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U.S. Army Toxic and Hazardous Materials Agency

Testing to Determine Chemical Stability, Handling Characteristics, and Reactivity of Energetic-Fuel Mixtures

Compiled by

M. E. Lackey

April 1988

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Prepared by

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
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TESTING TO DETERMINE CHEMICAL STABILITY, HANDLING CHARACTERISTICS, AND REACTIVITY OF ENERGETIC-FUEL MIXTURES

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Engineering Technology Division

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The U.S. Army generates approximately 2.5 million pounds of waste explosives each year as a result of explosives production and the loading of ordnance. In addition, the U.S. Army currently stores >200,000 tons of obsolete munitions. The current alternative to storage is open-air burning, open-air detonation, or incineration. The U.S. Army Toxic and Hazardous Materials Agency is currently developing methods and procedures for the utilization of energetic materials blended with fuel oil as supplemental fuel in Army industrial combustors.

A series of tests were conducted to evaluate the chemical compatibility, reactivity, and handling characteristics of energetic-fuel mixtures. The energetics studied were TNT, RDX, and Composition B. Results indicated that under specific conditions of energetic content, energetic/fuel oil preparation, and system design, energetic/fuel oil mixtures can successfully be used as supplemental fuel in Army industrial combustors.

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Accelerating Rate Calorimetry
particle size distribution
nozzle plugging characteristics
propagation of detonation
energetic-fuel mixture
energetic-fuel slurry
solubility
viscosity

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EXECUTIVE SUMMARY

This study had two primary objectives: first, to determine the stability and reactivity of energetic-fuel mixtures that might be used in U.S. Army industrial combustors, and second, to investigate the handling characteristics of these mixtures. The data gathered will allow for the safe design and operation of systems that use energetic materials as a supplemental fuel.

Extensive testing evaluated the chemical stability, reactivity, and handling characteristics of energetic-fuel mixtures. These tests were conducted with laboratory-prepared single-phase and slurry samples of energetics (TNT, RDX, and Composition B) mixed with liquid fuels and solvents.

Chemical stability (determined by differential thermal analysis, vacuum thermal stability, and accelerating rate calorimetry) of the energetics was not affected by mixing them with fuels. Handling characteristics were favorable. Specifically, the particle size of the energetic resulting from the fuel preparation method will not plug a conventional burner nozzle. Furthermore, TNT from a saturated fuel oil-TNT solution did not plate the container walls. RDX cannot plate out of solution because it is insoluble in fuel oil.

Reactivity was evaluated by means of a series of static and dynamic propagation tests in a 2-in. schedule 40 pipe. Single-phase mixtures of TNT and fuel did not propagate at <65 wt % TNT. Similar tests showed that slurry mixtures of TNT did not propagate at <55 wt % TNT.

Static tests of RDX and No. 2 fuel oil slurry in a horizontal test pipe resulted in propagation at concentrations as low as 5 wt % RDX. These propagations were caused by RDX that had settled along the bottom side of the horizontal pipe and formed a fuselike tail, demonstrated by limited damage to the witness plate. Dynamic testing of RDX and No. 2 fuel oil was not practical because RDX rapidly settled to the bottom of the vertical pipe. Adding a gelling agent prevented settling, thus facilitating dynamic tests. RDX particles suspended in No. 2 fuel oil did not propagate at <22 wt % RDX in either the static or dynamic tests. Tests run with RDX and No. 5 fuel oil led to the following results: for static conditions, concentrations as low as 5 wt % RDX propagated detonations, and for dynamic conditions, concentrations of <15 wt % RDX did not propagate detonations.

Details of the experimental procedures and discussions of the results are provided in the following appendixes:

A. Particle-Size and Viscosity Characteristics of TNT and Composition B Fuel Oil Mixtures,

B. Plugging Characteristics of Nozzles with Slurries,

C. Compatibility of TNT and Composition B Fuel Oil Mixtures, and

D. Propagation of Detonation Testing of Energetic-Fuel Mixtures.

Specifically, Appendixes A and B deal with handling characteristics, C with compatibility, and D with reactivity.

The following recommendations, based on the test results, apply to the design and operation of the energetic-fuel process.

1. The system design should include continuous recirculation of the energetic-fuel mixtures to prevent settling in process equipment.

2. Propagation blocks, such as doglegs in the process piping, should be used to isolate the fuselike reactivity of settled RDX.

3. Three limits, based on the system design and explosive level present, apply to energetic-fuel reactivity for 2-in. and smaller pipe:

- TNT/fuel single-phase — if the TNT content is <65 wt %, the mixture is not reactive;
- TNT/fuel oil slurry — if the TNT content is <55 wt %, the mixture is not reactive; and
- RDX/fuel oil slurry — for a flowing system, if the RDX content is <15 wt %, the mixture is not reactive.

TESTING TO DETERMINE CHEMICAL STABILITY, HANDLING CHARACTERISTICS, AND REACTIVITY OF ENERGETIC-FUEL MIXTURES

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ABSTRACT

The U.S. Army generates ~2.5 million pounds of waste explosives each year as a result of explosives production and the loading of ordnance. In addition, the U.S. Army currently stores >200,000 tons of obsolete munitions. The current alternative to storage is open-air burning, open-air detonation, or incineration. The U.S. Army Toxic and Hazardous Materials Agency is currently developing methods and procedures for the utilization of energetic materials blended with fuel oil as supplemental fuel in Army industrial combustors.

A series of tests were conducted to evaluate the chemical compatibility, reactivity, and handling characteristics of energetic-fuel mixtures. The energetics studied were TNT, RDX, and Composition B. Results indicated that under specific conditions of energetic content, energetic/fuel oil preparation, and system design, energetic/fuel oil mixtures can successfully be used as supplemental fuel in Army industrial combustors.

1. INTRODUCTION

The U.S. Army generates ~2.5 million pounds of waste explosives each year [primarily TNT and Composition B (Comp. B)] as a result of explosives production and the loading of ordnance.[1] In addition, the U.S. Army currently stores >200,000 tons of obsolete munitions.[2] The current alternative to storage is open-air burning, open-air detonation, or incineration.[1,3,4] The U.S. Army Toxic and Hazardous Materials Agency is currently developing methods and procedures for the utilization of energetic materials blended with fuel oil as supplemental fuel in Army industrial combustors.

Burning energetic materials such RDX, TNT, and Comp. B in an oil-fired industrial combustor presents several difficult engineering problems. Among these are the interrelated problems of the feed preparation and the burner design. Efficient burning of fuel oil requires atomization before combustion. The primary functions of the burner are to atomize the oil and to prepare it for mixing with the combustion air. Oil burner designs are generally divided into four main groups: (1) the spray type, which divides the oil into a fine spray by the action of an atomizer agent (steam or air); (2) the rotary-type, which divides the oil by action of a centrifugal force produced by a revolving cone or plate; (3) the mechanical type, which divides the oil by the use of an orifice; and (4) the vapor type, which vaporizes the oil by heat. The spray and mechanical burner designs are most often used in combustors that are used for the production of process or high-pressure steam. The rotary burner design is used for low-load and low-pressure furnaces, and the vapor burner design is used with forging, annealing, and tempering furnaces. For the purpose of this discussion, the spray and mechanical burner designs are considered as standard. Both designs use small holes to effect the atomization of the fuel oil before combustion.

Because of the general limitations in solubility of the energetic materials in fuel oil [TNT has a solubility of ~8 wt % at ~57°C (135°F)], they must be burned as a slurry with the fuel oil. Because the energetic material to be burned is usually in the form of scrap, shell washout, or unacceptable end items, a large portion of the material must be reduced in size before combustion. Current state-of-the-art size-reduction methods use a mechanical grinding process with a water medium containing ~10% energetic material. A typical size-reduction facility is located at the Radford Army Ammunition Plant.[4] This facility employs a rotary knife grinder and produces a product with a maximum particle size of ~4760 μm (0.1875 in.) and a mean particle size of ~2540 μm (0.1 in.). Surveys of the literature and explosives manufacturers did not reveal any experiences with the grinding of energetic materials with oil as the grinding medium. Because of the relatively small holes required to effect the atomization of the oil, these large particle sizes preclude the use of standard oil burner technology.

The production of smaller particle sizes for energetics such as TNT, RDX, and Comp. B can be accomplished by dissolution in a solvent. The solution subsequently would be burned or mixed with fuel oil. If the solution is mixed with fuel oil, the solvent then can either be burned with the mixture or removed from the mixture by evaporation and reused in the dissolution step. If evaporation is used, crystallization of the energetic can occur. The particle size of the crystallized energetic will be dependent on the concentration of the energetic in the solvent, the mixing method with the fuel oil, the evaporation rate of the solvent, and the solubility of the energetic in the fuel oil.

The system envisioned for utilizing the energy content of energetic materials is an oil-fired industrial combustor fueled with a mixture of fuel oil and energetic material. The energy released in the combustion process then can be either transferred to an existing steam-generator tube matrix for the generation of process steam or utilized in a combined gas-turbine—steam cycle. In the combined cycle, the combustion gas would power a gas turbine generator set to produce electricity while the heat content of the turbine exhaust would be used for process steam generation. A simplified block diagram for a typical system is shown in Fig. 1.

However, before implementing such a process, important safety concerns must be addressed. For this reason, a series of tests has been conducted to determine the extent of potential safety hazards, thereby developing operating conditions necessary for the safe operation of this process. The tests, described in this document, include examinations of the handling characteristics of energetic-fuel oil mixtures, compatibility of these mixtures, and the potential for propagation of detonation within these mixtures.

The first series of tests was conducted to determine the handling characteristics of energetic-fuel mixtures. The major variables that affect the fuel handling are the solubility of the energetic in the fuel; the viscosity of the energetic-fuel mixture; and in the case of energetic-fuel slurries, the energetic particle-size distribution. The effect of the energetic particle-size distribution on the plugging of nozzles was also determined.

The second series of tests was conducted to determine chemical compatibility of energetic-fuel mixtures to ensure that the mixtures are not more sensitive than the virgin components. For compatibility, samples were analyzed by Differential Thermal Analysis (DTA), Vacuum Thermal

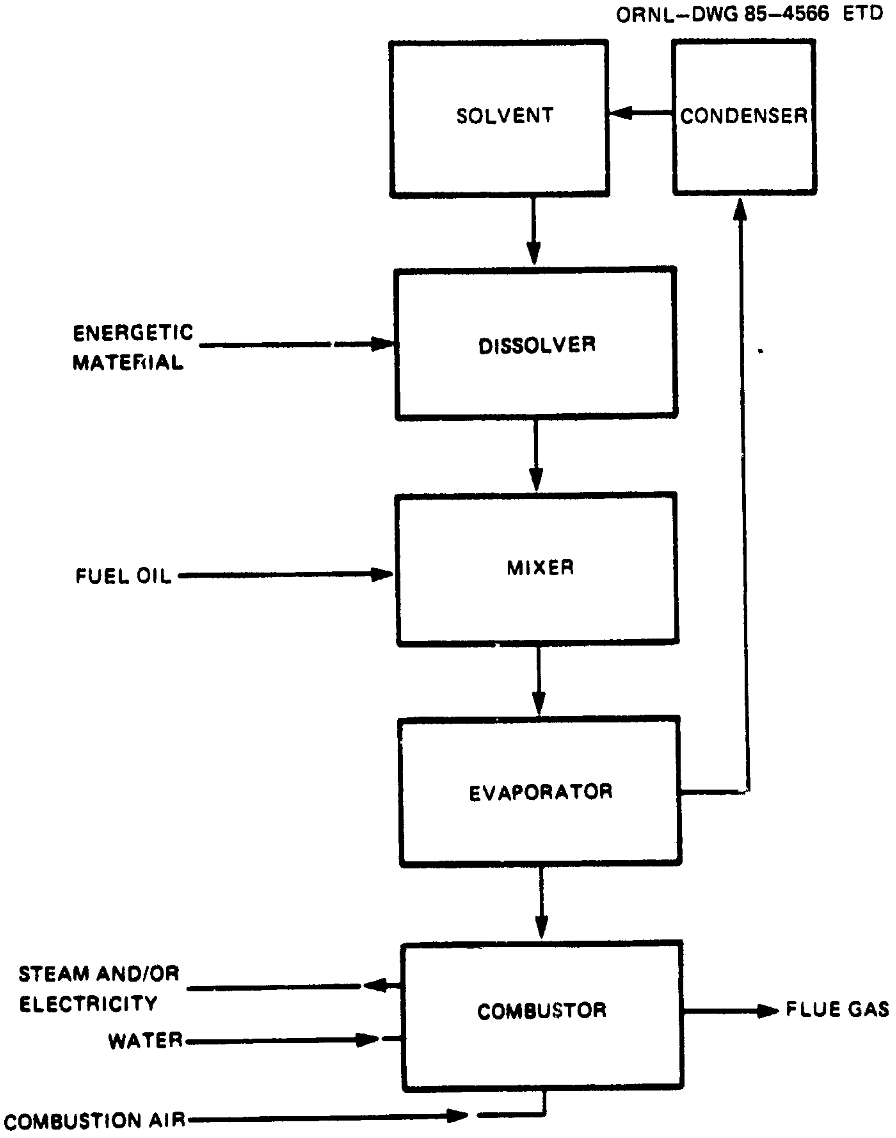

Fig. 1. Flow sheet for a combustion system fired by fuel oil and energetics.

Stability (VTS), and Accelerating Rate Calorimetry (ARC) tests. The results of these tests involving mixtures of fuel and energetics were compared with those of individual mixture components to determine the comparative mixture stability.

The third series of tests was conducted to determine the propagation of detonation characteristics of energetic-fuel mixtures. These tests were run to determine the maximum allowable concentrations of energetic materials that can be safely transported in process piping.

2. DISCUSSION OF RESULTS

The following sections discuss the results of the handling, compatibility, and propagation of detonation tests conducted with energetic-fuel mixtures.

2.1 HANDLING CHARACTERISTICS TESTS

Before beginning the chemical compatibility and propagation of detonation testing, the handling characteristics of the energetic-fuel mixtures were determined. For the particle-size distribution and viscosities studies, three explosives (TNT, RDX, and Comp. B) and two fuel oils (Nos. 2 and 5) were used (see Appendix A). Based on the results of the particle-size distribution study, nozzle-plugging characteristics were determined for typical distributions observed (see Appendix B). The solubility of TNT in fuel oils [~8 wt % at ~57°C (135°F)] could, in time, result in the deposition of TNT on container walls, which could be difficult to remove. Plating characteristics were determined for TNT and fuel-oil mixtures (see Appendix C). Because no solubility of RDX in fuel oil was observed, no plating tests with RDX were performed.

2.1.1 Particle-Size Distribution

Particle-size distribution of energetics in fuel oil was determined by dissolving the energetic (TNT, RDX, and Comp. B) in acetone and adding the solution to the fuel oil (Nos. 2 and 5) and evaporating the acetone to produce a dispersion of crystallized energetic in the fuel oil.

2.1.1.1 TNT particle-size distribution

The particle-size distribution for TNT was not determined. The crystallized TNT had a particle size of <20 μm for a concentration in the oil of <30 wt % (highest value investigated). The TNT was partially soluble in the fuel oil [~8 wt % at a temperature of ~57°C (135°F)]. Mixtures of TNT and fuel oil with TNT contents greater than the saturation value at room temperature produced typical needle crystals of TNT when allowed to cool to room temperature without agitation. Cooling with agitation maintained the initial fine-sized particles, which allow operation of an atomizing nozzle with small holes without plugging.

Before solvent evaporation, mixtures of TNT, fuel oil (both Nos. 2 and 5), and solvent (both acetone and toluene) were observed to form multiphase mixtures. A typical single-phase boundary is shown in Fig. 2. If multiphase mixtures are fired without solvent evaporation, constant agitation of the feed is required to ensure a constant composition feed to the burner.

