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Testing to Determine Relationship Between Explosive Contaminated Sludge Components and Reactivity

(Task Order Number 1)

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Introduction. Explosives manufacture and ammunition load, assembly and pack (LAP) operations result in the generation of explosives-contaminated wastewater. Over the years, the Department of the Army has used lagoons for treatment of these wastewaters by evaporation/percolation. These lagoons contain the remaining explosives-contaminated sludges (i.e., mixtures of explosives, water and soil). These explosives-contaminated waters and sludges are listed as hazardous wastes under Federal regulations promulgated under the Resource Conservation and Recovery Act (RCRA). The basis for this listing is the assumed explosive reactivity of these wastes if subjected to a strong initiating source or if heated under confinement (Refer to 40 CFR 261.23)[2] Presently, tests to determine the explosive reactivity of wastes are not specified. Different tests have been under consideration.
Objective. The objective of this study was to investigate and define the reactivity of explosive contaminated soils to flame and shock as a function of explosive composition using the Bureau of Mines (BOM) Zero Gap (shock) and Deflagration to Detonation

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Transition (DDT flame) tests. The resultant technical data will be used to define the reactivity of explosive contaminated soil on the basis of compositional analyses rather than the time consuming and expensive BOM protocols.

Summary and Conclusions. Extensive testing was conducted by Hercules Incorporated, Radford Army Ammunition Plant (RAAP) under subcontract to Arthur D. Little, Inc., contractor to USATHAMA to investigate and define the reactivity of explosive-contaminated soils to flame and shock stimuli. These tests were conducted with laboratory prepared, water-wet and dry samples of the explosives RDX or TNT mixed with sand. Shock sensitivity tests determined that explosive-contaminated soils containing ≤15% explosive will not react positively to induced shock inthe BOM Zero Gap test. Flame sensitivity tests determined that explosive-contaminated soils containing ≤12% explosive will not react explosively when subjected to submerged flame initiation in BOM DDT test confinement. This study provides additional data for the development of a technical data base suitable for use as reactivity criteria (see Figure 1) for assessing the explosive reactivity of contaminated soils to flame and shock stimuli on the basis of soil compositon. Verification tests conducted with predicted 0.5% reactive compositions resulted in 20 consecutive negative results indicating ≤0.5% reactivity at the 90% confidence level.

Sample composition may be used as the criteria to assess the explosive reactivity of U.S. Army lagoon soils containing principally secondary explosives such as TNT, RDX, HMX and others having equal or less sensitivity to shock and flame. Explosive-contaminated soil containing significant (≥0.1%) amounts of more initiation sensitive materials including those of primary explosives (e.g., lead styphnate, lead azide, etc.) and/or ingredients will require vertification testing using the BOM flame and shock test protocols.

From these tests, it is also concluded that explosive-contaminated soils can be diluted with virgin soil to reduce the total explosive content of ≤12% and result in a composition which is not reactive in the BOM flame and shock tests.

In themselves, the BOM Zero Gap and DDT tests are expensive and time consuming to perform. As screening tests, both are considered to be more severe than needed for assessing the explosive reactivity of contaminated soils. Moreover, special safety tooling and constructed facilities or remote test locations are necessary to conduct these tests in a safe manner and to protect personnel from delayed reactions and accompanying shrapnel. It is concluded, therefore, that more economical tests and/or criteria for reactivity should be considered. If sample composition is not adopted as recommended above, then the relatively inexpensive and quick tests for reactivity originally proposed by U.S. Environmental Protection Agency in the SW-846 Report should be reevaluated for adoption.

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5.0 EXECUTIVE SUMMARY

5.1 Objective

The objective of this study was to investigate and define the reactivity of explosive contaminated soils to flame and shock as a function of explosive composition using the Bureau of Mines (BOM) Zero Gap (shock) and Deflagration to Detonation Transition (DDT flame) tests. The resultant technical data will be used to define the reactivity of explosive contaminated soil on the basis of compositional analyses rather than the time consuming and expensive BOM protocols.

5.2 Summary and Conclusions

Extensive testing was conducted by Hercules Incorporated, Radford Army Ammunition Plant (RAAP) under subcontract to Arthur D. Little, Inc., contractor, to USATHAMA to investigate and define the reactivity of explosive-contaminated soils to flame and shock stimuli. These tests were conducted with laboratory prepared, water-wet and dry samples of the explosives RDX or TNT mixed with sand. Shock sensitivity tests determined that explosive-contaminated soils containing ≤15% explosive will not react positively to induced shock in the BOM Zero Gap test. Flame sensitivity tests determined that explosive-contaminated soils containing ≤12% explosive will not react explosively when subjected to submerged flame initiation in BOM DDT test confinement. This study provides additional data for the development of a technical data base suitable for use as reactivity criteria (see Figure 1) for assessing the explosive reactivity of contaminated soils to flame and shock stimuli on the basis of soil composition. Verification tests conducted with predicted 0.5% reactive compositions resulted in 20 consecutive negative results indicating ≤0.5% reactivity at the 90% confidence level.

Sample composition may be used as the criteria to assess the explosive reactivity of U.S. Army lagoon soils containing principally secondary explosives such as TNT, RDX, HMX and others having equal or less sensitivity to shock and flame. Explosive-contaminated soil containing significant (>0.1%) amounts of more initiation sensitive materials including those of primary explosives (e.g., lead styphnate, lead azide, etc.) and/or ingredients will require verification testing using the BOM flame and shock test protocols.

From these tests, it is also concluded that explosive-contaminated soils can be diluted with virgin soil to reduce the total explosive content to ≤12% and result in a composition which is not reactive in the BOM flame and shock tests.

In themselves, the BOM Zero Gap and DDT tests are expensive and time consuming to perform. As screening tests, both are considered to be more severe than needed for assessing the explosive reactivity of contaminated soils. Moreover, special safety tooling and constructed facilities or remote test locations are necessary to conduct these tests in a safe manner and to protect personnel from delayed reactions and accompanying shrapnel. It is concluded, therefore, that more economical tests and/or criteria for reactivity should be considered. If sample composition is not adopted as recommended above, then the relatively inexpensive and quick tests for

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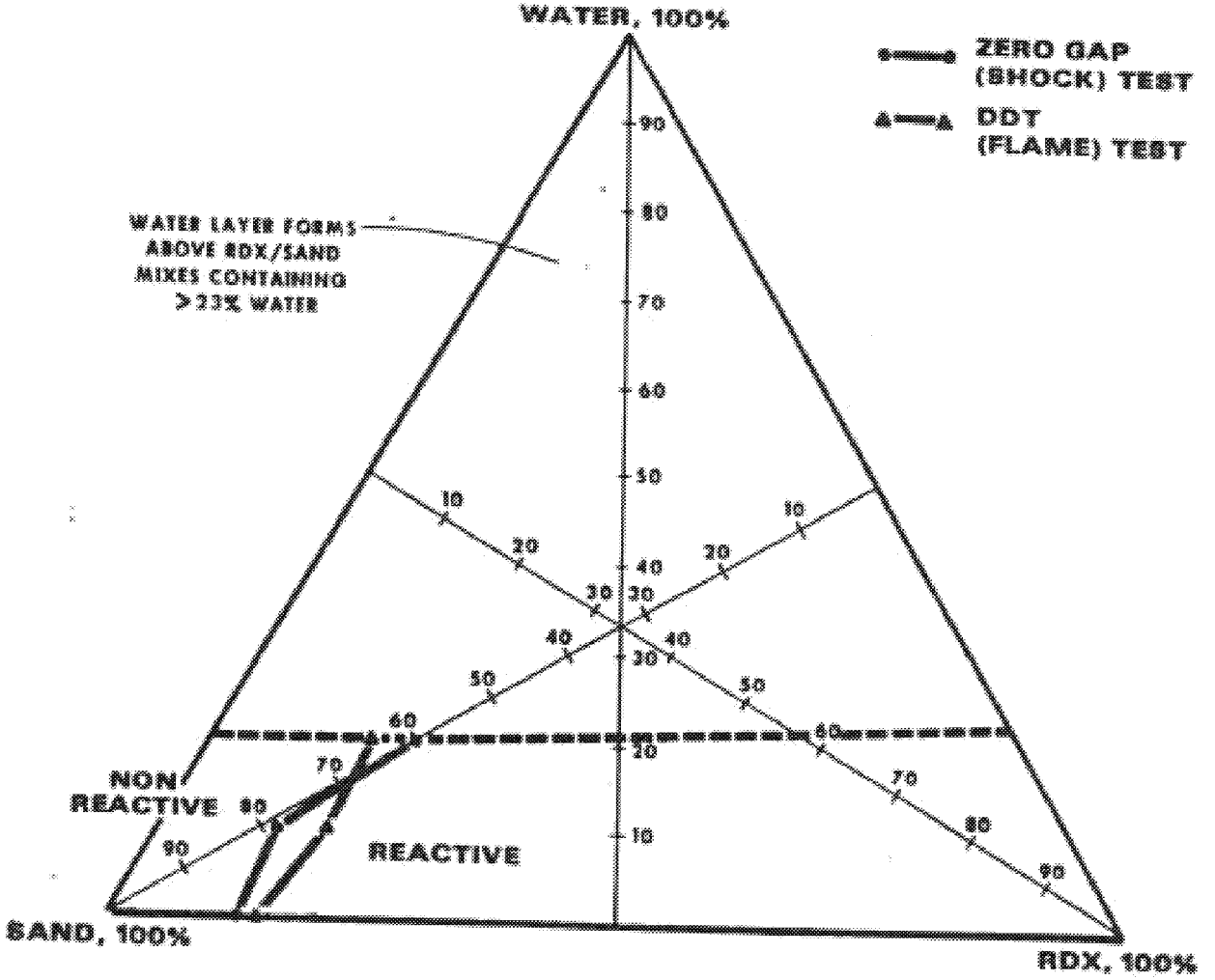

Figure 1. Combined Results of DDT and Zero Gap Tests

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reactivity originally proposed by U.S. Environmental Protection Agency in the SW-846 Report[1] should be reevaluated for adoption.

5.3 Recommendations

1. Base the determination of sample reactivity of contaminated soils on the more quantitative and economic chemical analysis of samples for explosives content rather than the qualitative and expensive BOM Zero Gap and DOT tests.

2. Adapt the criterion of sample composition as a measure of contaminated soil reactivity based on the explosive level present:

- If explosive content in samples is ≤12%, the sample is not reactive.
- If explosive content in samples is >12%, the sample is reactive.

3. If the criterion for reactivity will require explosive testing, then investigate the use of less expensive and time consuming tests for establishing if explosive-contaminated soils are explosively reactive.

1.0 INTRODUCTION

Explosives manufacture and ammunition load, assembly and pack (LAP) operations result in the generation of explosives-contaminated wastewater. Over the years, the Department of the Army has used lagoons for treatment of these wastewaters by evaporation/percolation. These lagoons contain the remaining explosives-contaminated sludges (i.e., mixtures of explosives, water and soil). These explosives-contaminated waters and sludges are listed as hazardous wastes under federal regulations promulgated under the Resource Conservation and Recovery Act (RCRA). The basis for this listing is the assumed explosive reactivity of these wastes if subjected to a strong initiating source or if heated under confinement (Refer to 40 CFR 261.23).[2] Presently, tests to determine the explosive reactivity of wastes are not specified. Different tests have been under consideration. Two test series are discussed in the following.

The first series of tests are similar to those used by the Department of Transportation (DOT) to determine the shipping classifications for hazardous materials. These inexpensive, small-scale tests determine if a material will burn or explode when subjected to an elevated temperature of 167°F for 48 hours, flame, shock of a No. 8 blasting cap, and BOE Impact Apparatus at 10 and 4-inch drop heights. These tests were listed in U.S. Environmental Protection Agency SW-846 (1980) "Test Methods for Evaluating Solid Waste."

Another series of tests were developed by the BOM in cooperation with DOT to assist the United Nations (UN) Group of Experts on Explosives in

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preparing recommendations for the international transport of dangerous goods. These test protocols are known as the Zero Gap shock and Deflagration to Detonation Transition (DDT) flame tests. This series of tests is more expensive and time consuming than the EPA SW-846 tests mentioned previously. One advantage of these tests is that test samples are subjected to greater shock and flame energy in stronger steel confinement than in EPA SW-846 tests and therefore test results are more safety conservative.

In order to provide a technical data base and investigate the Zero Gap and DDT tests for determining the explosive reactivity of explosive contaminated sludges, USATHAMA funded this project for the purpose of investigating and defining the relationship between explosive-contaminated soil reactivity to BOM flame and shock tests, and explosive content. This study provides additional data for the development of a technical data base that may be used to predict the reactivity of explosive contaminated soils to flame and shock stimuli on the basis of compositional analyses of explosive(s) content. Substitution of laboratory analyses of explosive contaminated sludges for Zero Gap and DDT testing of sludge compositions would result in lower costs for determining reactivity of contaminated soils. Hercules Incorporated at RAAP, Radford, VA, was subcontracted to conduct this investigation because of their expertise and experience in handling explosives safely and securely, and the availability of explosive test facilities suitable for conducting BOM flame and shock tests.

2.0 DISCUSSION OF RESULTS

The following sections discuss the results of the critical diameter, flame and shock sensitivity tests conducted with RDX/sand/water mixtures and the results of the flame and shock confirmatory tests conducted with TNT/sand mixtures.

2.1 Critical Diameter (C_d) Screening Tests

Before beginning Zero Gap shock tests, C_d tests were conducted to define RDX/sand/water mixtures which would be reactive or non-reactive in the 1.44-inch diameter steel Zero Gap test confinement (see Appendix A). RDX/sand mixtures containing more than 20% water were not tested because a water layer forms above the settled solids. A settled, water-wet RDX or RDX/sand mixture will react explosively to induced explosive shock regardless of how much water is present in the water layer.

C_d test results for dry and wet RDX and RDX/sand mixtures are summarized in Table 1 and shown in Figure 2. A typical C_d/pipe diameter curve has been included in Figure 2 showing the effect of substituting RDX for ammonium nitrate in a composite propellant. Individual trial results are listed in Appendix A, Table A1. As can be seen from these data, the C_d varies inversely with explosive content of the wet or dry RDX/sand mixtures. Figure 2 indicates that 18% to 25% RDX in wet or dry RDX/sand mixtures should not react explosively in Zero Gap shock tests. Knowing that differences between the more severe Zero Gap and C_d test configurations (greater container burst strength, use of Pentolite pellets instead of Composition C-4 donor

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Table 1

Summary of Critical Diameter for Explosion Test Results

Composition Tested, %			Average Bulk Density, g/cc	Critical Dimension[a] for Explosive Propagation (C_d), in.
RDX[b]	Sand	Water		
100[c]	0	0	1.05	≤ 0.25
25	75	0	1.21	0.5
25	75	0	1.22	1.0
20	80	0	1.26	1.5
15	85	0	1.25	2.0
35	55	10	1.29	0.5
30	60	10	1.23	1.0
25	65	10	1.28	1.5
20	70	10	1.20	2.0
25	55	20	1.75	0.5
20	60	20	1.71	1.0
15	65	20	1.82	1.5
10	70	20	1.81	2.0

[a] C_d – Confined material dimension above which sustained propagation of an explosive reaction can be expected. Nominal size of schedule 40 pipe shown. Refer to Appendix A, Table A1 for complete listing of tests.

[b] Type II, Class 1 except where otherwise noted.

[c] Type II, Class 5.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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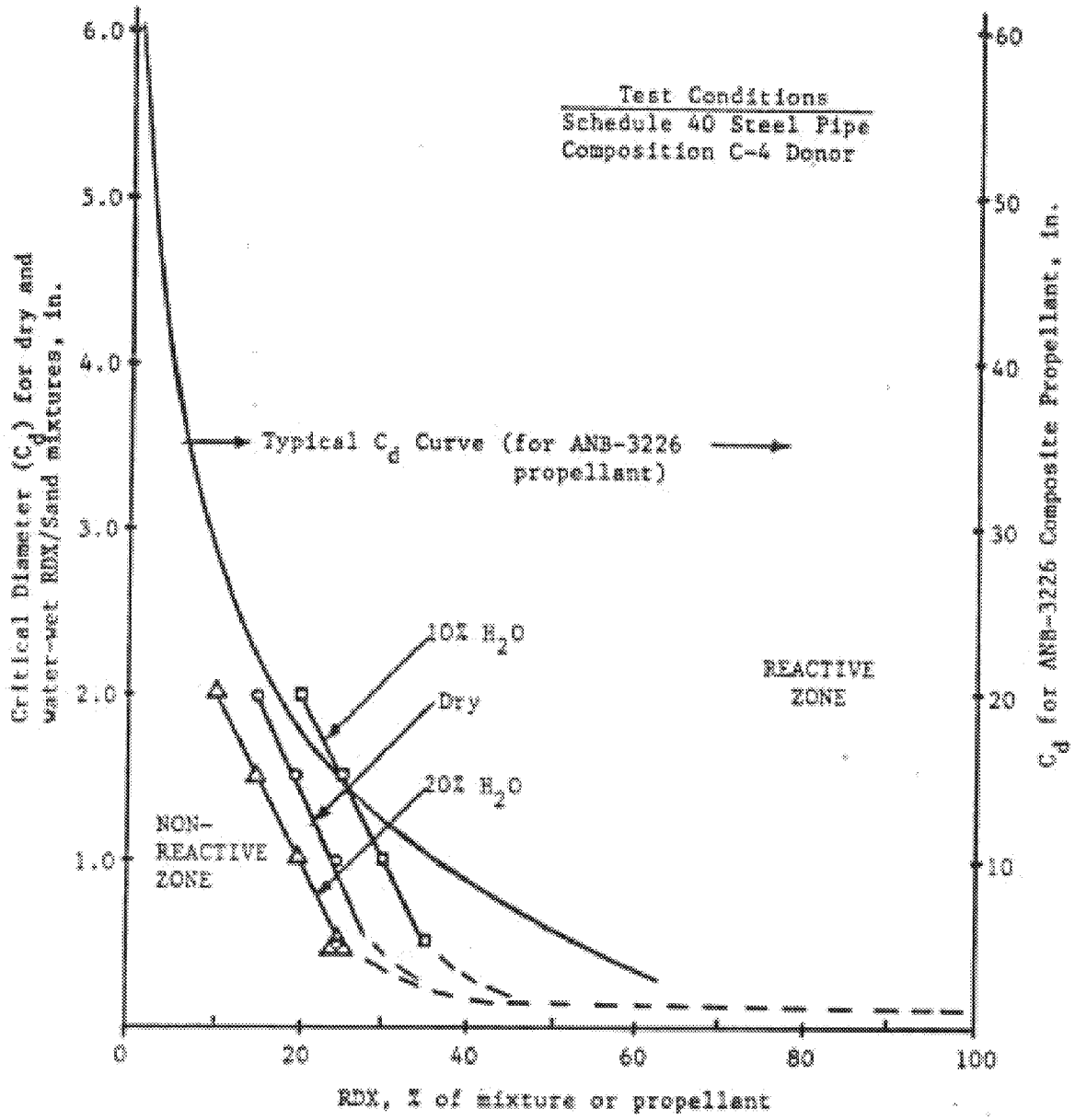

Figure 2. Critical Diameter for Explosion Propagation of RDX/Sand Mixtures.

