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Friction, Impact, and Electrostatic Discharge Sensitivities of Energetic Materials

Pu Sen Wang and Genola F. Hall

May 31, 1985

MOUND

Miamisburg, Ohio 45342

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MONSANTO RESEARCH CORPORATION

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U. S. DEPARTMENT OF ENERGY

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Contents

	<u>Page</u>
ABSTRACT	3
INTRODUCTION	3
EXPERIMENTAL	4
Impact Sensitivity Testing	4
Friction Sensitivity Testing	5
Electrostatic Discharge Testing	6
Safety Precautions for the Tests	8
Samples	8
RESULTS AND DISCUSSION	8
Statistics and Accuracy	8
Sensitivities of Explosives	9
Sensitivities of Pyrotechnics	13
SUMMARY	15
REFERENCES	19
DISTRIBUTION	21

Abstract

Impact, friction, and electrostatic discharge sensitivities of energetic materials (explosives and pyrotechnics) used or manufactured at Mound were tested by the "one-shot" method. The Bruceton statistical method was used to derive 50% initiation levels, and the results were compared. The materials tested include: PETN, HMX, Plastic Bonded Explosives (PBX), CP, TATB, RX26BB, RX26BH, barium styphnate, LX-15, LX-16, Ti/KClO_4 , $\text{TiH}_{0.65}/\text{KClO}_4$, $\text{TiH}_{1.65}/\text{KClO}_4$, Fe/KClO_4 , $\text{TiH}_{1.79}/\text{B/CaCrO}_4$, Ti/B/CaCrO_4 , B/CaCrO_4 , $\text{TiH}_{0.65}/2\text{B}$, $\text{TiH}_{0.65}/3\text{B}$, 2Ti/B , $\text{TiH}_{1.67}/2\text{B}$, Ti/2B , $\text{TiH}_{1.67}/3\text{B}$, Ti/B , and Ti/3B . Some samples were investigated for aging effects, physical variables, and the effect of manufacturing parameters on sensitivities. The results show that in both friction and impact tests, CP and barium styphnate are the most sensitive; $\text{TiH}_{1.65}/\text{KClO}_4$, LX-15, TATB and its related materials are the least sensitive; and other materials such as PETN and HMX are in the mid-range. In the electrostatic tests of Ti-based pyrotechnics, a decrease of sensitivity with increasing hydrogen concentration was observed.

Introduction

Understanding sensitivities of energetic materials under specific initiation modes is critical not only to the material's design application, but also to the safe handling of the material during development, production, and storage. While it is essential that we understand the conditions under which the material is certain to initiate so that the application can be properly designed, it is equally important to know how an unexpected initiation can be avoided.

In planning for safe handling of energetic materials, two major approaches have to be considered: establishing the conditions necessary to prevent a premature initiation, and limiting the degree of personnel and property damage that would result if an accident were to occur. Prevention of any accident is by far the most effective safety program. Because of the wide variety of conditions under which an accident could occur during handling of highly energetic materials, simulation of all such

conditions and their various combinations in laboratory tests is unlikely. Consequently, emphasis on minimizing the damage in case of an accident should never be neglected.

A premature initiation could be caused by impact, friction, electrostatic discharge, physical shock, and heat, or a combination of these and other factors. The sensitivities of energetic materials to these factors are therefore critical values in accident prevention. In this paper, we report impact, friction, and electrostatic discharge sensitivities of some energetic materials Mound has produced or handled. These materials include: PETN, HMX, CP, TATB, RX26BB, RX26BH, plastic bonded forms of PETN and HMX (PBX), barium styphnate, LX-15, LX-16, Ti/KClO_4 , $\text{TiH}_{0.65}/\text{KClO}_4$, $\text{TiH}_{1.65}/\text{KClO}_4$, Fe/KClO_4 , $\text{TiH}_{1.79}/\text{B/CaCrO}_4$, Ti/B/CaCrO_4 , B/CaCrO_4 , $\text{TiH}_{0.65}/2\text{B}$, $\text{TiH}_{0.65}/3\text{B}$, 2Ti/B , $\text{TiH}_{1.67}/2\text{B}$, Ti/2B , $\text{TiH}_{1.67}/3\text{B}$, Ti/B , Ti/3B . Some of the tests involved investigation of aging effects and processing parameters, as well as the sensitivity dependence of other physical characteristics.

