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Developing small-scale tests to predict explosivity

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Abstract Materials which release significant heat upon decomposition are energetic materials. Some of these are also explosives. Seeking a correlation with detonability of large quantities of energetic materials, four laboratory tests were used. The characteristics considered indicative of detonability were ability to fragment a metal casing, when initiated by a detonator, and ability to produce large quantities of gas and heat. The best developed of these tests is differential scanning calorimetry. It has already been pioneered by other researchers. A limitation of this study is that large-scale detonability remains unknown for a number of materials examined; thus, it is difficult to sufficiently evaluate the success of the small-scale analyses.

Keywords Explosives · Prediction · DSC · Hazards

Introduction

The general criteria for an energetic material to be explosive are that in addition to release of heat it also releases gas, the working fluid; and these releases happen extremely rapidly, fast enough to support a detonation front. Because support of a detonation front is scale-dependent (vide infra), it is difficult to know if a raw material or intermediate is explosive at the size, then it is commonly handled and stored. Recognizing potential explosivity is of great concern to chemical manufacturers as well as to those concerned with counterterrorism initiatives.

To mitigate the potentially severe consequences from the inadvertent or purposeful release of this potential energy, one must employ adequate layers of protection and safe operating procedures. All the aspects of safety require that chemicals with explosive potential be recognized to prevent a large-scale detonation. Perhaps, if the explosivity of un-fueled ammonium nitrate had been adequately appreciated, then the accident in Toulouse (France) would have been avoided [1].

Without testing, it is difficult to know whether a compound is explosive or not. Scale is an important factor when it comes to assessing degree of hazard. Explosives have a critical diameter, the minimum diameter at which a charge of explosive can sustain a high-order, steady-state detonation. When a forward-traveling shock wave hits the sides of the charge, the shock wave is reflected as water hitting the wall of a swimming pool. The bounced back waves (rarefaction waves) degrade the original shock wave so that at such edges the shock waves are slowed and an overall curvature to the wave develops. If the width of the explosive is narrow, then the rarefaction waves may be sufficient to kill the shock wave entirely; this is the critical diameter (Dcr) or failure diameter. The Dcr of a material can vary significantly with changes in its density, particle size, confinement, and initial temperature of the sample [2].

Rarely do chemicals undergo hazards testing on the scale that they will be prepared and stored. For example, a compound or formulation may pass the United Nations (UN) and U.S. Department of Transportation series 1 tests (required for screening for potential explosivity), but still be detonable at scales larger than the one or two pounds required to be tested in series 1 [3]. Critical diameter, relative importance of side reactions, and the inability of bulk samples to effectively dissipate heat mean that while a small sample may be safe, a large one is detonable. While

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the best way to determine if one ton of material is detonable is to test one ton of the material, the approach is not practical, economic, nor safe. Furthermore, if one ton of the material proves non-detonable, this gives no assurance about 10 tons of that material.

The objective of this study is to determine whether there are small-scale test results which can be correlated with large-scale explosivity. To that end, four lab-scale tests were chosen to evaluate explosivity. These tests evaluate heat release, differential scanning calorimetry (DSC), gas released upon slow and fast decomposition, and the response to the input of a shock wave.

Experimental section

Military explosives were obtained from government laboratories. Reagent chemicals were purchased from Sigma-Aldrich. All were used without further purification.

Manometer

A small sample (0.5–1.5 mg) was weighed into one end of a sealed glass capillary tube (1.5–1.8 × 90 mm), and the other end was flame sealed. The sealed capillary tubes were heated at defined temperatures 200 or 300 °C for 2–3 days. After heating, the capillary tube was cooled to ambient temperature and broken in a mercury manometer. The ambient temperature and pressure (P) and the volume (V) of gas released were recorded. Number of moles (n) of gas released was calculated using the ideal gas equation: $n = PV/RT$ (where T is temperature in Kelvin and R is the ideal gas constant)

Differential scanning calorimetry

The DSC is used to evaluate the heat-releasing or adsorbing nature of a formulation. It is routinely run to determine whether the material is thermally stable. Metrics of DSC are the amount of heat released and the rate (in terms of temperature at a given scan rate) at which the heat is released. A sample (0.1–0.6 mg) was weighed into a glass capillary tube (1.5-mm O.D. 0.28-mm wall thickness), with one end pre-sealed. The sample was kept cool, while the second end of the tube was sealed. DSC analyses were performed with TA instruments (DSC 2910 or DSC Q100). Samples were heated at 20 °C min^{-1} ramp rate from 50 to 450 °C.

