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LA-UR-89-431

LA-UR--89-431

DE89 007742

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SUBMITTED TO: Ninth Symposium (International) on Detonation
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ELECTROSTATIC SENSITIVITY TESTING OF EXPLOSIVES AT LOS ALAMOS

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An electrostatic sensitivity test for determining the handling hazards associated with both new and established explosives has been developed at Los Alamos and is now in routine use. The apparatus is a moving electrode device similar to that described by Kusler and Brown.[1] The energy stored in selected capacitors of a capacitor bank is discharged through the sample of explosive. A unique system of confining the samples with lead foil allows one to measure various degrees of sample response to changes in the electrostatic stimulus. Varying the foil thickness provides information about both the "sensitiveness" and the "explosiveness" of the sample. The lead-foil-confinement technique eliminates the subjective description of the response of a secondary explosive to a marginal stimulus as is common in many explosives tests on secondaries. Variables studied included: particle size, sample weight, electrode material, series resistance, temperature, voltage, sample volume, and degree of confinement.

INTRODUCTION

In any organization engaged in research and development on explosives, energetic materials, propellants, etc., it is necessary to develop small scale sensitivity tests to evaluate their hazards and establish safe handling conditions to ensure personnel safety. These small scale tests do not necessarily provide exact scientific values but rather relative ones that depend upon the testing conditions employed. We must rely on knowledgeable, experienced personnel to interpret even the relative values in any safety assessment.

It is important in any sensitivity test, especially small scale, to avoid the temptation to attribute a greater scientific content to the results than is really present.

Most of these small tests will not scale to either larger circumstances or slightly different stimuli, so their results must be used with caution. For example, the ERL drop weight impact machine does not distinguish between PBX 9404 and PBX 9501. Yet in a large scale skid test the PBX 9404 has a 50% drop height of 1.5 m while the value for PBX 9501 is 8 m. It required an accident in the UK to bring about development of the skid test.

Even the well characterized gap test, which determines shock sensitivities of explosives, can have faults. If one tests explosives with very short duration shocks, one observes differences and details that cannot be found in gap test results.[2]

Another problem with sensitivity tests is the demand for standardization. Standardization can result in many problems, accidents, blind acceptance of numbers, and neglect of important parameters, especially if one standardizes on the "wrong" test. The French ESD test indicates clearly that we have neglected some parameters in our testing.

To further complicate matters, secondary explosives are more difficult to test for sensitivity characteristics than are primary explosives. The primary explosives give clear cut responses to low level stimuli. (Yes, they explode or No, they don't.) With secondary explosives the response is proportional to the stimulus up to a point where the reaction becomes self sustaining. For example, if one hits a small sample of PETN with a hammer, it goes "bang"; if hit harder, it goes "bang" louder. Thus, in the sensitivity testing of secondary explosives one is forced to make a decision as to what level of response is significant.

DESCRIPTION OF APPARATUS AND TEST VARIABLES

The method used to determine the sensitivity of an explosive to spark initiation is, in general, to subject it to a single discharge from a condenser that has been charged to a high voltage. The energy of the discharge is varied, and by an up and down procedure, the energy producing initiation of the sample in 50% of the trials is estimated. (Bruceton method)[1]

A variable (0-15 kV) power supply is used to charge the selected condensers in a condenser bank.[4] Any total value of capacitance from 2×10^{-4} to 3 µF may be obtained by a switching arrangement that allows one to connect any of the 18 condensers in the bank in parallel.[4] The condenser output, in turn, is connected to a moving electrode device, similar to that of Brown, Kusler, and Gibson.[5] It may be described as a spring-loaded phonograph needle chuck or perhaps more simply as a single-stroke sewing machine. The apparatus is cocked, and a metal phonograph needle placed in the chuck. When the spring is released, the needle moves downward 31.75 mm (1-1/4 in.) and returns. The duration of this stroke is approximately 0.04 s. In all tests carried out so far, the needle has been positively charged, and the spark produced passes through the explosive sample to ground. By keeping the needle positive, corona losses are avoided.

The spark energy is taken to be the energy stored in the selected condensers, $E = 1/2\ CV^2$, where E is the spark energy, C the capacitance, and V the applied voltage. Fifty tests were performed in which the voltage on the condensers was measured immediately after spark discharge. The voltage was found to be anywhere from less than 10 to 390 V. The condensers had originally been charged to 5000 V in the tests. Therefore, less than 0.6% of the energy remains after discharge.

