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SMALL-SCALE EXPLOSIVITY TESTING

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ABSTRACT

In the area of energetic materials testing, correlation of small- and large-scale test results is a frequently sought and seldom achieved goal. We have experimented using the cartridge test to obtain a relative ranking of the explosivity of energetic materials. The cartridge test attempts to detonate a 2 gram sample of energetic material confined in a .303" brass cartridge case with a number 8 blasting cap. Violence of an event was judged by the weight of the main body of the casing remaining attached to the base after detonation.

INTRODUCTION

Testing performance of energetic materials must be done on large-scale (100-1000lb) for reasons involving run-up-to-detonation and critical diameter. For low-energy materials, such as gun powders or ammonium nitrate formulations, confinement issues become critical. Nevertheless, the scientist working in energetic material formulation or testing frequently has a need for a quick small-scale performance evaluation. This is

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especially true when only small quantities of the material are available. Therefore we have adapted and tested a explosivity test developed by the British.^{1,2}

EXPERIMENTAL SECTION

Brass .303" cartridge cases (one hole in base) were purchased from Value Bullet Comp., Pudsey, West Yorkshire, UK. Each case was weighed before 2g of energetic material was packed into it. Since each material has a different density, the volume occupied by each fill in the cartridge varied. This is an experimental difficulty discussed below. The height of the material from the open end of the cartridge was recorded, and a detonator was inserted on top of the material and taped into place. For the tests described below, number 8 detonators were used. These contain 19 mg lead azide, 100 mg lead creosol, and 450 mg PETN. [Previous studies have used number 6 detonators,^{1,2} but these are not available in North America. For tests where we wish to perform chemical analysis on the explosive residue, smaller detonators (RP3) containing only 28 mg of PETN, were employed. In such tests scrupulous cleaning of the detonation chamber and initial sampling of it are necessary.] In our laboratory, the detonation chamber is a heavy-walled, bolted-closure, stainless-steel vessel (Figure 1). The lid was held on with eight bolts; the detonation gases were vented to the outside through small holes in the lid. The .303" brass cartridge is tapered, with the base thicker-walled than the open end. To calibrate explosivity, after initiation of the energetic material, the metal fragments were removed from the detonation chamber, and the cartridge base only was washed with water and acetone, dried, and weighed. The violence of the event was assessed by the weight of

metal still attached to the cartridge base. We made no attempt to follow the subjective observations used by previous researchers.^{1,2}

RESULTS

Since setting off the detonator, even in an empty cartridge, would result in some damage to the casing, it was necessary to calibrate "no response." For this baseline test, the cartridge was filled with 2 g of the NaCl. To compare to the response of a high-explosive 2g of HMX was used. Previous researchers simply reported the weight of base remaining.^{1,2} For HMX they reported 2.21 g of base remaining; we find 2.22g or 2.04g of base remaining, depending on the batch of .303" cartridges used (Table I). Because the initial weight of the cartridges vary slightly (see column 2, Table I), we think more meaningful than reporting base weight remaining is reporting the fraction of base remaining. In Table I, column 7 shows the fraction of base remaining after initiation, column 8, the average fraction of base remaining; and column 9 the standard deviation in that average. Reproducibility among duplicate or triplicate tests was high; standard deviation was usually less than 0.02. However, for small sample sets (less than 20), the confidence factors depend not only on the confidence interval but also on the number of samples.³ Using Student's t factors for a 95% confidence limit, the uncertainties in column 10 were calculated.

The results of all cartridge tests are shown in Table I. After initiation of the detonator in NaCl about 0.60 of the cartridge remained attached to the base. With HMX only 0.20 of the original case weight remained attached to the base. Column 11 in Table I expresses the results in terms of explosive power of the test material relative to HMX, where the power of HMX is set at 100%. The meaning of this number may be a little more intuitively obvious than the fraction of base remaining.