Note: See reference 8 for additional information on ANB-3226 propellant testing.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

charge, etc.) could affect Zero Gap test results, initial shock tests were conducted starting with 25% RDX in sand compositions.

Figure 2 also shows that the addition of 10% moisture to RDX/sand mixes moderates (increases) the C_d level by ~0.75 inch; but at the 20% moisture level the C_d is lower than dry RDX/sand mixtures. It is likely that the observed shifts in C_d caused by the addition of water can be explained by mixture bulk density. Experiments by others have demonstrated that, for a given explosive in cylinders of large diameter, the detonation velocity is nearly a linear function of the initial bulk density.[3] A more recent report of C_d studies with loose, crystalline explosives concluded that increase of the explosive charge density as a result of pressing (charge consolidation) or filling voids with water decreases the charge air content, improves the conditions for shock wave propagation in a given medium and results in lower C_d.[4] An examination of the measured bulk densities of test mixtures shows that the bulk density of dry and 10% water-wet RDX/sand mixtures were essentially the same and averaged 1.2 g/cc. As one would expect, an increase in the percent of inert material with no change in bulk density resulted in a less reactive mixture as reflected by an increase in the diameter of pipe necessary to sustain propagation of an explosive reaction (Figure 2). However, the bulk density of 20% water-wet RDX/sand mixtures was significantly higher and averaged 1.7 g/cc. The higher bulk density apparently caused the observed C_d shift between the 10% and 20% moisture parameters shown in figure 2.

On the basis of the above, it is concluded that an increase in RDX content in the mixture will reduce the sample diameter capable of propagating an explosive reaction. In contrast, an increase in sand content increases C_d; and an increase in water content has little effect upon C_d. The C_d tests indicate that wet or dry mixtures of sand and 25% RDX are likely to be non-reactive in BOM Zero Gap tests.

2.2 Zero Gap Shock Test Results

Wet and dry RDX/sand mixtures were tested to define mixture shock reactivity as a function of RDX content. Testing was conducted using the BOM developed Zero Gap test described in Appendix B and shown in Figure B1. In this test, samples were confined in 1.44-inch diameter steel tubing and subjected to an explosive shock wave induced at one end by two Pentolite pellets. RDX/sand/water compositions reacting explosively were identified using BOM test protocols. Standard probit techniques[5] were used to establish an RDX level in wet and dry sand that has a low (0.5%) probability of reacting explosively to shock in the BOM test configuration.

2.2.1 Initial Trials

Initial trials were conducted using 100% RDX, 100% sand, 100% water and an 80% sand/20% water mixture to verify that the Zero Gap shock test is capable of identifying material samples reactive or non-reactive to shock. These test results are presented in Appendix B, Table B1 and show that the test is capable of identifying samples reactive or non-reactive to shock in the BOM test configuration.

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Trials with RDX produced a positive result and demonstrated RDX reactivity to shock. In both the water and sand trials (three each), end-to-end pipe fragmentation occurred during one trial. Both materials also transmitted a fairly stable shock wave in one or more trials at velocities just below the >1,500 m/s criterion for an explosive reactive material. Water and probably any continuous phase (liquid or solid) material should be expected to transmit the donor induced shock wave effectively to the end of the comparatively short, 16-inch long pipe. It is suspected that much longer pipes would be required to detect shock wave degradation (decaying reaction) in continuous phase materials. Although sand is not a continuous phase material (contains air in granular interstices), another mechanism is thought to have caused the test container to fragment into long strips or appear to propagate the shock wave (positive results). In one sand trial, sand remaining within the undamaged portion of the pipe had been compressed and wedged into the pipe. It is theorized that in other trials with sand, a slug of tightly compressed sand was driven up the steel tube with sufficient force to rupture and fragment the tube and indicate propagation of a shock wave to the end of the 16-inch long test container. It is not likely that both tube fragmentation and indication of a shock wave by mechanical force of sand on the velocity probe would occur at the same time. A plug of sand hard enough to rupture the pipe would be expected to push the velocity probe out ahead of it and no velocity trace would result.

Zero Gap tests with 20% water filling spaces between sand granules gave indications of a pressure wave propagation velocity of ≤770 m/s. None of the sand and/or water (inert) trials transmitted sufficient shock to puncture the 1/8-inch thick, mild steel witness plate.

Zero Gap tests with inerts (sand and water) indicate that positive velocity and/or fragmentation results may occur with inerts in the BOM test configuration. It is speculated that this is why the BOM protocols require at least 2 of 3 different reaction criteria (velocity, pipe fragmentation and/or hole in the witness plate) be met before declaring a positive test result. If a trial with inert material resulted in a positive test result, the resulting data and test conclusions would be safety conservative. It appears unlikely that a shock sensitive material would not react positively in the Zero Gap test.

2.2.2 RDX/Sand/Water Trials

Zero Gap tests were conducted with 0, 10 and 20% water-wet RDX/sand mixtures containing 15-25% RDX. These test results are also presented in Appendix B, Table B1.

Test results summarized in Table 2 and shown in Figure 3 indicate that dry RDX/sand mixes containing 15% RDX are not reactive to induced shock in the BOM test configuration at the 0.5% reactivity level. Twenty consecutive trials with 15% RDX in sand tested negatively and verified at the 90% confidence level that this RDX/sand composition is unreactive at the 0.5% reactivity level.

Zero Gap tests with 20% water-wet RDX/sand mixes determined that mixes containing 16.0% RDX are also 0.5% reactive at the 90% confidence level. A comparison of 0 and 20% water-wet test results indicate that the

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Table 2

Summary of Zero Gap Shock Test Results

Composition Tested, %			Average Bulk Density, g/cc	No. Trials	Average Shock Propagation Rate,[b] m/s	Positive Reactions[c] %
RDX	Sand[a]	Water				
20	80	0	1.342	11	2,220	73
18.75	81.25	0	1.294	10	3,030	20
17.5	82.5	0	1.339	10	2,670	20
15	85	0	1.345	20	1,864	0
25	65	10	1.285	5	2,550	100
23.5	66.5	10	1.262	10	2,760	60
22	68	10	1.289	10	2,480	30
19	71	10	1.273	10	2,620	10
18.5	61.5	20	1.760	2	3,960	100
17	63	20	1.752	10	3,120[d]	50
16.5	63.5	20	1.746	10	1,140	10
16	64	20	1.768	20	887	0

[a]Sand 0.8 to 0.2% water-wet.

[b]Shock propagation rate recorded by velocity probe in the upper half of the test sample (shock was induced into the bottom of the test sample).

[c]Two of the three following positive test result criteria are recorded: (A) Clean hole punched through, 1/8-in. thick steel plate; (B) Pipe fragmented along its entire length; (C) Stable propagation velocity > 1,500 m/s. Refer to Appendix B, Table B1 for complete listing of tests.

[d]Five trials averaged. Others were decaying reactions (variable rates).

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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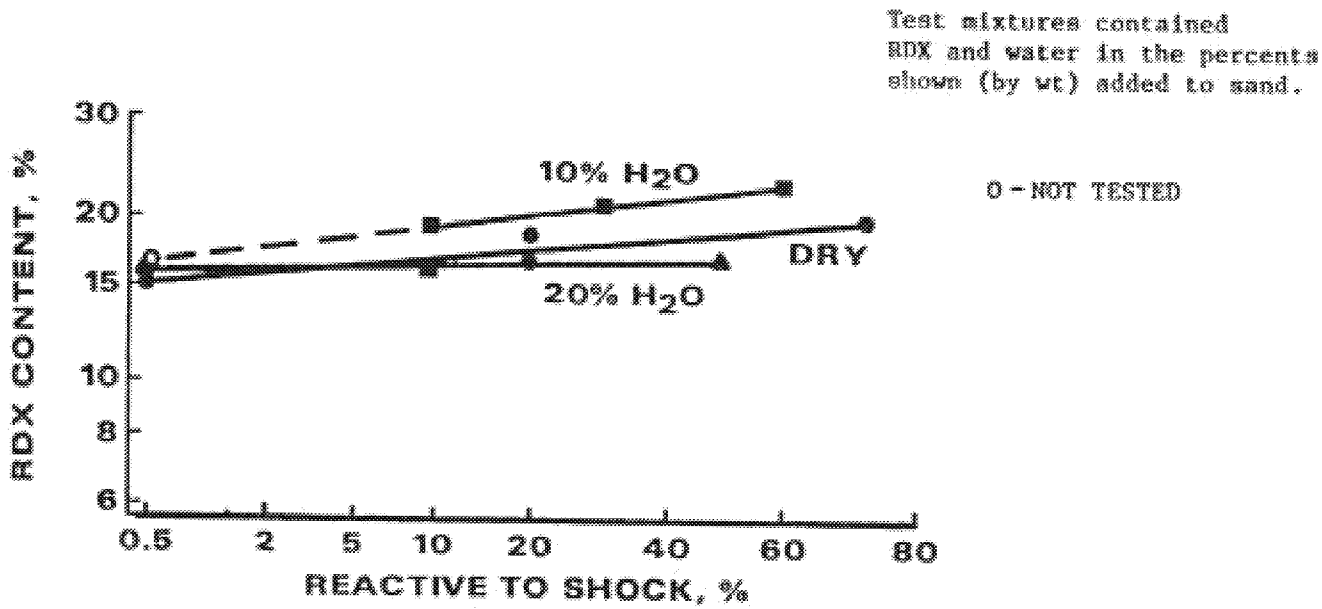

Figure 3. RDX/Sand/Water Shock Reactivity in Zero Gap Tests.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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substitution of up to 20% sand with water has little effect upon sample reactivity at the 0.5% reactivity level.

As anticipated from previously discussed Zero Gap tests, the predicted 0.5% reactive RDX concentration (16.5%) in 10% water-wet RDX/sand mixes was nearly the same at those obtained at the 0 and 20% water-wet levels. Figure 3 shows the results of all RDX/sand samples tested in the Zero Gap test configuration.

Comparing the results of RDX/sand Zero Gap tests at higher reactivity levels (Figure 3), it can be seen that substitution of 10% sand with water reduces sample reactivity. However, substitution of an additional 10% sand with water (20% water content) has the opposite effect. The reason for these results is likely the same (changes in bulk density) as discussed for C_d test results.

It is concluded that water-wet or dry RDX/sand mixtures containing ≤15% RDX are not likely to sustain propagation of a shock wave in the BOM Zero Gap test. In contrast, RDX contaminated soils containing >15% RDX may be desensitized to shock stimuli by adding uncontaminated soil to reduce the RDX content to ≤15% RDX.

2.2.3 Statistical Analyses

A statistical analysis was conducted to determine if there was a relationship between shockwave propagation velocity and sample composition. Analysis details are presented in Appendix C. The findings of this analysis, for the narrow range of compositions tested, indicate that:

1. Velocity is dependent upon the level of RDX in the RDX/sand/water mixture.

2. Sand and water do not have equal effects upon velocity.

3. There is no effect upon velocity due to changes in sand content if the difference between RDX and water do not change.

The first two "findings" agree with overall Zero Gap test results and indicate that the reactivity of RDX/sand/water mixtures increase when the RDX content is increased. However, the third finding is apparently true only for the narrow range of compositions in the statistical analysis. Extrapolation of the third finding leads to the conclusion that a test sample containing no RDX, 1.5% water and 98.5% sand should react explosively and sustain a shock wave equivalent to that obtained by a 18.5% RDX/20% water/61.5% sand mixture (in both cases, the difference between RDX and water contents is 1.5%). Since this finding is clearly not a valid one outside of the range of compositions tested, its application is very limited and of questionable value in determining the explosive reactivity of soils.

2.3 Deflagration to Detonation Transition (DDT) Test Results

Wet and dry RDX/sand mixtures were also tested to define mixture flame reactivity as a function of RDX content. Testing was conducted using the BOM

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DDT test described in Appendix B and shown in Figure B2. In this test, samples are confined in 3-inch, schedule 80 steel pipe and subjected to flame from a 20-gram igniter. RDX/sand/water compositions reacting explosively were identified using BOM test protocols. Standard probit analysis techniques[5] were used to establish an RDX level in wet and dry sand that has a low (0.5%) probability of reacting to flame in the BOM DDT test configuration.

2.3.1 RDX/Sand/Water Trials

The DDT flame test results are summarized in Table 3 and plotted in Figure 4. All individual trial results are listed in Appendix B, Table B2 for reference. The DDT tests were conducted with 0, 10 and 20% water-wet RDX/sand mixes containing 12 to 28% RDX. Figure 4 shows that dry RDX/sand mixes containing ≤13% RDX should not react explosively when subjected to submerged flame initiation in the BOM test configuration. Twenty consecutive trials with 13% RDX in sand gave negative results, and verified at the 90% confidence level that this RDX/sand composition is unreactive at the ≤0.5% reactivity level.

DDT tests with 10% water-wet RDX/sand mixtures reacted about the same as tests with dry RDX/sand mixtures. Twenty consecutive trials with 10% water-wet RDX/sand mixes containing 12% RDX gave negative results, and verified at the 90% confidence level that this RDX/sand/water composition is also unreactive at the 0.5% reactivity level.

DDT tests conducted with 20% water-wet RDX/sand mixtures determined that these mixtures are not as reactive to flame as other moisture levels tested. Figure 4 indicates that a 20% RDX/60% sand/20% water composition should be 0.5% reactive in the BOM DDT test configuration. Verification tests were not conducted since previous verification tests have consistently been successful in demonstrating low (≤0.5%) reactivity for projected low reactivity compositions. However, all DDT trials conducted with 20% water-wet RDX/sand mixtures containing 25% RDX generated sufficient pressurization to rupture the schedule 80 pipe. Many pipes were split end-to-end and flattened. It is apparent that the 25% RDX/55% sand/ 20% water composition is reactive to flame in the steel pipe confinement, but that water at the 20% level moderated (slowed down) and prevented a DDT reaction most of the time. Fragmentation of the pipe or cap into two or more separate pieces (BOM criteria) occurred in only three of 10 trials conducted (30% reactive).

During DDT testing, 2 out of 10 trials were negative for dry 25% RDX/75% sand samples. This result is not in agreement with 20% RDX/80% sand tests resulting in 10 positive results out of 10 trials, or the correlation between RDX/sand compositions and percent positive reactions shown in Figure 4. A review of test records show nothing abnormal to indicate the cause of the two negative results. It is concluded that these results may be indicative of test variability.

As determined during Zero Gap tests, the bulk density of 20% water-wet RDX/ sand mixtures averaged 1.8 g/cc and was greater than that of dry and 10% water-wet mixtures which ranged from 1.3 to 1.4 g/cc. The effect of increased density upon the sensitivity of RDX/sand mixtures to flame initiation is not clear based upon DDT test results. It is suspected that the decrease in RDX/sand mixture reactivity experienced with 20% water-wet

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Table 3

Summary of DDT Test Results for RDX/Sand Mixtures

Composition Tested, %			Average Bulk Density g/cc	No. Trials	Positive Reactions,[b] %
RDX	Sand[a]	Water			
50	50	0	–	1	100
30	70	0	–	1	100
25	75	0	1.28	10	80
20	80	0	1.32	10	100
17.5	82.5	0	1.34	10	70
15	85	0	1.33	10	10
13	87	0	1.43	20	0
19	71	10	1.34	3	100
15	75	10	1.41	10	30
12	78	10	1.49	20	0
28	52	20	1.70	10	80
26.5	53.5	20	1.71	4	25
25	55	20	1.73	10	30
13	67	20	1.77	5	0

[a]Sand = 0.25% water wet.

[b]Pipe and/or at least one end cap fragmented into two distinct pieces.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

Refer to Appendix B, Table B2 for complete listing of tests.