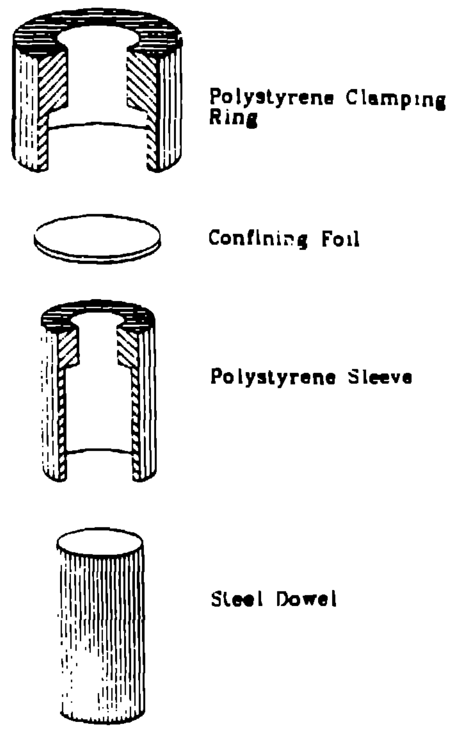

FIGURE 1. EXPLODED VIEW OF SAMPLE HOLDER

SAMPLE HOLDERS AND DEGREE OF REACTION

During preliminary experiments using this apparatus, it became evident that it was very difficult to describe the results of subjecting secondary explosives to low-energy discharges in any quantitative fashion. Therefore, special sample holders were designed that would allow the reproducible detection of a limited amount of reaction in the sample.

Such a sample holder is shown in Figure 1. A polystyrene sleeve is cemented around a steel dowel pin, leaving a space 4.76 mm diam x 6.35 mm (3/16 in. diam x 1/4 in.) high to contain the sample. A circular piece of lead foil is placed over this opening to confine the sample. The polystyrene clamping ring, which holds the foil by the outer edges, is then clamped down over the polystyrene sleeve.

In using these sample holders, the needle punctures the lead foil and a spark is discharged through the explosive sample. A "Go" (positive event) is indicated by a ruptured foil, while a "No Go" is evidenced by a punctured, but otherwise intact foil. The degree of reaction can be changed by changing the foil thickness because as one increases the confining lead foil thickness, it should take a greater degree of reaction to provide sufficient pressure build up to rupture the foil.

PETN was chosen as the material to study the variables involved in this test. Figure 2 shows the results of a number of experiments with several types of PETN and several degrees of confinement.

These results suggest that two types of reactions may be occurring:

a. In the low energy region, the amount of reaction appears to be proportional to the energy.

b. As the energy is increased beyond a certain value, the amount of reaction is no longer proportional to the energy because a self sustaining reaction occurs. The pressure from this reaction is sufficient to rupture the thickest foils tested. In fact, many of these reactions destroy the plastic part of the sample holder. Sample destruction is tabulated as per cent explosions (% Expl.).