In the real world, it is impossible to separate impact, friction, and electrostatic influences in an operation. An accident most likely would occur as a result of a combination of these factors in various degrees. Consequently, the objective of this report is to compare the sensitivities of various energetic materials so that precautions can be taken to prevent an accident and the resulting damage to personnel and property.

Experimental

2.1 Testing Apparatus and Procedures

2.1.1 Impact Sensitivity Testing

(A) Impact Sensitivity Tester

A Technoproducts drop-weight tester was used for the impact sensitivity tests (Figure 1). This tester permits varying the drop-weight over 0 to 50 cm in 0.5 cm increments, and from 1 to 5 kg in 0.1 kg increments.

(B) Test Principle and Procedure

Figure 2 shows the structure of a sample holder. A sample weighing 0.020 g was placed into a 7.94 mm diameter brass cup. The cup with sample was then placed on the anvil, and the plunger tip was inserted into the cup. A weight was dropped onto the plunger from a certain height, and a positive test was indicated by explosion (for explosives) or ignition (for pyrotechnics) of the sample.

Figure 1. The Technoproducts Drop-Weight Tester

SOLID SAMPLE HOLDER

Figure 2. Configuration for a sample holder for the drop-weight tester.

With various combinations of weights and heights, impact sensitivity can be recorded in cm-kg units. For easy comparison of sensitivities of different samples, however, our tests were always dropped from the maximum height (50 cm) with various load weights.

2.1.2 Friction Sensitivity Testing

(A) Friction Sensitivity Tester

A large BAM (Bundesanstalt fur Materialprufung) tester manufactured by Julius Peters K. G., Berlin, Germany was used for friction testing (Figure 3). The tester has nine different positions on the arm

Figure 3. BAM Tester for friction sensitivity characterization

to give a weight charge from 0.5 to 36 kg. Samples were tested between a porcelain pin and a porcelain plate of standardized roughness by a path of 10 mm length (Figure 4).

Figure 4. Configuration of sample, porcelain pin, and plate in a BAM tester.

(B) Testing Procedures

A sample weighing 0.010 g was placed onto the porcelain plate which was then mounted on the slide of the friction apparatus so that the "sponge mark" was transverse to the direction of pin motion. The major part of the sample must be ahead of the pin so that it finds its way under the pin when the plate moves. The charged arm must be lowered slowly, and the pin set on the sample. After triggering the switch, an explosion (for explosives) or inflammation (for pyrotechnics) is recorded as positive; no reaction or discoloration is recorded as negative.

2.1.3 Electrostatic Discharge Testing

(A) Electrostatic Discharge Sensitivity Tester

The electrostatic discharge sensitivity tester (Figure 5) consists of a two-bay console with the test chamber, a Thelco Model 19 vacuum oven, located in the right-hand bay. The test chamber can be evacuated or pressurized. Chamber temperature can be varied from ambient to 200°C. A pulse circuit containing 500 ohms series resistance discharges a 600 pF capacitor through the sample placed on the lower electrode (a circular steel block with a 9.5 mm diameter, 0.8 mm deep depression to hold the powder) located inside the oven (Figure 6). The potential across the capacitor was varied from 0 to 50 kV.

(B) Testing Procedures

Samples weighing 0.050 g were placed into the sample container which was then placed into the tester. Operating parameters such as sample size, geometry, temperature, gap width, humidity, and electrode surface condition were kept as constant as possible. For the tests of environmental dependence, premixed gaseous atmospheres were introduced via the vacuum inlet and exit

Fig. 5. Electrostatic discharge sensitivity tester

Fig. 6. Electrode configuration for electrostatic discharge sensitivity tester

ports. A voltage potential (in kV) was chosen for the test; and again, the Bruceton method was used to increase or decrease the potential according to the firing condition.