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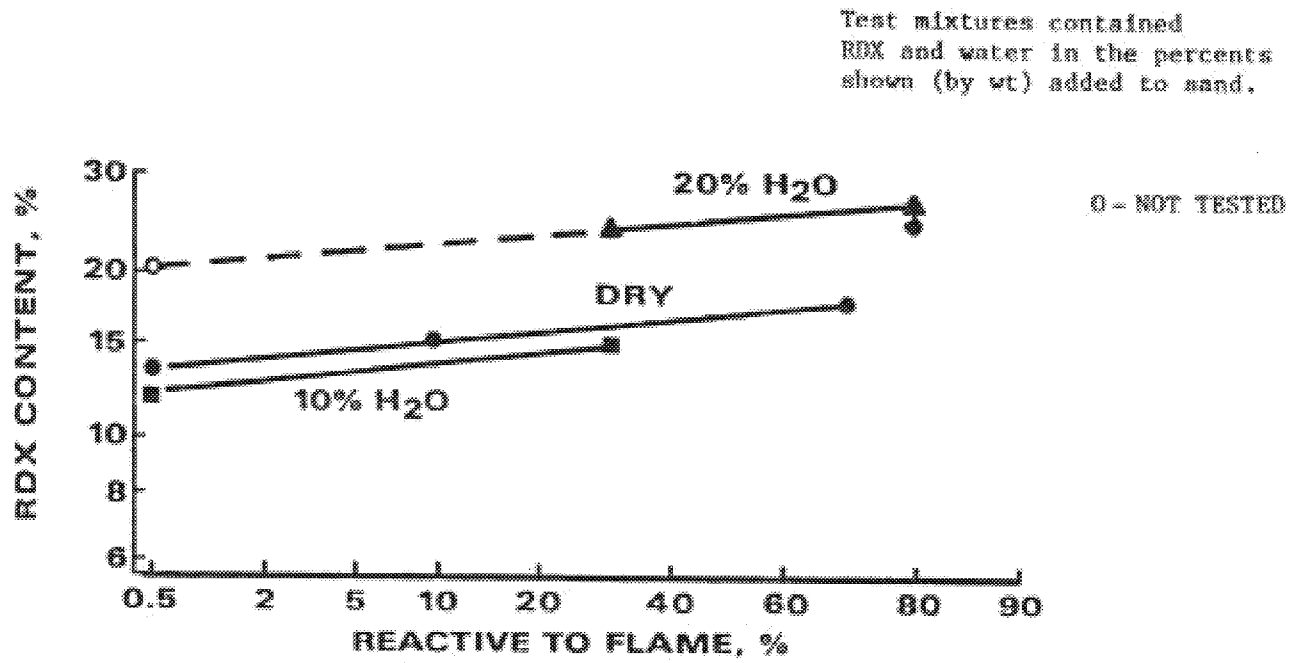

Figure 4. RDX/Sand/Water Flame Reactivity in DDT Tests.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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mixtures is due primarily to the flame quenching effect of the water rather than increased bulk density.

DDT tests at the predicted 0.5% reactive composition levels resulted in "no reactions" in 20 consecutive trials and verified that wet or dry mixtures of RDX/sand containing ≤12% RDX are not flame sensitive in the BOM DDT test. Likewise, the DDT test results also show that reactive RDX contaminated soils containing >12% RDX may be desensitized to flame by adding uncontaminated soil and reducing the RDX content to ≤12% RDX.

2.4 Reactivity Criteria

Predicted 0.5% reactive RDX/sand/water compositions for both the Zero Gap and DDT tests are also plotted on the trimodal plot in Figure 1. This plot identifies dry and settled RDX/sand compositions not reactive to flame and shock in the BOM tests. A dotted line has been drawn to show the maximum percent of water which will be present in settled RDX/sand mixtures and the limits of this study. However, it is likely that any RDX/sand/water composition not reactive to BOM tests in the settled state will also be non-reactive if the same weights of an RDX/sand mixture are suspended in greater amounts of water.

The trimodal plot serves as a quick means to identify explosive-contaminated soils which are reactive or non-reactive to the BOM flame and shock tests based primarily on sample composition. Using this reactivity criteria, comparatively quick and inexpensive chemical analysis of Army lagoon soil samples may be used instead of the more time consuming and expensive BOM Zero Gap and DDT tests to establish the reactivity of soils containing secondary explosives contaminates such as RDX, HMX, TNT, etc.

2.5 Confirmatory Tests with TNT

Dry TNT/sand mixtures were prepared and tested in the BOM DDT and Zero Gap tests to confirm that TNT is no more reactive in these tests (Figure 5) than RDX. Test results are presented in Tables 4 and 5 and discussed in the following.

Zero Gap tests were conducted with a mixture of 19% TNT fines in sand. This composition was selected for comparison with a 19% RDX/81% sand mixture determined previously to react positively to shock 50% of the time in the Zero Gap test configuration (see Figure 5). Test results for this TNT/sand mixture are listed in Table 4 and show that no positive reactions occurred in 10 consecutive Zero Gap trials. It is concluded that additional (>19%) TNT must be added to TNT/sand mixtures to achieve a reactivity level (50%) equivalent to a 19% RDX/81% sand mixture in the BOM Zero Gap shock test.

Likewise, DDT tests were conducted with a mixture of 17% TNT fines in sand. This composition was selected for comparison with a 17% RDX/83% sand mixture determined previously to react positively to flame initiation 50% of the time in the DDT test configuration (see Figure 5). Test results for this TNT/sand mixture are listed in Table 5 and show that no positive reactions occurred in 10 consecutive trials. It is concluded that TNT is less reactive in the BOM DDT flame initiation test than RDX.

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Table 4

Summary of Zero Gap Shock Test Results for TNT/Sand Mixtures

Trial No.	Composition, % TNT[a]	Sand[b]	Water	Loading Density, g/cc	Shock Propagation Rate Thru Sample,[c] m/s	BCM Test Criteria[d] Velocity > 1,500 m/s	Hole in Plate	End-to-End Pipe Fragmentation	Type Reaction[e]
1	19	81	0	1.26	8	+			+
2	19	81	0	1.31	8	+	-	+	-
3	19	81	0	1.26	f	-	-	-	-
4	19	81	0	1.29	3,796	+	-	-	-
5	19	81	0	1.30	2,179	+	-	-	-
6	19	81	0	1.26	1,072	-	-	-	-
7	19	81	0	1.26	1,419	-	-	-	-
8	19	81	0	1.26	1,166	-	-	-	-
9	19	81	0	1.24	1,473	-	-	+	-
10	19	81	0	1.27	2,032	+	-	-	-
11	19	81	0	1.27	2,748	+	-	-	-
				Averages = 1.271	1,921				

[a]Type II, Class 1.

[b]Moisture in sand = 0.25%.

[c]16-in. long steel tubing; 1.46-in. I.D.; 0.22-in. wall thickness.

[d]"+" indicates positive result. "-" indicates negative result. See Appendix B for further description of BCM criteria.

[e]"+" indicates positive result; 2 or 3 criteria are positive and therefore the test indicates sustained propagation of the shock wave through the sample.
"-" indicates negative result. See Appendix B for further description of BCM criteria.

[f]Decaying reaction. No steady state velocity in sample.

[g]Propagation rate not recorded - Oscilloscope trigger did not function.

[h]Insufficient criteria to determine if reaction was positive or negative.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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Table 5

Summary of DDT Test Results for TNT/Sand Mixtures

Trial No.	Composition, % TNT[a]	Sand[b]	Water	Loading Density, g/cc	Type Reaction[c]
1	17	83	0	1.32	-
2	17	83	0	1.28	-
3	17	83	0	1.32	-
4	17	83	0	1.33	-
5	17	83	0	1.28	-
6	17	83	0	1.29	-
7	17	83	0	1.32	-
8	17	83	0	1.32	-
9	17	83	0	1.32	-
10	17	83	0	1.30	-
			Average =	1.309	

[a]Type II, Class 1.

[b]Sand = 0.25% water wet.

[c]"+" indicates positive result - that the pipe or an end cap fragmented into 2 or more distinct pieces; "-" indicates negative result. See Appendix B for further description of BOM criteria.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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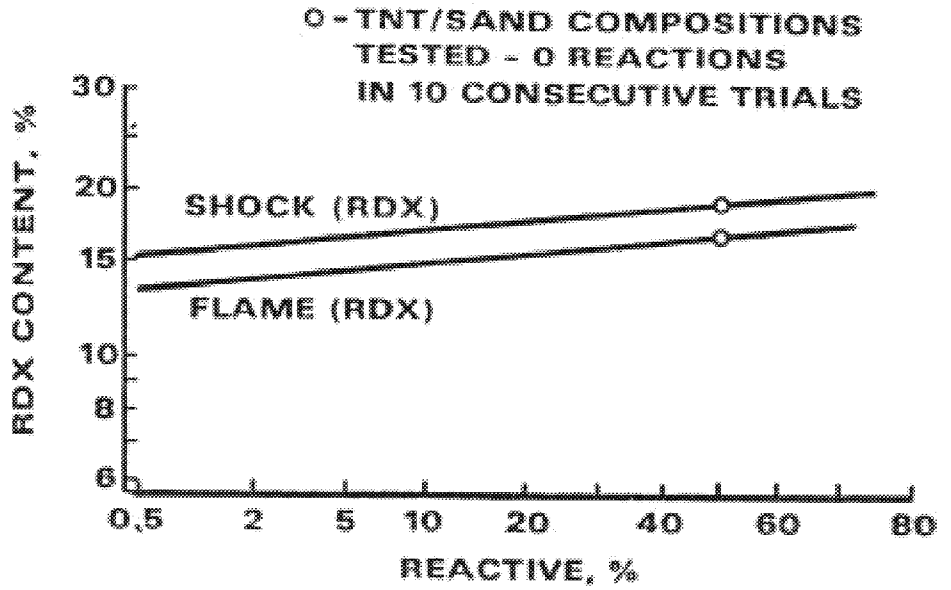

Figure 5. Dry RDX/Sand vs Dry TNT/Sand Reactivity

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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DDT and Zero Gap tests with TNT verified that TNT is actually less reactive than RDX used to establish figure 1 reactivity criteria. This study's findings further confirm that the sample reactivity based on compositional analyses can be used to predict the reactivity of contaminated soils in BOM flame and shock tests.

3.0 Experimental

The following sections describe the test plan, selection of test materials, mix preparation and subsequent uniformity testing, C_d tests, BOM Zero Gap tests and BOM DDT tests.

3.1 Overall Test Plan

Major explosive contaminates in Army lagoons were identified from available analyses (see Table 6). The initiation sensitivity and explosive reactivity of the major solid explosive components were assembled from Hercules data files and the literature and compared to establish which are more sensitive/reactive than the others (Table 7). Based upon these analyses and data, explosive and inert test materials were selected for BOM flame and shock tests. Initial tests were conducted with various compositions of these materials using the standard critical diameter for explosive propagation test protocols to: (1) identify compositions which should be unreactive in the BOM shock test and (2) establish the relationships between composition, reactivity and pipe diameter. Laboratory prepared compositions were then tested using BOM Zero Gap test protocols to determine compositions which were reactive and non-reactive in this test. Various compositions were then tested using BOM DDT test protocols to determine compositions reactive and non-reactive in this test. Test results were evaluated statistically and presented for use in determining explosive-contaminated soil compositions which can be classified as reactive or non-reactive to the BOM tests based upon chemical analysis.

3.2 Selection of Test Sample Materials

3.2.1 General

The reactivity of Army lagoon sludges will depend upon the type of explosive present, its concentration in the non-reactive (inert) components and the degree of confinement afforded by the inerts in handling and storage containers. Typical soil analyses from two Army lagoons are shown in Table 6. The data is based upon chemical analyses of explosives-contaminated sludges from Savanna Army Depot (SAD) and Louisiana Army Ammunition Plant (LAAP)[6] - see Appendix D. These analyses show that the principal solid explosives present are TNT, RDX and HMX. Other solid components include water, sand, clay and low ($\leq$0.1%) concentrations of other explosives and heavy metals.

3.2.2 Explosive Component

A review of initiation sensitivity and explosive reactivity data summarized in Table 7 shows that RDX and HMX exhibit similar initiation characteristics when subjected to mechanical, electrostatic and thermal

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Table 6

Typical Army Lagoon Sludge Compositions[a]

Component	Range, % (Dry Basis)[b]
A. Explosive:	
1. TNT	5-41
2. RDX	0.1-10
3. HMX	0.5-1.5
4. TNB, DNB, 2-Amino, DNT	ND -0.1
Total Explosives Content	9-41
B. Inerts:	
1. Sand 2. Clay	≥ 52

[a]Based upon analyses shown in Appendix D.

[b]Moisture content ranged from 11 to 30%.

ND - None Detected

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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Table 7

Comparison of RDX, HMX and TNT Initiation, Flame and Shock Sensitivity Characteristics[a]

Initiation Stimuli	Units	Test Conditions	TNT	RDX	HMX
1. Mechanical					
a. Impact, TIL[b]	ft-lb/in.^{2}	Steel/steel	10.2	13.3	3
b. Sliding Friction, TIL[b]	psi @ 8 fps	Steel/steel	70,000	21,000	23,000
2. Electrostatic Spark Discharge, TIL[b]	Joules	N/A	0.025	0.024	0.065
3. Thermal					
a. Differential Thermal Analysis	°C	–	300	232	~ 280
b. Explosion Temperature	°C	Ignition in 1 s	520	316	327 (in 5 s)
4. Flame					
a. Critical Height to Explosion		Schedule 40 Steel Pipe			
2-in. diameter	In.		12	2	3
4-in. diameter	In.		≥ 24	5	7
5. Shock					
a. Detonation Velocity	m/s	–	6,825	8,180[c]	9,124
b. Critical diameter for explosive propagation	In.	Schedule 40 Steel Pipe	≤ 0.27	≤ 0.27	≤ 0.27
c. Rifle Bullet Impact	N/A	30 caliber	40% Expl. 60% Unaff.	100% Expl.	–

[a]See Glossary in Appendix G for definitions and test criteria.

[b]Lowest values included only. Higher values available reflect effect of sample thickness, particle size, density, etc.

[c]Pressed pellet; density = 1.65 g/cc.

NA – not applicable

Source: RAAP materials sensitivity laboratory files and AMC Pamphlet 706-177, "Explosive Series, Properties of Explosives of Military Interest," March 1967.

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stimuli. When confined and subjected to submerged flame initiation (critical height test), each transits from burning to an explosion reaction at low sample heights. Both materials sustain a detonation reaction and have critical diameters for explosive propagation of ≤0.27 inch in schedule 40 steel pipe. For purposes of this study, it is concluded that RDX and HMX are equivalent in initiation sensitivity and explosive reactivity.

A comparison of RDX, TNT and HMX initiation sensitivity and explosive reactivity data in Table 7 shows that TNT reacts similarly to impact and electrostatic discharge stimuli. However, TNT is much Less sensitive to sliding friction and thermal stimuli as it requires greater energy for initiation. Flaked TNT is also less likely to transit to detonation as evidenced by a critical height of ~24 inches in 4-inch diameter confinement. In contrast, RDX and HMX have critical heights of 5 and 7 inches, respectively, in the same confinement.

TNT, RDX and HMX are all capable of detonation in small diameters (≤0.27 inch). The TNT shock wave propagation rate is slower (6,825 m/s) than those of RDX and HMX (8,180 and 9,120 m/s, respectively). From this comparison, it is concluded that TNT is no more initiation sensitive and a less reactive explosive than RDX and HMX.

It is concluded that the selection of either RDX or HMX, rather than TNT, for BOM flame and shock testing will result in a conservative estimate of explosive reactivity for compositions containing TNT or other secondary explosives of equal sensitivity in these tests. Since typical lagoon analyses indicate that there is up to 6 times more RDX than HMX in the lagoons, RDX was selected as the candidate explosive for use in this study. The presence of small concentrations (≤0.1%) of explosives other than TNT, RDX or HMX will have a negligible effect upon the overall reactivity of sludge.

Type II, Class 1 RDX[7] was purchased from Holston Defense Corporation for use in this study. A Holston analysis of the RDX is shown in Appendix E. A RAAP chemical analysis of the Type II RDX determined that it contained 8.6% HMX and 2.8% of other nitramine variations formed during RDX manufacture.

Limited testing was also conducted with TNT fines obtained from the RAAP TNT Plant. Chemical analysis determined it to contain 99.84% 2,4,6 TNT, 0.1% 2,3,4 TNT, and small amounts (0.06% total) of DNT and water. The TNT particle size distribution was determined microscopically by measuring 200 particles and plotting the data to form a distribution curve (Figure 6). The distribution curve indicates that most TNT particles fall in the range of 3 μm to 200 μm (average ~14 μm). Some of the larger particles measured were agglomerates instead of single crystals.

3.2.3 Inert Components

3.2.3.1 Soil

Soil samples from SAD and LAAP were characterized as shown in Table 8. Using U. S. Bureau of Public Roads soil-classification protocol (Figure 7), the LAAP soil was identified as loamy sand and the SAD soil as sand.

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Table 8

Summary of Soil Characterization Tests

	Bulk Density, g/cc	Sieve Analysis Sieve, μ	Sieve Analysis % Retained	% Organic Matter	Type of Soil[a]
LAAP	0.99	420	30.48	6.06	Loamy Sand
		105	20.27		
		75	37.05		
		45	12.02		
		45	0.14[b]		
SAD	1.39	420	13.95	c	Sand
		105	78.48		
		75	5.06		
		45	1.93		
		45	0.58[b]		
RAAP Sand					
Sample No. 1	1.38[d]	420	17.52	1 to 4	Sand
		105	78.48		
		75	2.43		
		45	1.48		
		45	0.61[b]		
Sample No. 2	-	420	13.61	c	Sand
		105	66.94		
		75	8.43		
		45	6.83		
		45	4.19[b]		

[a]Based upon U. S. Bureau of Roads protocol (see Figure 7).