DISCUSSION

The results of eight-five cartridge tests are shown in Table I. Inert NaCl (60% case intact) and high explosive HMX (20% case intact) define the bounds of the expected result. A number of smokeless powder, improvised explosives, and ammonium nitrate formulations were examined. Bullseye (tests 10-16), with 40% nitroglycerin (NG) and nitrocellulose (NC) in its composition was the most powerful material examined, other than HMX. With Bullseye only 27% of the casing remained together. In contrast, Pyrodex (tests 17-19), a black powder substitute using chlorate, left 43% of the case intact. Not surprisingly, improvised formulations of perchlorate (tests 20-22) or chlorate (tests 23-28) and table sugar produced similar results 42-41% of the case intact.

A number of ammonium nitrate (AN) formulations were examined (Table I). The differentiation in cartridge-case destruction among AN formulations with fuel and those without was clear. Those with fuel (tests 29-43) left on average 39% of the cartridge attached to its base, while ammonium nitrate left on average 45% of the cartridge attached.

Since “inert” NaCl left 60% of the case intact, it is clear at this end of the scale there is not a clear differentiation among 10-50% AN formulations. However, from Table II it can be seen that as the amount of energetic (AN) decreased, the percent of cartridge remaining attached increased. Tests 83-85 (Table I) examined the performance of a 2/1 mixture of $\text{Ca}(\text{NO}_3)_2 / (\text{NH}_4)_2\text{HPO}_4$ which left 63% of the case intact. This was comparable to a mixture (tests 53-55) which stoichiometrically should have been identical 2/1 AN/Ca HPO_4 which left 62% of the case intact. Further discussion of the AN formulations can be found in references 4 and 5.

As with most small-scale explosive tests, a number of objections can be raised. These involve the concept of critical diameter and initiation issues. High explosives such as HMX have critical diameters less than 1 mm,⁶ whereas ammonium nitrate has a cited critical diameter about 13 cm.⁷ Below its critical diameter a material does not propagate a detonation wave; applying a detonator to such a material may scatter it, but does not cause a detonation of the material. Confinement decreases critical diameter, but the extent to which it decreases it is not calibrated. The mass of the energetic material in the cartridge was held constant at 2 g, but since the samples did not have identical densities the depth to which the detonator protruded into the cartridge varied from 3.5 mm to 2.4 mm. Since the deeper the detonator was placed in the cartridge, the more metal would be blasted free of the base, this could result in systematic variations in the results, but we found no way to remedy this problem. A second problem was that since only 60% of the casing remained attached to the base when the test material was an inert, the differentiation between a good

explosive and a weak one was reduced. A possible solution would be to use smaller detonators. Reducing the amount of explosive in the detonator would reduce the damage to the cartridge/inert system, expanding the low-reactivity end of the test. However, an energetic material which is marginally detonable, especially if it is used in small amounts, is sensitive to the power of the initiation pulse. Decreasing the power of the detonator would increase the likelihood that insensitive explosives like ammonium nitrate-based materials would not propagate the detonation wave. For this reason and for cost reasons number 8 detonators were used in all explosivity studies. Initial results suggest the cartridge tests will be quite useful in comparing the power of various formulations and testing the explosivity of an unknown material.

CONCLUSIONS

A laboratory-scale explosivity testing device was found useful in estimating the power of explosive samples. The "cartridge" testing device correlates explosivity with the amount of cartridge remaining attached to its base after 2g of sample had been detonated in it. This study shows a general correlation between the power of a formulation and the amount of casing left attached to its base. Samples containing a high explosive such as Bullseye or HMX left only 27% to 20%, respectively, of the case intact. Improvised explosives, such as sugar chlorate mixtures and ammonium nitrate with fuel, left 44%-35% of the casing attached. Mixtures of AN without fuel and other mixed oxo-salts left 58% to 70% of the casing attached, comparable to the

response of "inert" NaCl (60% casing attached). The test would be more useful for low-power formulations if the amount of the base remaining for the control were larger, i.e. if more than 60% of the case remained attached to the base upon the "initiation" of inert NaCl. One way to lower the degree of fragmentation with the inert is to reduce the size of the initiating charge. Lowering the size of the initiating charge must be balanced against achieving initiation of the sample. Our investigations of this technique are ongoing.