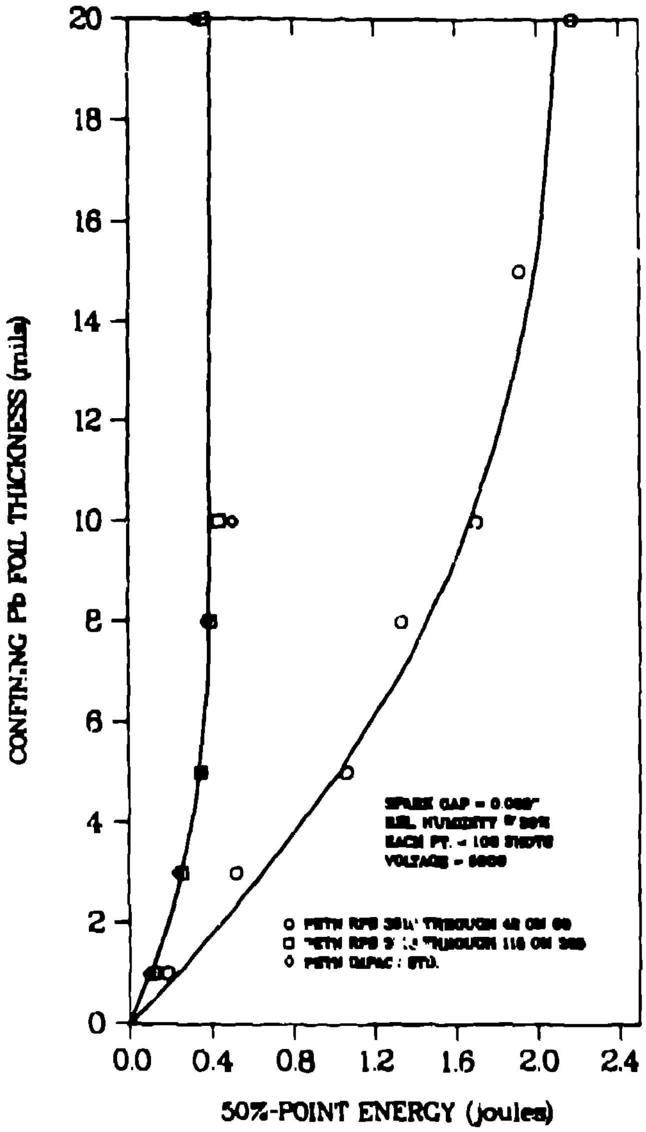

FIGURE 2

It can also be seen that while the fine PETN is only slightly more sensitive to a spark in the minimum reaction region, it is very much more sensitive than the coarse PETN in the violent region.

PARTICLE SIZE EFFECTS

In preliminary experiments, it appeared that the fines in a sample governed its sensitivity. To study this effect of the particle size of the sample in greater detail, one lot of PETN was sieved, and each fraction was tested. Table 1 shows that, as the particle size decreases, the sensitivity increases. It is interesting to note that for the minimum reaction region, the sensitivity has only increased about 1.5 times, but for the more violent reactions, the sensitivity has increased about ten fold as the particle size was decreased.

Detonator grade PETN having a large surface area appears to be slightly less sensitive than fine crystalline PETN. A possible explanation is that detonator material forms a kind of mat, and the spark is forced to take a longer more circuitous path through the material that results in a lower energy density in the spark.

TABLE 1. PETN: PARTICLE SIZE EFFECTS

Sample Particle Size[a]	50%-Point Energy (joules) 1-mil Foil	10-mil Foil	% Expl.
On 35	0.162	4.00	0
Through 35 on 42	0.150	2.42	0
Through 42 on 60	0.165	1.83	0
Through 60 on 80	0.138	1.23	0
Through 80 on 115	0.135	1.00	15
Through 115 on 325	0.098	0.408	33

[a] U.S. Standard Sieve Series
Tested with steel phonograph needles

SERIES RESISTANCE

Other investigators[b] have reported that increasing the series resistance in the circuit resulted in an increase in the sensitivity of the explosives. Their studies were carried out using only primary explosives.

A series of tests was carried out using the following explosives: PETN, RDX, HMX, Pentolite, Tetryl, and TNT. Resistances of 0.1, 0.51, 1.0, 5.1, 10, 51, and 100 kΩ have been used individually in series with the spark gap. Each of the listed explosives was tested at energies of 1, 5, and 10 J. PETN, the most sensitive explosive, was also tested at 25 J. All samples were confined with a 1 mil Pb foil. In no case did a "Go" occur. When using resistances of 51 and 100 kΩ (long RC times), an examination of the PETN samples after the spark discharge, clearly indicated that the samples were fused and that some melting had taken place.

The 50% point energies for the above materials with no added resistance range from 0.19 to 0.54 J. Thus, secondary explosives behave opposite to primary explosives in that adding resistances to the discharge circuit decreases the secondaries' sensitivities very markedly.

SAMPLE WEIGHT

A series of experiments was performed with samples of PETN that weighed 30, 40, 50, 60, 75, 100, and 110 mg. The samples were confined with 1- and 10-mil Pb foils. The sensitivity of the minimum reaction samples decreased by a factor of 2, while the severe reaction samples' sensitivity increased by a factor of ~3.5. These results were explained on the basis of two competing effects of increasing the sample weight; namely, the decrease in free volume of the container, and the greater inertia of the material over the site of the ignition. The first was presumed to predominate with the thicker foil, the second with the thin foil. This explanation was at least partially confirmed by fabricating special sample holders in which a free volume of 0.086 cm^3 was maintained above the bulk sample at each sample weight (lengthening the polystyrene sleeve). The same sample weights mentioned above were tested with the modified sample holders. The variation of the results with sample weight were reduced considerably in these tests, indicating that our explanations were confirmed, and that a constant free volume in the loaded sample holder was desirable. As a result of this set of experiments, we chose to standardize on constant volume samples.