[b]Percent passing through sieve.

[c]Not determined.

[d]Average of 5 measurements in 16-in. long, 1.5-in. steel pipe (430 ml volume).

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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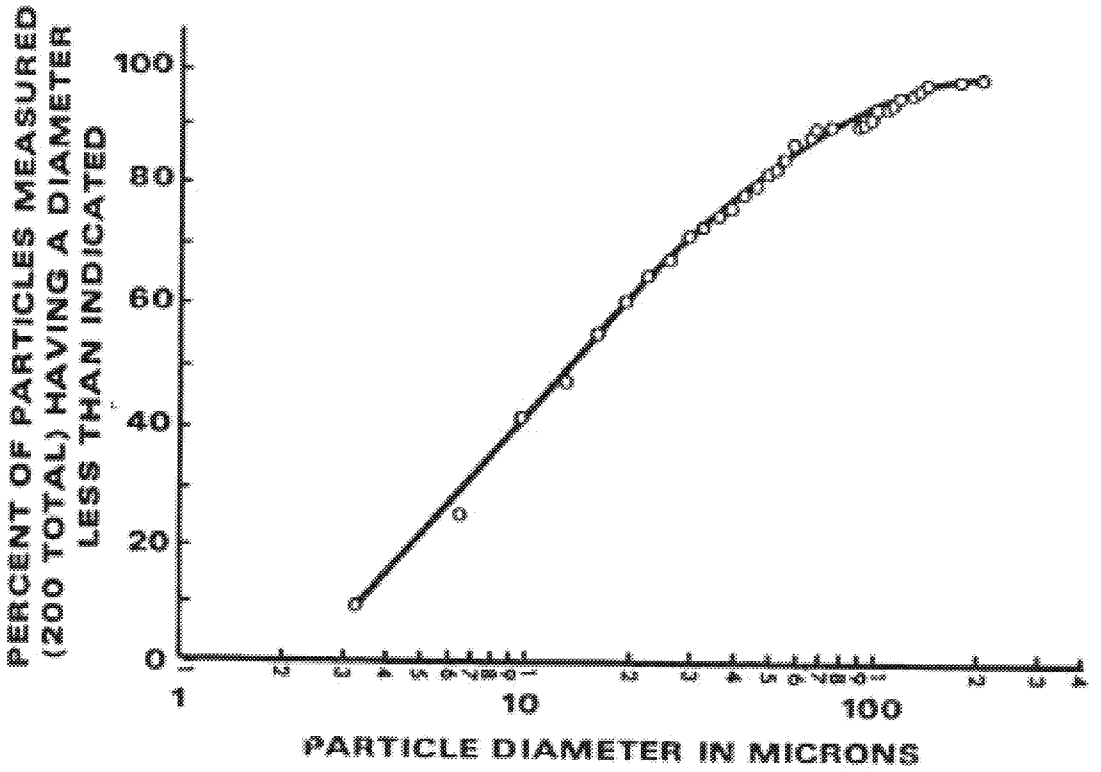

Figure 6. TNT Fines Particle Size Distribution

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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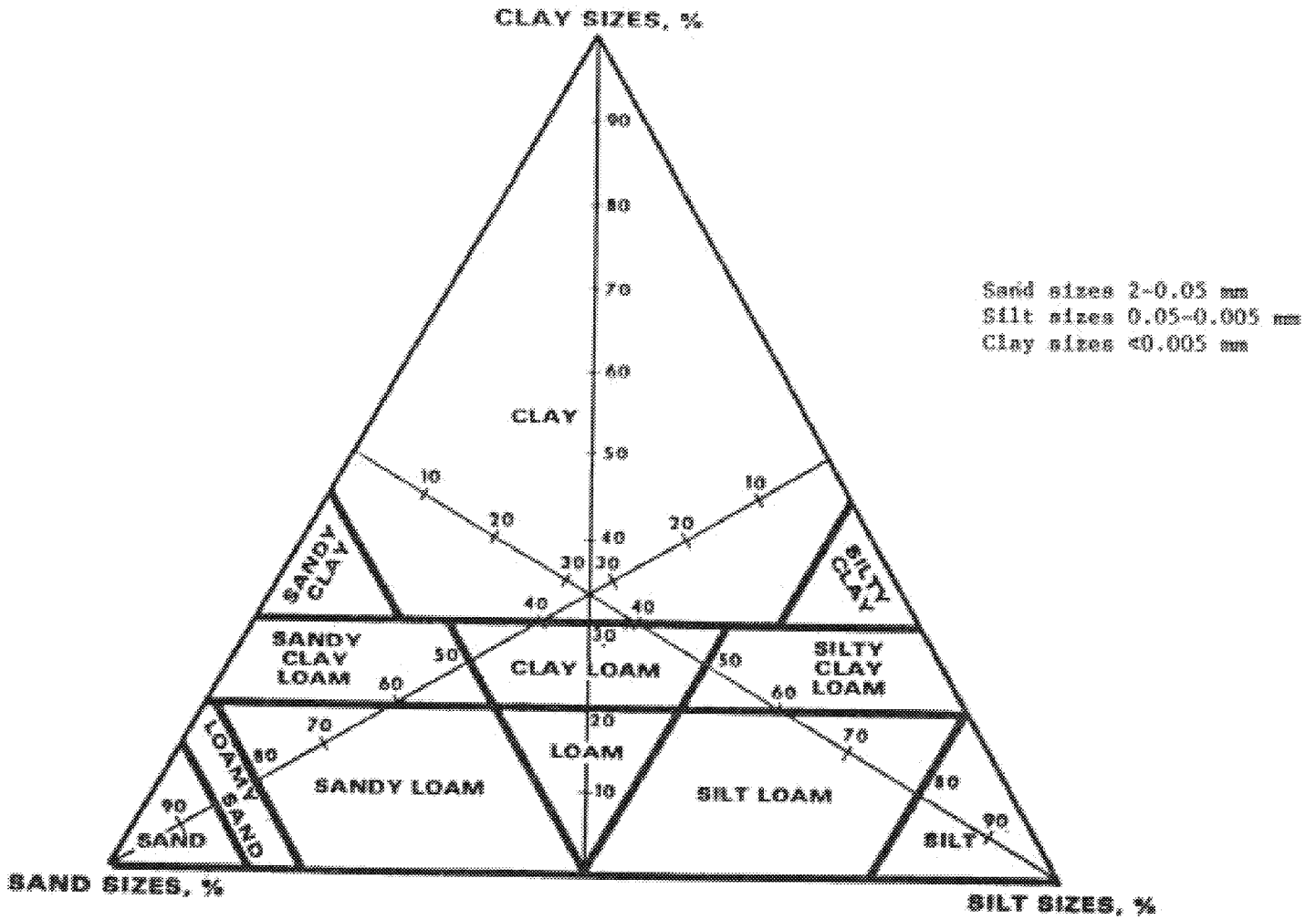

Sand sizes 2-0.05 mm
Silt sizes 0.05-0.005 mm
Clay sizes <0.005 mm

Figure 7. Soil Classification by Particle Size

Source: Adopted from U.S. Bureau of Public Roads

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Several graded and ungraded sand and soil samples taken and analyzed at RAAP identified a New River sand bar sample which closely matches the SAD soil sample (see Table 8). Approximately 2,000 lb of New River ungraded sand was placed in cotton bags, air dried at 140°F for 48 hours, passed through a 20-mesh screen to remove foreign material (grass, branches, roots, rocks) and used in this study.

3.2.3.2 Water

Since Army lagoon sludges also contain up to 30% water (Table 6), both water-wet and dry RDX/sand mixtures were investigated in this study. Support laboratory tests conducted with a one liter graduated cylinder and beam balance determined that settled beds of sand or Type II, Class 1 RDX in water contain 20.0% and 22.9% (wt. basis) water, respectively. The addition of more water results in a layer of water above the settled RDX/sand mixture (two phases). The presence of a water head above a settled RDX/sand/water mixture should have little effect upon the reactivity of the settled RDX/sand mixture to flame or shock. Furthermore, Zero Gap and DDT test configurations are not very well suited for testing two phase systems. Since most flame and shock tests were conducted with RDX/sand mixtures containing more sand than RDX, all trials conducted with settled RDX/sand in water mixtures were conducted with 20% (wt) water added. Visual inspection of 20% water-wet RDX/sand mixtures after loading into test pipes showed a thin water layer on top of samples indicating that all intergranular voids were full of water. Partly water-wet beds of RDX/sand mixtures were also tested with 10% water added.

3.3 Mix Preparation

3.3.1 Blending

Portions of RDX or TNT, sand and water (when required) were weighed to ±1 gram and manually tumbled together to achieve a uniform mixture immediately before loading in test pipes. Mixes weighing up to 30 lb were prepared in sealed, conductive plastic bags in contact with a grounded, conductive surface to minimize the risk of electrostatic initiation of the explosive. Mixes were kept sealed in the plastic bags until used in tests to preclude loss of moisture by evaporation.

3.3.2 Mix Uniformity

A number of RDX/sand/water mixes were sampled to verify proper composition and mix uniformity. Sample analyses were conducted as described in Appendix F, are summarized in Table 9 and show that mix moisture contents were within ±1% of the desired moisture content in all 45 samples (40 separate mixes). The moisture content measured in "dry" RDX/sand mixes was introduced by the slightly moisture-wet (0.8-0.2%) sand added to each mix. It is concluded that sample preparation techniques employed yielded acceptable levels and uniformity of moisture content.

Duplicate samples were taken from five mixes (see Table 9) and analyzed for chemical composition. Inspection of these data shows that the Type II RDX (RDX/HMX/etc.) content varied between mix samples by ≤1.05%.

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Table 9

RDX/Sand/Water Mix Compositional Analyses

Prepared Sample Composition RDX-Sand[a]-Water, %	Mix wt. lb	Chemical Analysis,[b] % RDX[c]	Sand	Water	Difference Between Analyses and Prepared Compositions, % RDX[c]	Water
10-90-0	3	9.73	89.57	0.68	-0.23	-0.68
10-70-20	6	9.64	70.95	19.41	-0.36	-0.59
10-70-20	12	9.19	71.33	19.56	-0.81	-0.44
15-85-0	6	14.03	85.04	0.91	-0.95	0.91
15-85-0	12	13.66	85.36	0.98	-1.34	0.98
15-85-0	24	15.32	84.47	0.21	0.32	0.21
15-85-0	*	15.38	84.20	0.22	0.38	0.22
15-65-20	6	13.61	67.15	19.24	-1.39	-0.76
15-65-20	4	14.13	66.69	19.19	-0.88	-0.81
15-65-20	8	13.52	67.05	19.43	-1.48	-0.57
20-80-0	12	18.45	80.85	0.70	-1.55	0.70
20-80-0	*	19.30	79.93	0.77	-0.70	0.77
20-80-0	24	19.26	80.54	0.20	-0.74	0.20
20-80-0	*	18.70	81.11	0.19	-1.30	0.19
20-70-10	3	20.34	69.57	10.09	0.34	0.09
20-70-10	12	19.76	70.24	9.98	-0.24	-0.02
20-68-12	3	19.38	68.69	11.93	-0.62	-0.07
20-60-20	4	18.60	61.27	20.13	-1.40	0.13
20-60-20	8	19.07	61.62	19.31	-0.93	-0.69
20-60-20	4	18.86	61.78	19.36	-1.14	-0.64
20-60-20	2	18.79	61.90	19.31	-1.21	-0.69
20-34.2-23.8	4	18.87	36.11	25.02	-1.13	-0.78
25-75-0	3	22.83	76.34	0.83	-2.17	0.83
25-75-0	2	24.42	74.91	0.67	-0.58	0.67
25-75-0	4	23.52	75.93	0.55	-1.48	0.55
25-75-0	1	23.34	76.17	0.49	-1.66	0.49
25-75-0	24	25.86	74.02	0.12	0.14	0.12
25-75-0	*	25.06	74.77	0.17	0.06	0.17
25-75-0	24	24.96	74.83	0.21	-0.04	0.21
25-75-0	*	26.01	73.78	0.21	1.01	0.21
25-65-10	3	24.88	65.19	9.93	-0.12	-0.07
25-55-20	2	24.60	55.76	19.64	-0.40	-0.36
25-55-20	2	23.67	56.69	19.64	-1.33	-0.36
30-70-0	1	29.42	70.14	0.44	-0.58	0.44
30-60-10	3	30.43	59.64	9.93	0.43	-0.07
30-60-10	1	29.62	60.78	9.60	-0.38	-0.40
30-60-10	2	29.90	59.98	10.12	-0.10	0.12
30-60-10	1	28.01	62.94	9.05	-1.99	-0.95
30-50-20	2	29.15	51.59	19.26	-0.85	-0.74
35-65-0	1	34.39	65.27	0.34	-0.61	0.34
35-55-10	1	33.29	57.42	9.29	-1.71	-0.71
35-55-10	1	33.17	57.28	9.55	-1.83	-0.45
35-55-10	1	33.74	56.26	9.44	-1.26	-0.56
40-50-10	1	38.13	52.68	9.19	-1.87	-0.81
50-50-0	3	50.16	49.42	0.41	0.16	0.42
				Max.	* 1.01	0.98
				Min.	* -2.17	-0.95
				Average	* -0.76	-0.04
				Variance	* 0.554	0.298
				Std. Dev.	* 0.744	0.546

*Duplicate sample from preceding mix.

[a]Analyses show that moisture in the sand varied from - 0.8% to + 0.20% during testing.

[b]Analysis Techniques: (a) "Dry" Samples - Moisture determined using Gas Chromatography. HMX and RDX dissolved into acetonitrile. Sand filtered out. HMX and RDX concentrations determined using Liquid Chromatography; (b) "Wet" Samples - Samples air dried + 3 days. Moisture determined by weight difference before and after drying. HMX and RDX concentrations determined with LC as described for "Dry" samples.

[c]Corrected to include 88.4% RDX, 8.6% HMX and 2.8% of other nitramine variations resulting from the RDX manufacturing process and present in the Type II RDX from Holston Defense Corporation.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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This data also indicates that the sample preparation technique used yielded an acceptable level of mix uniformity. However, a comparison between laboratory analyses and the sample compositions prepared shows that the RDX analyses ranged from 1.0% greater to 2.2% lower than expected values. Most RDX analyses are lower and average 0.76% less than expected. Inspection of Table 9 indicates that the greatest RDX analysis-to-expected variability generally occurred in small mixes containing no water. A review of mix weighing records indicates that the proper weights of RDX and sand were added to the mixes. The apparent shift in analysis-to-expected compositions may be caused by errors introduced by small, non-representative samples, analysis techniques, RDX impurities or other. Further investigation to determine the cause(s) of the analysis-to-expected differences was not pursued further because it was small and not expected to affect sample reactivity test results significantly.

Analyses of TNT and sand mixtures are presented in Table 10. These analyses were also an average of 0.64% lower in expected explosive (TNT) content. Chemical analysis of duplicate samples from four mixes show that TNT/sand mix uniformity is not quite as good as RDX/sand mix uniformity, but is acceptable for the tests conducted (Table 10).

3.4 Critical Diameter (C_d) Screening Tests

For all explosive materials, there is a dimension which is too small to sustain propagation of a shock wave through the explosive. Generally, the more reactive the explosive the smaller the critical dimension capable of propagating an explosive reaction. Critical diameter is dependent upon confinement, density, composition, etc. Stronger test container confinements are expected to reduce the explosives critical diameter. Critical diameter tests are normally conducted in 24-inch long, schedule 40 steel pipe as described and shown in Appendix A. Use of schedule 40 steel pipe generates critical diameter data useful in evaluating the risk of sustained explosive reactions in typical explosive processing and storage operations.

3.5 Zero Gap Shock Tests

3.5.1 General

Wet and dry RDX/sand mixtures were tested to define mixture reactivity to shock as a function of RDX content using the BOM Zero Gap shock test protocol described in Appendix B. Water-wet and dry mixtures of RDX and sand were confined in steel tubing (Figure B1) and subjected to induced shock of two Pentolite pellets. The RDX content in wet and dry sand mixtures was varied to identify RDX levels which react explosively to shock as defined by the BOM test protocol.

3.5.2 Analysis

Standard Probit analysis techniques[5] were used to establish an RDX level in wet and dry sand mixtures that has a low (0.5%) probability of reacting to shock in the BOM Zero Gap test configuration. Ten test trials were conducted for each wet and dry RDX/sand composition tested to obtain percent reaction data; i.e., <u>some</u> of the trials reacted positively to induced shock. Since only 10 trials were conducted at each RDX level, resulting

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Table 10

TNT/Sand Compositional Analyses

Sample No.	Prepared Sample Composition, TNT-Sand,[a] %	Mix wt. lb	Chemical Analysis,[b] % TNT[c]	Chemical Analysis,[b] % Sand	Difference Between Lab Analyses and Prepared TNT Composition, %
1	17-83	30	15.79	84.21	-1.21
2	17-83	*	15.70	84.30	-1.30
3	17-83	30	17.41	82.59	0.41
4	17-83	*	16.18	83.82	-0.82
5	19-81	17	18.08	81.92	-0.92
6	19-81	*	19.79	80.21	0.79
7	19-81	17	18.33	81.67	-0.67
8	19-81	*	17.56	82.44	-1.44

Max. = 0.79
Min. = -1.44
Average = -0.64
Std. Dev. = 0.763

[a]Sand = 0.25% water-wet.