Henkin time-to-explosion

Samples (40 mg) of the chemicals of interest were weighed into an empty aluminum blasting-cap shell. The lid was pressed to a set depth in the shell using a Carver press. The entire assembly was immersed in a constant-temperature Wood's metal bath. Time-to-explosion (as judged by an audible "pop") was measured. The temperature of the bath was adjusted until a temperature was found at which explosion occurred 50% of the time. Maximum time at any temperature was 120 min. The data recorded in this test were the lowest temperature at which explosion would occur and time-for-explosion. This test has been used to calculate Arrhenius values and critical temperatures, the lowest temperature at which a given size and shape of energetic material can initiate runaway self-heating.

Small-scale explosive device testing [1]

The three previous tests probed material response to thermal input. This test probed response to shock input. Brass .303" cartridge cases (one hole at base) were purchased from Value Bullet Comp., Pudsey, and West Yorkshire, UK. Each case was weighed before packing in 2.0 g of the chemical to be tested. For comparison, it was desirable that exactly 2.00 g of chemical be used in every test, but due to the low density of some materials, this was not always possible. The exact weight and depth of the chemical from the cartridge mouth were measured. A detonator (RP-3), containing of 29 mg of PETN, was inserted into the mouth of the cartridge such that it directly touched the material; it was taped in place. The entire assembly was placed in the detonation chamber—a heavy-walled, stainless-steel vessel of about 1 L inside volume [4–6]. The lid of the chamber was held in place with eight bolts; detonation gases were vented to the outside through small holes in the lid. After initiation of the detonator, the metal cartridge fragments were removed from the chamber; the base of the cartridge was collected, washed in water and acetone, dried, and weighed. The fraction of base remaining after the explosion was used to evaluate the violence of the event. The smaller the amount of the case remaining attached to the base, the more the violent the event became. No response by chemicals, such as NaCl, produced a cartridge with a hole in the side (from the detonator), but all the cartridge remained attached to the base.

Results and discussion

Initial choice of energetic materials was based on the work of Bodman [7]. He used as the standard for materials which detonate the results of the Koenen test. The Koenen test is one of the three tests in the UN test series 1. In this test, 25 g of material is packed in a cylindrical steel vessel; the

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[1] The small-scale explosivity device (SSED) is adapted from the British Cartridge test [4, 7, 8]

vessel is completely sealed, save for a 1-mm-diameter hole in the lid. The material is heated from four sides with Bunsen burner flames until there is no material left or until the vessel ruptures. The metric of the test is the violence of the event as judged by the number of fragments created by vessel rupture. Bodman [7] used the results of the Koenen test because large-scale test results were not available, but this medium-scale test has not been directly linked to detonability. This study used Bodman's targeted compounds and additional compounds known to be high explosives, explosive salts and peroxides, and other highly energetic chemicals. Using high explosives, we were assured to be studying the behavior of detonable materials. To be consistent with Bodman's scale, we assumed these high explosives would perform violently in the Koenen test. This may not be the case.

Given the description of the Koenen test, it was expected it might correlate with the total amount of gas released by a compound. From Table 1, it can be seen that all the known high explosives produce at least 2 moles of gas per mole of compound or about 0.01 mol of gas per gram of compound. For the most part, the other materials produce less gas, although it is notable that TNT, a known high explosive and 3,5-dinitrobenzoic acid, thought to be non-explosive, both produce about 0.01 mol gas per gram. Ammonium Nitrate (AN), which does not support a violent event in the Koenen test, produces similar volumes of gas to the high explosive and is known to detonable on the ton scale, though its performance is less than 50% that of TNT [8]. Evidently AN produces gas so slowly that it does not violently rupture the Koenen device violently, but as pointed out above, there is no sure correlation of Koenen results with explosivity.

The small-scale explosivity device (SSED) was designed to determine a material's response to explosive shock [9, 10]. The metric used is the amount of brass remaining attached to the cartridge base. We have already shown that the performance of certain smokeless powders in the SSED can be correlated with their fragmentation effect in pipe bombs [9]. From Table 1, it can be seen that the high explosives leave less than 50% of the brass cartridge attached to its base. In this test, AN performed like a high explosive. The only other chemical which fragments the casing is 2,4 dinitrophenylhydrazine, and it also ruptures the Koenen test violently. However, at the scale of this test (2 g of test material); a weakly energetic material may have too large a Dcr to detonate. Furthermore, some high explosives such as ANFO (ammonium nitrate with fuel oil) are such poor explosives that they require a booster as well as detonator to initiate detonation.