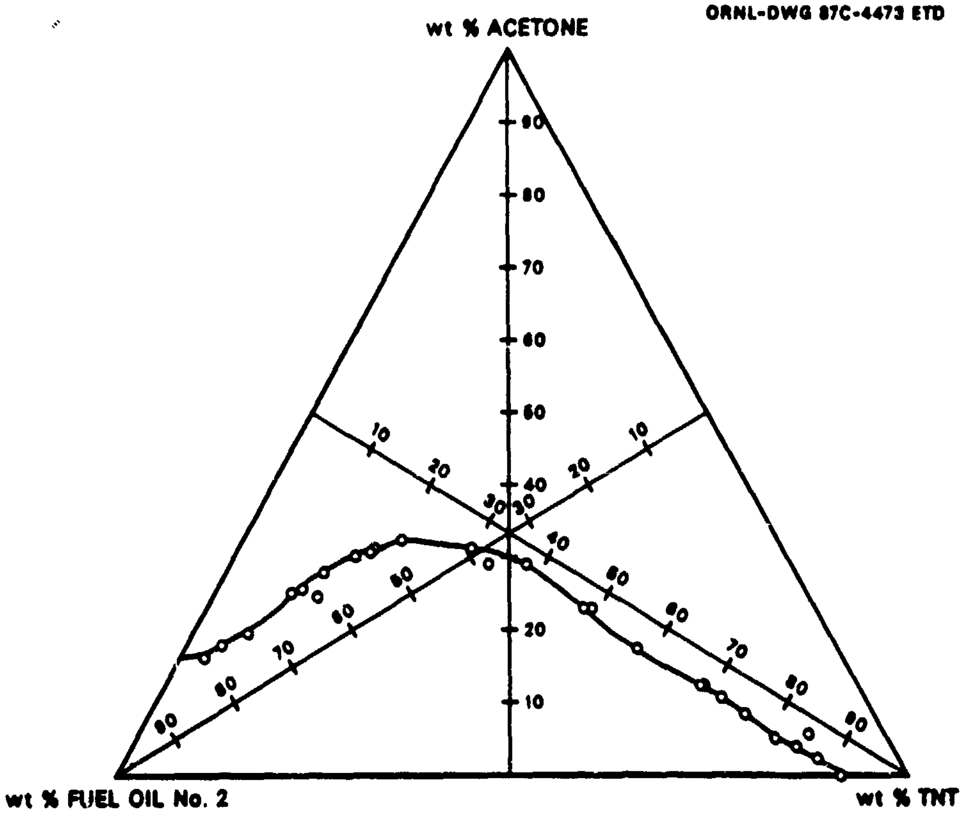

Fig. 2. Single-phase boundary for mixtures of TNT, No. 2 fuel oil, and acetone at ~57°C (135°F)

2.1.1.2 RDX and Comp. B particle-size distribution

The particle-size distribution of recrystallized RDX from acetone solution in the presence of fuel oil was found to be a function of the concentration of the RDX in the acetone before evaporation. Typical mean particle sizes of the recrystallized RDX produced from the treatment of Comp. B with acetone at a concentration of 8.3 wt % RDX in the acetone (equivalent to ~75% of the saturation value) are shown in Table 1. Also shown in Table 1 are the particle sizes of RDX derived from Comp. B treated with toluene both with and without the presence of fuel oil.

Table 1. Mean particle size of RDX from recrystallization in the presence of fuel oil[a]

	Case number				
	1	2	3[b]	4	5
Fuel oil type	No. 2	No. 5		No. 5	No. 5
Solvent	Acetone	Acetone	Toluene	Toluene	Toluene
Temperature, °C (°F)	~57 (135)	~57 (135)	~57 (135)	~57 (135)	~63 (145)
RDX mean particle size, μm	24	38	78	75	67

[a] The RDX was obtained by treating Comp. B with sufficient solvent to give a solution that is ~75% saturated with respect to RDX dissolved in acetone.

[b] Case 3 was run to indicate the effect of the absence of oil on the mean particle size of the RDX content of Comp. B.

As can be seen from the data in Table 1, the mean particle size of the RDX produced by the recrystallization from an acetone solution in the presence of fuel oils was reduced in size by 50 to 70% compared with the size of the RDX in the original Comp. B. This reduction in size allows the operation of an atomizing nozzle with small holes without plugging (see Sect. 2.1.3).

2.1.2. Viscosity

To obtain atomization in an oil burner, it has been determined that the viscosity of the oil should not exceed a range of 20 to 30 centistokes at the burner tip.[5,6] At a temperature of ~38°C (100°F) the Nos. 2 and 5 neat fuel oils indicated viscosities of 3.7 and 167 centistokes, respectively. At a temperature of ~60°C (140°F), the No. 5 oil viscosity was 37 centistokes.

To determine the effects of the TNT concentration on the fuel oil viscosity, the filtrates from the various TNT/solvent/fuel oil tests were used. The viscosities of TNT and No. 2 fuel oil mixtures of 10, 15, and 20 g of TNT per 100 mL No. 2 fuel oil measured at ~38°C (100°F) were 4.2, 4.4, and 4.7 centistokes, respectively. The viscosities of TNT and No. 5 fuel oil mixtures of 10, 20, and 30 g of TNT per 100 mL of No. 5 fuel oil measured at ~60°C (140°F) were 56, 75, and 106 centistokes, respectively. The filtrates of Comp. B slurries stripped of RDX gave viscosities proportional to the TNT concentration that would have resulted from the completed dissolution of the TNT from the Comp. B.

The increase in viscosity of the fuel oils resulting from the dissolved TNT, which could result in atomization difficulty for the more viscous oils, can be reduced by the addition of small amounts of the solvent that was used for the explosive dissolution. The solvent addition could also have the added advantage of lowering the recrystallization temperature of the TNT.

2.1.3. Nozzle-Plugging Characteristics

To avoid plugging the small holes used in the burner atomizing nozzles when firing energetic slurries, a series of tests was performed to determine the plugging characteristics of slurries (see Appendix B). Table 2 shows the plugging results when using a dilute concentration of narrow-range-sized particles. As can be seen from the data, a ratio of hole size to mean particle size greater than approximately 3 : 1 was required to prevent plugging.

The particle-size distribution of RDX generated from Comp. B by evaporation of toluene in the presence of No. 5 fuel (see Table 3) was simulated with closely sized coal particles to determine the effects of distribution and loading on the plugging characteristics. The plugging results for the mixed sizes are given in Table 4. For each nozzle plate tested, the plugging characteristics were independent of solids concentration over the range of 10 to 30 g solid per 100 g of water and for air volume fractions of zero and 0.27. As can be seen from the data in Table 4, for the mixed particle sizes tested, a ratio of hole size to mean size particle of ~4.5 was required to prevent plugging.

Table 2. Particle size range required for nozzle plugging at a concentration of 1.5 wt %

Nozzle hole size [μm (in.)]	Particle size (μm)			Ratio of hole size to mean particle size
	Minimum	Maximum	Mean	
~406 (0.016)	106	150	128	3.18
~508 (0.020)	150	180	165	3.08
~635 (0.025)	180	212	196	3.24
~737 (0.029)	212	250	231	3.19
~1067 (0.042)	300	425	362.5	2.94

Table 3. Particle size and weight fraction of RDX simulant

Particle size (μm)		Weight fraction (%)
Maximum	Minimum	
0	45	21
45	75	26
75	106	23
106	150	27
150	180	2.2
180	212	0.5
212	250	0.2
250	300	0.07
300	425	0.03

Table 4. Plugging characteristics of ~737- and ~1067-μm (0.029- and 0.042-in.) nozzle plates with mixed particle sizes

	Plate number	
	4	5
Number of holes	12	6
Hole diameter, μm (in.)	~737 (0.029)	~1067 (0.042)
Ratio of hole size to mean top size of particle		
Plugging	3.76	3.88
No plugging	4.46	4.62

2.2 COMPATIBILITY OF ENERGETICS AND FUEL OIL TESTS

A series of explosive compositions and commercial fuel oils was prepared by dissolution of the explosive in a solvent and subsequently evaporating the solvent from the mixture. Various mixtures of three explosives (TNT, RDX, and Comp. B) and two fuel oils (Nos. 2 and 5) were tested. The resultant mixtures were then analyzed for stability by DTA, VTS, and ARC tests. In addition to these standard tests, a 6-month test was performed to determine the plating characteristics of energetic-fuel mixtures (see Appendix C).

2.2.1 Differential Thermal Analyses

DTA tests were run separately on the Nos. 2 and 5 fuel oils, TNT, RDX, and Comp. B (Ref. 7). Mixtures of the two oils with the explosives were also run. No extra thermal excursions were noted on the mixtures when compared with the controls. The exotherms of the mixtures occurred at the temperature at which the explosives alone normally decompose.

2.2.2 Vacuum Thermal Stability

VTS tests were run on mixtures of Nos. 2 and 5 fuel oils and TNT, RDX, and Comp. B. For the No. 2 fuel oil, a test temperature of 120°C was used; for the No. 5 fuel oil, test temperatures of 120, 100, 80, and 60°C were used. At a test temperature of 120°C, the gas evolution from the mixture of No. 2 fuel oil and the three explosives was comparable to virgin energetics. The gas evolution from the No. 5 fuel oil and TNT mixture was also comparable to virgin TNT at the four test temperatures of 120, 100, 80, and 60°C. These results indicate that the presence of the fuel oil does not affect the decomposition of the energetics.

For the test temperature range, the gas evolution for all the materials tested followed a first-order reaction as indicated by the straight line produced when the logarithm of the specific reaction rate was plotted against the reciprocal of the absolute temperature. The first order reaction and the comparable gas evolution indicated chemical stability of the test components over the test temperature range.

2.2.3 Accelerating Rate Calorimetry

The accelerating rate calorimetry tests indicated that the energetic-fuel oil mixtures of TNT and Comp. B behaved like a solid in solution. The energetics in the presence of the oil exhibited self-heating characteristics comparable to virgin energetics, which indicated that the fuel oil—energetic mixtures are chemically compatible.

2.2.4 Plating Tests

The plating tests indicated that a very thin film is deposited on the surfaces of stainless steel containers when exposed to mixtures of TNT and fuel oil (Nos. 2, 5, and 6) for at least 6 months at temperatures up to ~79°C (175°F). The results also indicated that any surface buildup was easily removed by flushing with warm acetone.

2.3 PROPAGATION OF DETONATION TESTS

A series of tests has been performed to establish the propagation of detonation characteristics of solvent solutions and fuel oil slurries of TNT and RDX in 2-in. schedule 40 stainless steel pipes. The solutions and slurries were prepared on a weight basis. Testing was conducted using the propagation of the detonation test described in Appendix D and shown in Figs. D.1 and D.2.

2.3.1 Single-Phase Test

Single-phase mixtures of fuels, solvents, and energetics were tested to define mixture reactivity as a function of energetic content. The solvents considered were acetone, toluene, 90 : 10 wt % acetone and water, and 80 : 20 wt % acetone and No. 2 fuel oil. The energetics considered were TNT and RDX. The test results are presented in Appendix D, Tables D.1 to D.6.

Test results summarized in Table 5 indicate that these solvent solutions are not reactive in regard to propagation of detonations in the TNT concentration range of >65 wt % when tested in the static mode. In general, examination of the data in Appendix D indicates that higher concentrations are allowable for the dynamic mode.

Table 5. Summary of propagation of detonation results of single-phase static tests

Energetic material	Solvent type	Concentration (wt %)	
		No propagation	Full propagation
TNT	Acetone	75	80
TNT	Toluene	65	70
TNT	90 : 10 wt % acetone and water	75	*a*
TNT/RDX	Acetone saturated with RDX at 50°C	70/3.4	77/2.6
TNT	80 : 20 wt % acetone and No. 2 fuel oil	78	ND

[a] Not determined.

2.3.2 Slurry Tests

Slurries of fuels and energetics were tested to define mixture reactivity as a function of energetic content. The fuels considered were Nos. 2, 5, and 6 fuel oils, and the energetics considered were TNT and RDX. Test results are presented in Appendix D, Tables D.7 to D.11.

The test results summarized in Table 6 indicate that TNT slurries were not reactive in regard to propagation of detonations in the concentration range of 55 wt %. The witness plates from the RDX static tests with No. 2 fuel oil indicate that the RDX that settled in the pipe formed a fuselike trail along the bottom of the pipe, which, when initiated, propagated a detonation. Cabosil™ (a thickening agent that gave a viscosity of ~3000 centistokes at a concentration of 8 wt %) was added to the No. 2 fuel to prevent the rapid settling out of the RDX and allow both dynamic and static testing. These data (Table 6) indicate that RDX slurries were not reactive in regard to propagation of detonations in the concentration range of 22 wt % in the dynamic operational mode and ~15 wt % in the static operational mode.

Table 6. Summary of propagation of detonation results of slurry tests

Energetic material	Test type	Slurry medium	Concentration (wt %)	
			No propagation	Full propagation
TNT	Static	No. 6 fuel oil	55	65
RDX	Static	No. 2 fuel oil	[a]	5.3
RDX	Static	92 : 8 wt % No. 2 fuel oil and cabosil	15	26
RDX	Dynamic	92 : 8 wt % No. 2 fuel oil and cabosil	22	26
RDX	Dynamic	No. 5 fuel oil	15	20

[a] Not determined.

Table 7. Propagation of detonation test results for energetic-water slurries

Sample material	Slurry type	Pipe size[a] (in.)	Concentration (wt %)		Reference
			No propagation	Full propagation	
Comp. B	Gelled	2	30	40	Table IV[b]
Comp. B	Settled	2	35	45	Table V[b]
TNT	Gelled	2	40	60	Table III[b]
TNT	Settled	2	40	55	Table VI[b]
RDX	Gelled	2	25	30	Table 1[c]
RDX	Settled	2	15	20	Table 6[c]

[a] Confined material dimension above which sustained propagation of detonations can be expected. Nominal size of schedule 40 pipe size shown.

[b] *Source*: G. Petino, Jr., and D. Westover, *Detonation Propagation Tests on Aqueous Slurries of TNT, Composition B, M-9 and M-10*, Technical Report No. 4584, Facilities and Protective Technology Division, Manufacturing Technology Directorate, Picatinny Arsenal, N.J., November 1973.

[c] *Source*: G. Petino, Jr., and D. Westover, *Detonation Propagation Tests on Aqueous Slurries of RDX, HMX, M-1, and Nitrocellulose*, Contractor Report ARLCD-CR-77002, Large Caliber Weapons Systems Laboratory, Dover, N.J., April 1977.

For comparison, Table 7 shows the propagation of detonations data for 2-in. schedule 40 pipes containing water slurries of energetic RDX, TNT, and Comp. B for use in the design and operation of the feed system for propellant and explosive incinerators such as those employed by Radford Army Ammunition Plant.[8,9]

The indicated lower limits for the oil and water data under comparable conditions (i.e., static and settled) are 55 and 40 wt % for TNT and 15 and 15 wt % for RDX, respectively. These values indicate that the slurry mediums of oil and water did not contribute significantly to the propagation of detonation characteristics of TNT and RDX.

3. EXPERIMENTAL PROCEDURES

The following sections describe the test plan, selection of test materials, mix preparation, and subsequent testing.

3.1 OVERALL TEST PLAN

Explosives that require disposal by the U.S. Army primarily consist of TNT, RDX, and Comp. B (~60% RDX, 39% TNT, and 1% wax).[1,2] To determine the potential safety hazards and to specify conditions necessary for safe operation when using these energetic materials as a supplemental fuel in an industrial combustor, a test series was developed.

The combustion process involves the use of (1) solvents and fuel oils in the feed preparation systems that must be compatible and (2) fuel atomizing nozzles that could be subject to plugging if the energetic particle sizes are not small enough. In addition, the process piping must be designed to prevent propagation of detonations. Based on these requirements, three series of tests were developed to supply safety and design information involving the energetic-fuel mixture handling, compatibility, and propagation of detonation characteristics.

3.2 SELECTION OF TEST SAMPLE MATERIALS

3.2.1 General

The use of energetics as a fuel supplement in industrial combustors will involve the use of solvents (acetone, acetone and water solutions, and toluene), fuel oils (Nos. 2, 5, and 6), and energetics (TNT, RDX, and Comp. B). The use of these materials in various combinations will depend on safety, environmental, and operability considerations as well as on availability and process economics. The solvents and oils will be commercial grades, and the energetics will be material generated by explosive production, by ordnance loading, or from ordnance washout.

3.2.2 Solvent Components

Laboratory CP grades of toluene and acetone were used for all laboratory and propagation of detonation sample preparation. The 90 wt % acetone and water mixture was prepared from CP grade acetone and distilled water.

3.2.3 Fuel Oil Components

Commercial grades of Nos. 2, 5, and 6 fuel oils were obtained from utility supply brokers. A typical analysis of these oils is shown in Table 8.

Table 8. Analysis of fuel oils

	Grade number		
	2	5	6
Element			
Carbon, %	86.35	85.60	85.70
Hydrogen, %	12.72	11.70	10.55
Oxygen, %	0.82	0.80	0.92
Nitrogen, %	[a]	0.33	0.50
Sulfur, %	0.10	1.52	2.25
Ash, %	0.01	0.05	0.08
Density, lb/gal at ~16°C (60°F)	7.208	7.941	8.215
Higher heat value, Btu/lb	19,500	18,650	18,250
Viscosity, centistokes at 38°C (100°F)	3.7	167	180

[a] Trace.