2.2 Safety Precautions for the Tests

Since some of the tests involved development materials that had little or no background information in literature to help with safety precautions, the tests were carried out with extreme caution. All tests were performed in Cell 5, Building 27, which has a collapsible wall on the south side of the cell. Before each test, the cell was emptied of any other energetic materials. Proper signs were posted at various parts of the building to provide warnings to the occupants of other laboratories. The north door of the cell was closed, and all samples were transported through the south door. Only a limited sample quantity was allowed in the cell as determined according to its chemical properties; for example, 0.25 g maximum for barium

styphnate monohydrate. The principle investigator made sure that all the samples were in the Velostat container until the tests, all instruments were properly grounded, and all conductive equipment was set up (wrist straps, conductive footwear, safety glasses, etc.). All other personnel were cleared from the building before tests began. No test was carried out when thunderstorms were possible. Testing personnel were separated from the testers by safety shields. Ventilation pipes (12.7 cm diameter) above each tester were turned on. All "unfired" samples were destroyed by a shot under heavy load. For some very sensitive materials such as CP or barium styphnate, only wood spatulas were used.

2.3 Samples

The sources, batch numbers, and physical characteristics of samples will be described together with results in the next section.

Results and Discussion

3.1 Statistics and Accuracy

Each test was carried out 20 to 40 times, and the Bruceton statistical method was used to calculate the threshold of 50% probability for initiation [1]. A specific "charge" (weight or voltage) is used to give a one-shot test on a sample. An "x" is recorded if initiation occurs; the charge is reduced by a specific increment, and the test repeated. A failure to fire is recorded by "0". This up-and-down test is repeated 20 to 40 times until the threshold can be

determined. Although Bruceton statistics can indicate the "all-fire" and "no-fire" levels, people have taken the Bruceton test to task for its admitted inaccuracy in predicting the standard deviation [2]. In addition, Mother Nature's uncertainty and the equipment dependence of the specific test account for relatively few values reported in literature [3-12]. For our purposes of comparison among the energetic materials that we have experience with (such as PETN), the 50% probability threshold extracted from Bruceton data can be of service. Figure 7 shows an example of the Bruceton data for an LX-16 sample (ER 7364) of 96% PETN (ER 7350, 4110 cm²/g surface area) and 4% FPC 461. The 50% threshold deduced from these data is 7.7 kg for the friction test (the increment used was 0.8 kg). The uncertainty of this result is within 10%.

3.2 Sensitivities of Explosives

3.2.1 Friction and Impact

Sensitivities of PETN, HMX, and Their PBX Forms

Results of friction and impact sensitivity tests are given in Table 1. Note that the larger the charge, the less sensitive the sample is. Two PETN samples of different surface areas were tested for comparison. ER 7350, which has a surface area of 4,110 cm²/g, was found to be less friction sensitive than ER 7366 powder, which has a surface area of 31,000 cm²/g. This is expected because the higher the surface area, the stronger the friction the sample experiences in the tester. On the contrary, a lower surface area sample tends to have a larger particle size which would suffer more impact from the drop-weight test. This is just what we observed: 4,110 cm²/g (ER 7350) powder is more sensitive to impact than 31,000 cm²/g (ER 7364). Coating of ER

Weight Charge (kg)	Test Number																							
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
8.8			X					X		X										X		X		X
8.0	0		X		X		0		0		X		X		X				0		0			0
7.2					0		0						0		0		X		0					
6.4																					0			

50% probability: 7.7 kg

Fig. 7. An example of Bruceton data for friction sensitivity test (LX-16, ER 7364)

Table 1. FRICTION AND IMPACT SENSITIVITIES OF PETN, HMX AND THEIR PBX FORMS
(Weight charges for 50% initiation probability)

<u>Explosive</u>	<u>Friction (kg)</u>	<u>Impact (kg-cm)</u>	<u>Batch</u>	<u>Physical Characteristics</u>
PETN	8.1	124	ER 7350	4110 cm ² /g surface area
PETN	7.0	170	ER 7366	31000 cm ² /g surface area
LX-16 (PBX of PETN)	7.7	120	ER 7364	96 wt % ER 7350 and 4 wt % FPC 461
HMX	6.2	160	ER 5344	6320 cm ² /g surface area, before baking
HMX	6.1	176	ER 5344 -34	ER 5344 after baking at 100°C for 100 hr.
PBX of HMX	6.1	178	ER 5397	96 wt % of ER 5344-34 and 4 wt % FPC 461
PBX of HMX	7.2	170	ER 5396	96 wt % of a 930 cm ² /g HMX (ER 5393) and 4 wt % FPC 461

7350 PETN with 4 wt. % FPC 461 did not seem to change the sensitivities significantly.

The HMX powders and plastic bonded HMX (PBX) we tested show friction and impact sensitivities in the same range as those of PETN and its PBX (Table 1). No significant change was observed in friction sensitivity of 6,320 cm²/g HMX as a result of baking at 100°C for 100 hr.