Both the Henkin test, using 40 mg of sample, and the DSC, using less than 1 mg of sample, assess temperature at which thermal decomposition of a material is rapid enough to rupture the Henkin holder or to register on the DSC thermocouple. Table 1 suggests these temperatures happen to coincide. DSC, which compares heat flow differences between a sample and a reference as both are heated, allows rapid screening of thermal response with only a few milligrams of material. Heat flow (positive or negative) accompanies melting, crystal-phase transition, and chemical reaction; therefore, DSC is used for a variety of chemicals–explosives, pharmaceuticals, and polymers. DSC is the most commonly used in the programmed mode which raises the temperature of the sample and reference at a pre-set rate. DSC is used on energetic materials to quantify the heat released during decomposition and to determine how readily decomposition occurs. The position of the exothermic decomposition moves to lower temperatures as the programmed heating rate of the DSC decreases, which means for thermal scans to be comparable they must be run at the same programmed heating rate. The quantity of heat released is absolute for thermal decomposition though it is generally only half of the heat of detonation. (Detonation reduces the atom groupings to the most thermodynamically stable products—CO or CO_2, N_2, and H_2O.)

In the far right column in Table 1 is our attempt to combine the most important properties of the energetic materials as measured by the four laboratory tests. The metric combines heat and temperature of decomposition with the amount of gas produced and the remaining cartridge in the SSED test as follows:

$$\frac{\text{DSC heat of decomposition} * \text{moles gas of decomposition}}{\text{DSC exothermic temp.} * \%\ \text{brass remaining on SSED base}}$$

While this may not be the best way to combine these values, it can be seen that the metric combination roughly mirrors the known explosivity of the materials.

Yoshida [11, 12] attempted to develop an empirical correlation between explosive hazard and DSC results alone. Bodman [7] continued in that vein, using the heat of decomposition and the temperature of the onset of the exothermic maximum. Both plotted the logarithm of the temperature of the onset of the exotherm ($T_{\mathrm{oe}} - 25$) versus the logarithm of the quantity of heat released (H_{d}) and drew a line separating the explosives from the non-explosives. Table 2 gives Bodman's results for 22 chemicals, and the log terms are used for the plot given in Fig. 1 which includes Bodman and Yoshida linear plots of explosion propagation (EP). It is evident their line equations are slightly different for their lines. Table 3 shows the DSC metrics we used to construct a plot like Yoshida [11, 12] and Bodman [7]. Unlike the heat of melting, the heat of decomposition of energetic materials is not very repeatable; it varies from run to run by as much as 25%. One rationale for this is that heat