3.2.4 Explosive Components

The TNT was obtained from the Radford Army Ammunition Plant and contains typically 99.84% 2, 4, 6 TNT; 0.1% 1, 3, 4 TNT; and small amounts (0.06% total) of DNT and water. The Comp. B-3 (60% RDX and 40% TNT) and Type II, Class 1, RDX was obtained from the Holston Defense Corporation. The Type II, Class 1, RDX typically contains ~88.6% RDX, 8.6% HMX, and 2.8% of other nitramine variations formed during the RDX manufacture.

3.3 MIX PREPARATION

3.3.1 Laboratory Tests

Portions of energetics (RDX, TNT, or Comp. B), solvents (acetone, acetone and water, or toluene), and fuel oils (Nos. 2, 5, or 6) were weighed to ±1 mg in predetermined combinations as specified by the test procedure. Mixing was accomplished by use of air-driven agitators, and heat was supplied by water or oil baths with temperatures maintained at ±0.1°C. Special laboratory preparation techniques are described in Appendixes A and C.

3.3.2 Plugging Tests

The sized coal was prepared by wet screening ground coal with a series of Tyler screens to give a rough size cut. Particles were rescreened using adjacent screen sizes as shown in Appendix B until all particles passed the top-size screen and none passed the bottom-size screen. Mixes up to ~22.7 kg (50 lb) were prepared; solids were weighed to ±0.10 g, and water was weighed to ~±28 g (±1 oz).

3.3.3 Propagation of Detonation Tests

3.3.3.1 Single phase

Portions of TNT and RDX (when required) and solvent (toluene, acetone, 90 wt % acetone and water mixture or fuel oil and acetone mixture) were weighed to ±0.1 g and mixed together to yield the required percentage called for in the test. The mix was agitated with an air-driven agitator until dissolution of the solids was complete.

3.3.3.2 Slurry

The slurries of RDX (both with and without a gelling agent) and No. 2 fuel oil were prepared by slowly adding portions of RDX weighed to ±0.1 g to the liquid medium. The slurry was mixed with an air-driven agitator until visual inspection indicated that the mixture was uniform.

Because of the high viscosity of No. 6 fuel oil, portions of TNT weighed to ±0.1 g were added to the No. 6 fuel oil and the mixture was heated to a temperature of 85°C to allow the TNT to melt and uniformly mix with the No. 6 fuel oil. The mix was agitated with an air-driven agitator until visual inspection indicated that the mixture was uniform. Then the mixture of TNT and No. 6 fuel oil was charged into the test pipes at 70°C, at which temperature the viscosity of the No. 6 fuel oil is ~25 centistokes.

3.4 PARTICLE-SIZE AND VISCOSITY TESTS

Because of the general insolubility of energetics in fuel oil, mixtures of energetics and fuel oil must be burned as slurries. Particle-size distributions and mix viscosities were determined for various mixes of TNT, RDX, and Comp. B with fuel oils. The particle-size distribution was determined by wet screening of the slurries, and the viscosities were measured using a Saybolt viscometer according to ASTM-D88-73 specifications. These test procedures are described in Appendix A.

3.5 NOZZLE-PLUGGING TEST

The plugging of small holes in an atomizing nozzle during the firing of energetics in an industrial combustor would require that the system be shut down and cleaned. To decrease the frequency of this operation, design data concerning the plugging characteristics of nozzles with slurries containing particle-size distributions corresponding to those obtained from energetics (see Appendix A) were obtained (see Appendix B).

3.6 COMPATIBILITY TESTS

Chemical stability of energetics (TNT, RDX, and Comp. B) and fuel oil (Nos. 2, 5, and 6) mixtures were determined by DTA, VTS, and ARC tests. In addition to the chemical stability tests, a plating test was run to determine the tendency of energetic materials to plate on metal surfaces. The description and results of these tests are given in Appendix C.

3.7 PROPAGATION OF DETONATION TESTS

For all explosive materials at a fixed diameter, an explosive concentration exists that is too small to sustain propagation of a shock wave through the explosive. In general, the more reactive the explosive, the smaller the concentration required to propagate the explosive reaction.

Single-phase and slurry mixtures were tested to define the mixture sensitivity to shock as a function of the energetic content using the propagation of detonations protocol described in Appendix D. Single-phase and slurry mixtures are confined in schedule 40 stainless steel pipes (Figs. D.1 and D.2) and subjected to an induced shock from a combination booster consisting of PBX 9407, PBX 9205, and TNT. Use of schedule 40 stainless pipe generates data useful in evaluating the risk of sustained explosive reaction in typical process piping systems. The results of these tests are given in Appendix D.

4. WARRANTY AND DISCLAIMER

Within the scope of work, Los Alamos National Laboratory and Oak Ridge National Laboratory warrant that they have exercised their best efforts in performing the hazards analysis and testing reported herein; but they specifically disclaim 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 Los Alamos National Laboratory or Oak Ridge National Laboratory are adopted.

REFERENCES

1. J. D. Pinkerton, R. F. Tobias, and R. Scola, *Energy Recovery from Army Ammunition Plant Solid Waste by Pyrolysis*, ARLCD-CR-78030, TRW Defense and Space Systems, Redondo Beach, Calif., March 1979.

2. Comp. JCAP Demilitarization and Disposal Task Group, *Joint Conventional Ammunition Program Coordination Group, Volume I, Demilitarization/Disposal Inventory*, Rock Island Arsenal, Rock Island, Ill., June 30, 1983.

3. R. Scola and J. Santos, *Fluidized Bed Incinerator for Disposal of Propellants and Explosives*, ARLCO-TR-78032, ARRADCOM Large Caliber Weapon Systems Laboratory, Dover, N.J., October 1978.

4. D. E. Rolison, R. L. Dickenson, and R. Scola, *An Evaluation of an Incinerator for Waste Propellants and Explosives*, Technical Report 4984, Radford Army Ammunition Plant, Hercules Inc., Radford, Va., December 1976.

5. J. H. Perry, ed., *Chemical Engineers' Handbook*, 3d ed., McGraw-Hill, New York, 1950.

6. R. T. Haslam and R. P. Russell, *Fuels and Their Combustion*, McGraw-Hill, New York, 1926.

7. S. W. Dudiel and J. Baytos, *Differential Thermal Analysis Unit*, LAMS-2988, Los Alamos Natl. Lab., Los Alamos, N.M., December 1963.

8. G. Petino, Jr., and D. Westover, *Detonation Propagation Tests on Aqueous Slurries of TNT, Composition B, M-9 and M-10*, Technical Report No. 4584, Facilities and Protective Technology Division Manufacturing Technology Directorate, Picatinny Arsenal, Dover, N.J., November 1973.

9. G. Petino, Jr., and D. Westover, *Detonation Propagation Tests on Aqueous Slurries of RDX, HMX, M-1, and Nitrocellulose*, Contractor Report ARLCD-CR-77002, ARRADCOM Large Caliber Weapons Systems Laboratory, Dover, N.J., April 1977.

APPENDIX A

PARTICLE-SIZE AND VISCOSITY CHARACTERISTICS OF TNT AND COMPOSITION B FUEL OIL MIXTURES

INTRODUCTION

Burning energetic materials such as TNT and Composition B (Comp. B) in an oil-fired industrial combustor presents several difficult engineering problems. Among these are the interrelated problems of the feed preparation and the burner design. Efficient burning of the fuel oil requires atomization before combustion. The primary functions of the burner are to atomize the oil and to prepare it for mixing with the combustion air.

Because of the general solubility limitations of some of the energetic materials in fuel oil, they must be burned as a slurry with the fuel oil. The energetic material to be burned is usually in the form of scrap, shell washout, or unacceptable end items. A large portion of the material must be reduced in size before combustion. The current state-of-the-art size-reduction methods use a water medium of ~10 g of water per gram of energetic material. A typical size-reduction facility is located at Radford Army Ammunition Plant.[1] This facility employs a rotary knife grinder and produces a product with a maximum particle size of ~4760 µm (0.1875 in.) and a mean particle size of 2540 µm (0.1 in). Because of the relatively small holes required to effect the atomization of the oil, these large particle sizes preclude the use of standard oil burner technology.

The production of smaller particle sizes for an energetic such as TNT and Comp. B can be accomplished by dissolution in a solvent followed by mixing with fuel oil. The solvent then can either be burned with the mixture or removed from the mixture by evaporation and reused in the dissolution step. The resulting particle-size distribution will be a function of the solubility of energetic material in both the solvent and the fuel oil. To determine the optimum strategy for burning energetic materials in industrial combustors, the particle-size distributions for TNT and Comp. B resulting from energetic-fuel and energetic-solvent mixtures were determined by J. F. Baytos and J. O. Martinez of the Los Alamos National Laboratory* (LANL). In addition to the particle-size

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*Los Alamos National Laboratory, subcontract 41X-90448V, May 15, 1984.

distribution, LANL also determined the viscosity of TNT and Comp. B fuel oil mixtures.

PROCEDURES

PARTICLE-SIZE DISTRIBUTION

A general laboratory procedure was developed to determine particle-size distribution resulting from the admixture of a solution of either TNT and/or RDX in a solvent with fuel oil and subsequent evaporation of the solvent. The general procedure follows.

Fifty mL of fuel oil in a beaker was placed in a hot water bath and agitated with an air-driven stirrer. After the fuel oil reached a temperature of ~57°C (135°F), a solvent-explosive solution (50 mL at ~50°C (122°F) was added and mixed with the fuel oil. The mixture was stirred at a constant temperature of ~57°C (135°F) until the solvent evaporated. The completeness of solvent evaporation was indicated by an absence of solvent odor and confirmed by a mass balance calculation. The energetic material, if soluble in the fuel oil, remained in solution as the solvent was evaporated; or if insoluble in the fuel oil, crystallized as the solvent was evaporated.

The oil-energetic mixture was passed through a series of hot [~57°C (135°F)] sieves of decreasing pore size. The oil passed and left oil-coated crystals of energetic material on the grids. The oil was washed off the sieves with a solvent in which the oil has a high solubility and the energetic material a very low solubility. The energetic material retained on the various sieves and the catch pan was then determined by weight to give the particle-size distribution.

This procedure was used with Nos. 2 and 5 fuel oil. No. 2 fuel oil is a distillate oil used by installations not equipped with preheating facilities. It has a specific gravity of 0.865 at ~16°C (60°F) and a viscosity of 3.7 centistokes at ~38°C (100°F). No. 5 fuel oil is a residual-type oil used by burner installations equipped with preheating facilities. It has a specific gravity of 0.953 at ~16°C (60°F) and a viscosity of 167 centistokes at ~38°C (100°F). These oils were chosen for the experiments as being representative of the types of fuel oils burned in industrial combustors.

The energetic materials chosen were TNT, RDX, and Comp. B. These materials represent a large fraction of the energetics in the Demil account[2] and the current waste from propellants, explosives, and pyrotechnics (PEP) production.[3] The effect of energetic concentration upon the particle-size distribution resulting from the solvent evaporation and crystallization of the energetic material in the presence of the fuel oil was studied. Energetic concentrations of 5, 10, 15, and 20% of the oil were investigated.

Three solvents were used: toluene, acetone, and 90 : 10 volume ratio of acetone and water. The 90 : 10 acetone and water solvent was chosen to account for the presence of some water in the energetic that might result from a wet washout demilling process.

VISCOSITY

Viscosity, which is a measure of the force required to shear a fluid at unit rate, was measured with a Saybolt viscometer that allows the measurement of the time required for a definite quantity of fluid to flow through a fixed resistance. The Saybolt viscosity tests were run according to ASTM-D88-73 specifications, using the universal [~18-mm-diam (0.0697-in.)] orifice, and the efflux measured in Saybolt Universal seconds. The Saybolt Universal seconds were then converted to centistokes.

RESULTS AND DISCUSSION

NO. 2 FUEL OIL

The discussion for No. 2 fuel oil pertains to the results obtained with Comp. B, TNT, and RDX for concentrations of 5, 10, 15, and 20 g of energetic per 100 mL of the oil using 100% acetone as the solvent for the energetic material.

The TNT did not form particles larger than 20 μm after the acetone was stripped from the No. 2 oil mixture as indicated by 100% of the mixture passing the 20-μm screen. At room temperature the TNT crystallized from an undisturbed mixture into red needles, and the slurry gelled, indicating that part of the TNT was soluble in the fuel oil. The solubility of the TNT in the No. 2 fuel oil could be dependent on the source of the crude oil [e.g., Venezuela and the United States (Pennsylvania and Texas)] or the method of solution. If source-dependent, the TNT particle-size distribution, as with the RDX, would be dependent on crystallization conditions.

Shown in Table A.1 are the particle-size distributions of RDX obtained from the slurry process tests with No. 2 fuel oil. Shown also in Table A.1 is the as-received particle-size distribution of RDX (Lot 253) for comparison to the treated distribution.

The data in Table A.1 indicate that the particle-size distribution of the RDX that results from crystallization upon evaporation of the acetone is a function of the degree of saturation of the RDX in the acetone before admixing with the No. 2 fuel oil. Two of the cases shown in Table A.1 are above the saturation value of 12.8 g of RDX per 100 g of

Table A.1. Particle-size distribution of RDX from slurry process tests with No. 2 fuel oil

Energetic concentration (g/100 mL oil)	RDX retained on sieve (wt %) by sieve size (μm)								
	From Comp. B/acetone/No. 2 fuel oil extraction								
	250	177	125	80	62	45	30	20	<20
5	0	0	0	0	0	0	0	9.0	91.0
10	0.7	0.3	0.2	0.3	0.8	1.2	10.8	19.6	66.1
15	0.2	0.3	0.9	3.6	9.4	12.5	0.4	39.7	33.0
20	14.0	18.8	14.4	14.0	8.2	7.1	3.3	2.6	17.6
	From RDX (Lot 253)/acetone/No. 2 fuel oil extraction								
	250	177		88	62	45	30	20	<20
5	0.3	0.5	1.3	3.5	23.7	31.3	23.1	10.7	6.7
10	2.3	3.8	10.9	22.8	27.6	10.1	3.8	1.1	17.6
15	55.3	21.7	7.2	2.5	3.1	2.5	1.4	0.8	5.5
	From Lot 253, as received								
	250	177	125	88	62	45	30	20	<20
	27.5	33.5	16.7	7.9	3.7	1.8	0.4	0.4	8.1

acetone at ~50°C (122°F). The two cases are 15 g of RDX per 100 mL of oil and 20 g of Comp. B per 100 mL oil (the Comp. B contains ~60 wt % RDX). For these two cases, a particle-size distribution was obtained that was assumed to have resulted from starting with a saturated solution of RDX in acetone. The 100% saturation distribution was obtained by assuming that only the smallest particles were dissolved when the quantity of RDX exceeded 12.8 g per 100 g of acetone. The remaining particles were assumed to be unchanged and subtracted from the particles found. The remaining particles were redistributed to give the 100% saturation particle-size distribution. Other degrees of saturation were obtained by expressing the quantity of RDX dissolved in the acetone as a percentage of 12.8 g of RDX per 100 g of acetone. Shown in Figs. A.1 and A.2 are the particle-size distributions of RDX as a function of the degree of saturation in the acetone solvent.