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Table I: Cartridge Test Results

#	303 Brass wt	Tested Material	Sample Weight (gm)	Depth (cm)	Base wt (g) Remaining	Fraction Base Remaining	Average Fract. Base Rem.	Std. Dev.	±1.95% Confide	% HMX power
1	10.947	NaCl	2.000	3.3	6.543	0.60				
2	10.999	NaCl	2.000	3.3	6.428	0.58				
3	10.815	NaCl	2.029	3.2	6.589	0.61	0.60	0.01	0.05	34%
4	10.911	HMX	2.010	3.2	2.214	0.20				
5	10.945	HMX	2.017	3.2	2.203	0.20				
6	10.920	HMX	2.041	3.2	2.248	0.21	0.20	0.00	0.01	100%
7	11.192	HMX (BC2)	2.006	3.2	2.000	0.18				
8	11.122	HMX (BC2)	2.006	3.2	1.951	0.18				
9	11.257	HMX (BC2)	2.006	3.2	2.172	0.19	0.18	0.01	0.04	112%
10	11.011	Bullseye	2.002	1.6	3.278	0.30				
11	10.953	Bullseye	2.003	1.5	2.678	0.24				
12	10.979	Bullseye	2.001	1.6	2.841	0.26				
13	11.014	Bullseye	2.007	1.4	3.674	0.33				
14	11.034	Bullseye	2.005	1.5	2.590	0.23				
15	11.020	Bullseye	2.001	1.5	2.940	0.27				
16	11.019	Bullseye (NC+NG+EtCentralite+Rosin)	2.009	1.5	2.873	0.26	0.27	0.03	0.08	75%
17	11.110	HODGDON Pyrodex	2.008	1.6	4.880	0.44				
18	11.079	HODGDON Pyrodex	2.005	1.6	4.725	0.43				
19	10.940	HODGDON Pyrodex	2.008	1.6	4.756	0.43	0.43	0.01	0.03	47%
20	10.942	71.5% KClO ₄ + 28.5% sucrose	2.005	3.5	4.659	0.43				
21	10.881	71.5% KClO ₄ + 28.5% sucrose	2.006	3.5	4.536	0.42				
22	11.030	71% KClO ₄ + 29% sucrose	2.005	3.5	4.688	0.43	0.42	0.00	0.02	48%
23	10.986	71.35 % NaClO ₃ + 28.65 % sucrose	2.002	3.3	4.636	0.42				
24	10.980	71.35 % NaClO ₃ + 28.65 % sucrose	2.004	3.1	4.557	0.42				
25	11.028	71 % NaClO ₃ + 29% sucrose	2.002	3.3	4.701	0.43	0.42	0.01	0.02	48%
26	11.039	74.14% KClO ₃ + 25.86 %sucrose	2.003	3.3	4.801	0.42				
27	11.028	74.14% KClO ₃ + 25.86 %sucrose	2.003	3.3	4.651	0.42				
28	10.995	74% KClO ₃ + 26% sucrose	2.003	3.3	4.404	0.40	0.41	0.01	0.05	49%

Table I: Cartridge Test Results (cont.)