[b]Analysis Techniques - see Appendix F.

[c]TNT fines from Radford AAP TNT Plant. Chemical analysis showed it to be 99.94% pure TNT.

*Duplicate sample from preceding mix.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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probabilities of a positive reaction ranged from 10 to 90% in increments of 10. The percent reactive data was plotted on probability paper to convert a logarithmic function between the probability of a positive reaction in the Zero Gap test, and the RDX content in dry and moisture-wet samples tested to a straight line. Then a straight line was drawn through the data and extrapolated to the 0.5% reactive level. The RDX level expected to react positively at the 0.5% level was determined from the extrapolated plot and tested to verify that the wet or dry RDX/sand composition has a low level of reactivity in the BOM Zero Gap test. Verification testing was accomplished by conducting 20 confirmatory trials with the predicted 0.5% reactive composition. Statistically, there was a 90% chance of achieving 0 positive reactions in 20 consecutive trials. Achievement of no reactions in 20 consecutive trials was accepted as proof of low composition reactivity.

3.6 Deflagration to Detonation Transition (DDT) Tests

3.6.1 General

Wet and dry RDX/sand mixtures were tested using the BOM DDT test described in Appendix B and shown in Figure B2. In this test, samples were confined in 3-inch, schedule 80 steel pipe and subjected to flame from a 20-gram igniter. RDX/sand moisture compositions reacting explosively were identified using BOM test protocols.

3.6.2 Analysis

Standard probit analysis techniques[5] were used to establish an RDX level in wet and dry sand that has a low (0.5%) probability of reacting to flame in the BOM DDT test configuration. The testing scheme was conducted the same as described previously in the Shock Test Plan.

3.6.3 Test Container Assembly

Assembly of the DDT test container includes installation of steel caps on both ends of threaded, schedule 80 steel pipe. Installation of the second cap is performed after the pipe is filled with the explosive RDX/sand test sample. This operation produces frictional heating between the mating metal cap and pipe threads. The potential exists for sample initiation if the threads could become contaminated. Ignition of sample in the pipe threads during manual torquing operations could result in propagation of hot decomposition gases or incandescent particles to the bulk test sample inside the pipe. Ignition of the highly confined RDX/sand test sample could result in an explosive reaction and possible personnel injury. Although the test container assembly procedure is designed to minimize thread contamination, the potential for operator injury during a manual pipe cap torquing operation was an unacceptable risk.

Consequently, prior to beginning the flame DDT tests, the special test fixture shown in Figure B was designed and fabricated to remotely torque pipe caps on loaded pipes. The torquing fixture protects personnel from the consequences of an accidental initiation of explosive during pipe cap installation. The pipe cap torquing fixture consists of a chain vise to hold the loaded test container stationary, a roller cam lock cap gripping assembly, an air operated impact wrench to turn the cap gripping assembly, and

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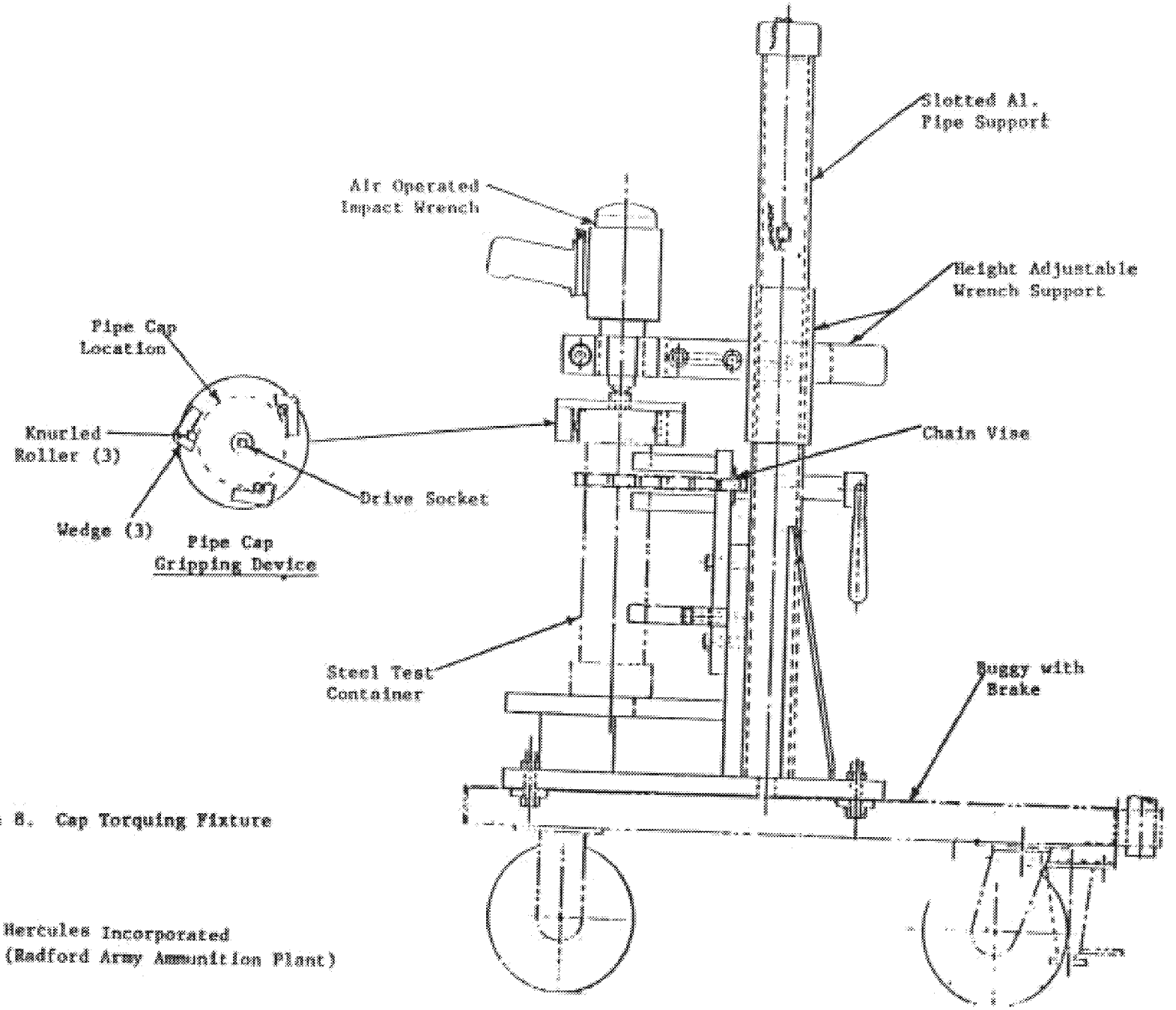

Figure 8. Cap Torquing Fixture

Source: Hercules Incorporated
(Radford Army Ammunition Plant)

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supporting members. No accidental initiations occurred during the remote container DDT test assembly operations.

3.6.4 Test Container Disassembly

Operating procedures were developed to protect test personnel from possible delayed thermal cookoff reactions in the case of no sample reactions. Test trials in which the sample was not sufficiently energetic to rupture the pipe or cap, posed the risk of an explosive reaction during subsequent disposal operations. Manual removal of a pipe cap from the closed container was an unacceptable risk due to the possibility of a delayed thermal cookoff reaction in the test sample. Previous procedures required a 24 hour waiting period before entering the barricaded test area. To enhance personnel safety and minimize waiting times, a Composition C-4 destruct charge was taped to the outside of the test container at setup time. If a negative sample reaction (no explosion) was ascertained, the Composition C-4 destruct charge was initiated remotely to punch a hole through the pipe wall and vent the test container. The RDX/sand mixture was then washed out of the pipe via the hole before manual removal of pipe caps from the remaining pipe section(s). Testing conducted with sand filled, capped pipe determined that a 0.5 lb Composition C-4 charge weight and hollow cone configuration were sufficient to punch a hole through the pipe wall. Subsequent DDT tests demonstrated that this safety technique reduced test time and did not affect DDT test results - even reactive DDT trials with RDX/sand mixtures did not initiate the Composition C-4 charge. It is likely that very reactive samples could initiate the Composition C-4 charge, but the test result would not be changed by the Composition C-4 reaction since the very reactive sample would test positive to flame initiation anyway.

3.6.5 Bulk Density Measurements

The void volume in the schedule 80 steel pipe test fixtures was variable due to dimensional variation of the pipe and pipe cap threads. Some caps would screw down more than others and decrease the void volume. Bulk densities were calculated using the weight of sample required to fill the test unit and estimated void volume determined by measurement of unit components. Average bulk densities for the RDX/sand compositions tested are comparable to bulk densities obtained for similar compositions during Zero Gap testing.

4.0 WARRANTY AND DISCLAIMER

Within the scope of work, Hercules warrants that it has exercised its best efforts in performing the hazards analysis and testing reported herein, but specifically disclaims any warranty, expressed or implied, that hazards or accidents will be completely eliminated or that any particular standard or criterion of hazard or accident elimination has been achieved if the findings and recommendations of Hercules Incorporated are adopted.

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5.0 REFERENCES

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6 J. W. Noland, J. R. Marks, P. J. Marks, "Task 2, Incineration Test of Explosives Contaminated Soils at Savanna Army Depot Activity, Savanna, Illinois," Roy F. Weston, Inc., Final Report, DRXTH-TE-CR-84277, April 1984.

7 Department of Defense, "Military Specification RDX," MIL-R-398C with Int. Amendment 5, 1 May 1978.

8 R. B. Elwell, et. al., "Project SOPHY - Solid Propellant Hazards Program," Aerojet-General Corporation, Technical Report AFRPL-TR-67-211-VOL I, August 1967.

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APPENDIX A

CRITICAL DIAMETER (C_d)

TEST DESCRIPTION

AND RESULTS

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APPENDIX A

Critical diameter (C_d) for propagation

OBJECTIVE. To determine if a material will propagate an explosive reaction when subjected to induced shock and to establish the critical dimension for nonpropagation .

OPERATING PRINCIPLE. Materials are purposely shocked by pressures of a detonating high-energy donor to determine if a material dimension is capable of propagating an explosive reaction. The dimensions of the material are varied under specific environmental process conditions to establish the critical nonpropagating dimension.

TEST DESCRIPTION. The test arrangement for determining the critical non-propagating diameter for wet and dry KDX/sand mixtures is shown in Figure A1. Schedule 40 steel containers were charged with the material to be tested and subjected to induced shock produced by a high-energy donor material. The explosive donor diameter was equal to that of the test specimen and had a minimum length over diameter ratio (L/D) equal to 3:1 plus one inch for the initiating cap. This minimum ratio presumably allows the donor-induced shock wave to achieve constant velocity and maximum radius of curvature at the sample interface.

A pressure activated velocity probe and visual inspection of pipe remains after the test were used to ascertain that a material dimension propagated an explosion reaction in any particular test trial. A resistance wire probe was used to monitor the reaction velocity the entire sample length. In principle, the pressure front accompanying the reaction collapses a metal tube onto a resistance wire, producing a change in the circuit resistance and a corresponding change in the magnitude of the input voltage signal to an oscilloscope. The voltage signal, interpreted as distance (material length) and expressed as a function of time, provides a continuous velocity profile for studying the reaction rate through the entire sample length. Visual inspection of pipe remains provides a go-no go indication of propagation. If the pipe is fragmented from one end to the other, propagation occurred. The test container diameter is normally varied in 0.25 inch increments until a diameter is reached at which the material fails to propagate an explosion reaction. A minimum of three trials is performed at this level to establish the nonpropagating material diameter. Three trials are sufficient to establish C_d because C_d is a unique and sharply defined property of explosives and explosive compositions.

TEST ANALYSIS AND LIMITATIONS. Critical diameter data are reported as the material diameter (inches) at which an explosion reaction will not be propagated. Degree of confinement can influence test results and thus must be considered in applying the data.

Reference: Hercules Aerospace Company (RAAP)

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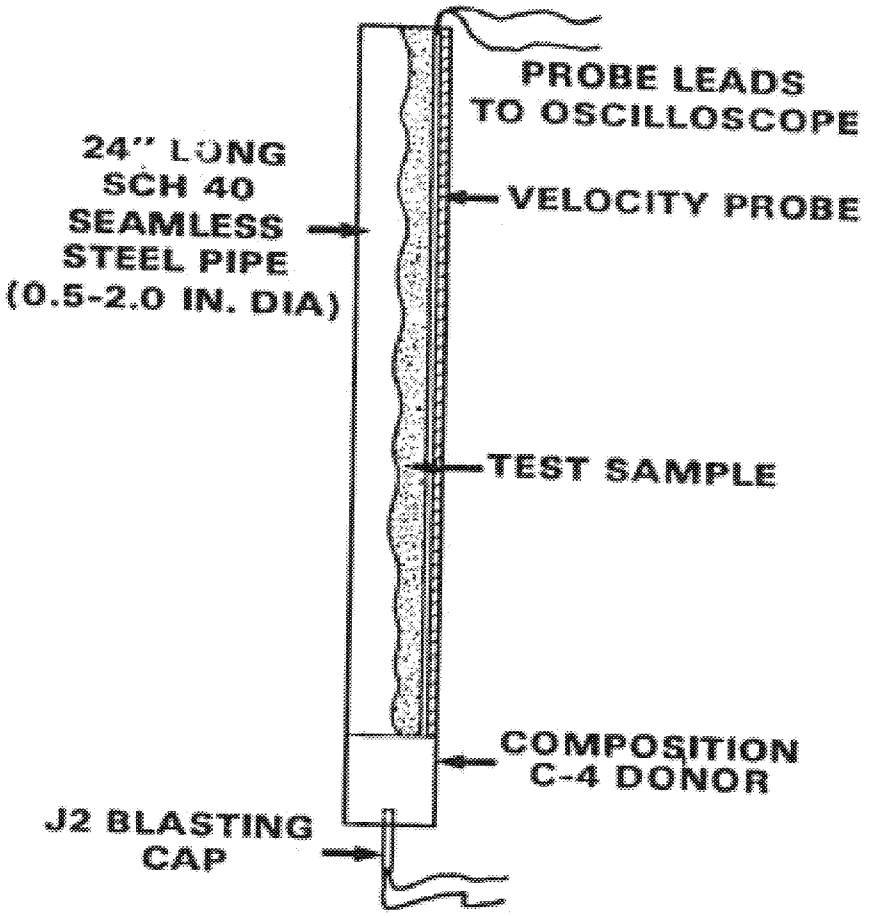

Figure A1

C_d TEST ARRANGEMENT

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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Table A1

Critical Diameter for Explosion Test Results

Trial No.	Composition, % RDX[a]	Sand[b]	Water	Loading Density, g/cc	Nominal Sch. 40 Pipe Size,[c] in.	End-to-End Pipe Fragmentation[d]	Shock Propagation Velocity Thru Sample, m/s
1	75	25	0	1.12	0.5	+	-
2	50	50	0	1.04	0.5	+	-
3	35	65	0	1.25	0.5	+	4,314
4	30	70	0	1.21	0.5	+	-
5	25	75	0	1.22	0.5	-	-
6	25	75	0	1.18	0.5	-	-
7	25	75	0	1.22	0.5	-	-
8	30	70	0	1.31	1.0	+	-
9	25	75	0	1.25	1.0	-	-
10	25	75	0	1.19	1.0	-	-
11	25	75	0	1.21	1.0	-	-
12	50	50	0	1.17	1.5	+	3,648
13	25	75	0	1.23	1.5	+	-
14	20	80	0	1.19	1.5	-	-
15	20	80	0	1.32	1.5	-	-
16	20	80	0	1.27	1.5	-	-
17	10	90	0	1.25	1.5	-	-
18	20	80	0	1.32	2.0	+	-
19	15	85	0	1.28	2.0	-	-
20	15	85	0	1.15	2.0	-	-
21	15	85	0	1.31	2.0	-	-
22	40	50	10	1.192	0.5	-	-
23	40	50	10	1.295	0.5	+	-
24	35	55	10	1.280	0.5	-	1,072
25	35	55	10	1.221	0.5	-	-
26	35	55	10	1.369	0.5	-	-
27	30	60	10	1.176	0.5	-	-
28	35	55	10	1.219	1.0	-	-
29	35	55	10	1.188	1.0	+	3,300
30	30	60	10	1.261	1.0	-	-
31	30	60	10	1.177	1.0	-	-
32	30	60	10	1.240	1.0	-	-
33	30	60	10	1.207	1.5	+	3,366
34	25	65	10	1.231	1.5	-	-
35	25	65	10	1.324	1.5	-	-
36	25	65	10	1.287	1.5	-	-
37	20	70	10	1.347	1.5	-	-
38	25	65	10	1.073	2.0	-	-
39	25	65	10	1.300	2.0	+	2,255
40	20	70	10	1.037	2.0	-	-
41	20	70	10	1.246	2.0	-	-
42	20	70	10	1.315	2.0	-	-
43	35	45	20	1.408	0.5	+	-
44	30	50	20	1.251	0.5	-	-
45	30	50	20	1.668	0.5	+	4,512
46	25	55	20	1.727	0.5	-	-
47	25	55	20	1.697	0.5	-	-
48	25	55	20	1.816	0.5	-	-
49	30	50	20	1.668	1.0	+	-
50	25	55	20	1.642	1.0	-	-
51	25	55	20	1.709	1.0	-	-
52	25	55	20	1.678	1.0	+	-
53	20	60	20	1.673	1.0	-	-
54	20	60	20	1.741	1.0	-	-
55	20	60	20	1.704	1.0	-	-

Table A1 (CONT)

Trial No.	Composition, % RDX[a]	Sand[b]	Water	Loading Density, g/cc	Nominal Sch. 40 Pipe Size,[c] in.	End-to-End Pipe Fragmentation[d]	Shock Propagation Velocity Thru Sample, m/s
56	20	60	20	1.860	1.5	+	4,127
57	20	60	20	1.736	1.5	+	-
58	15	65	20	1.794	1.5	-	-
59	15	65	20	1.834	1.5	-	-
60	15	65	20	1.821	1.5	-	-
61	20	60	20	1.818	2.0	+	3,839
62	15	65	20	1.908	2.0	+	3,844
63	10	70	20	1.811	2.0	-	-
64	10	70	20	1.793	2.0	-	-
65	10	70	20	1.819	2.0	-	-
66	-	100	-	1.380	2.0	-	-
67	-	-	100	0.973	2.0	-	-

[a]Type II, Class 1. Bulk density averaged 1.11 g/cc.