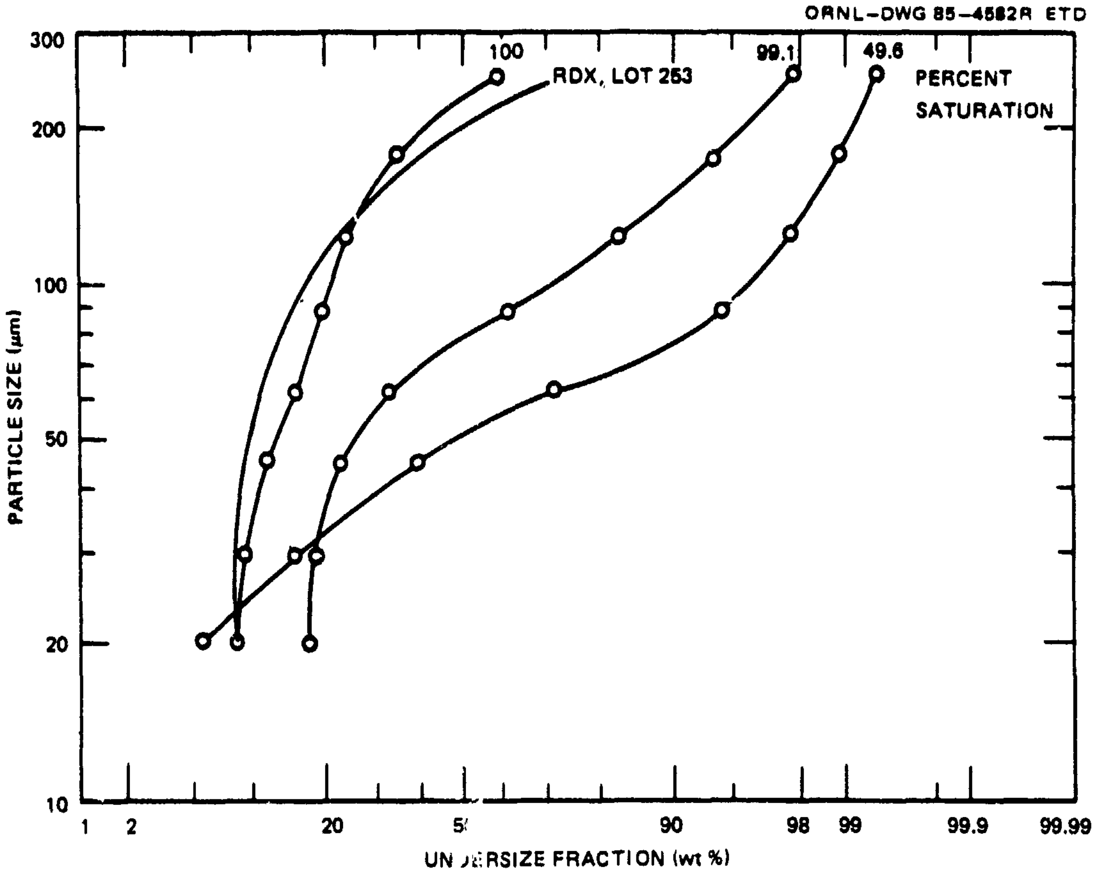

Fig. A.1. Particle-size distribution of RDX (Lot 253) generated by evaporation of acetone in the presence of No. 2 fuel oil.

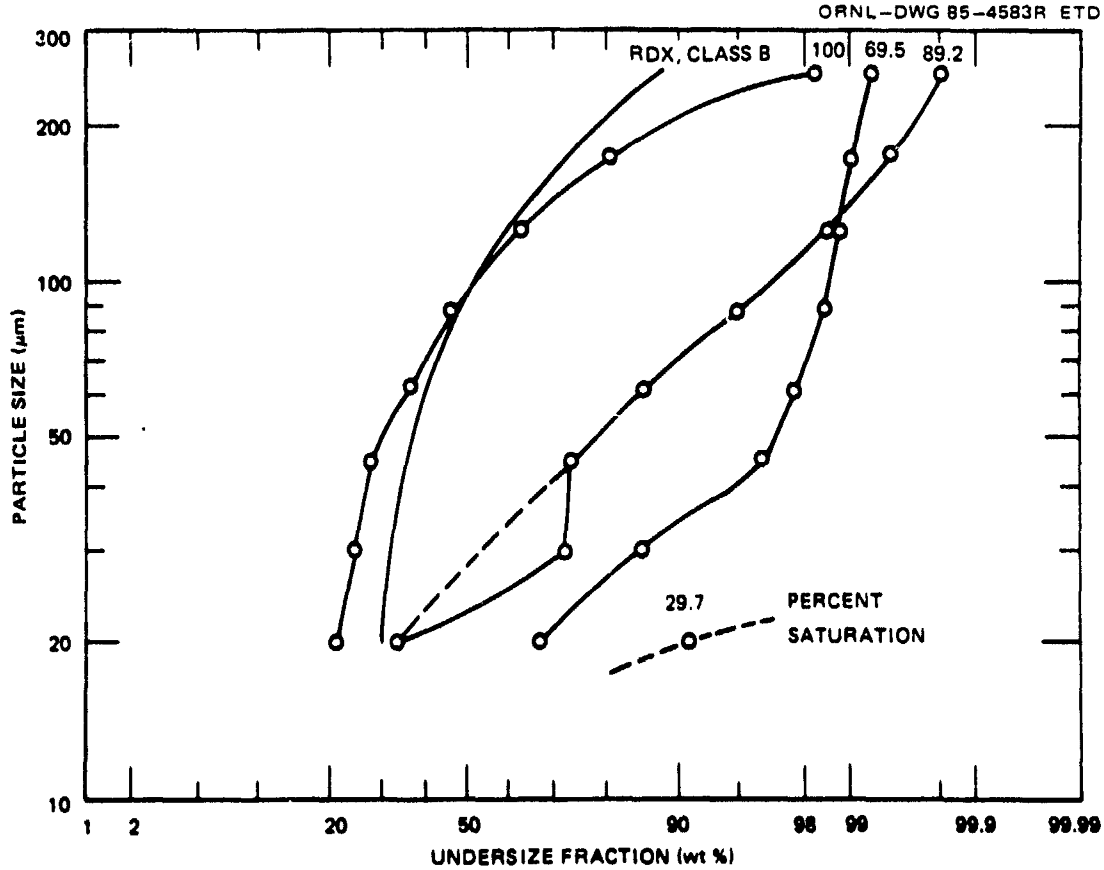

Fig. A.2. Particle-size distribution of RDX (Comp. B, Class B) generated by evaporation of acetone in the presence of No. 2 fuel oil.

NO. 5 FUEL OIL

The discussion for No. 5 fuel oil pertains to the results obtained with Comp. B and TNT for concentration of 10, 15, 20, and 30 g of energetic per 100 mL of the oil using 100% acetone, a 90-to-10 volume ratio of acetone and water, and 100% toluene as the solvent for the energetic material.

The slurry procedure was performed at a temperature of ~57°C (135°F) with TNT and No. 5 fuel oil using acetone, acetone/water (90 : 10 vol %), and toluene. An additional test was run with the toluene at a temperature of ~63°C (145°F). For each of these tests, concentrations of 10, 20, and 30 g of TNT per 100 mL of No. 5 fuel were investigated. After the solvent was stripped, the TNT and No. 5 fuel oil mixture was transferred to a hot sieve at test temperature. All except ~3 wt % of the TNT readily passed through the smallest sieve. No particles of TNT could be detected by sight or feel. Upon cooling to room temperature, the TNT crystallized in a manner similar to the No. 2 fuel oil. However, unlike the No. 2 fuel oil, which produced needlelike TNT crystals ~10 mm (~0.4 in.) long and ~1 mm (~0.04 in.) in diameter, the crystals from the No. 5 fuel oil were much smaller. This size difference was probably related to the much higher room temperature viscosity of the No. 5 fuel oil. As was the case with the No. 2 fuel oil, the solubility of the TNT in the No. 5 oil could be dependent upon the source of the crude or the method of solution. If source dependent, the TNT particle-size distribution, as with the RDX, would be dependent upon crystallization conditions.

For the No. 5 fuel oil, the particle-size determination of RDX from Comp. B was made using two solvents, 100% acetone and a 90 : 10 volume ratio of acetone and water. The 90 : 10 acetone and water solvent was chosen to allow the presence of some water in the energetic that might be present from a wet washout demilling process. The RDX in Comp. B, after being dissolved in acetone (and acetone and water) and slurried with hot No. 5 fuel oil [~57°C (135°F)], crystallizes after the solvent is evaporated. The resultant slurry thickened, which indicated a change in the viscosity caused by the solubility of the TNT from the Comp. B (similar observations were made for the No. 2 fuel oil slurries). The No. 5 fuel oil slurry did not pass the sieves as easily as the No. 2 fuel oil. Some of the No. 5 oil passed and left the RDX and thickened oil on the sieve surface. When no more oil passed and the residue cooled, a thick gummy deposit remained. To separate the oil from the RDX, cold toluene (RDX is only slightly soluble in toluene) was used to wash the oil through the sieves leaving a dark brown residue of RDX. The remaining oil was washed from the RDX with petroleum ether.

Shown in Table A.2 are the particle-size distributions of RDX obtained from the slurry process tests with the No. 5 fuel oil using 100% acetone and a 90 : 10 volume ratio of acetone and water as solvents. Shown also in Table A.2 are the particle-size distributions of RDX extracted with toluene from two lots of Comp. B for comparison with the treated distribution. The data in Table A.2 indicate that the particle-

Table A.2. Particle-size distribution of RDX from slurry process tests with No. 5 fuel oil

Energetic concentration (g/100 mL oil)	RDX retained on sieve (wt %) by sieve size (μm)								
	From Comp. B/acetone/No. 5 fuel oil extraction								
	250	177	125	88	62	45	30	20	<20
Lot 11-65									
10	0	0.1	0.7	0.0	0.5	5.1	40.7	21.2	31.7
15	27.8	21.1	16.2	9.9	4.0	3.5	3.8	1.9	11.8
20	3.9	18.0	32.8	18.9	8.7	5.2	3.2	1.2	8.1
Lot K461									
10	0.1	0.1	0.0	0.0	0.2	0.2	4.4	30.5	64.5
15	0.4	0.3	0.7	3.0	8.4	21.8	30.7	13.6	21.1
20	0.2	2.7	14.9	25.3	17.1	12.4	6.6	3.6	17.2
	From Comp. B/acetone/water 90/10/No. 5 fuel oil extraction								
	250	177	125	88	62	45	30	20	<20
Lot 11-65									
10	0.1	0.1	0.2	1.9	13.7	30.1	29.0	4.6	20.3
15	0.5	0.5	2.9	23.2	26.2	16.6	7.8	3.5	18.8
20	0.2	8.4	44.3	23.0	8.5	2.7	1.6	1.6	9.7
	From Comp. B by toluene, dried, and then sieved without further treatment								
	250	177	125	88	62	45	30	20	<20
Lot 11-65	0.1	0.9	12.1	28.7	22.0	15.1	10.1	3.4	7.6
Lot K461	0.2	0.6	12.4	21.7	19.6	17.9	14.1	3.8	9.7

size distribution of the RDX resulting from crystallization upon evaporation of the solvent is a function of the degree of saturation of the RDX in the solvent before admixing with the No. 5 fuel oil (a similar observation was made for the No. 2 fuel oil). The solubility of RDX is ~12.8 g of RDX per 100 g for 100% acetone and 11 g of RDX per 100 g for a 90 : 10 volume ratio of acetone and water at ~50°C (122°F). For the cases in Table A.2 that exceed these values, a particle-size distribution was obtained that was assumed to have resulted from starting with a saturated solution of RDX in the solvent. The 100% saturation curve for both solvents was obtained by assuming that only the smallest particles were dissolved when the quantity of RDX exceeded saturation. The remaining particles were assumed to be unchanged and subtracted from the particles found. The remaining particles were redistributed to give the 100% saturation particle-size distribution. Other degrees of saturation were obtained by expressing the quantity of RDX dissolved in the solvent as a percentage of the saturation value. Shown in Figs. A.3 and A.4 are the particle-size distributions of RDX as a function of the degree of saturation in the acetone solvent and in the acetone and water solvent in the 90 : 10 volume ratio, respectively.

To determine if the presence of the No. 5 fuel oil had an effect on the particle-size distribution of RDX, toluene was used to dissolve the

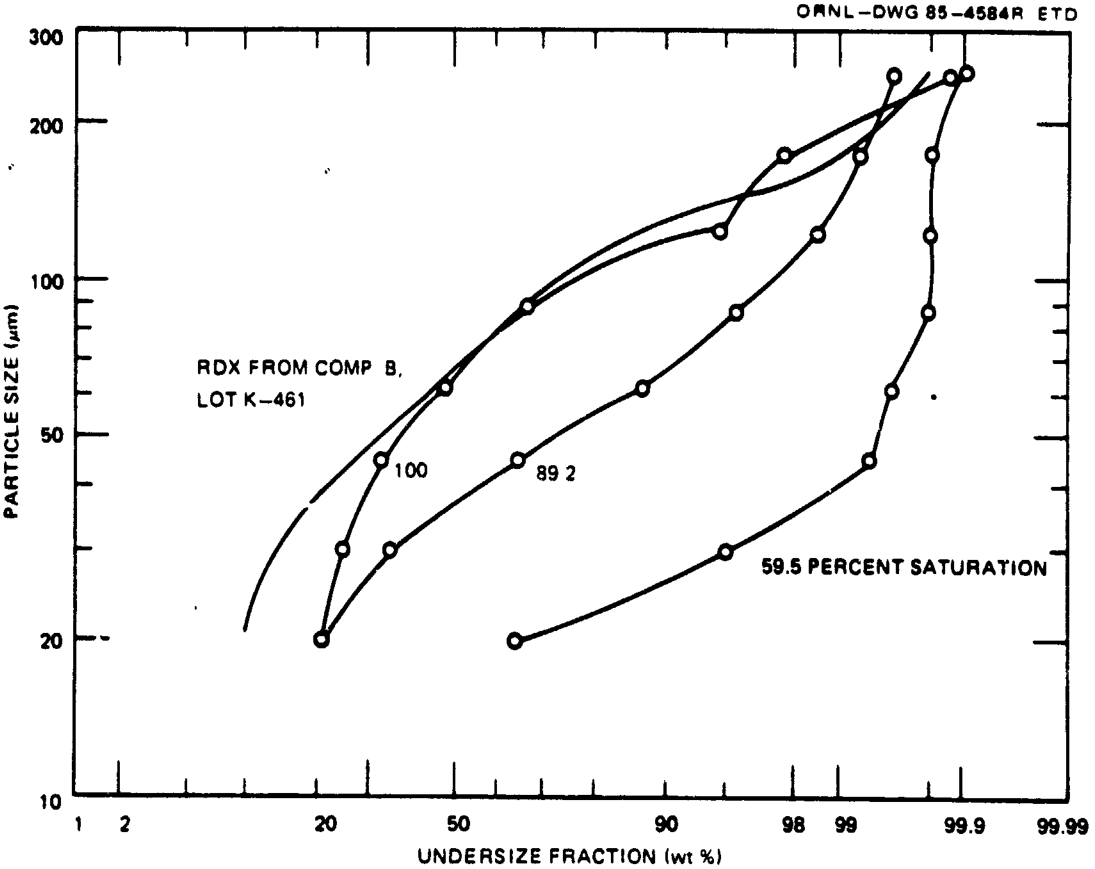

Fig. A.3. Particle-size distribution of RDX (Comp. B, Lot K-461) generated by evaporation of acetone in the presence of No. 5 fuel oil.

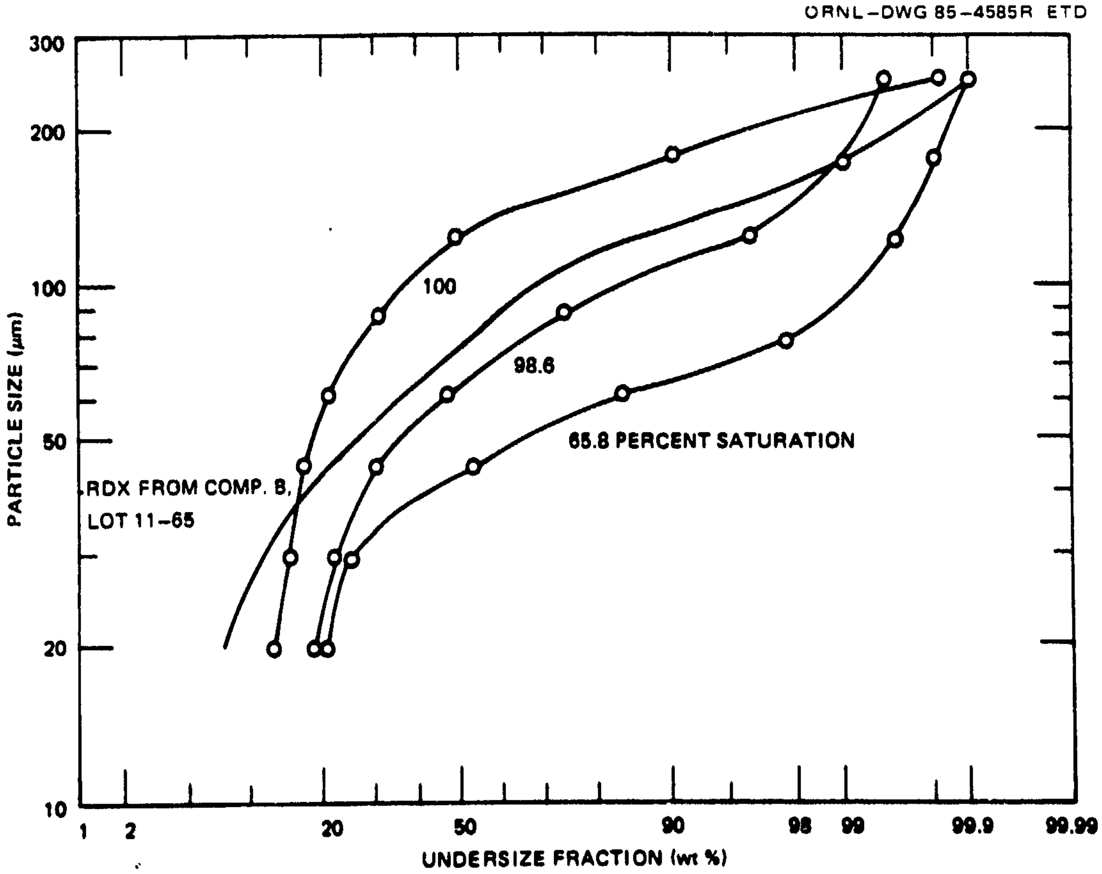

Fig. A.4. Particle-size distribution of RDX (Comp. B, Lot 11-65) generated by evaporation of a mixture of acetone and water (90 : 10) by volume in the presence of No. 5 fuel oil.