Table 1 Small-scale performance tests on energetic materials

Sample name	Chemical formula	Molecular weight	DSC T_{oe}/°C	DSC H_d/Jg^{-1}	Henkin runaway temp	Time-to-run/sec	/moles-gas moles compound-1	/moles-gas gram compound-1	Temp/°C for gas analysis	SSED/ %	Koenen	$\frac{\text{Heat * gas}}{\text{°C*SSED}}$
HMX	$C_4H_8N_8O_8$	296	268	4028	276	2824	4.3	0.015	220	20	Violent	324
RDX	$C_3H_6N_6O_6$	222	217	4556	223	4203	3.9	0.017	220	23	Violent	354
PETN	$C_5H_8N_4O_{12}$	316	195	3095	193	4007	5.2	0.016	220	20	Violent	412
TNT	$C_7H_5N_3O_6$	227	317	3995	320	3488	2.2	0.010	220	36	Violent	77
HMTD	$C_6H_{12}N_2O_6$	208	152	2359	150	2469	5.0	0.024	160	38	Violent	204
TATP	$C_9H_{18}O_6$	222	187	4059	186	2645	3.2	0.014	230	42	Violent	165
2,4-Dinitrophenylhydrazine	$C_6H_6N_4O_4$	198	218, 371	1178, 422	224	19	0.9	0.005	200	34	Violent	19
4-Nitrophenylhydrazine	$C_6H_7N_3O_2$	153	181,374	2803, 2593	225	5	0.7	0.005	200	99	Violent	21
Benzoyl peroxide (97%)	$C_{14}H_{10}O_4$	242	107	631	109	18	0.8	0.003	200, 100	93	Violent	5
2,4-Dinitrotoluene (97%)	$C_7H_6N_2O_4$	182	339	4454	>250	N/A	1.2	0.007	300	98	Modest	16
NH4NO3	NH_4NO_3	80	286	1586	329		2.0	0.025	300	40	Modest	28
2-Chloro-5-nitrobenzoic acid	$C_7H_4ClNO_4$	202	370	1015							Modest	
2-Br-2-nitro-1,3-propanediol	$C_3H_6BrNO_4$	200	233	1320	>250	N/A	1.6	0.008	200	102	Low	9
3,5-Dinitrobenzoic acid	$C_7H_4N_2O_6$	212	381	2438			2.2	0.010	300	98	Low	14
2-NH2-4-chloro-5-nitrophenol	$C_6H_5ClN_2O_3$	189	234	525	210	398	0.7	0.004	200	97	Low	2
Black powder	KNO_3, S, C		308	723						100	Low	0
Thiosemicarbazide (99%)	CH_5N_3S	91	185	770	>250	N/A	0.3	0.003	200	100	No	1
Malononitrile	$C_3H_2N_2$	66	307	1685							No	
3-Nitrobenzenesulfonic acid sodium	$C_6H_4NNaO_5S$	225	422	1120							No	
Ca hypochlorite 70%	$CaCl_2O_2$	143	162	115								

Table 2 Bodman's original data [7]

Material	DSC T_{oe}/°C	Log (DSC H_d)	Log (DSC T_{oe} − 25/°C)	Explosion propagation EP (Yoshida)	Explosion propagation EP′	Actual rank	Koenen	Time pressure	UN gap
2,4-Dintrophenyl hydrazine	210, 340	2.91	2.40	0.33	0.36	A	Violent	NA	Yes
2,4-dinitrotoluene	345	2.93	2.51	0.31	0.34	A	Medium	Slow	Yes
4-Nitrophenylhydrazine 100%	182, 327	2.75	2.33	0.19	0.23	A	Violent	Rapid	Yes
3,5-dintrobenzoicacid	383	2.84	2.55	0.20	0.23	A	Low	NA	Yes
2-Bromo-2-nitropropane-1,3-diol	209	2.71	2.26	0.18	0.23	A	Low	No	Yes
2,2′-Dithiobis(4-methyl-5-nitrothiazole)	223	2.72	2.30	0.18	0.22	A	Medium	Slow	Yes
Benzoyl peroxide 100%	107	2.46	1.91	0.06	0.13	A	Violent	Rapid	Yes
Benzoyl peroxide 70% with H_2O	104	2.34	1.90	−0.05	0.01	A	Violent	Rapid	Yes (No)
2-Chloro-5-nitrobenzoic acid	366	2.61	2.53	−0.02	0.01	A	Medium	No	Yes
t-Butyl peroxybenzoate	124	2.50	2.00	0.07	0.14	B	Violent	Slow	No
2-Diazo-1-naphthol-5-sulphochloride	133	2.31	2.03	−0.13	−0.07	B	Violent	NA	No
4-Nitrophenylhydrazine 76% with H_2O	156, 331	2.73	2.29	0.19	0.23	C	Medium	No	No
2-Amino-4-chloro-5-nitrophenol	225	2.61	2.30	0.06	0.10	C	Low	Slow	No
Di-t-butyl peroxide	161	2.48	2.13	−0.004	0.048	C	No	Slow	No
1-Phenyl-5-mercapto tetrazole	159	2.47	2.13	−0.01	0.04	C	Low	Slow	No
Organic perchlorate#1	224	2.47	2.30	−0.07	−0.03	C	Low	No	No
Benzenediazonium, 2-methoxy-4-(phenylamino)-sulfate (1:1)	178	2.30	2.18	−0.20	−0.15	C	Low	No	No
3-Nitrobenzenesulfonic acid sodium salt	368	2.42	2.54	−0.21	−0.19	C	No	Slow	No
Malononitrile	276	2.65	2.40	0.06	0.10	D	No	No	No
Organic perchlorate#2	229	2.54	2.31	−0.003	0.04	D	No	No	No
Dilauroyl peroxide	88	2.24	1.80	−0.12	−0.05	D	No	No	No
3-Thiosemicarbazide	176	2.34	2.18	−0.16	−0.11	D	No	No	No

