and Performance Tests for the Qualification of Explosives, Department of Defense.
3. SNL-SSST-20150707. Phillips, J. (2015) Overview of ESD Sensitivity Testing. Sandia National Laboratories: Albuquerque, NM USA.
4. Fronabarger, J. and Williams, M. Pacific Scientific Energetic Materials Company LLC: Personal communication, May 2021.