[b]Moisture in sand ranged from 0.8 to 0.1%. Sand bulk density averaged 1.40 g/cc.

[c]Typical pipe inside diameter for nominal sch. 40 pipe: 0.5-in. Nom. = 0.622 in.; 1.0-in. Nom. = 1.049 in.; 1.5-in. Nom. = 1.610 in.; 2.0-in. Nom. = 2.067".

[d]"+" designates positive results; Pipe fragmented entire length of 24-in. long pipe. "-" designates negative result; Sample did not sustain shock wave propagation.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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APPENDIX B

PROCEDURES FOR THE CLASSIFICATION OF EXPLOSIVE SUBSTANCES AND TEST RESULTS

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APPENDIX B

Procedures for the classification of explosive substances

These tests determine whether the substance is explosive. Two tests are used to determine the response of the substance under test to a strong shock wave and to a strong thermal stimulus: The Bureau of Mines Gap Test and the Bureau's Deflagration/Detonation Transition (DDT) Test. The Gap Test subjects the substance to a strong shock from a pentolite donor charge and indicates whether the substance is able to propagate the detonation. In the DDT test, the substance is ignited inside a steel pipe bomb and an observation is made of whether it will continue to burn or will transit to detonation.

DESCRIPTION OF TESTS

1. GAP TEST FOR SOLID MATERIALS

The experimental arrangement used for the gap test is shown in figure E1. The test sample is contained in a cylinder consisting of a 40.6 cm (16-inch) length of cold-drawn seamless carbon steel "mechanical" tubing 4.76 cm (1.875 inches) in outside diameter with a thickness of 0.56 cm (0.219 inch) and inside diameter of 3.65 cm (1.438 inch). The sample in this test is a granular solid at room temperature that is loaded to the density attained by tapping the cylinder until further settling becomes imperceptible or clay tamped gently into place. The bottom of the cylinder is closed with two layers of 0.0076-cm (0.003-inch) thick polyethylene sheet tied on with gum rubber bands and polyvinyl chloride electrical insulating tape. The sample is subjected to the shock wave generated by the detonation of two cast pentolite density 1.65 g/cm^3 (50/50 pentaerythritol tetranitrate PETN/TNT) pellets 5.08 cm (2 inches) in diameter and 2.54 cm (1 inch) thick. The pellets will be in direct contact with the bottom of the sample tube ("zero gap"). The pentolite pellet is initiated by a U.S. Army Engineers special detonator having a base charge of 0.935 gram (14.4 grains) of the PETN and a primary charge of 0.35 gram (5.4 grains) of diazo dinitrophenol which is butted against the bottom surface of the pentolite pellets and held in place by a cylinder of wood or a metal chip. Instrumentation consists of a continuous rate probe made of a thin aluminum tube with an inner diameter of 0.051 cmx (0.02 inch) and a wall thickness of 0.0038 cm (0.0015 inch) with an axial nylon (skip wound) resistance wire of 0.0079-cm (0.0031 inch) diameter, having a resistance of 3.0 ohms/cm (7.52 ohms/inch). The outer tubing is crimped against the innter wire at the lower end, form a resistor. When this assembly is inserted in a medium that transmits a shock wave, the outer wall crushes against the inner wire as the wave moves up the tubing, shortening the effective

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length and changing the resistance. If a constant current (usually 0.06 ampere) is made to flow between the outer and inner conductors, the voltage between them is proportional to the effective length and can be recorded as a function of time using an oscilloscope. The slope of the oscilloscope trace is thus proportional to the velocity of the shock wave.

Criteria. Results of this test are considered to be positive if a stable propagation velocity greater than 1.5 km/sec is observed. Additional diagnostic information is provided by a mild steel witness plate 15.24 cm (6 inches) square and 0.3175 cm (0.125 inch) thick, mounted at the upper end of the sample tubing and separated from it by spacers 0.16 cm (0.063 inch) thick. A hole punch cleanly through the plate is an indication of a positive result.

A third source of diagnostic information is the fragmentation of the sample tube. The results of the test are considered to be positive only if the tube is fragmented along its entire length. The fragments range, depending on the material tested, from a few long strips to nearly a hundred small fragments; bulging, cracking, or "banana-peeling" of the acceptor is not considered a positive result.

In most cases, the results of the above three diagnostic methods agree. In some they do not, particularly with low-energy material, e.g., benzoyl peroxide, in which the witness plate is not punched through, but the tube is fragmented; also with certain propellants, the witness plate is punched, but little damage is done to the tube, evidently indicating a localized explosion at the upper end of the tube. In such cases, since there are essentially three criteria (witness plate, tube fragmentation, and rate probe), the result is assessed on the basis of the two criteria that agree; i.e., if any two criteria indicate a detonation, the result is considered positive, but not so if only one indicates a detonation. Some cases of doubtful propagation can also be resolved by using a longer sample tube. As applied in zero gap test, a negative result in this test is interpreted to mean that the substance does not have significant explosive properties.

2. DDT Test

The experimental arrangement for the DDT Test is shown in Figure E2. The sample of material to be tested is contained in a 45.7 cm (18 inch) length of 3-inch diameter schedule 80 carbon steel pipe with inside diameter of 7.37 cm (2.9 inches) and wall thickness of 0.75 cm (0.30 inch), capped at both ends with "3000 pound" forged steel pipe caps.

The sample is subjected to the thermal and pressure stimulus generated by an igniter consisting of a mixture of 50 percent RDX and 50 percent grade FFF_g black powder located at the center of the sample vessel. The igniter assembly consists of a cylindrical container 2.06 cm (0.81 inch) in diameter and of variable length, which is made from 0.0254 cm (0.01 inch) thick cellulose acetate held together by two layers of nylon-filament-reinforced cellulose acetate tape. The length of the igniter capsule is 0.32 cm (0.125 inch) for each gram of igniter material. The igniter capsule contains a small loop formed from a 2.54 cm (1 inch) length of nickel-chromium alloy resistance wire 0.03 cm

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(0.012 inch) in diameter lead wires 0.066 cm (0.026 inch) in diameter; the overall wire diameter including insulation is 0.127 cm (0.05 inch). These lead wires are fed through small holes in a brass disc approximately 1 cm (0.4 inch) in diameter and 0.08 cm (0.03 inch) thick, which is soldered to the end of 23 cm (9 inch) length of "1/8 inch" steel pipe having a diameter of 1.03 cm (0.405 inch); this pipe is threaded at the outer end and screwed into a threaded hole on the inside of one of the pipe caps. This pipe supports the igniter capsule and serves as channel for the igniter wires. The igniter is fired by a current of 15 amperes obtained from a 20-volt transformer.

Criteria. The criterion currently used in the interpretation of this test is that for a positive result either the pipe or at least one of the end caps be fragmented into at least two distinct pieces, i.e., results in which the pipe is merely split or laid open or in which the pipe or caps are distorted to the point at which the caps are blown off are considered to be negative results. Although it may be argued that a small number of fragments does not indicate the development of a detonation, it at least indicates a very rapidly rising pressure which in a larger sample could lead to development of detonation.

DOT Testing using a 20-gram (308-grain) igniter provides a strong thermal stimulus. Substances that yield a negative result with a 20-gram (308-grain) igniter are interpreted to have no significant explosive properties.

SOURCE: J. Edmund May, Richard W. Watson, and Richard J. Mainiero, U.S. Bureau of Mines, Department of the Interior, Pittsburgh, PA 15236.

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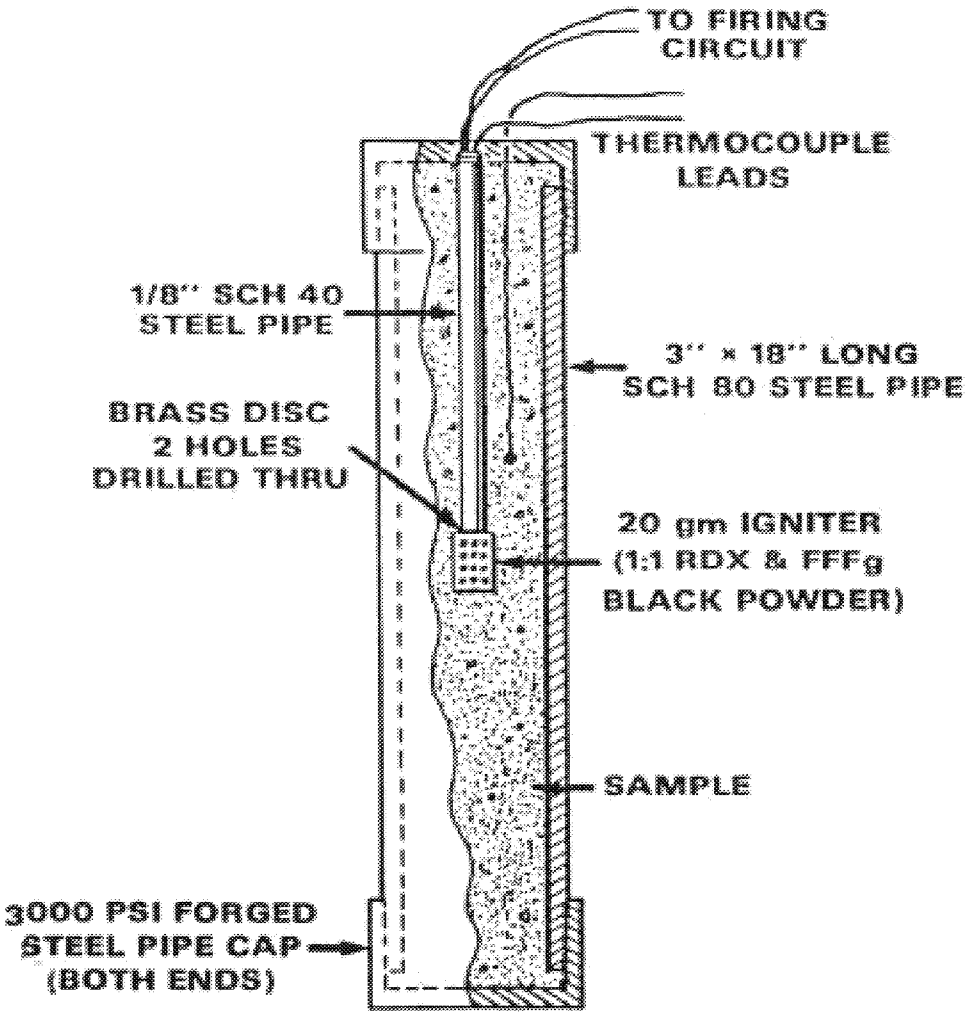

DDT TEST

Figure B2

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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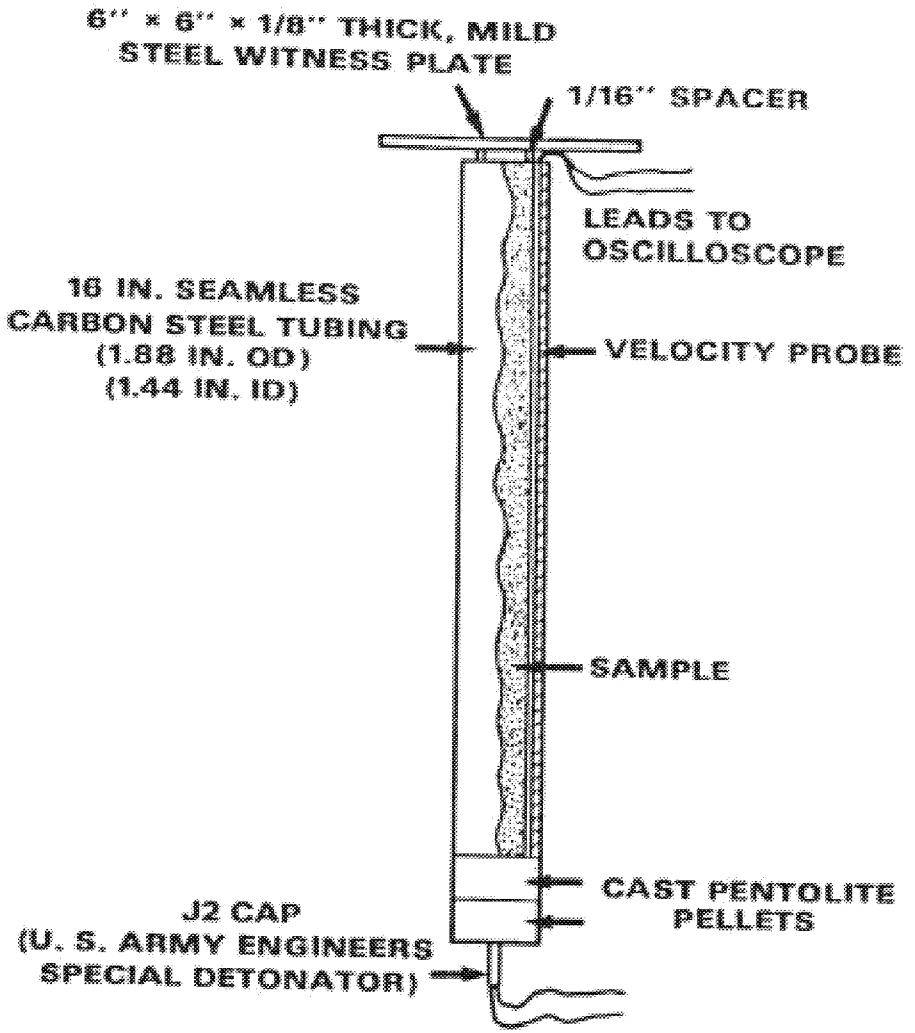

ZERO GAP SHOCK TEST†

Figure B1

Source: U.S. Bureau of Mines, Department of the Interior

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Table 21

BOM Zero Gap Shock Test Results - RDX/Sand Mixtures

Trial No.	Composition, % RDX[a]	Sand[b]	Water	Loading Density, g/cc	Shock Propagation Rate Thru Sample,[c] m/s	BOM Test Criteria[d]: Velocity > 1,500 m/s	Hole in Plate	End-to-End Pipe Fragmentation	Type Reaction[e]
1	100	0	0	1.088	6,110	+	+	+	+
2	100	0	0	1.096	5,780	+	+	+	+
3	100	0	0	1.191	6,475	+	+	+	+
4	100	0	0	1.079	6,882	+	+	+	+
5	100	0	0	1.088	6,882	+	+	+	+
6	0	100	0	1.422	1,215	-	-	-	-
7	0	100	0	1.446	f	-	-	-	-
8	0	100	0	1.417	f	-	-	+	-
9	0	0	100	0.997	1,364	-	-	-	-
10	0	0	100	0.981	1,364	-	-	-	-
11	0	0	100	0.981	1,419	-	-	+	-
12	0	80	20	1.879	766	-	-	-	-
13	0	80	20	1.834	f	-	-	-	-
14	0	80	20	1.862	724	-	-	-	-
15	50	50	0	1.207	3,362	+	+	+	+
16	30	70	0	1.306	1,826	+	+	+	+
17	20	80	0	1.394	3,790	+	-	+	+
18	20	80	0	1.352	2,788	+	-	+	+
19	20	80	0	1.352	1,783	+	-	+	+
20	20	80	0	1.372	1,859	+	-	+	+
21	20	80	0	1.347	2,304	+	-	+	+
22	20	80	0	1.310	> 2,500	+	-	+	+
23	20	80	0	1.347	1,763	+	-	-	+
24	20	80	0	1.335	2,101	+	-	+	+
25	20	80	0	1.347	1,413	-	-	+	+
26	20	80	0	1.352	1,826	+	-	-	+
27	20	80	0	1.352	2,029	+	-	+	+
28	18.75	81.25	0	1.288	1,337	+	-	-	-
29	18.75	81.25	0	1.277	3,240	+	-	-	-
30	18.75	81.25	0	1.286	3,644	+	-	+	+
31	18.75	81.25	0	1.273	3,644	+	-	-	-
32	18.75	81.25	0	1.282	3,644	+	-	-	-
33	18.75	81.25	0	1.331	1,839	+	-	-	+
34	18.75	81.25	0	1.261	4,129	+	-	+	+
35	18.75	81.25	0	1.286	4,129	+	-	-	-
36	18.75	81.25	0	1.339	1,419	-	-	-	-
37	18.75	81.25	0	1.306	2,256	+	-	-	-
38	17.5	82.5	0	1.339	> 3,900	+	-	+	+
39	17.5	82.5	0	1.339	> 2,800	+	-	-	-
40	17.5	82.5	0	1.343	f	f	-	-	-
41	17.5	82.5	0	1.343	f	f	-	-	-
42	17.5	82.5	0	1.327	f	f	-	-	-
43	17.5	82.5	0	1.323	2,253	+	-	-	-
44	17.5	82.5	0	1.364	1,892	+	-	-	-
45	17.5	82.5	0	1.335	2,101	+	-	+	+
46	17.5	82.5	0	1.347	3,000	+	-	-	-
47	17.5	82.5	0	1.337	2,594	+	-	-	-
48	15	85	0	1.339	1,313	-	-	-	-
49	15	85	0	1.384	1,471	-	-	-	-
50	15	85	0	1.310	> 3,400	+	-	-	-
51	15	85	0	1.380	> 2,500	+	-	-	-
52	15	85	0	1.331	> 3,000	+	-	-	-
53	15	85	0	1.337	3,891	+	-	-	-
54	15	85	0	1.364	765	-	-	-	-
55	15	85	0	1.319	3,951	+	-	-	-
56	15	85	0	1.393	1,701	+	-	-	-
57	15	85	0	1.372	f	f	-	-	-
58	15	85	0	1.389	f	f	-	-	-
59	15	85	0	1.368	f	-	-	-	-
60	15	85	0	1.347	f	f	-	+	-