TNT from Comp. B (RDX is only slightly soluble in toluene); the resultant mixture was added to No. 5 fuel oil, and the toluene evaporated. Two evaporation temperatures, ~57°C (135°F) and ~63°C (145°F), were used. Shown in Table A.3 are the particle-size distributions of RDX obtained from the slurry process tests with No. 5 fuel oil and toluene. The particle-size distribution of the RDX in the Comp. B (Lot 11-65) is given in Table A.2. Shown in Figs. A.5 and A.6 are the particle-size distribution of the RDX as a function of the quantity of Comp. B added to the No. 5 fuel oil. As can be seen from Figs. A.5 and A.6, the particle-size distributions of the RDX was very nearly the same for each condition and similar to the RDX distribution in the untreated Comp. B (Lot 11-65).

Table A.3. Particle-size distribution of RDX from slurry process tests with No. 5 fuel oil: Comp. B (Lot 11-65)/toluene/No. 5 fuel oil extraction

Energetic concentration (g/100 mL oil)	RDX retained on sieve (wt %) by sieve size (μm)								
	Extraction at ~57°C (135°F)								
	250	177	125	88	62	45	30	20	<20
10	0.1	0.3	3.5	18.2	30.1	21.9	11.5	2.2	12.2
15	1.0	0.4	6.0	26.9	31.0	18.4	8.2	1.2	6.9
20	0.2	0.3	5.0	22.4	33.8	20.5	9.6	1.5	6.7
	Extraction at ~63°C (145°F)								
	250	177	125	88	62	45	30	20	<20
10	0.2	0.4	7.0	24.9	34.2	18.9	6.8	1.2	6.4
15	0.3	0.4	2.9	18.1	34.4	22.2	10.5	2.2	8.7
20	0.2	0.3	1.8	2.6	57.2	18.3	9.8	1.9	7.9

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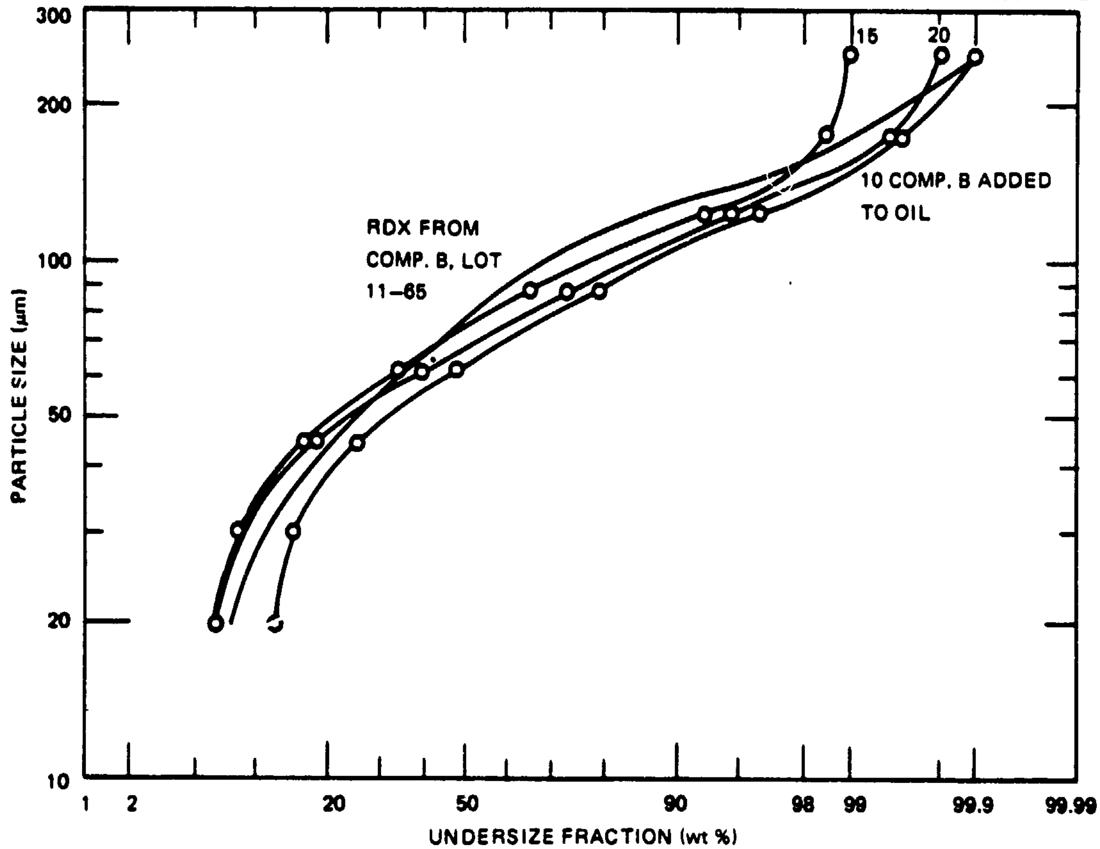

Fig. A.5. Particle-size distribution of RDX (Comp. B, Lot 11-65) generated by evaporation of toluene in the presence of No. 5 fuel oil at ~57°C (135°F).

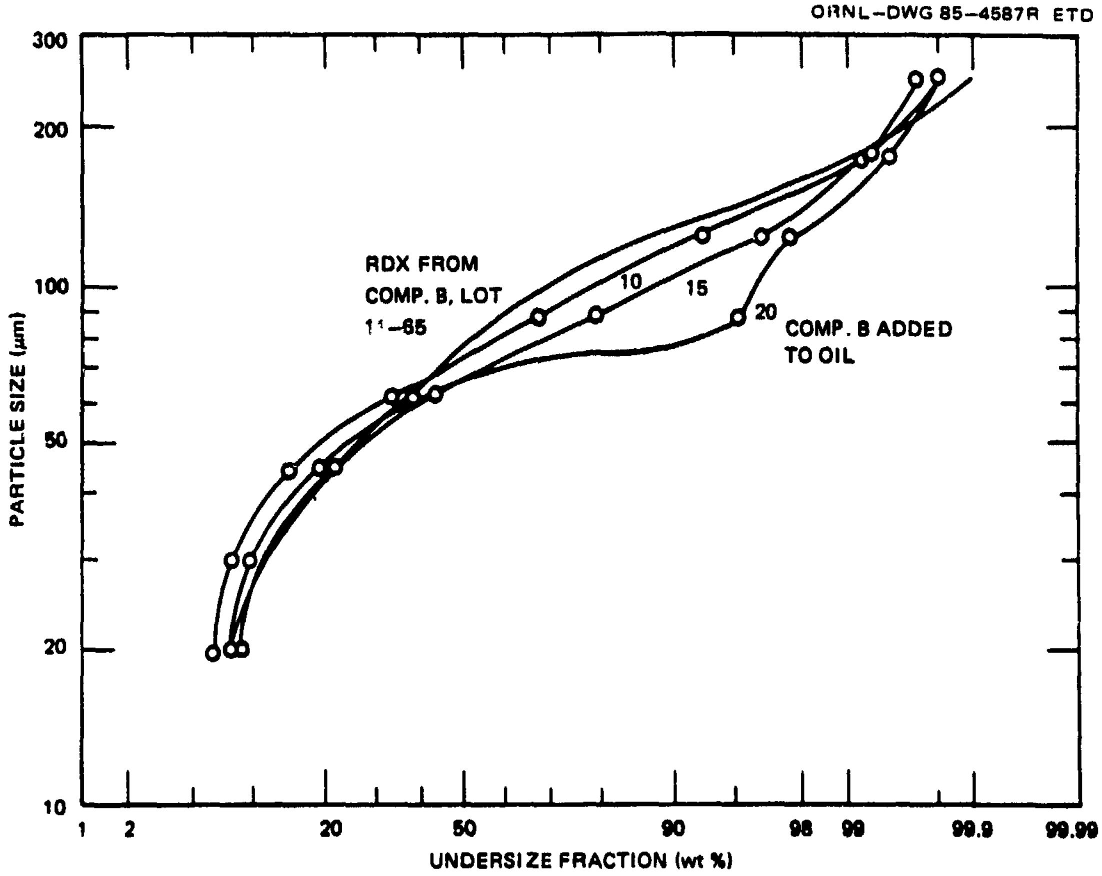

Fig. A.6. Particle-size distribution of RDX (Comp. B, Lot 11-65) generated by evaporation of toluene in the presence of No. 5 fuel oil at ~63°C (145°F).

VISCOSITY

To obtain atomization in an oil burner, it has been determined that the viscosity of the oil should not exceed a range of 20 to 30 centistokes at the burner tip.[4,5] To determine the effects upon viscosity of the TNT solute, the filtrates from the various TNT/solvent/fuel oil tests were used to measure the effects of the TNT concentration on the fuel oil viscosity. At a temperature of ~38°C (100°F), the Nos. 2 and 5 fuel oils indicated viscosities of 3.7 and 167 centistokes, respectively. At a temperature of ~60°C (140°F), the No. 5 oil viscosity was 37 centistokes.

The viscosities of TNT and No. 2 fuel oil mixtures of 10, 15, and 20 g of TNT per 100 mL of No. 2 oil measured at a temperature of ~38°C

(100°F) were 4.2, 4.4, and 4.7 centistokes, respectively. The viscosities of TNT and No. 5 fuel oil mixtures of 10, 20, and 30 g of TNT and 100 mL of No. 5 fuel oil measured at a temperature of ~60°C (140°F) were 56, 75, and 106 centistokes, respectively. The filtrates of Comp. B slurries stripped of RDX gave viscosities proportional to the TNT concentration that would have resulted from the completed dissolution of the TNT from the Comp. B.

CONCLUSIONS

- The production of a fuel oil and energetic slurry composed of No. 2 or No. 5 fuel oil and of RDX or RDX from Comp. B has been demonstrated for an acetone solvent and an acetone and water solvent in a 90 : 10 volume ratio at RDX concentrations in the upper range of 90 to 95% of saturation to have particle sizes small enough to be used in industrial combustors using standard oil burner technology.
- The solubility and small particle size of TNT in the Nos. 2 and 5 fuels oils will require that the oils be maintained at a temperature of at least ~57°C (135°F) or in an agitated state to prevent recrystallization of the TNT into large particle sizes.
- The solubility and small particle size of TNT in the Nos. 2 and 5 fuel oils affords greater ease in the handling of the mixture.
- The increase in viscosity of the fuel oils resulting from the dissolved TNT, which could result in atomization difficulty for the more viscous oils, can be reduced by the addition of small amounts of the solvent that is used for the explosive dissolution. The solvent addition could also have the added advantage of lowering the recrystallization temperature of the TNT.

REFERENCES

1. D. E. Rolison, R. L. Dickenson, and R. Scola, *An Evaluation of an Incinerator for Waste Propellants and Explosives*, Technical Report 4984, Radford Army Ammunition Plant, Hercules Inc., Radford, Va., December 1976.

2. Comp. JCAP Demilitarization and Disposal Task Group, *Joint Conventional Ammunition Program Coordination Group, Volume I, Demilitarization/Disposal Inventory*, Rock Island Arsenal, Rock Island, Ill., June 30, 1983.

3. J. D. Pinkerton, R. F. Tobias, and R. Scola, *Energy Recovery from Army Ammunition Plant Solid Waste by Pyrolysis*, ARLCD-CR-78030, TRW Defense and Space Systems, Redondo Beach, Calif., March 1979.

4. J. H. Perry, ed., *Chemical Engineers' Handbook*, 3d ed., McGraw-Hill, New York, 1950.

5. R. T. Haslam and R. P. Russell, *Fuels and Their Combustion*, McGraw-Hill, New York, 1926.

APPENDIX B

PLUGGING CHARACTERISTICS OF NOZZLES WITH SLURRIES

INTRODUCTION

Burning of energetic materials such as TNT and Comp. B in an oil-fired industrial combustor presents several difficult engineering problems. Among these are the interrelated problems of the feed preparation and the burner design. Efficient burning of the fuel oil is accomplished by atomization before combustion. The primary functions of the burner are to atomize the oil and to prepare it for mixing with the combustion air. Because of the general solubility limitations of the energetic materials in fuel oil, they must be burned as a slurry with the fuel oil.

A simplified schematic of an energetic-fuel oil combustor is shown in Fig. B.1. The primary area of concern is the size of the nozzle required to produce atomization of the energetic-fuel oil slurry and the maximum size of the energetic particles that can be handled without plugging of the nozzle. The purpose of these tests is to determine the effects of particle size and particle-size distribution on the plugging characteristics of burner nozzles. The effects of particle size and

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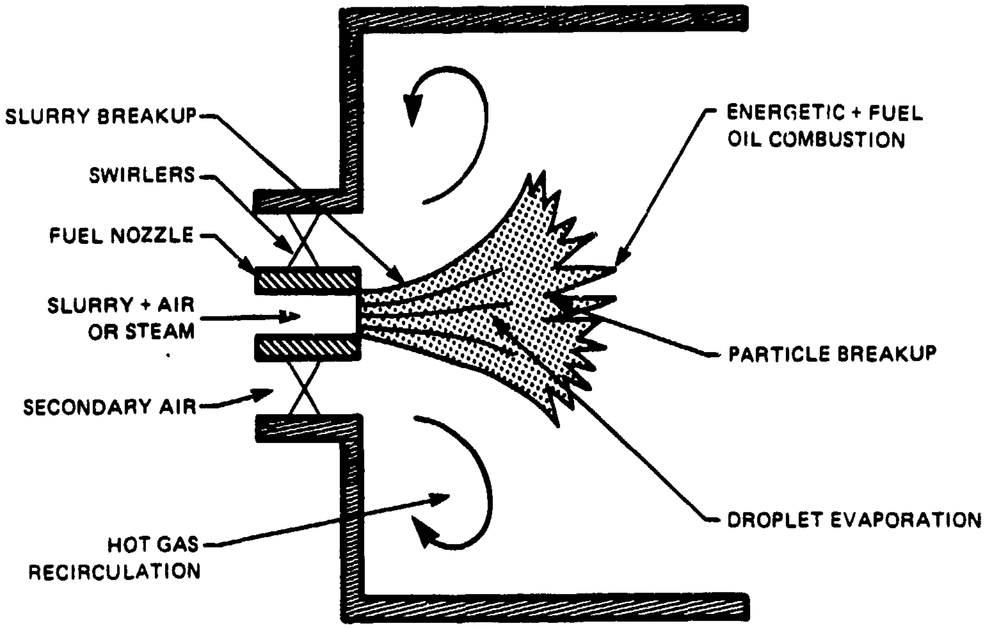

Fig. B.1. Schematic of an energetic-fuel oil combustor.

particle-size distribution on fuel atomization will be determined in a proof-of-principle burn test to be conducted at the Los Alamos National Laboratory.

SYSTEM DESCRIPTION

Shown in Fig. B.2 is a schematic of the test apparatus used to determine the plugging characteristics of burner nozzles operating under simulated combustor feed conditions. The system consisted of a slurry supply tank fitted with a variable speed agitator, an air-operated double-diaphragm slurry pump, an expansion tank, a burner nozzle holder, and a spray chamber.