The baked powders, however, seemed to be less impact sensitive than the unbaked, as baking may cause crystal-relaxation. Coating of the baked powders with 4 wt. % FPC 461 does not change their sensitivities. A different PBX (ER 5396) derived from larger crystal parents (ER 5396) does seem to have lower friction sensitivity and higher impact sensitivity.

3.2.2 Friction and Impact Sensitivities of CP, Styphnates, IATB, RX26BB, RX26BH, and LX-15

CP and barium styphnate are very sensitive to both friction and impact. Extreme care should be taken when handling either of them. The shockwave caused by CP explosive is unusually strong and damaging. Two CP samples from Unidynamics were tested (EL58633 and EL47344), and both powders were found to be much more sensitive than PETN or HMX. EL58633 powder is more sensitive than EL47344, and the reason is unknown (the manufacturing parameters were not known). Barium styphnates are almost as sensitive as CP to friction and impact. We found Sandia powders are slightly less sensitive than Chemtronics powders (Table 2).

Table 2. FRICTION AND IMPACT SENSITIVITIES OF CP, BARIUM STYPHNATE, TATB, RX26BB, and RX26BH

(Weight Charge for 50% Initiation Probability)

<u>Explosive</u>	<u>Friction (kg)</u>	<u>Impact (kg-cm)</u>	<u>Batch #</u>	<u>Physical Characteristics</u>
CP	0.9	66	EL58633	Unidynamics Powders
CP	1.3	105	EL47344	Unidynamics Powders
Barium Styphnate	1.7	113		Chemtronics
Barium Styphnate	2.0	122		Sandia (Albuquerque)
TATB	>36	>250	LLL-B-592	Lawrence Livermore Powders
RX26BB	13	>250	ER 5809	50% HMX, 50% TATB
RX26BH	>36	>250	ER 5809BH	ER 5809 blended with 0.1% graphite and 0.1% calc. stearate
LX-15	>36	>250	JS-70-022-LLL(A)	95 wt % HNS-1, 5 wt % Kel-F 800 large crystal size
LX-15	>36	>250	9113-170-01	Fine powders

On the other hand, TAIB was found to be less sensitive (in both friction and impact) than PETN or HMX. With maximum loads of our testers (36 kg for BAM and 250 kg-cm for impact) none of 40 shots fired. Mixing TATB with HMX does increase the sensitivity of TAIB, however, and thus decreases the sensitivity of HMX. RX26BB, which is a mixture of 50 wt % HMX and 50% TATB, shows a 13 kg charge requirement for 50% initiation in the friction test; although the impact test is still out of our range. Lubricating the surface of this mixture with 0.1 wt % calcium stearate and 0.1 wt % graphite reduces the friction sensitivity out of our test range again, as can be seen from Table 2.

LX-15 is composed of 95 wt % HNS-I and 5 wt % Kel-F 800. We have tested two powders of LX-15 (batch #JS-79-022-LLL(A) and 9113-170-01). The

first powder is larger in particle size. With the maximum load in our friction tester (36 kg), only 3 of 10 shots showed partial detonation. In the impact test, with maximum force of 250 kg-cm, only 2 out of 11 shots exploded. For 9113-170-01 powders, only 4 out of 11, and 4 out of 18 shots detonated with the maximum load of 36 kg and 250 kg-cm for friction and impact, respectively.

These results reflect the insensitivity of HNS. One unexpected detonation, however, occurred on October 14, 1983 in I-Building during the pressing of LX-15 pellets. This serves as a classic example of how an accident might happen.