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Table B1 (cont)

Trial No.	Composition, % RDX[a]	Sand[b]	Water	Loading Density, g/cc	Shock Propagation Rate Thru Sample,[c] m/s	RDM Test Criteria[d]: Velocity > 1,500 m/s	Hole in Plate	End-to-End Pipe Fragmentation	Type Reaction[e]
61	15	85	0	1.327	E	E	+	+	h
62	15	85	0	1.327	E	-	+	+	-
63	15	85	0	1.335	E	-	+	-	+
64	15	85	0	1.323	E	E	-	-	+
65	15	85	0	1.332	E	-	-	-	-
66	15	85	0	1.319	E	-	-	-	-
67	15	85	0	1.327	2,029	+	+	+	-
68	15	85	0	1.327	E	-	+	-	-
69	15	85	0	1.319	E	-	+	-	+
70	25	65	10	1.261	3,644	+	-	+	+
71	25	65	10	1.269	1,894	+	-	+	+
72	25	65	10	1.310	2,236	+	-	+	+
73	25	65	10	1.335	2,693	+	-	+	+
74	25	65	10	1.249	2,236	+	-	+	+
75	23.5	66.5	10	1.306	2,236	+	-	+	+
76	23.5	66.5	10	1.269	3,240	+	-	+	+
77	23.5	66.5	10	1.269	4,114	+	-	+	+
78	23.5	66.5	10	1.286	1,829	+	-	+	+
79	23.5	66.5	10	1.265	3,502	+	-	-	-
80	23.5	66.5	10	1.265	2,604	+	-	+	+
81	23.5	66.5	10	1.224	2,890	+	-	-	-
82	23.5	66.5	10	1.257	1,765	+	-	-	-
83	23.5	66.5	10	1.265	2,490	+	-	+	+
84	23.5	66.5	10	1.219	E	E	-	-	-
85	22	68	10	1.273	3,502	+	-	-	+
86	22	68	10	1.277	3,337	+	-	+	-
87	22	68	10	1.339	2,337	+	-	-	-
88	22	68	10	1.287	1,473	-	-	-	-
89	22	68	10	1.269	3,502	+	-	-	-
90	22	68	10	1.228	3,235	+	-	-	-
91	22	68	10	1.277	1,641	+	-	-	-
92	22	68	10	1.319	2,417	+	-	-	-
93	22	68	10	1.294	1,763	+	-	+	+
94	22	68	10	1.327	2,594	+	-	-	-
95	19	71	10	1.261	3,790	+	-	+	-
96	19	71	10	1.306	1,526	+	-	+	-
97	19	71	10	1.306	2,689	+	-	-	-
98	19	71	10	1.302	1,113	-	-	+	-
99	19	71	10	1.310	3,115	+	-	-	-
100	19	71	10	1.249	1,473	-	-	-	-
101	19	71	10	1.265	1,166	-	-	-	-
102	19	71	10	1.236	4,957	+	-	-	-
103	19	71	10	1.249	2,896	+	-	-	-
104	19	71	10	1.244	3,367	+	-	-	-
105	18.5	61.5	20	1.755	3,957	+	+	+	+
106	18.5	61.5	20	1.764	3,957	+	+	+	-
107	17	63	20	1.784	E	E	+	+	+
108	17	63	20	1.759	3,957	+	+	+	+
109	17	63	20	1.780	3,957	+	+	+	+
110	17	63	20	1.714	603	-	-	-	-
111	17	63	20	1.751	3,441	+	-	+	+
112	17	63	20	1.784	E	-	-	+	-
113	17	63	20	1.677	E	-	-	+	-
114	17	63	20	1.776	E	-	-	+	-
115	17	63	20	1.731	E	-	-	-	-
116	17	63	20	1.764	3,644	+	-	+	+

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Table B1 (cont)

Trial No.	Composition, % RDX[a]	Sand[a]	Water	Loading Density, g/cc	Shock Propagation Rate Thru Sample,[c] m/s	BCM Test Criteria[d] Velocity > 1,500 m/s	Hole in Plate	End-to-End Pipe Fragmentation	Type Reaction[e]
117	16.5	63.5[b]	20	1.739	564	-	-	-	-
118	16.5	63.5	20	1.722	893	-	-	-	-
119	16.5	63.5	20	1.731	724	-	-	-	-
120	16.5	63.5	20	1.751	766	-	-	-	-
121	16.5	63.5	20	1.743	g	g	-	-	-
122	16.5	63.5	20	1.743	564	-	-	-	-
123	16.5	63.5	20	1.784	4,129	+	+	+	+
124	16.5	63.5	20	1.739	684	-	-	-	-
125	16.5	63.5	20	1.751	855	-	-	-	-
126	16.5	63.5	20	1.753	1,219	-	-	+	-
127	16	64	20	1.753	643	-	-	-	-
128	16	64	20	1.764	981	-	-	-	-
129	16	64	20	1.804	850	-	-	-	-
130	16	64	20	1.751	1,313	-	-	-	-
131	16	64	20	1.776	808	-	-	-	-
132	16	64	20	1.772	643	-	-	-	-
133	16	64	20	1.751	766	-	-	-	-
134	16	64	20	1.743	523	-	-	-	-
135	16	64	20	1.753	564	-	-	-	-
136	16	64	20	1.817	808	-	-	+	-
137	16	64	20	1.780	1,072	-	-	+	-
138	16	64	20	1.776	1,116	-	-	+	-
139	16	64	20	1.755	g	g	-	-	-
140	16	64	20	1.947	1,215	-	-	+	-
141	16	64	20	1.764	f	-	-	+	-
142	16	64	20	1.776	f	-	-	+	-
143	16	64	20	1.768	g	g	-	-	-
144	16	64	20	1.764	g	g	-	-	-
145	16	64	20	1.788	937	-	-	+	-
146	16	64	20	1.764	1,072	-	-	+	-

[a]Type II, Class 1.

[b]Moisture in sand ranged from 0.8 to 0.3%.

[c]16-in. long steel tubing; 1.46-in. ID; 0.25-in. wall thickness.

[d]"+" indicates positive result. "-" indicates negative result. See Appendix B for further description of BCM criteria.

[e]"+" indicates positive result: 2 or 3 criteria are positive and therefore the test indicates sustained propagation of the shock wave through the sample. "-" indicates negative result. See Appendix B for further description of BCM criteria.

[f]Decaying reaction. No steady state velocity in sample.

[g]Propagation rate not recorded - Oscilloscope trigger did not function.

[h]Insufficient criteria to determine if reaction was positive.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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Table B2

BOM Deflagration to Detonation Transition Test Results - RDX/Sand Mixtures

Trial No.	Composition, % RDX[a]	Sand[b]	Water	Loading Density, g/cc	Type Reaction[c]
1	50	50	0	d	+
2	30	70	0	d	+
3	25	75	0	1.23	+
4	25	75	0	1.39	+
5	25	75	0	1.19	-
6	25	75	0	1.28	+
7	25	75	0	1.17	-
8	25	75	0	1.35	+
9	25	75	0	1.37	+
10	25	75	0	1.26	+
11	25	75	0	1.29	+
12	25	75	0	1.28	+
13	20	80	0	1.42	+
14	20	80	0	1.31	+
15	20	80	0	1.29	+
16	20	80	0	1.32	+
17	20	80	0	1.31	+
18	20	80	0	1.33	+
19	20	80	0	1.30	+
20	20	80	0	1.32	+
21	20	80	0	1.32	+
22	20	80	0	1.28	+
23	17.5	82.5	0	1.29	-
24	17.5	82.5	0	1.33	+
25	17.5	82.5	0	1.35	+
26	17.5	82.5	0	1.33	+
27	17.5	82.5	0	1.34	+
28	17.5	82.5	0	1.36	+
29	17.5	82.5	0	1.34	+
30	17.5	82.5	0	1.35	+
31	17.5	82.5	0	1.33	+
32	17.5	82.5	0	1.34	+
33	15	85	0	1.26	-
34	15	85	0	1.38	-
35	15	85	0	1.34	+
36	15	85	0	1.32	+
37	15	85	0	1.32	-
38	15	85	0	1.32	-
39	15	85	0	1.34	-
40	15	85	0	1.36	-
41	15	85	0	1.30	-
42	15	85	0	1.35	+
43	13	87	0	1.44	+
44	13	87	0	1.43	+
45	13	87	0	1.44	+
46	13	87	0	1.44	+
47	13	87	0	1.47	+
48	13	87	0	1.46	+
49	13	87	0	1.44	+
50	13	87	0	1.40	+
51	13	87	0	1.43	+
52	13	87	0	1.37	+
53	13	87	0	1.42	+
54	13	87	0	1.46	+
55	13	87	0	1.39	+
56	13	87	0	1.39	+
57	13	87	0	1.47	+
58	13	87	0	1.39	+
59	13	87	0	1.45	+
60	13	87	0	1.48	+
61	13	87	0	1.46	+
62	13	87	0	1.35	+

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Table 32 (cont)

Trial No.	Composition, % RDX[a]	Sand[b]	Water	Loading Density, g/cc	Type Reaction[c]
63	19	71	10	1.32	+
64	19	71	10	1.37	+
65	19	71	10	1.32	+
66	15	75	10	1.33	+
67	15	75	10	1.43	+
68	15	75	10	1.42	+
69	15	75	10	1.36	+
70	15	75	10	1.45	+
71	15	75	10	1.43	+
72	15	75	10	1.46	+
73	15	75	10	1.41	+
74	15	75	10	1.43	+
75	15	75	10	1.42	+
76	12	78	10	1.47	+
77	12	78	10	1.51	+
78	12	78	10	1.50	+
79	12	78	10	1.45	+
80	12	78	10	1.51	+
81	12	78	10	1.51	+
82	12	78	10	1.50	+
83	12	78	10	1.44	+
84	12	78	10	1.53	+
85	12	78	10	1.52	+
86	12	78	10	1.50	+
87	12	78	10	1.48	+
88	12	78	10	1.44	+
89	12	78	10	1.48	+
90	12	78	10	1.50	+
91	12	78	10	1.47	+
92	12	78	10	1.55	+
93	12	78	10	1.47	+
94	12	78	10	1.44	+
95	12	78	10	1.53	+
96	28	52	20	1.72	+
97	28	52	20	1.71	+
98	28	52	20	1.69	+
99	28	52	20	1.72	+
100	28	52	20	1.74	+
101	28	52	20	1.71	+
102	28	52	20	1.70	+
103	28	52	20	1.67	+
104	28	52	20	1.67	+
105	28	52	20	1.70	+
106	26.5	53.5	20	1.70	+
107	26.5	53.5	20	1.74	+
108	26.5	53.5	20	1.68	+
109	26.5	53.5	20	1.73	+
110	25	55	20	1.74	+
111	25	55	20	1.66	+
112	25	55	20	1.77	+
113	25	55	20	1.74	+
114	25	55	20	1.74	+
115	25	55	20	1.75	+
116	25	55	20	1.71	+
117	25	55	20	1.76	+
118	25	55	20	1.70	+
119	25	55	20	1.71	+
120	13	67	20	1.85	+
121	13	67	20	1.82	+
122	13	67	20	1.79	+
123	13	67	20	1.66	+
124	13	67	20	1.72	+

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Table B2 (cont)

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[a]Type II, Class 1.

[b]Sand = 0.25% water wet.

[c]"+" indicates positive result - that the pipe or an end cap fragmented into two or more distinct pieces; "-" indicates negative result. See Appendix B for further description of BOM criteria.

[d]Not determined.

Source: Hercules Incorporated (Radford Army Ammunition Plant)

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APPENDIX C

STATISTICAL ANALYSIS

OF

ZERO GAP TEST DATA

ED_001691B_00013247

HERCULES

RADFORD ARMY AMMUNITION PLANT

Safety is part of your job.

Memorandum

August 15, 1986

TO: F. T. Kristoff, Manager
Hazards Analysis

FROM: D. W. Hall, Statistician
Quality Engineering

The final analysis has been written for the zero-gap data. Appendices for tabled results and a glossary for statistical terms are included.

A stepwise regression was done for the entire set of zero-gap data to determine if there was a relationship between shockwave propagation velocity and sample composition components (% RDX, % Sand and % H_2O).

A. The independent variables RDX, Sand and H_2O were entered into the stepwise procedure. The results indicated that 43% (r^2 = .431589) of the variability in velocity could be explained by RDX and H_2O. The variable Sand was removed from the model which indicated that it was not important in relationship to velocity in this model (Appendix I).

   a. Due to the fact that the above stepwise procedure forced Sand out of the model. A regression was run again forcing Sand into the procedure. The results indicated that 43% (r^2 = .431589) of the variability in velocity could be attributed to RDX and Sand. Note that the estimated coefficients (beta's) were equal and opposite for Sand (37.87) and H_2O (-37.87). The explainable variability in velocity (r^2 = .43) was equal whether Sand and RDX or RDX and H_2O were the variables remaining in the regression model (Appendix I).

B. In order to show an analysis of variance for regression further analysis was done on the entire data set using the multiple regression approach. A regression could only be run on RDX and Sand (Case 1) and on RDX and H_2O (Case 2, Appendix II).

   1. (RDX and Sand) Model: $y = b_0 + b_1 X_1 + b_2 X_2$
      Velocity = -1980.14 + 80.67 RDX + 37.87 Sand

   2. (RDX and H_2O) Model: $y = b_0 + b_1 X_1 - b_3 X_3$
      Velocity = 1807.06 + 42.80 RDX - 37.87 H_2O

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The addition of any single variable to a regression system will increase the regression sum of squares and thus reduce the error sum of squares. A decision must be made as to whether the increase in regression is sufficient to warrant using the variable in the model. Using unimportant variables can reduce the effectiveness of the prediction equation by increasing the variance of the estimated response. This point can be pursued by using the t-distribution to test: H_0: $B_j = 0$ H_1: $B_j \neq 0$. If B_j does not significantly differ from 0, it may be justifiable to remove the X variable (in question) from the model.

1. (<u>RDX and Sand Model</u>) - A t-test was run for the variables RDX, Sand and the constant term (b_0). All were significant at the $\alpha = .05$ level and should remain in the model. The f-test in the analysis of variance for the regression yielded an f-ratio of 37 which was significant at the $\alpha = .05$ level for the model (Appendix II). R-squared was .43159 for both variables in the model.

2. (<u>RDX and H_2O Model</u>) - A t-test was run for the variables RDX, H_2O and the constant term b_0. All were significant at the $\alpha = .05$ level. The f-ratio for the analysis of variance was 37 which was significant at the $\alpha = .05$ level. R-squared was .43159 for both variables in the model (Appendix II).

C. An additional approach was made in an attempt to further analyze the data. The goals for three component chemical ingredients X_1, X_2, X_3 are:

a. $X_1 + X_2 + X_3 = 100$ for all experimental conditions.

The classic multiple regression model is $y_i = b_0 + b_1 X_{1i} + b_2 X_{2i} + b_3 X_{3i} + e_i$. As long as $X_1 + X_2 + X_3$ add up to a constant value, the least squares solution to estimate the b's has no unique solution. There is an entire set of values that yield the same fit. This is supported by Case 1 and Case 2 in the multiple regression procedures in Part B of this memo which provides the same model fit with the equations:

1. Velocity = -1980.14 + 80.67 RDX + 37.87 Sand and
2. Velocity = 1807.06 + 42.80 RDX - 37.87 H_2O.

The classic model for plotting any experimental conditions in a three component chemical composition system is with triangular paper with 100% composition of each component represented by the apexes of the triangle. Since the graph is a flat plane, this corresponds to a two variable cartesian coordinate system.

A model using these two coordinates can be calculated from the original composition using the following transformation: X_2 = Sand and $X_3 = [1/\sqrt{3}\ (RDX - H_2O)]$. At that point, the model (Velocity = $b_0 + b_1[1/\sqrt{3}\ (RDX - H_2O)] + b_2$ Sand) is valid as a predictor of a flat response surface above or through a triangular plane. Another model using two coordinates was also calculated. The model (Velocity = $b_0 + b_1 [1/\sqrt{3}\ (RDX - Sand)] + b_2\ H_2O$ is also a valid predictor of a flat response surface above or through a triangular plane.