STANDARDIZATION AND RESULTS

As a result of our studies, we chose a set of conditions that were used as a routine version of the spark sensitivity test. These conditions are listed below:

a) Tests would be run using two different foil thicknesses, a thin foil (3 mils) and a thicker foil (10 mils). The data from the thin foil confinement would be used for the evaluation of hazards, while the test using the thicker foil confinement would provide information about the severity of the reaction.

b) Brass pins would be used as the upper electrode rather than steel phonograph needles. Experiments showed that the variation in the sensitivity of PETN with sample weight is less when brass pins are used as the upper electrode.

c) Experiments with PETN have shown that its spark sensitivity is very dependent upon the particle size of the sample. Therefore, it may be necessary to specify particle size when comparing a series of explosives. On the other hand, in evaluating a material for hazards it should be tested "as received", because it is handled in this form.

d) Samples are scooped to a constant volume rather than weighed. Results in the last section indicate that maintaining a constant free volume in the sample holder results in less dependence of the sensitivity upon the sample weight.

e) A voltage of 5,000 is standard. Energy is taken as $1/2\ CV^2$ and is changed by varying the capacitance. Limited studies on PETN showed that energy, not voltage, was the important quantity.

We have tested our "impact standard" explosives over the years in our routine version of the test. Table 2 gives typical results.

TABLE 2. COMMON EXPLOSIVES

Material	50%-Point Energy (joules)		% Expl.
	3-mil Foil	10-mil Foil	
PETN (DuPont)	0.19	0.75	8
RDX (Impact Std)	0.21	0.96	0
HMX (Impact Std)	0.23	1.42	23
Tetryl (Impact Std)	0.54	3.79	42
TNT (Impact Std)	0.46	3.75	0
PYX	1.18	9.00	0
DATB (Lot 11426)	1.48	10.79	0
TATB (X 398)	4.25	18.14	0

max. human static charge ≃ 0.015 joules[1]

At Los Alamos, we use many molding powders that are pressed into large pieces. Electrostatic discharges have been thought to be less of a hazard with consolidated charges than with powdered explosives. (This may no longer be valid in view of the past year's experience with propellants.) Therefore, we prefer to test an explosive in the most sensitive form in which it is handled. In the case of many molding powders, the agglomerates or pellets are too large to fit into our sample holders. Therefore, we decided to test these materials in the form of chips and turnings from machined charges. This is actually a commonly occurring condition, since there are still facilities where these materials are machined dry. The materials were tested under our standard conditions, previously outlined. Samples were scooped to a constant volume, where possible, and yielded samples weighing 27-30 mg. Otherwise, 30 mg samples were weighed and loaded. These results are given in Table 3.

TABLE 3. MOLDING POWDERS
(MACHINED TURNINGS)

Material	50%-Point Energy (joules) 3-mil Foil	10-mil Foil	% Expl.
Pentolite	0.32	1.96	15
75/25 Cyclotol	0.38	3.29	23
PBX 9404	0.42	3.13	0
PBX 9205	0.55	1.37	42
Comp A	0.63	4.38	0
PBX 9407	0.77	1.50	50
PBX 9010	0.79	1.53	54
Octol	0.82	4.63	17
PBX 9501	0.84	2.52	78
LX-04	1.04	2.58	38
PBX 9011	1.09	2.77	33

The materials are listed in order of decreasing sensitivity as determined by the rupture of a 3 mil Pb foil. This is a minimum type of reaction. If the 10 mil Pb foil results were used, an entirely different order would result, with the RDX based explosives being the most sensitive materials.

HEATED SAMPLES

In some cases it is desirable to test materials at temperatures above room temperature. This allows one to evaluate hazards that may exist during the processing of these materials (for example, molding powders at their preheat temperatures). In this variation of the test, each sample holder is fitted with a heat reservoir, which consists of a steel block 25.4 mm diam x 19.1 mm (1 in. diam x 3/4 in.) high drilled to receive the dowel pin of the standard sample holder. The sample holder/heat reservoir assembly is heated to the desired temperature in an oven, then rapidly transferred to the firing chamber and tested. It was found that when the sample holder/heat reservoir assembly was removed from an oven at 160°C, the temperature dropped at a rate of 0.2°C/s for the first several minutes. The average time from removal from the oven to firing is about 15 s. In elevated temperature testing the polystyrene sample holders are replaced by identical Teflon holders.