3.2.3 Aging Effect on Friction and Impact Sensitivities for HMX

We are also interested in the aging effect on friction and impact

sensitivities of explosives and their PBX forms. Such experiments were carried out for HMX and its PBX for up to 9 weeks at 100°C in open air. The data are tabulated in Table 3 and plotted on Figure 8. From these results, it seems that accelerated aging at 100°C for up to 9 weeks does not

alter the friction and impact sensitivities in most of our samples, probably with the exception of ER 5344 (the unbaked HMX). In this case, aging causes the powder to lose its friction sensitivity, possibly through surface reduction and relaxation.

Table 3. FRICTION AND IMPACT SENSITIVITIES OF HMX AND ITS PBX AFTER ACCELERATED AGING AT 100°C

Days Aged	HMX ER-5344		HMX ER-5344-34		PBX-ER-5397		PBX ER-5396	
	Friction (kg)	Impact (kg-cm)	Friction (kg)	Impact (kg-cm)	Friction (kg)	Impact (kg-cm)	Friction (kg)	Impact (kg-cm)
0	6.2	160	6.1	176	6.1	178	7.2	170
7	8.1		6.3		6.2		6.8	
14		166		176		172		173
21	7.5		6.2		6.4		7.2	
28		170		172		168		188
35	7.4		6.1		6.3		6.7	
49	6.3		5.8		6.4		7.0	
63	6.6		6.3		6.0		6.5

Fig. 8. Aging effect on friction sensitivities for HMX and its PBX

3.2.4 Friction Sensitivity Tests and Specimen Shapes

In order to further define the effect of variables on BAM test results, the shapes of the explosive powder piles placed on the tester were investigated. For this investigation, two sample shapes (round and rectangular) of same weight (PEIN ER 4054) were tested 20 times each. No deviation in the results was found.

3.3 Sensitivities of Pyrotechnics

3.3.1 Friction, Impact, and Electrostatic Discharge Sensitivities of $TiH_x/KClO_4$ Pyrotechnics

We used $Ti/KClO_4$ (PB-98), $TiH_{0.65}/KClO_4$ (PA-70), and $TiH_{1.65}/KClO_4$ (PA-89) in a friction, impact, and electrostatic discharge sensitivity comparison, which would give the effect of hydrogen content on sensitivity.

The results are tabulated in Table 4. The tests were carried out in open air; and we had to pay special attention to the toxic chlorine products present after ignition. The ignition phenomena of pyrotechnics are quite astonishing in the fired shots: one observes large

fire-balls coupled with violent explosion when ignition occurs, as well as the smell of chlorine products of reaction. Table 4 shows that in general, all three tests show a hydrogen dependence in these sensitivities. The higher the hydrogen concentration, the safer the pyrotechnic materials are. This agrees well with our studies, which show a hydrogen diffusion before the oxygen diffusion into the fuel; this hydrogen outward diffusion requires additional activation energies for pyrotechnic reaction [13-20].

3.3.2 Electrostatic Discharge Sensitivities of TiH_x/YB

Upon the request of Sandia National Laboratories, Livermore (SNLL), eight blends of TiH_x mixed with various amounts of boron were tested for electrostatic sensitivities. The results are tabulated in Table 5. From this table we can conclude:

- (1) The higher the boron content, the less sensitive the blend.
- (2) $TiH_{0.65}$ blends are more sensitive than $TiH_{1.67}$ blends, as we expected.

Table 4. FRICTION, IMPACT, AND ELECTROSTATIC DISCHARGE SENSITIVITIES OF Ti-BASED PYROTECHNICS

50% Initiation	$Ti/KClO_4$	$TiH_{0.65}/KClO_4$	$TiH_{1.65}/KClO_4$
Batch No.	PB-98	PA-70	PA-89
Friction (kg)	6.3	7.2	15.8
Impact (kg-cm)	225	>250	>250
Electrostatic Discharge (kV)	9.5	45	>50