Nozzle plates with hole sizes ranging from ~406 to 1067 µm (0.016 to 0.042 in.) were mounted in the nozzle holder. The number of holes in each nozzle size was varied so as to pass a flow rate of ~4.4 L/min

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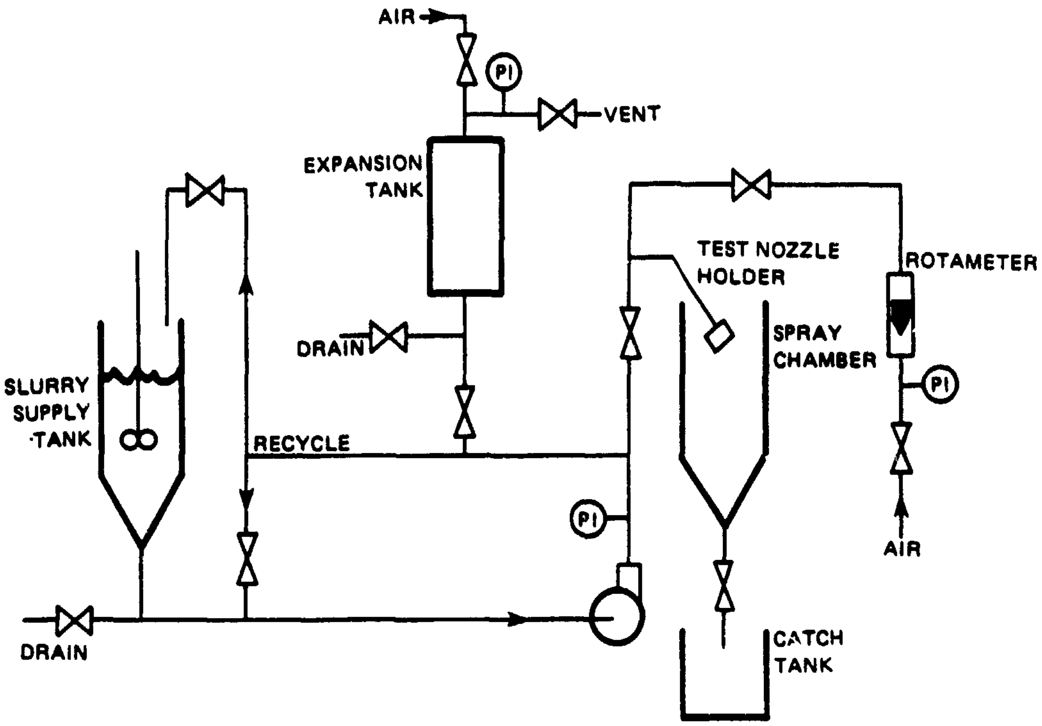

Fig. B.2. Schematic of nozzle-plugging test system.

(~1 gal/min) with a pressure drop of ~0.138 MPa (20 psi) (Table B.1). These values were chosen to simulate a combustor with a heat load of ~9.5 × 10^9 J/h (9 × 10^6 Btu/h).

Table B.1. Nozzle plate dimensional data

Plate number	Number of holes	Hole diameter [μm (in.)]
1	45	406.4 (0.016)
2	25	508 (0.020)
3	16	635 (0.025)
4	12	736.6 (0.029)
5	6	1066.8 (0.042)

The test to determine the plugging characteristics of the burner nozzles consisted of two main parts. One concerned the plugging characteristics of a two-phase mixture of solid particles and a liquid transport fluid. The second part considered the use of an atomizing gas (air) to produce a three-phase mixture of solid particles and a transport fluid consisting of a liquid and a noncondensible gas. The flow (nozzle-loss coefficient) and plugging characteristics were determined for each of these conditions using closely sized particles and particles with a wide range of sizes.

TEST APPARATUS AND PROCEDURES

Shown in Fig. B.3 and B.4 is the test apparatus used to determine the nozzle-plugging characteristics. Figure B.3 depicts the complete assembly, and Fig. B.4 presents it with the spray chamber removed to show the position of the nozzle holder. Shown in Fig. B.5 are the five nozzle plates used to evaluate the effects of hole diameter and particle-size distribution on the plugging characteristics. The head and flow characteristics of the air-driven, double-diaphragm, slurry feed pump are given in Fig. B.6 as a function of the air supply pressure.

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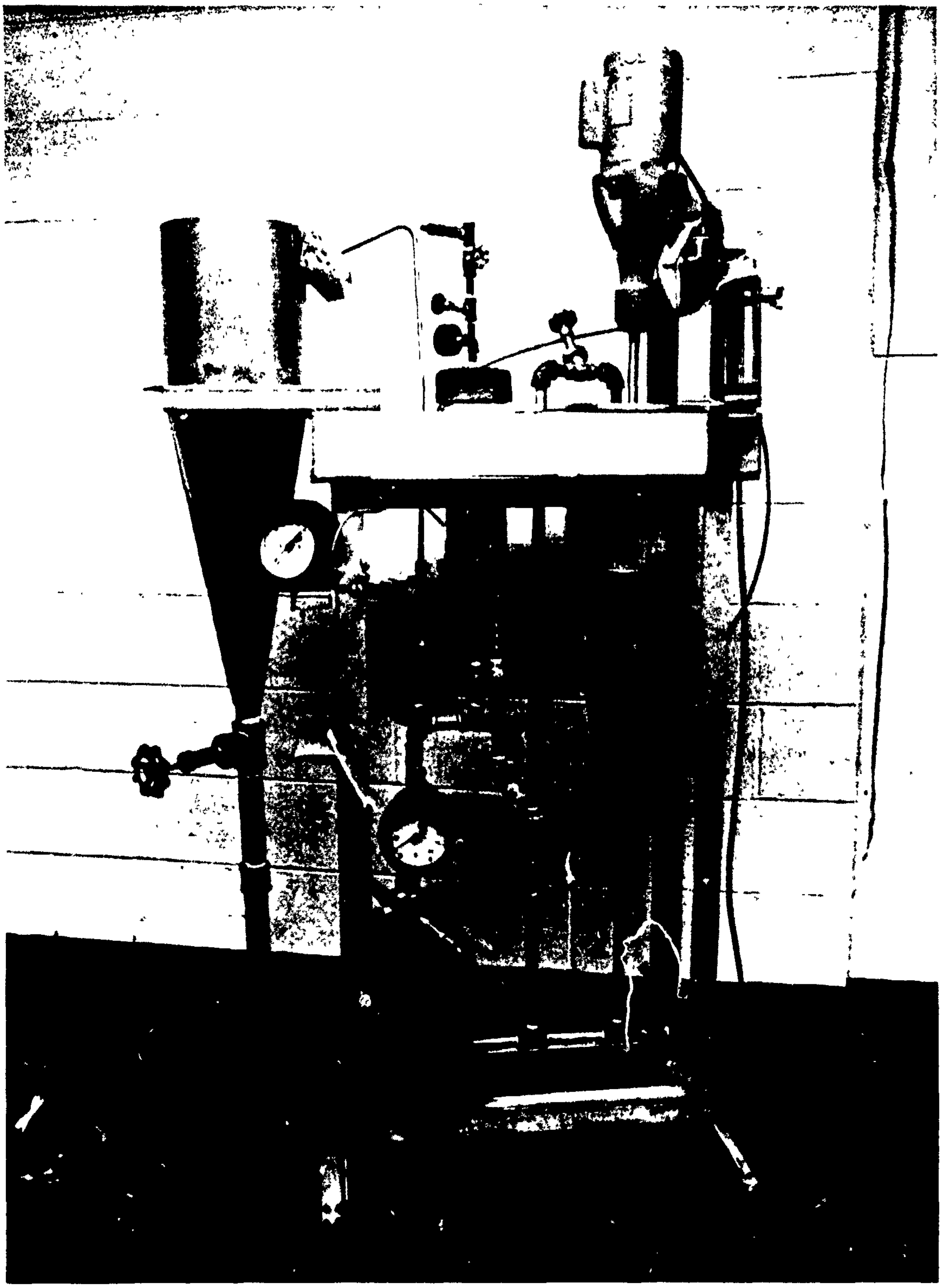

Fig. B.3. Test assembly for determining nozzle-plugging characteristics.

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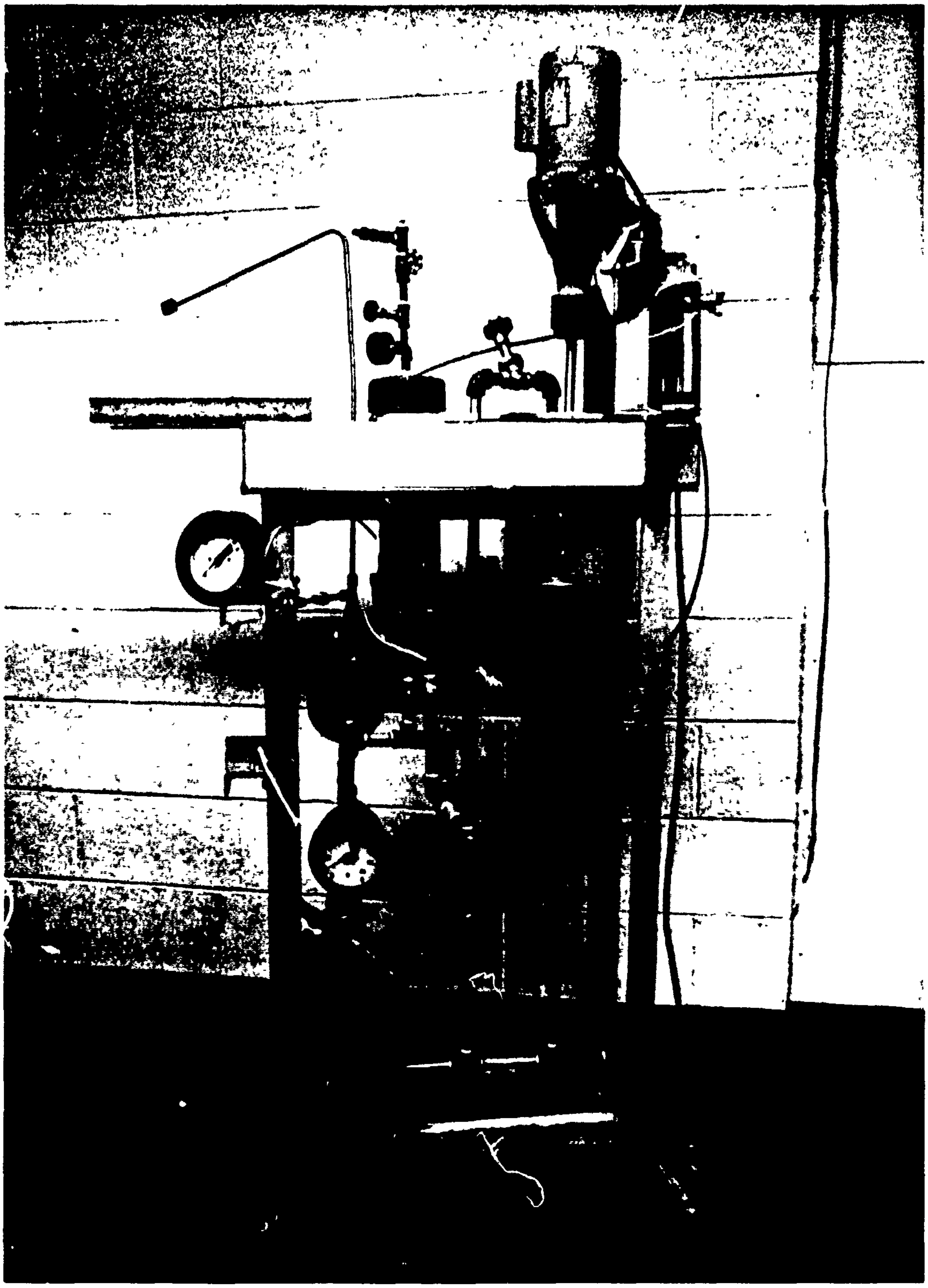

Fig. B.4. Test assembly with spray chamber removed.

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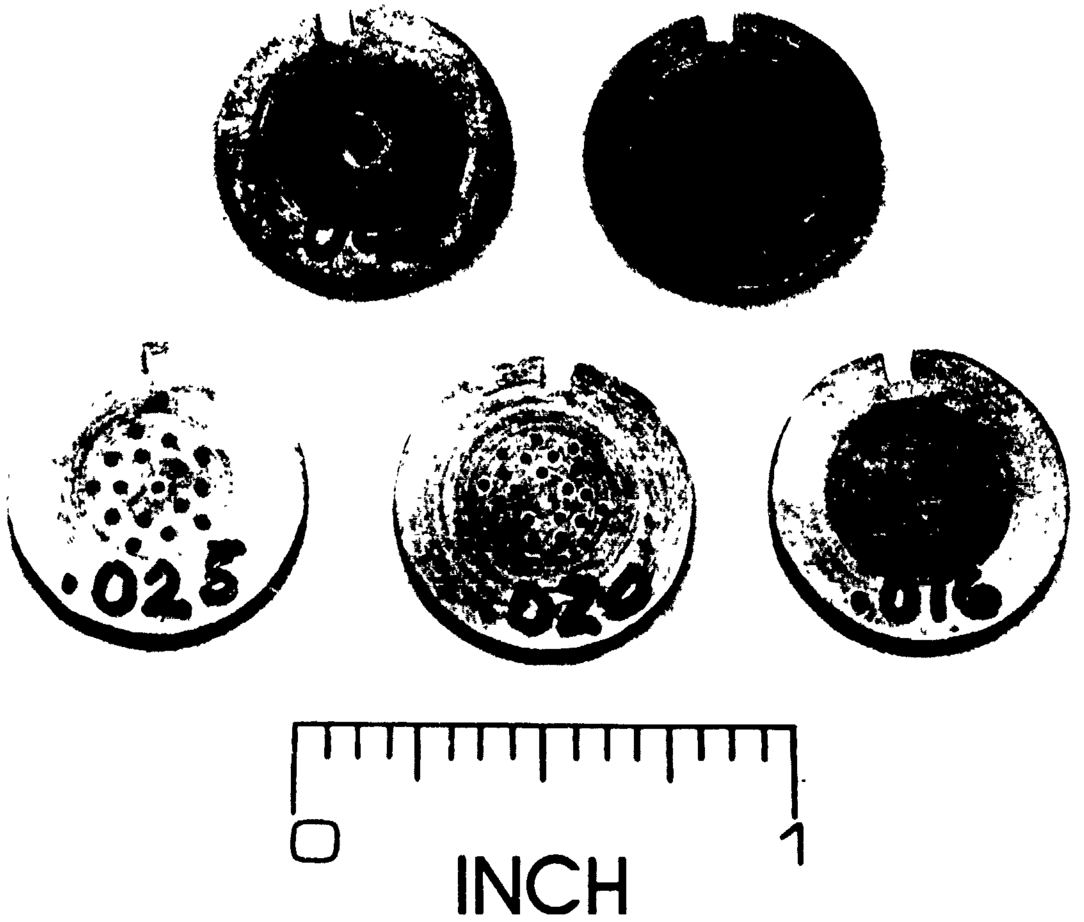

Fig. B.5. Nozzle plates with hole sizes from ~406 to ~1067 μm (0.016 to 0.042 in.).

The general experimental procedure consist of the following steps:

1. A test nozzle plate was placed in the nozzle holder.

2. The expansion tank was isolated and pressurized to ~0.239 MPa (20 psig).

3. The loop was filled with water.

4. Flow was started in the loop by applying an air pressure of ~0.342 MPa (35 psig) to the slurry pump.

5. The nozzle flow was adjusted to a value of ~4.4 L/min (~1 gal/min) and the loop by-pass was adjusted to give a nozzle pressure drop of ~0.138 MPa (20 psi).