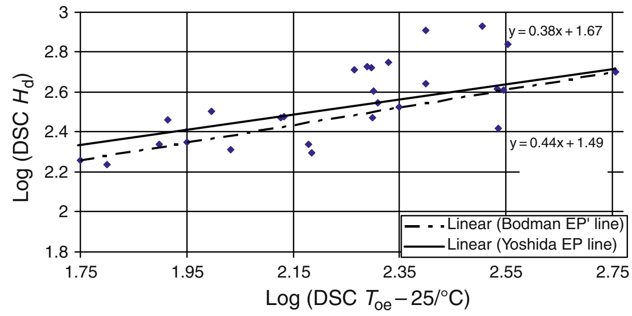

Fig. 1 Bodman and Yoshida plots replotted from data in reference [7]

is released so rapidly by energetic materials that the instrument has difficult in responding. The temperature at which the exothermic maximum is observed is a function of the DSC scan rate; therefore, a scan rate of 20 °C min^{-1} was maintained for all the runs. Following the protocol of Bodman, the exothermic onset, rather than the maximum, temperature was used.

Figure 1 shows the plot Bodman constructed using the Yoshida correlation. Figure 2 is our plot using some of the compounds Bodman suggested (other) as well as a number of military, high explosives (HE), and known explosive compositions. We do not find a clear line separating explosives from non-explosives. Of course, it is not clear whether the supposed non-explosives would not detonate if tested on sufficiently large scale. The addition of high explosives makes clear that the high explosives area is found in the upper left quadrant. Explosivity results when a great deal of heat is released at relatively low temperatures. There appears to be some quantitative correlation between position in the chart and degree of explosivity. All the high explosives, including the known explosive mixture of hydrogen peroxide, are in the upper left quadrant; the dinitroarenes are in the upper right quadrant, with a few near the line. Just above the line are the highly insensitive

Table 3 DSC onset of exotherm temperature (T_{oe}) and Heat of Decomposition (H_d)

	T_{oe}/°C	H_d/J g^{-1}	H_d/cal g^{-1}	Log (H_d)	Log ($T_{oe} - 25$/°C)
CL20	251	4550	1087	3.04	2.35
DADP	132	3860	923	2.96	2.03
HMTD	150	2469	590	2.77	2.10
HMTD	152	2359	564	2.75	2.10
HMX	276	2824	675	2.83	2.40
HMX	268	4028	963	2.98	2.39
Nitrocellulose (NC)	210	1418	339	2.53	2.27
Nitroglycerine (NG)	202	879	210	2.32	2.25
Picric acid	318	3259	779	2.89	2.47
PETN	193	4007	958	2.98	2.23
PETN	195	3095	740	2.87	2.23
RDX	223	4203	1004	3.00	2.30
RDX	217	4556	1089	3.04	2.28
TATB	356	2026	484	2.68	2.52
TATP	186	2645	632	2.80	2.21
TATP	187	4059	970	2.99	2.21
Tetryl	203, 287	1279, 2110	810	2.91	2.25
TNAZ	255	4747	1135	3.05	2.36
TNAZ	258	4066	972	2.99	2.37
TNT	320	3488	834	2.92	2.47
TNT	317	3995	955	2.98	2.47
Smokeless powder (SP)	203	797	190	2.28	2.25
Black powder (BP)	308	723	173	2.24	2.45
Guanidine nitrate (GN)	344	1979	240	2.38	2.50
Urea nitrate (UN)	219, 384	1495, 1193	642	2.81	2.29
NH_4ClO_4 (AP)	364	1280	306	2.49	2.53
NH_4NO_3 (AN)	286	1586	379	2.58	2.42
Ca hypochlorite 70% (Cahypo)	162	115	27	1.44	2.14
Benzoyl peroxide (97%)	107	631	151	2.18	1.91
70% H_2O_2 ethanol	184	2950	705	2.85	2.20
Peroxy laundy soap	149	500	120	2.08	2.09
di-t-butyl peroxide $(tBuO)_2$	180	690	165	2.22	2.19
2,4-Dinitrotoluene (DNT)	339	4454	1065	3.03	2.50
3,5-Dinitrobenzoic acid (DNBA)	381	2438	583	2.77	2.55
1,3-DNB	422	5747	1373	3.14	2.60
1,4-DNB	297	1297	310	2.49	2.43
2,4-DNT	347	4468	1068	3.03	2.51
2,6-DNT	358	2696	644	2.81	2.52
3,4-DNT	342	6519	1558	3.19	2.50
2,4-Dinitrophenylhydrazine (97%)	218, 371	1178, 1938	282,463	2.87	2.29
4-Nitrophenylhydrazine	181, 374	2803, 2593	670,620	2.83	2.19
2-Chloro-5-nitrobenzoic acid	372	1266	303	2.48	2.54
2-Bromo-2-nitro-1,3-propanediol (98%)	233	1320	315	2.50	2.32
Dicyandiamide	246	966	231	2.36	2.34
Thiosemicarbazide (99%)	185	770	184	2.26	2.20
Malononitrile	307	1685	403	2.61	2.45
3-Nitrobenzenesulfonic acid sodium	422	1120	268	2.43	2.60
2-Amino-4-chloro-5-nitrophenol (98%)	234	525	125	2.10	2.32