Table 5. ELECTROSTATIC DISCHARGE SENSITIVITIES OF TiH_x WITH VARIOUS AMOUNTS OF BORON

<u>Sample</u>	<u>Batch No.</u>	<u>No. of Tests Performed</u>	<u>50% Fine (kV)</u>
$TiH_{0.65}/2B$	AC-309-1	26	6.1
$TiH_{0.65}/3B$	AC-309-1	26	9.6
2Ti/B	AC-301-1	24	45.4
$TiH_{1.67}/2B$	AC-310-1	27	48.6
Ti/2B	AC-303-1	26	49.7
$TiH_{1.67}/3B$	AC-311-1	10	>50
Ti/B	AC-302-1	10	>50
Ti/3B	AC-304-1	12	>50

(3) To our surprise, the Ti/B blend is less sensitive than $TiH_{0.65}/2B$ and $TiH_{0.65}/3B$. This may be the result of different TiO_2 surface films in Ti and $TiH_{0.65}$.

the Fe surface to the environment, and its tendency to corrode. Results are shown in Table 6. Fe/ $KClO_4$ blends are, in general, insensitive, except FEK-017. They also have larger batch-to-batch variations.

3.3.3 Friction, Impact, and Electrostatic Discharge Sensitivities of Fe/ $KClO_4$.

3.3.4 Electrostatic Sensitivity of $TiH_x/B/CaCrO_4$ and B/ $CaCrO_4$ as a Function of Milling Time

Fe/ $KClO_4$ is a very difficult case, and results are harder to interpret, possibly because of the sensitivity of

Five B/ $CaCrO_4$ mixtures with 0, 0.5, 1, 5, and 50-hr treatment by ball milling were tested for electrostatic

Table 6. FRICTION, IMPACT, AND ELECTROSTATIC DISCHARGE SENSITIVITIES OF Fe/ $KClO_4$

<u>Batch No.</u>	<u>Fe/$KClO_4$ Ratio</u>	<u>Electrostatic Discharge (kV)</u>			<u>Friction (kg)</u>	<u>Impact (kg-cm)</u>
		<u>Open Air</u>	<u>Dry Argon</u>	<u>Vac</u>		
FEK-017	80/20	15	>50	>50	32-36	>250
FEK-018	84/16	37	>50	>50		
FEK-019	80/20	27	>50	>50		
FEK-008	84/16	30				
8400 IB-GE	84/16	43				
1254-GE	88/12	>50				

Table 7. ELECTROSTATIC SENSITIVITIES OF $\text{TiH}_x/\text{B}/\text{CaCrO}_4$ AND B/CaCrO_4 AS A FUNCTION OF BALL-MILL TREATMENTS

SAMPLE	TIME OF BALL-MILLING TREATMENT (hr)	ELECTROSTATIC SENSITIVITY (kV)
B/CaCrO ₄	0	>50
B/CaCrO ₄	0.5	42
B/CaCrO ₄	1	30
B/CaCrO ₄	5	16
B/CaCrO ₄	50	9.5
Ti/B/CaCrO ₄	-	29
TiH _{1.79} /B/CaCrO ₄	-	>50

sensitivity. The results show >50, 42, 30, 16, and 9.5 kV, respectively, were required for 50% ignition probability. An increase in sensitivity occurred with increased milling time.

These results are tabulated in Table 7 and plotted in Figure 9. Results of Ti/B/CaCrO₄ and TiH_{1.79}/B/CaCrO₄ were also listed.

Figure 9. Plot of electrostatic discharge sensitivities of B/CaCrO₄ vs. sample milling times.

Summary

Impact, friction, and electrostatic discharge sensitivities of energetic materials used or manufactured at Mound were tested by the "one-shot" method, with 50% initiation levels derived by Bruceton statistics from 20-40 shots tested for each sample. In some cases, the results were found to be dependent on sample characteristics and process parameters. The relative sensitivities obtained in this project, however, are valid only for comparison purposes for the specific samples tested. The results are plotted in Figures 10-12 for friction, impact, and electrostatic discharge sensitivities.

Figure 10. Comparative friction sensitivities of the energetic materials tested at Mound. (Note: Different sample parameters may give different results.)

Figure 11. Comparative impact sensitivities of the energetic materials tested at Mound. (Note: Different sample parameters may give different results.)

Figure 12. Comparative electrostatic discharge sensitivities for pyrotechnic materials tested at Mound. (Note: Different sample parameters may give different results.)

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