#	303 Brass wt	Tested Material	Sample Weight (gm)	Depth (cm)	Base wt (g) Remaining	Fraction Base Remaining	Average Fract. Base Rem.	Std Dev	+1.95% Confide	% HMX power
29	10.884	AN + 5%MO	2.021	2.7	4.625	0.42				
30	10.969	AN + 5%MO	2.005	2.7	4.620	0.42				
31	10.871	AN + 5%MO	2.005	2.7	4.576	0.42	0.42	0.00	0.01	48%
32	11.321	94%AN+6% FO (BC2)	2.005	2.7	5.443	0.49				
33	11.256	94%AN+6% FO (BC2)	2.001	2.7	4.733	0.42				
34	11.180	94%AN+6% FO (BC2)	2.000	2.7	4.551	0.40	0.44	0.05	0.21	46%
35	11.011	90%AN+10% Icing sugar (BC2)	2.004	2.6	3.768	0.34				
36	11.388	90%AN+10% Icing sugar (BC2)	2.003	2.6	4.122	0.36				
37	11.278	90%AN+10% Icing sugar (BC2)	2.001	2.6	3.793	0.34	0.35	0.01	0.06	59%
38	10.991	96.5%(79%AN+Ca/MgCO3) + 3.5% MO	2.004	3	3.940	0.36				
39	11.040	96.5%(79%AN + Ca/MgCO3) + 3.5% MO	2.003	2.9	4.054	0.37				
40	10.984	96.5%(79%AN + Ca/MgCO3) + 3.5% MO	2.002	3.1	4.024	0.37	0.36	0.00	0.02	56%
41	10.846	87.7%(79%AN + Ca/MgCO3) +12.3% sucrose	2.005	2.4	4.236	0.39				
42	11.030	87.7%(79%AN + Ca/MgCO3) +12.3% sucrose	2.003	2.4	4.328	0.39				
43	10.775	88%(79%AN + Ca/MgCO3) +12.3% sucrose	2.004	2.5	4.033	0.37	0.39	0.01	0.04	53%
44	10.839	AN	2.001	2.3	4.628	0.43				
45	10.948	AN	2.001	2.3	4.913	0.45	0.44	0.02	0.20	46%
46	10.981	79%AN +Ca&MgCO3	2.009	2.8	5.878	0.54				
47	10.920	79%AN +Ca&MgCO3	2.007	2.9	4.822	0.44				
48	11.014	79%AN +Ca&MgCO3	2.003	2.7	5.138	0.47				
49	11.674	79%AN +Ca&MgCO3	2.006	2.6	5.072	0.43	0.47	0.05	0.15	43%
50	10.969	2:1 AN/CaCO3	2.009	2.9	7.230	0.66				
51	10.997	2:1 AN/CaCO3	2.007	2.9	6.855	0.62				
52	10.962	2:1 AN/CaCO3	2.009	2.9	6.892	0.63	0.64	0.02	0.08	32%
53	11.018	2:1 AN/CaHPO3	2.007	2.9	7.083	0.64				
54	10.966	2:1 AN/CaHPO3	2.006	3	6.444	0.59				
55	10.953	2:1 AN/CaHPO3	2.008	3	6.772	0.62	0.62	0.03	0.12	33%

FO = fuel oil; MO = mineral oil

Table I: Cartridge Test Results (cont.)

#	303 Brass wt	Tested Material	Sample Weight (gm)	Depth (cm)	Base wt (g) Remaining	Fraction Base Remaining	Average Fract. Base Rem.	Std Dev	±0.5% Confide	% HMX power
56	10.826	9:1 AN /NaNO3	2.006	2.7	6.025	0.56				
57	11.008	9:1 AN /NaNO3	2.006	2.6	6.427	0.58				
58	10.941	9:1 AN /NaNO3	2.005	2.7	6.086	0.56	0.57	0.02	0.07	36%
59	11.021	1:1 AN /NaNO3	2.005	2.9	7.083	0.64				
60	11.017	1:1 AN /NaNO3	2.007	2.9	6.765	0.61				
61	10.933	1:1 AN /NaNO3	2.006	2.9	6.268	0.57	0.61	0.03	0.15	33%
62	10.959	1:9 AN /NaNO3	2.006	3.1	6.627	0.60				
63	11.075	1:9 AN /NaNO3	2.007	3.2	6.274	0.57				
64	10.936	1:9 AN /NaNO3	2.009	3.1	6.359	0.58	0.58	0.02	0.08	35%
65	11.022	1:1 AN /ASO4	2.002	2.6	6.626	0.60				
66	11.018	1:1 AN /ASO4	2.002	2.7	6.572	0.60				
67	11.005	1:1 AN /ASO4	2.003	2.6	6.390	0.58	0.59	0.01	0.05	34%
68	10.847	1:1 AN /Al2O3	2.004	2.8	7.128	0.66				
69	11.071	1:1 AN /Al2O3	2.005	2.8	7.412	0.67				
70	10.967	1:1 AN /Al2O3	2.005	2.8	7.015	0.64	0.66	0.02	0.06	31%
71	11.016	30 mol % AN + 10 mol % AS + 30 mol % CaCO3 + 30 mol % Urea	2.004	2.3	7.870	0.71				
72	10.993	30 mol % AN + 10 mol % AS + 30 mol % CaCO3 + 30 mol % Urea	2.002	2.3	7.308	0.66				
73	10.945	30mol% AN + 10mol% AS + 30mol% CaCO3 + 30mol% Urea	2.007	2.3	7.331	0.67	0.68	0.03	0.12	30%
74	11.035	30% AN + 10% AS + 30% CaCO3 + 30% urea	2.003	2.2	7.857	0.71				
75	10.998	30% AN + 10% AS + 30% CaCO3 + 30% urea	2.002	2.2	7.474	0.68				
76	10.967	30% AN + 10% AS + 30% CaCO3 + 30% urea	2.003	2.2	7.811	0.71	0.70	0.02	0.08	29%
77	11.009	10% AN + 30% urea + 30% CaCO3 + 30% ASO4	2.003	2.3	6.549	0.59				
78	11.000	10% AN + 30% urea + 30% CaCO3 + 30% ASO4	2.002	2.3	6.998	0.64				
79	10.917	10% AN + 30% urea + 30% CaCO3 + 30% ASO4	2.002	2.4	7.552	0.69	0.64	0.05	0.21	32%
80	10.912	10% AN + 30% urea + 30% Aph + 30% CaCO3	2.006	2	7.628	0.70				
81	10.878	10% AN + 30% urea + 30% Aph + 30% CaCO3	2.005	2.1	7.502	0.69				
82	11.043	10% AN + 30% urea + 30% Aph + 30% CaCO3	2.005	2.1	7.325	0.66	0.68	0.02	0.08	30%
83	11.001	1:1 Ca(NO3)2 / (NH4)2HPO4	2.005	2.7	6.993	0.64				
84	11.050	1:1 Ca(NO3)2 / (NH4)2HPO4	2.009	2.6	7.253	0.66				
85	10.915	1:1 Ca(NO3)2 / (NH4)2HPO4	2.001	2.6	6.448	0.59	0.63	0.03	0.14	32%