Tables 4 and 5 show the results obtained when testing several common military explosives and typical DOE molding powders as a function of increasing temperature. It can be seen that the sensitivity increases somewhat as a function of temperature. The major effect appears to be the severity of the reaction when confined with a 10-mil Pb foil. The per cent explosion increases, and the degree of reaction to shatter a Teflon holder is considerably greater than that required for destruction of one fabricated from polystyrene.

TABLE 4. HEATED EXPLOSIVES

Temperature (°C)	50%-Point Energy (joules) 3-mil Foil	10-mil Foil	% Expl.
	PETN (Trojan Barrel No. 1)		
22	0.25	0.70	50[a]
50	0.24	0.78	42[b]
75	0.21	0.70	15[b]
100	0.18	0.60	42[b]
125	0.26	0.79	40[b]
	RDX (Wabash Ground)		
22	0.27	1.88	8.3[a]
75	0.18	1.05	7.7[b]
125	0.18	0.93	23.0[b]
175	0.10	0.37	92.0[b]
	HMX (98-63)		
22	0.26	1.12	75[a]
75	0.26	1.03	0[b]
125	[illegible]	0.80	0[b]
175	0.12	0.52	25[b]
200	0.125	0.36	54[b]

[a] Polystyrene holders

[b] Teflon holders

MATERIALS EXHIBITING ANOMALOUS BEHAVIOR

Since testing was begun at Los Alamos, we have found a number of materials that behave somewhat differently than our usual explosives and molding powders. Some of these are listed in Table 6.

Cedesol 10 is a double cesium nitrate salt of decaborane. It always exploded at the lowest energies we could supply from our equipment. The value of 0.0025 J is ~1/6 of the energy that can be built up on a human. In fact, we set the material off by sliding out of a chair. It also had an impact sensitivity of 4.7 cm drop height. The material behaves like a primary explosive in these two tests. It is believed to be an ingredient of Hivelite, which has been implicated in several accidents.

TABLE 5. HEATED MOLDING POWDERS

Temperature (°C)	50%-Point Energy (joules) 3-mil Foil	10-mil Foil	% Expl.
	Composition A		
22	0.63	4.38	0[a]
50	0.42	4.75	0[b]
75	0.51	6.75	0[b]
125	0.58	5.25	0[b]
	9404 (94/3/3 - HMX/NC/CEF)		
22	0.42	3.13	0[a]
75	0.33	3.25	0[b]
125	0.30	2.50	0[b]
175	0.24	1.92	25[b]
	LX-04 (85/15 - HMX/Viton)		
22	1.04	2.58	38[a]
75	0.78	2.25	0[b]
125	0.73	2.10	42[b]
175	0.65	2.15	11[b]
	9407 (94/6 - RDX/Exon)		
22	0.77	1.50	50[a]
75	0.53	1.14	0[b]
125	0.45	1.01	35[b]
175	0.43	1.02	11[b]

[a]Polystyrene holders

[b]Teflon holders

The next three materials are best classed as pyrotechnics. One of their characteristics is that once a reaction starts, the entire sample is consumed. Both the heat powder and Ti/B igniter have sensitivity values similar to human electrostatic energies.

ZPCP, BTF, and pentanitroaniline all transform into very vigorous reactions with only a slight increase of energy. This behavior is more typical of the behavior of a primary explosive and care should be exercised with these materials. The latter two materials, BTF and pentanitroaniline, have threshold sensitivities similar to that of PETN.

TABLE 6. ANOMALOUS MATERIALS

Material	50%-Point Energy (joules) 3-mil Foil	10-mil Foil	% Expl.
Cedesol 10	(same value for 0 and 1 mil)	0.0025	-
Heat Powder 88/12	0.018	0.019	---
Ti/B	0.02	(same value unconfined)	
B/KNO_3	0.23	0.32	--
ZPCP	0.31 (17% Expl)	0.40	100
BTF (HNB)	0.14	0.19	85.7
Pentanitroaniline	0.21	0.31	75
4-Nitro-1 picryl 1,2,3,1H-triazole	0.24	0.23	100
KHND	0.51	0.43	67
K Picrate	0.73	0.54	100

KHND, potassium picrate, and the triazole are anomalous in that they require less energy to cause a reaction under heavier confinement. These reactions are also much more severe than those with light confinement, as shown by the sample holder destruction. While none of three materials are unduly sensitive, one would predict that in an accident, propagation would occur that could lead to serious results.

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