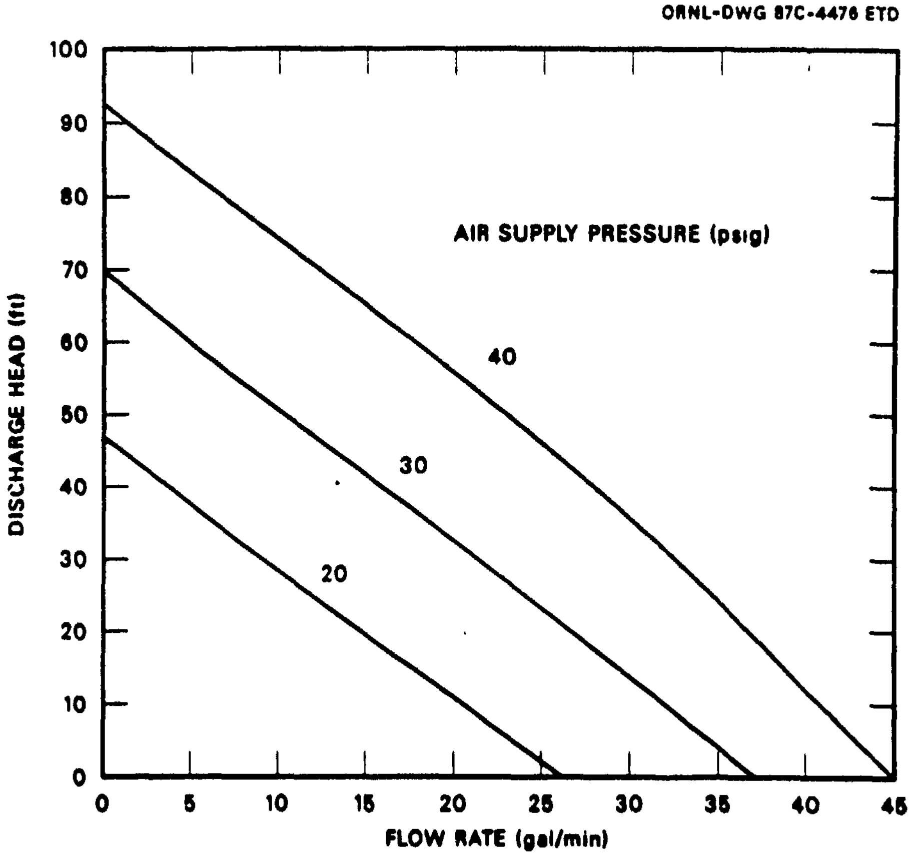

Fig. B.6. Performance characteristics of the double-diaphragm slurry-feed pump.

6. After the flow and pressure drop were established, the expansion tank was valved into the loop. The expansion tank reduces the loop pressure fluctuations from ~28 to 7 kPa (~4 to 1 psi).

7. The nozzle flow coefficient was determined by weighing the nozzle effluent and measuring the nozzle pressure drop. The coefficient was determined for mixtures of air and water for air volume fractions of 0, 0.09, 0.18, and 0.27 based on the nozzle inlet pressure.

8. After the nozzle coefficients were established for water and air mixtures, sized coal was added to the loop and the coefficients were redetermined for the air volume fractions listed in step 7.

9. A change of ~10% in the flow coefficient (a change of ~5% in flow) was taken as an indication of nozzle plugging. At this point the nozzle plate was removed and visually inspected for indication of plugging.

TEST RESULTS AND ANALYSIS

The nozzle-loss coefficient for each test was defined as the nozzle head loss according to the following equation.

$$K = 2g\rho\Delta PG^{-2} ,$$

where

K = nozzle loss coefficient,
g = gravitational constant (9.8 m/s^2),
ρ = slurry density (kg/m^3),
ΔP = nozzle pressure drop (kg/m^2)
G = nozzle mass flow rate ($kg/s\text{-}m^2$).

NOZZLE CALIBRATION

The nozzle-loss coefficients for the five nozzle plates determined with water and air mixtures are given in Table B.2.

Table B.2. Nozzle-loss coefficients

Nozzle plate number	Air volume fraction (%)			
	0	9	18	27
1	1.31	1.32	1.30	1.28
2	1.34	1.30	1.25	1.30
3	1.34	1.34	1.31	1.24
4	1.37	1.32	1.29	1.37
5	1.27	1.27	1.25	1.24

PLUGGING NARROW-RANGE PARTICLE SIZE

Coal particles with a narrow range of particle sizes were used to estimate the plugging characteristics. The particle size ranges investigated (as determined by Tyler screen sizes in microns) included

0/45, 45/75, 75/106, 106/150, 150/180, 180/212, 212/250, 250/300, and 300/425. A solids and water mixture containing one size range was tested for each nozzle and for each size range. A solids concentration of 1.5 wt % was used both with and without air addition. For each condition, tests were conducted for each particle size range beginning with the smallest size and increasing the size until plugging occurred.

The particle size ranges that resulted in plugging for each nozzle plate are given in Table B.3. The plugging characteristics were not affected by air addition up to a volume fraction of 27%.

Table B.3. Particle-size range required for nozzle plugging at a concentration of 1.5 wt %

Nozzle hole size [μm (in.)]	Particle size (μm)			Ratio of hole size to mean particle size
	Minimum	Maximum	Mean	
~406 (0.016)	106	150	128	3.18
~508 (0.020)	150	180	165	3.08
~635 (0.025)	180	212	196	3.24
~737 (0.029)	212	250	231	3.19
~1067 (0.042)	300	425	362.5	2.94

Shown in Fig. B.7 is a 20× photomicrograph of plugging resulting from 250/300 μm particles and an ~737-μm (0.029-in.) nozzle hole. Figure B.8 shows a section of Fig. B.7 at 100×. In this test the ratio of the hole diameter to the mean particle diameter was 2.68. As can be seen from the photomicrographs, the plug resulted from upstream particle bridging. The plug was easily dislodged by backwashing.

PLUGGING WIDE RANGE PARTICLE SIZES

Using the typical particle-size distribution of RDX extracted from Comp. B as a standard (see Fig. B.9) and plugging data given in Table B.3, the effect of the variation of the particle-size distribution on the nozzle-plugging characteristics was determined for nozzle plates 4 and 5 with hole sizes of ~737 and 1067 μm (0.029 and 0.042 in.), respectively. The RDX simulant was prepared by mixing various sizes of wet-screened

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Fig. B.7. Nozzle plate No. 4 [~737-μm (0.029-in.) hole size] plugged with 250- by 300-μm particles.

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Fig. B.8. Nozzle plate No. 4 [~737-µm (0.029-in.) hole size] plugged with 250- by 300-µm particles.

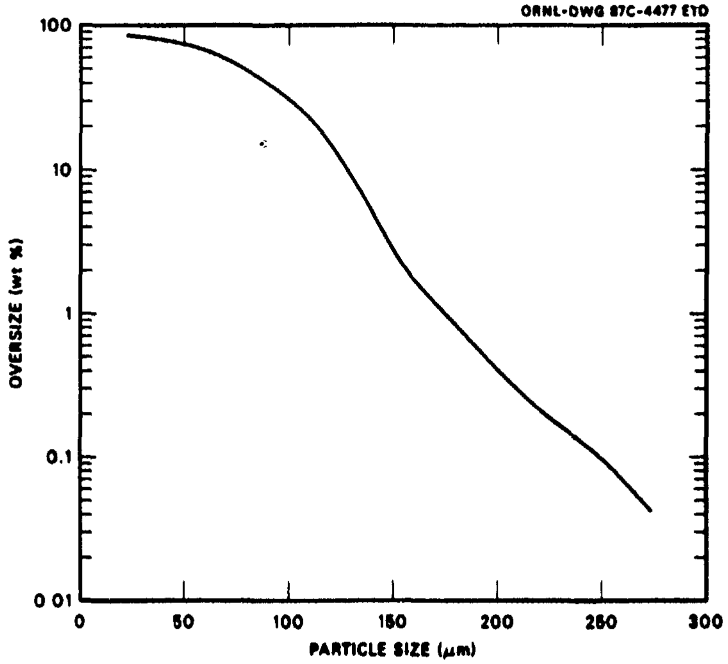

Fig. B.9. Particle-size distribution of RDX generated from Comp. B (Lot 11-65) by evaporation of toluene in the presence of No. 5 fuel oil.

coal particles to give the distribution indicated in Fig. B.9 for RDX extracted from Comp. B, Lot 11-65. The mix sizes and weight fractions are given in Table B.4.

Table B.4. Particle size and weight fraction of RDX simulant

Particle size (μm)		Weight fraction (%)
Minimum	Maximum	
0	45	21
45	75	26
75	106	23
106	150	27
150	180	2.2
180	212	0.5
212	250	0.2
250	300	0.07
300	425	0.03

Based on the plugging data for narrow-range particle sizes given in Table B.3, a top size of 150 μm was chosen as the starting point for the testing of nozzles plates Nos. 4 and 5 with hole sizes of ~737 and 1067 μm (0.029 and 0.042 in.), respectively. The cut point of 150 μm represents 97 wt % of the RDX simulant as given in Table B.4. Three total solids concentrations of 10, 20, and 30 g of solids per 100 g of water were evaluated for each nozzle plate to determine the effects of concentration on the plugging characteristics.

Each test was conducted in a similar manner by starting with the cut point particle distribution and adding the next larger particle size fraction (see Table B.4) until plugging occurred. For each nozzle plate tested, the plugging characteristics were independent of solids concentration over the range of 10 to 30 g of solids per 100 g of water and for air volume fractions of zero and 0.27.

Nozzle plate No. 4 [hole size ~737 μm (0.029 in.)] operated satisfactorily with the addition of the weight fraction for 150- to 180-μm particles and plugged with the addition of the fraction for 180- to 212-μm particles. Nozzle plate No. 5 [hole size ~1067 μm (0.042 in.)] operated satisfactorily with the addition of the fraction for 250- to 300-μm particles. Dimensional and plugging characteristics for nozzle plates Nos. 4 and 5 operating with mixed particle sizes are given in Table B.5.

Table B.5. Plugging characteristics of ~737- and ~1067-μm (0.029- and 0.042-in.) nozzle plates with mixed particle sizes

	Plate number	
	4	5
Number of holes	12	6
Hole diameter, μm (in.)	~737 (0.029)	~1067 (0.042)
Ratio of hole size to mean top size of particle		
Plugging	3.76	3.88
No Plugging	4.46	4.62

CONCLUSIONS

Based on the particle-size distribution of RDX (see Fig. B.9) and a 4.5 : 1 ratio, a nozzle hole diameter of ~1626 mm (0.064 in.) is required to prevent plugging with concentrations in the range of 10 to 30 g of solids per 100 g of water.

The nozzle pressure loss for multiphase flow can be determined from the nozzle loss coefficient and the mean density for compositions in the range of 0 to 30 g of solids per 100 g of water with an air volume fraction range of 0 to 0.27.

Closely sized particles in relatively dilute concentrations plug nozzles at a ratio of nozzle hole diameter to particle size that is approximately 3 : 1.

Mixed-size particles in a concentration range of 10 to 30 g of solids per 100 g of water plug nozzles at a ratio of nozzle hole diameter to top particle size that is approximately 4 : 1.

Mixed-size particles in a concentration range of 10 to 30 g of solids per 100 g of water indicated no nozzle plugging at a ratio of nozzle hole diameter to top particle size ratio that was approximately 4.5 : 1.

APPENDIX C

COMPATIBILITY OF TNT AND COMPOSITION B FUEL OIL MIXTURES

INTRODUCTION

A series of explosive compositions and commercial fuel oils was prepared by dissolution of the explosive in a solvent, mixing the solution with a commercial fuel oil, and subsequently evaporating the solvent from the mixture. Three explosives (TNT, RDX, and Comp. B) and three fuel oils (Nos. 2, 5, and 6) were used for the determination of the compatibility of energetics and fuel oils.

The samples were analyzed for compatibility by Differential Thermal Analysis (DTA), Vacuum Thermal Stability (VTS), and Accelerating Rate Calorimetry (ARC) tests. Results were compared with those of the untreated compositions to determine compatibility. In addition to the test for chemical compatibility, a long-term plating test was run to determine the effects of operating condition on the plating of TNT on container surfaces.

PROCEDURES

DIFFERENTIAL THERMAL ANALYSIS

Energy changes that accompany phase changes were measured by DTA (Ref. 1). The temperature differential between a sample and an inert reference material as a function of temperature was recorded. Samples ~10 mg in size were run on the DTA analyzer through a temperature range of 0 to 500°C at a rate of 11°C/min. For comparison, the DTA test was run on the fuel oil, the explosive, and the mixture of fuel oil and explosive. The DTA tests were run according to ASTM E-537-76.

VACUUM THERMAL STABILITY

The volume of gas evolved from a sample of material under vacuum at constant temperature over a specific time interval was measured by the VTS test. A 200-mg sample was placed in a gas heating tube and evacuated, and the evacuated tube was then inserted in a constant temperature bath. The gas that evolved after 40 h was measured and used to indicate the chemical stability of the explosive. For mixtures, a VTS test was run on each component, and the sum of gases that evolved was compared with the VTS test results for the mixture. The VTS tests were run according to the procedure given by the *Military Explosives Technical Manual 9-1300-214* (Ref. 2).

ACCELERATING RATE CALORIMETRY

An ARC test was used in a programmed mode to determine the stability of energetic materials as a function of heating rate and past time-temperature history. In operation a sample was hermetically sealed in a 1-in. spherical test cell suspended in the center of the ARC calorimetric cylinder. The calorimeter was heated to some starting temperature, allowed to dwell at this temperature, and search isothermally for a period of time to determine if self-heating of the sample were detected as determined by a 0.01°C/min temperature increase sustained for 20 min. If this temperature rise were not found, the program proceeded to the next temperature step, and the next wait-and-search period was repeated. When self-heating was detected, the isothermal calorimeter went adiabatic and the system reaction was allowed to run away. With explosives, the runaway is rapid. After the adiabatic excursion was completed, the system resumed the program mode and continued until another self-heating excursion was detected or the upper cutoff temperature was reached.

The ARC was calibrated in the normal manner, and then offset corrections were further "biased" to a slight negative drift so that time and changing external conditions could not cause a positive temperature drift. A further calibration was made with Comp. B and oil (50 : 50), which resulted in a typical explosive temperature of 216°C. The ARC tests were run according to the *ARC Operating Manual* (ROM SET 509, Part 851-9001) of Columbia Scientific Industries, Inc.[3]

SIX-MONTH PLATING TEST

The solubility of TNT in fuel oil [~8 wt % at ~57°C (135°F)] could, in time, result in the deposition of TNT on container walls, which would be difficult to remove. Static plating tests were performed with mixtures of fuel and TNT to determine if the TNT would plate on stainless steel container surfaces after a long (~6-month) storage period at constant temperature.

RESULTS AND DISCUSSION

DIFFERENTIAL THERMAL ANALYSES

DTA tests were run on the Nos. 2 and 5 fuel oils, TNT, RDX, and Comp. B separately. Mixtures of the two oils with the explosives were also run. Typical DTA curves for TNT, RDX, and Comp. B are shown in Figs. C.1 to C.3, respectively. No extra thermal excursions were noted

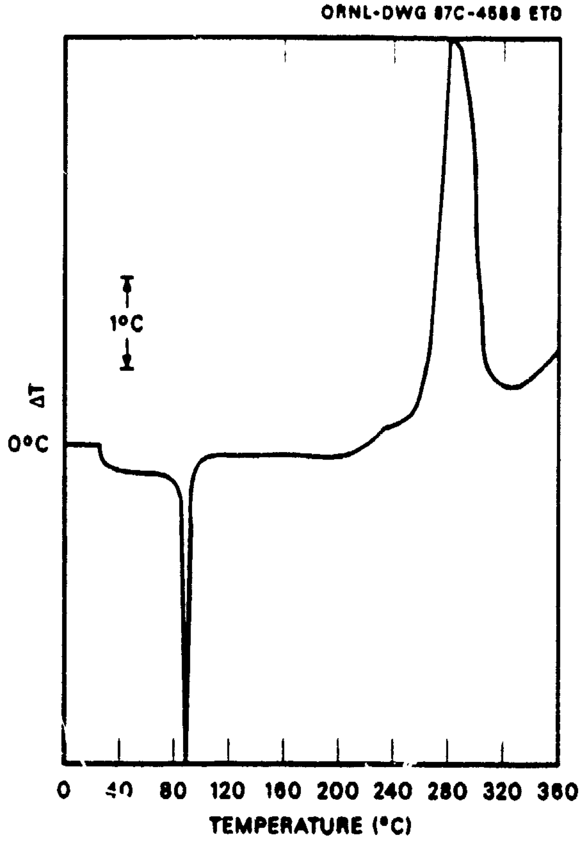

Fig. C.1. DTA curve of recrystallized TNT — melting point 81°C. *Source*: S. W. Dudiel and J. Baytos, *Differential Thermal Analysis Unit*, LAMS-2988, Los Alamos Natl. Lab., Los Alamos, N.M., December 1963, Fig. 18, p. 51.