sucrose = table sugar (C12H22O12); AN= (NH4)NO3; AS =(NH4)2SO4; Aph = (NH4)2HPO4

Table II
Ammonium Nitrate Diluted with Additives

% AN	% casing attached
94% + fuel	39%
100%	45%
90%	56%
67%	62%
50%	62%
30%	69%
10%	68%

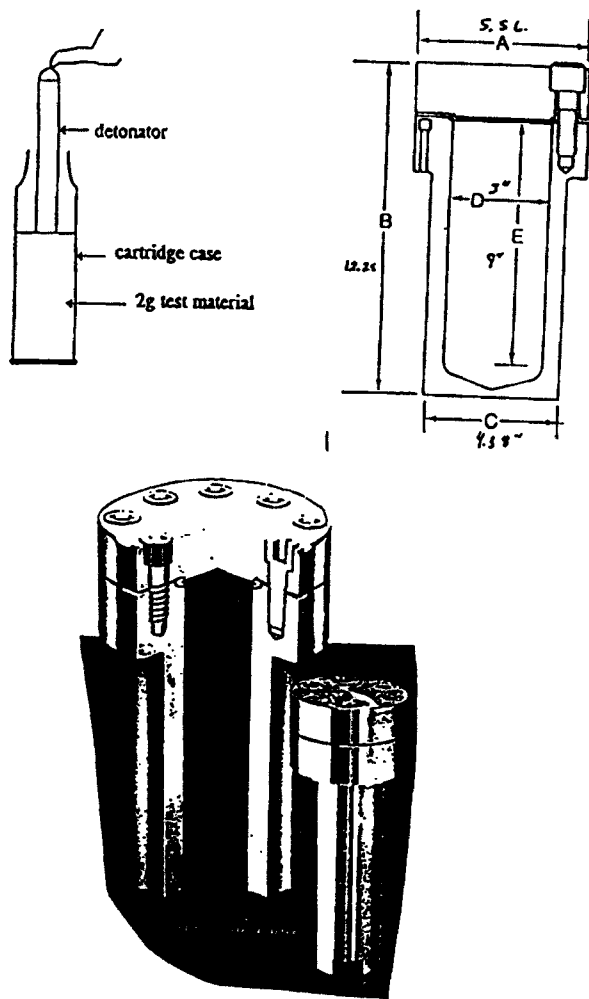


Figure 1 Schematic of the Cartridge Test Apparatus