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Case 1: Velocity = 2053.39 + 60.91 $[1/\sqrt{3}\ (RDX - H_2O)]$ - 2.46 Sand. The stepwise procedure indicated that Sand had no effect on velocity if there was no difference between RDX and H_2O. Forty-three percent of the variability in velocity could be explained by X_3 and Sand. A t-test was run on the variables X_3, Sand and the constant term (b_0). The constant term and X_3 were significant at the α = .05 level. The t-test was not significant for Sand which indicated that Sand should be removed from the model (Appendix III). The f-test for X_3 was significant as well.

Case 2: Velocity = 3947 + 37.09 $[1/\sqrt{3}\ (RDX - Sand)]$ - 59.27 H_2O. The variables X_2 = $[1/\sqrt{3}\ (RDX - Sand)]$ and H_2O remained in the stepwise model. A t-test was run on the variables X_2 and H_2O and the constant term (b_0). All were significant at the α = .05 level. The f-test was significant for both variables in the regression model (Appendix IV).

In conclusion, Sand and H_2O have similar effects on velocity. There is no effect on velocity due to Sand if the difference between RDX and H_2O does not change. For example, if test firings are made at a certain level of RDX and H_2O and an adjustment is made in such a manner that the percentage of Sand is decreased by 10% by adding 5% more RDX and 5% more H_2O, then the velocity will remain the same. I have used three different methods of looking at this data and all have given the same results.

These best fit models give the same information as the model in Part A (Stepwise Regression: RDX, Sand, H_2O), using only a constant term and two coefficients which is clearly a superior fit.

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Table C1

Stepwise Regression

Table Results (A)

r-squared = .431589

Variables in Model			Variables Not in Model		
Variable	Coefficient (b's)	F-Remove	Variable	Partial Corr.	F-Enter
1. RDX	42.79869	49.6113	2. Sand	.0000	.0000
3. H_2O	-37.87201	7.7154			

Table Results (a)

Variables in Model

Variables	Est. Coefficient (b's)	F-Remove
1. RDX	80.67070	37.0127
2. Sand	37.87201	7.7154

Table C2

Multiple Regression

Model Fitting Results (Case 1)

Variable	Coefficient	Stnd. Error	T-Value	Prob (> T)
Constant	-1980.137219	1219.115424	-1.6242	.1075
RDX	80.670702	13.259912	6.0838	.0000
Sand	37.872008	13.634473	2.7777	.0066

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	Prob (>F)
Model	82390851	2	41195425	37	0
Error	1.0851E0008	98	1.1072E0006		
Total (Corr.)	1.9090E0008	100			

r-squared = 0.431589
r-squared (Adj. for D.F.) = 0.419989
Standard error of estimation = 1052.26

Model Fitting Results (Case 2)

Variable	Coefficient	Stnd. Error	T-Value	Prob (> T)
Constant	1807.063624	232.815245	7.7618	.0000
RDX	42.798694	6.076314	7.0435	.0000
N_2O	-37.872008	13.634473	-2.777	.0066

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	Prob (>F)
Model	82390851	2	41195425	37	0
Error	1.0851E0008	98	1.1072E0006		
Total (Corr.)	1.9090E0008	100			

r-squared = 0.431589
r-squared (Adj. for D.F.) = 0.419989
Standard error of estimation = 1052.26

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Table C3

Stepwise (Case 1)

r-squared = 0.431068
r-squared (Adj.) = 0.425321 MSE = 1.09707E6 With 99 D.F.

Variables in Model			Variables Not in Model		
Variable	Coefficient	F-Remove	Variable	Partial Correlation	F-Enter
1. X_3	72.26252	75.0101	2. Sand	-.0303	.0900

Model Fitting Results

Variable	Coefficient	Stnd. Error	T-Value	Prob (> T)
Constant	2053.397882	639.178137	3.2126	.0018
X_3	69.905288	11.490392	6.0838	.0000
Sand	-2.463343	8.213033	-.2999	.7649

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	Prob (>F)
Model	82390851	2	41195425	37	0
Error	1.0851E0008	98	1.1072E0006		
Total (Corr.)	1.9090E0008	100			

r-squared = 0.431589 = 43% of the variability in velocity can be explained by X_3 and Sand.
r-squared (Adj. for D.F.) = 0.419989
Standard error of estimation = 1052.26

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Table C4

Stepwise (Case 2)

r-squared = 0.431589
r-squared (Adj.) = 0.419989 MSE = 1.1072E6 with 98 D.F.

Variables in Model			Variables Not in Model		
Variable	Coefficient	F-Remove	Variable	Partial Correlation	F-Enter
1. X_2	37.08726	49.6113			
2. H_2O	-59.27136	20.4688			

Multiple Regression

Model Fitting Results

Variable	Coefficient	Stnd. Error	T-Value	Prob (> T)
Constant	3946.998304	199.469712	19.7875	.0000
X_2	37.087256	5.265437	7.0435	.0000
H_2O	-59.271355	13.100827	-4.5242	.0000

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	Prob (>F)
Model	82390851	2	41195425	37	0
Error	1.0851E0008	98	1.1072E0006		
Total (Corr.)	1.9090E0008	100			

r-squared = 0.431589 43% of the variability in velocity can be explained by X_2 and H_2O
r-squared (Adj. for D.F.) = 0.419989
Standard error of estimation = 1052.26

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Glossary

Coefficient (b's) - estimates of the model coefficients for each independent variable ($y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3$);

F-to-Enter - enters a value for the F ratio above which variables will be entered into the model;

F-to-Remove - enters a value for the F ratio below which variables will be removed from the model;

Partial Correlation Coefficient - measures the relationship between two variables while controlling for the possible effects of other variables. These effects are controlled by removing the linear relationship with the other variables before calculating the correlation coefficients between the variables of interest. Partial correlation is useful for uncovering hidden relationships, identifying intervening variables and detecting spurious relationships;

Standard Error of Estimation - the standard deviation of the error in the model; it measures the amount of variability in the dependent variable not explained by the estimated model;

Mean Square - sum of squares divided by the degrees of freedom;

T-Value (Test Statistic) - calculated by dividing the coefficient term by its standard error;

P > /T/ - the probability that a larger t-value would occur if there were no marginal contribution from that variable;

P > F - the smaller the probability value, the more likely that a factor has had a significant effect on a response variable;

r-squared - represents the percentage of variability that can be explained by the variables that remain in the model after a regression has been run;

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APPENDIX D

CHEMICAL ANALYSES OF TYPICAL EXPLOSIVE CONTAMINATED ARMY LAGOON SOILS

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TABLE IX. SAVANNA ARMY DEPOT ACTIVITY SOIL ANALYSIS

Total Analysis

Parameter	Range of Values	Detection Limit[1]
Moisture, %	11.7 - 26.9	—
Ash, % as received	44.5 - 82.5	—
Ash, % dry basis	60.5 - 95.6	—
Heating Value, Btu/lb as received	ND[2] - 2,364	50

Elemental Analysis (Dry Weight Basis)

Parameter	Range of Values	Detection Limit
Sulfur, %	ND	0.01
Carbon, %	2.68 - 12.70	—
Hydrogen, %	0.28 - 0.79	—
Nitrogen, %	1.01 - 6.03	—
Total Chlorine %	ND - 0.72	0.01

Heavy Metals Content (Dry Weight Basis)

Parameter	Range of Values	Detection Limit
Barium (Ba), ppm	17 - 29	—
Cadmium (Cd), ppm	ND	3.9
Chromium (Cr), ppm	ND - 13	5.9
Copper (Cu), ppm	ND - 30	10.4
Lead (Pb), ppm	16 - 100	—
Zinc (Zn), ppm	32 - 160	—
Arsenic (As), ppm	ND	5.7
Selenium (Se), ppm	ND	5.0
Mercury (Hg), ppm	ND	0.5

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TABLE D1. (CONTINUED)
Explosives Analysis (Dry Weight Basis)

Parameter	Range of Values	Detection Limit
2,4,6-Trinitrotoluene (TNT), ppm	88,100 - 406,000	—
HMX, ppm	ND	15.9
RDX, ppm	28.6 - 145	—
1,3,5-Trinitrobenzene (TNB), ppm	90.7 - 256	—
1,3-Dinitrobenzene (DNB), ppm	ND - 35.1	7.39
Nitrobenzene (NB), ppm	ND	5.26
2-Amino-4,6-Dinitrotoluene (2-Amino), ppm	ND - 27.9	3.64
2,6-Dinitrotoluene (2,6-DNT), ppm	ND	5.03
2,4-Dinitrotoluene (2,4-DNT), ppm	ND	5.20

1/Detection limit listed only for parameters not detected.

2/ND - Not detected (i.e., sample concentration below the detection limit).

Source: J. W. Noland, J. R. Marks, P. J. Marks, "Task 2. Incineration Test of Explosives Contaminated Soils at Savanna Army Depot Activity, Savanna, Illinois," Roy F. Weston, Inc., Final Report, DRXTH-TE-CR-84277, April 1984.

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TABLE D2. LOUISIANA ARMY AMMUNITION PLANT SOIL ANALYSIS

Total Analysis

Parameter	Range of Values	Detection Limit[1]
Moisture, %	25.1 - 29.5	—
Ash, % as received	54.3 - 66.0	—
Ash, % dry basis	77.1 - 88.1	—
Heating Value, Btu/lb as received	562 - 1,172	—

Elemental Analysis (Dry Weight Basis)

Parameter	Range of Values	Detection Limit
Sulfur, %	ND[2] - 0.01	0.01
Carbon, %	5.08 - 7.66	—
Hydrogen, %	0.66 - 1.05	—
Nitrogen, %	2.52 - 6.72	—
Total Chlorine, %	ND - 0.37	0.01

Heavy Metals Content (Dry Weight Basis)

Parameter	Range of Values	Detection Limit
Barium (Ba), ppm	98 - 150	—
Cadmium (Cd), ppm	ND - 13	3.9
Chromium (Cr), ppm	17 - 23	—
Copper (Cu), ppm	42 - 65	—
Lead (Pb), ppm	100 - 160	—
Zinc (Zn), ppm	140 - 310	—
Arsenic (As), ppm	ND	5.7
Selenium (Se), ppm	ND	5.0
Mercury (Hg), ppm	2.2 - 3.4	—

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TABLE D2 (CONTINUED)

Explosives Analysis (Dry Weight Basis)

Parameter	Range of Values	Detection Limit
2,4,6-Trinitrotoluene (TNT), ppm	55,100 - 142,000	—
HMX, ppm	5,740 - 13,500	—
RDX, ppm	33,100 - 96,500	—
1,3,5-Trinitrobenzene (TNB), ppm	57.0 - 139	—
1,3-Dinitrobenzene (DNB), ppm	ND - 22.4	7.39
Nitrobenzene (NB), ppm	ND	5.26
2-Amino-4,5-Dinitrotoluene (2-Amino), ppm	ND - 558	3.64
2,6-Dinitrotoluene (2,6-DNT), ppm	ND	5.03
2,4-Dinitrotoulene (2,4-DNT), ppm	ND	5.20

1/Detection limit listed only for parameters not detected.

2/ND - Not detected (i.e., sample concentration below the detection limit).

Source: J. W. Noland, J. R. Marks, P. J. Marks, "Task 2, Incineration Test of Explosives Contaminated Soils at Savanna Army Depot Activity Savanna, Illinois," Roy F. Weston, Inc., Final Report, DRXTH-TE-CR-84277, April 1984.

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APPENDIX E

CHEMICAL/PHYSICAL ANALYSIS OF TYPE II, CLASS 1 RDX USED IN BOM TESTS

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Table E1.

HARSHAW DEFENSE CORPORATION
KINGSPORT, TENNESSEE

BOX, TYPE II, CLASS 1
MIL-B-398C, W/INT. AMEND. 5

HDL84HS15-064
REG. NO. 20919
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SPECIFICATIONS	MELTING POINT C	% ACIDITY AS ACETIC ACID	% ACETONE INSOLUBLES	% INORGANIC INSOLUBLES	INSOLUBLE PARTICLES ON USSS NO. 60	GRANULATION % PASSING USSS NO. 20	50	100	200
MINIMUM	190.0	-	-	-	-	96	80	30	5
MAXIMUM	-	0.02	0.05	0.03	5	100	100	90	45
BATCH NUMBER	ANALYTICAL RESULTS								
3AC14-278	198.0	0.01	0.00	<.03	0	100	84	33	15

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APPENDIX F

SUMMARY OF ANALYTICAL TECHNIQUES TO VERIFY SAMPLE COMPOSITION

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ANALYTICAL TECHNIQUES

A. Moisture Analysis of RDX/Sand Mixtures

1. Mixtures 10-20% wet

The total moisture content was determined by drying weighed samples to constant weight of 105°C. Samples weighing at least 25 g were used.

Reference: F. Welcher, Ph.D., Standard Methods of Chemical Analysis, D. Van Nostrand Co. Inc., Princeton, NJ 1963.

2. Mixtures <1% wet

The total moisture content was determined by extracting moisture from weighed subsamples into weighed portions of isopropanol and using standard gas chromatographic analysis techniques.

B. RDX Purity Analysis

Standard high pressure liquid chromatographic (HPLC) techniques were used. Four standards were prepared by dissolving weighed portions of high purity RDX and HMX in measured amounts of acetonitrile. The RDX/HMX content of Type II RDX was determined by dissolving samples into measured portions of acetonitrile, running sample and standards through the HPLC using the same conditions and comparing test results. Listed below are the HPLC conditions used for analysis of RDX in sand.

Instrument:	Hewlett Packard 1084B
Column:	Hewlett Packard RP-8; Length = 200 mm; I.D. = 4.6 mm; Packing Size = 10 μm
Oven Temperature:	40°C
Detector:	Variable wavelength, 254 nm
Mobile Phase Flow:	2.0 cc/min
Composition and Temp.	70% water at 80°C 30% methanol at 40°C
Sample Injection:	Automatic variable volume injector. 10 μl injection.

C. Analysis of RDX in Sand

RDX in RDX/sand mixtures was determined quantitatively using HPLC techniques. The same conditions and procedure were used as in the purity determination. Sample sizes and dilutions were based on the ratio of sand to RDX. Standard RDX mixtures were placed in acetonitrile and shaken overnight to assure complete solution of the RDX. The final volumes employed depended on the ratio of sand to RDX. Four samples of sand were spiked with

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known amounts of RDX to establish percent recovery of RDX from the sand. Four samples of sand alone were also analyzed to assure that impurities in the sand did not interfere with the RDX analysis.

D. TNT Purity Analysis

The purity of TNT was determined by HPLC. Samples of the TNT used to prepare sand/TNT mixtures was dissolved in acetonitrile and compared to high purity TNT standards in acetonitrile. Listed below are the HPLC conditions used for determination of TNT purity.

Instrument:	Hewlett Packard 1084B
Column:	Resolvex C-18; Length = 250 mm; I.D. = 4.6 mm; Packing Size = 10 µm
Oven Temperature:	50°C
Detector:	Variable wavelength, 254 nm
Mobile Phase Flow:	2.0 cc/min
Composition and Temp.	45% water at 80°C 55% methanol at 40°C
Sample Injection:	Automatic variable volume injector. 15 µl injection.

E. Analyses of TNT and Sand

TNT in TNT/sand mixtures was determined quantitatively using HPLC techniques. The same conditions and procedures were used as in the TNT purity determination. Sample sizes and dilutions were based on the ratio of sand to TNT. The final volumes employed depended on the ratio of sand to TNT.

F. Particle Size Distribution

The particle size distribution of TNT fines was measured microscopically. The microscope reticle was calibrated using a stage micrometer, 200 particles were measured and a distribution curve plotted.

G. Particle Size Distribution of Sand

The particle size distribution of the sand was established using a series of suitable mesh sieves. One hundred grams of sand were shaken in the nest of sieves and the percentage retained was determined.

H. Bulk Density of RDX/Sand Mixtures

Measured amounts of water were used to fill test containers to determine container volumes. Dry containers were weighed before and after loading with RDX/sand mixtures to determine the weight of sample in the container. Bulk densities of samples were calculated using determined container volumes and sample weights.

APPENDIX G

GLOSSARY

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Critical Diameter Test	See Appendix A
Critical Height to Explosion	Defined as the greatest material height tested in a given container diameter which did not result in transition from burning to an explosive reaction.
Deflagration to Detonation (DDT) Test	See Appendix B
Detonation Velocity	Rate at which a shock wave induced at one end of a sample travels through and is sustained by the sample.
Differential Thermal Analysis	A test used to determine at what temperature propellant and explosive samples begin to thermally decompose.
Electrostatic Spark Discharge	The maximum electrostatic discharge energy which will not ignite propellant or explosive samples.
Explosion Temperature	The temperature which produces an explosion, ignition or decomposition of a sample in 5 seconds.
Friction	The maximum frictional (sliding) energy which will not ignite propellant or explosive material.
HMX	Cyclotetramethylene-tetranitramine (also known as Homocyclomite or octagen).
Impact	The maximum impact (falling weight) energy which will not ignite propellant or explosive materials.
RDX	Cyclotrimethylene trinitramine (also known as Cyclonite, Hexogen or T4).
Rifle Bullet Test	Determines the reactivity of a sample loaded into a 3-inch pipe nipple and subjected to the impact of a caliber .30 bullet.
TNT	Trinitrotoluene
USATHAMA	United States Army Toxic and Hazardous Materials Agency.
Zero Gap Test	See Appendix B

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