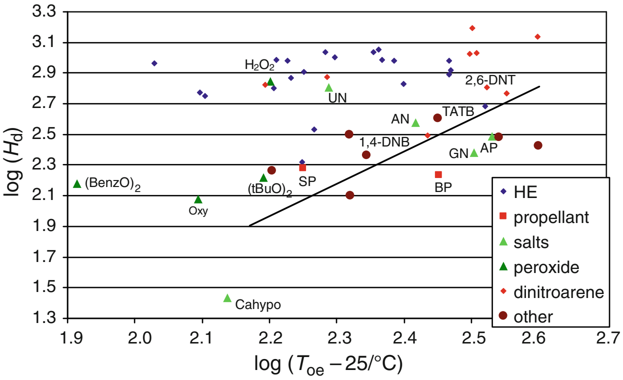

Fig. 2 Plotting DSC heat of decomposition versus exothermic maximum temperature [7, 11, 12]

explosives TATB and AN and smokeless powder, a propellant, which in some cases can be made to detonate. Just below the line are the insensitive explosives GN, AP, and the propellant, black powder (Fig. 2). Ammonium perchlorate (AP) is not classed as an explosive, though it detonates with sufficient quantity and confinement or sufficiently small particle size [13]. A possible reason clearly defined separation is not observed is that it is uncertain whether any non-explosive compounds have been tested. Possibly, all the materials plotted are explosive on a large enough scale, with sufficient confinement. Calcium hypochlorite, which is far below the line, may truly be a non-explosive. A number of fires have occurred in swimming pool supply warehouses where multi-tons of this oxidizer has been stored, and no detonation has resulted.

The discussion above suggests that in developing small-scale explosivity tests, there is a need to “know” which materials are detonable and which are not. The Koenen test is insufficient for this purpose. It is unclear whether pressure rupture using 25 g of material can be related to detonability. A 1998 National Research Council report [14] recommended a 12″ by 60″ cylinder test. For most materials this would require about 240 lb [2]. This pipe-bomb-like configuration is fairly large diameter (12″) and uses detonation velocity probes to determine whether the detonation wave, introduced into it by a 5-lb booster, is immediately quenched, is almost supported, or initiates a detonation in the test material. The pin response, the witness plate damage, and the pipe fragmentation can be used to judge material response. It is known that ANFO will detonate in this device, but un-fueled ammonium nitrate will not. ANFO can be made to detonate on the 50-lb scale, and un-fueled ammonium nitrate on the 1000-lb scale. With these extremely limited benchmarks, it appears that this test configuration is good for predicting detonability to at least a fivefold increase.

Conclusions

This preliminary study suggests that the chosen test, particularly the DSC results will be useful in predicting large-scale detonability.

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[2] The NRC-recommended explosivity test suggests a charge 12″ in diameter by 60″ in length in a mild-steel, schedule 40 pipe with a 24″ square witness plate ¼″ thick welded on the bottom end. Along the length of the pipe, starting 14″ from the open top and about every 12″ thereafter, are five piezoelectric pins (time-of-arrival) to measure detonation velocity. The fixture is placed in front of a zebra-stripped board (alternating black and white strips every inch), and a high-speed camera is used to record detonation velocity. The pipe is filled with about 240 lb of the test material, and a 5-lb Pentolite booster is used.