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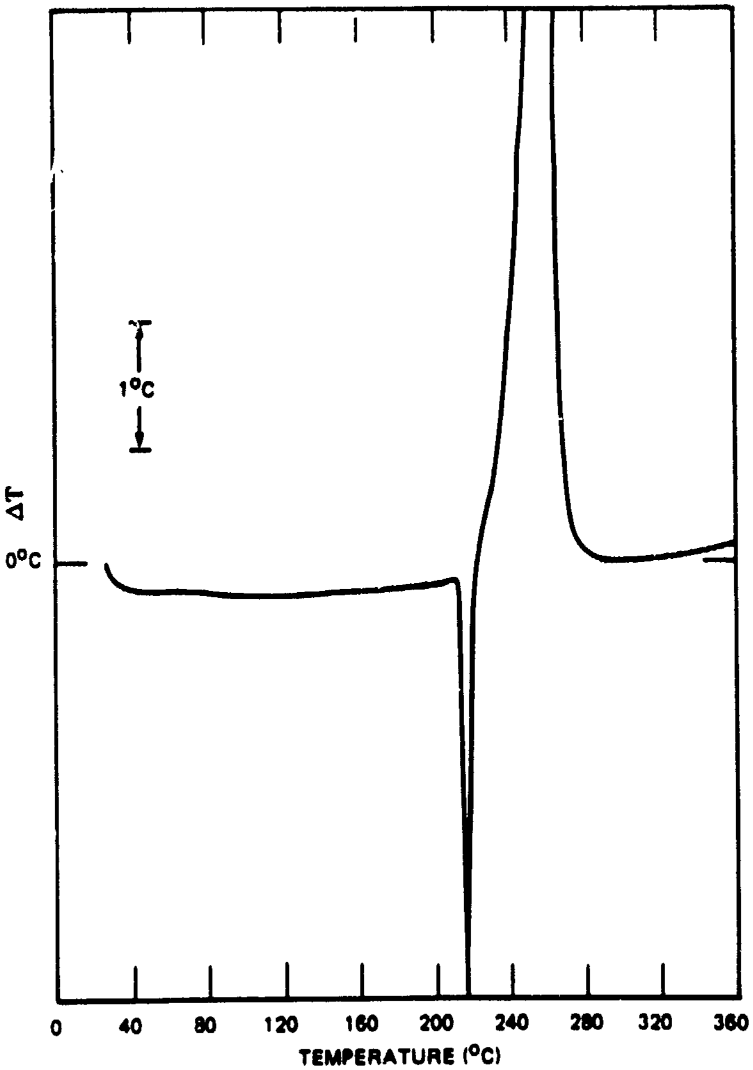

Fig. C.2. DTA curve of RDX (Holston Lot 3-68, 6-441, type A, class E, 99.8%) — melting point indicated at 211°C. *Source*: S. W. Dudiel and J. Baytos, *Differential Thermal Analysis Unit*, LAMS-2988, Los Alamos Natl. Lab., Los Alamos, N.M., December 1963, Fig. 20, p. 53.

on the mixtures when compared with the controls. The exotherms of the mixtures occurred at the temperatures where the explosives alone normally decompose.

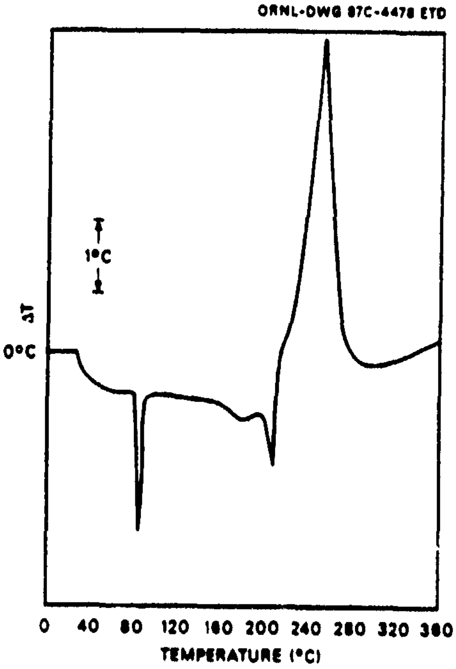

Fig. C.3. DTA curve of Comp. B-3 cast explosive. *Source*: S. W. Dudiel and J. Baytos, *Differential Thermal Analysis Unit*, LAMS-2988, Los Alamos Natl. Lab., Los Alamos, N.M., December 1963, Fig. 24, p. 57.

VACUUM THERMAL STABILITY

VTS tests were run on mixtures of Nos. 2 and 5 fuel oils and TNT, RDX, and Comp. B. For the No. 2 fuel oil, a test temperature of 120°C, was used; for the No. 5 fuel oil, test temperatures of 120, 100, 80, and 60°C were used. At a test temperature of 120°C, the gas evolution from the mixture of No. 2 fuel oil and the three explosives was comparable to the virgin energetics. The gas evolution from the No. 5 fuel oil and TNT mixture was also comparable to virgin TNT at the four test temperatures of 120, 100, 80, and 60°C. The gas evolution from the No. 5 fuel oil RDX and Comp. B mixtures was higher than the virgin energetics at the test temperatures of 120 and 100°C and below at the test temperature of 60°C.

For the test temperature range, the gas evolution for all the materials tested followed a first-order reaction as indicated by the straight line produced when the logarithm of the specific reaction rate was plotted against the reciprocal of the absolute temperature. The first-order reaction and the comparable gas evolution indicate chemical compatibility of the test components over the test temperature range.

ACCELERATING RATE CALORIMETRY

A sample of TNT, acetone, and No. 6 fuel oil was prepared in the ratio of 20 : 20 : 100 and stripped of acetone at 55°C to give a test sample of 20 g of TNT per 100 g of No. 6 fuel oil.

A 3-g sample of the mixture, which contained ~500 mg of the explosive (the upper explosive content limit for safe operation of the ARC) was used for the test. The sample was syringed into a tared lightweight Hastelloy cell and then reweighed. After the sample was hermetically sealed, the cell assembly was suspended in the center of the ARC. The test was started at 50°C and proceeded in the programmed mode to the 425°C cutoff temperature. Operational parameters are given in Table C.1,

Table C.1. Experimental parameters for ARC test 537 of No. 6 heavy fuel oil and TNT (100 : 20)

Parameters	Sample No. 6 oil/TNT
Cell material	Light weight Hastelloy
Cell weight (M_c), g	18.3917
System volume, cm^3	9
Sample weight, (M_s) (sp gr = 0.9 g/cm^3), g	3.235
Sample volume, cm^3	3.55
Head-space volume, cm^3	5.45
Head-space atmosphere	Air
Heat capacity of sample, Cv, cal/g°C	0.4
Heat-step start temperature, °C	50
Heat step, °C	5
Wait time, min	10
Preset end temperature, °C	425
Calorimeter rate threshold, °C/min	0.01
Thermal inertia,[a] ϕ	2.53

[a] WARNING: Calculation of ϕ is based on an assumed or estimated average sample heat capacity over the experimental temperature range [$\phi = 1 + (M_c \times Cp_{cell} + M_s \times Cv)$].

and instrument-derived data are shown in Table C.2. A plot of the real-time self-heating rate vs inverse temperature is shown in Fig. C.4.

The sample behaved like a solid in solution and did not give an explosive (off-scale) peak. The self-heating started at 150.4°C. The maximum heat rate was 0.117°C/min at 201°C for the first peak and 0.119°C/min at 226.4°C for the second peak. No rapid excursion took place in this test because the explosive was diluted to one in five parts in the oil, which attenuated the reaction. The self-heating terminated at 245.8°C, which gave an adiabatic temperature rise of 95.48°C. After the excursion exotherm was completed, the temperature was increased in the programmed mode to the 425°C cutoff temperature with no further reaction. The sample cell was allowed to cool to room temperature and was then removed from the calorimeter. The seal on the cell held all the pressure generated by the reaction. When the seal (0.25-mm-thick aluminum gasket) was removed, the contents blew out, indicating some pressure buildup. The residue was half of the original weight. The activation

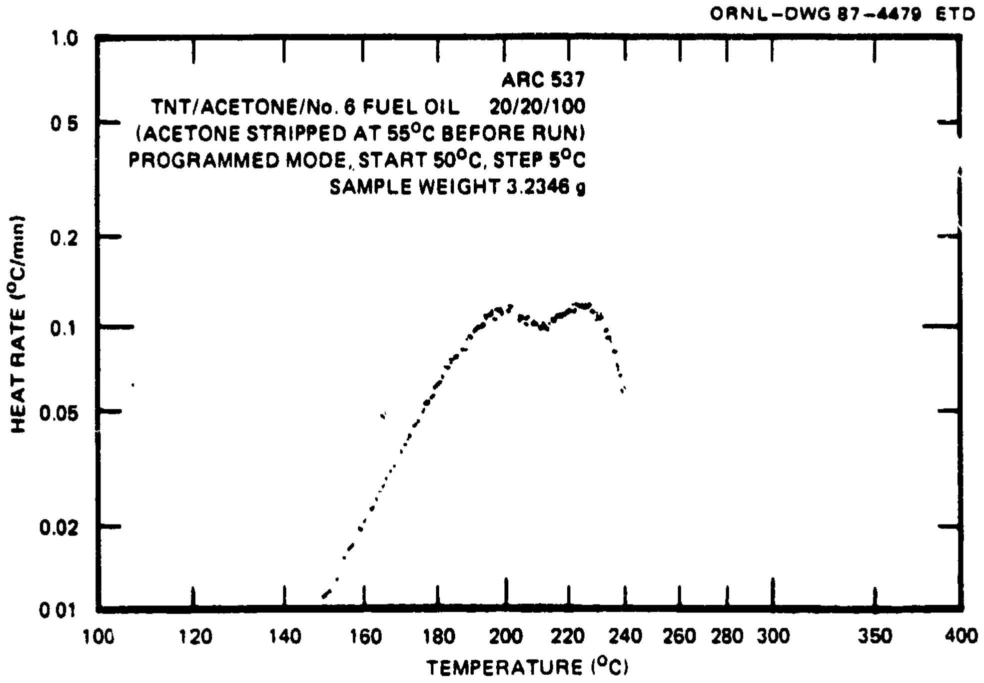

Fig. C.4. Self-heat rate vs temperature plot.

Table C.2. Experimental data generated by ARC test 537 of No. 6 heavy fuel oil/TNT (100 : 20)[a]

	Symbol	Peak I	Peak II	Peak Both
Observed initial reaction temperature, °C	T_o	150.41	211.25	150.41
Self-heat rate at T_o, °C/min	m_o	0.010	0.093	0.010
Maximum self-heat rate at T_m, °C/min	m_m	0.117	0.119	
Temperature at maximum self-heat rate, °C	T_m	201.33	226.44	226.44
Time to maximum self-heat rate, min (h)	$(\theta_m - \theta^\circ)$	1551 (~25.8)	144 (~2.4)	1790 (~29.8)
Time to maximum self-heat rate for the chemical system, min	(θ_m/ϕ)	613	57	707.5
Adiabatic temperature (final), °C	T_f	211.25	245.86	245.86
Adiabatic temperature rise $T_f - T_o$, °C	ΔTab	60.84	34.61	95.45
Adiabatic temperature rise for chemical system, °C	$(\phi \times \Delta Tab)$	153.9	87.6	241.5
Total heat of reaction $[-\phi(Cv)][\Delta Tab]$, cal/g	ΔH_{rx}			101.4
Activation energy, kcal/mol	$\Delta E_k{}^*$	26.4	26.5	

[a] All values apply the cell/sample system except where corrected for ϕ.

energy was 26.4 kcal/mol, which is similar to liquid reactions. The predicted heat rate curve vs inverse temperature calculated from the ARC data made one smooth arc on top of the experimental data. These data indicated no unusual material instabilities.

SIX-MONTH PLATING TEST

To simulate a range of operation conditions, 32 samples were tested in sets of four concentrations (0, 10, 20, and 30 g of TNT per 100 g of fuel oil) with three fuel oils (Nos. 2, 5, and 6). Twenty samples were run at ~57°C (135°F), eight at ~66°C (150°F), and four at ~79°C (175°F). The sample test conditions are given in Table C.3.

Table C.3. TNT—fuel oil plating test conditions

Fuel oil number	TNT concentration (g TNT/100 g oil)	Test temperature [~°C (°F)]
2	0, 10, 20, 30	57 (135)
5	0, 10, 20, 30	57, 66 (135, 150)
6	0, 10, 20, 30	57, 66, 79 (135, 150, 175)
5[a]	0, 10, 20, 30	57 (135)
6[a]	0, 10, 20, 30	57 (135)

[a] Sufficient acetone was added to the TNT and fuel oil mixture to give a viscosity of ~25 centistokes at ~57°C (135°F). The required acetone for the No. 5 oil and TNT mixtures was 4, 5, 9, and 13 g, and for the No. 6 oil and TNT mixtures was 1, 2, 5, and 9 g of acetone per 100 g of oil for the TNT concentrations of 0, 10, 20, and 30 g of TNT per 100 g of oil, respectively. The No. 2 oil requires no acetone addition.

The general results of these tests are discussed below as typical groups.

1. *Temperature of ~57°C (135°F) with Nos. 2, 5, and 6 oils.* The test mixtures poured easily both at ~57 (135°F) and room temperature. All test mixtures formed a thin, soft film on the container wall and a soft pasty sediment. For the TNT-containing tests, the sediment contained crystals of TNT. The film and sediment were easily removed with acetone at ~49°C (120°F).

2. *Temperature of ~66°C (150°F) and Nos. 5 and 6 oils.* The test mixtures poured both at ~66°C (150°F) and room temperature but were more viscous than the ~57°C (135°F) tests. All test mixtures formed a thin film on the container wall and a pasty sediment that contained TNT crystals for the TNT-containing tests. The film and sediment for these tests were harder than the corresponding ones for the ~57°C (135°F) test. The film and sediment were easily removed with acetone at ~49°C (120°F).

3. *Temperature of ~79°C (175°F) and No. 6 oil.* The test mixtures poured at temperatures of ~57 and ~79°C (135 and 175°F) but would not pour at room temperature. All test mixtures formed a thin, hard film on the container wall and a sediment that contained TNT crystals for the TNT-containing tests. Unlike the lower temperature test, the sediment formed from the TNT-containing test was hard packed instead of pasty. The film and packed sediment were easily removed with acetone at ~49°C (120°F).

4. *Temperature of ~57°C (135°F) and Nos. 5 and 6 oils (acetone addition to give a viscosity of 25 centistokes).* The test mixtures poured easily at ~57°C (135°F) and room temperature. All test mixtures formed a thin, soft film on the container wall and a soft pasty sediment. For tests including TNT, the sediment contained crystals of TNT. The film and sediment were easily removed with acetone at ~49°C (120°F). Listed below are several common characteristics exhibited by these tests.

a. Periodic examination of control samples indicated very little buildup on the inside surface. At the end of the tests the buildup was equally small for all samples.

b. Swipe tests of the film on the surface of the emptied containers gave a positive test for TNT for samples that contained TNT.

c. At the test temperature, all oil samples flowed.

d. After cooling to room temperature, all oil samples flowed [except No. 6 oil with a test temperature of ~70°C (175°F)].

e. All oil samples had a sediment at the conclusion of the tests.

f. All films and sediments were easily removed with acetone at a temperature of ~49°C (120°F).

The sediments were soft for the three oils at ~57°C (135°F) and increasingly harder for No. 5 and 6 oils at the higher temperatures of ~66 and ~79°C (150 and 175°F). The sediments from the tests of the No. 5 and 6 oils with acetone addition to give a viscosity of 25 centistokes were softer and more easily removed than those produced by the comparable acetone-free tests.

CONCLUSIONS

The DTA tests indicated that mixtures of Nos. 2 and 5 fuel oil with TNT, RDX, and Comp. B are compatible. The exotherms of the mixtures occurred at the temperatures where the energetics alone normally decompose.

The VTS tests indicated that the energetic-fuel oil mixtures follow a first-order reaction over the test temperature range and have gas evolution rates comparable to virgin material.

The ARC tests indicated that no unusual reaction occurred with energetic-fuel oil mixtures as compared with virgin materials.

The plating tests indicated that very little deposit builds up on the surfaces of a stainless steel container when exposed to oil and TNT mixtures for a period of time of at least 6 months at temperatures up to ~79°C (175°F). The results also indicated that any surface buildup is easily removed by flushing with warm acetone. The consistency of the sediments formed would indicate a preference for operation at the lower temperature of ~57°C (135°F) with acetone addition to give a viscosity of at least 25 centistokes.

REFERENCES

1. S. W. Dudiel and J. Baytos, *Differential Thermal Analysis Unit*, LAMS-2988, Los Alamos Natl. Lab., Los Alamos, N.M., December 1963.

2. *Military Explosives Technical Manual 9-1300-214*, U.S. Army Air Force, 1967.

3. Columbia Scientific Industries, Inc., *ARC Operating Manual, ROM SET* 509, Pt. 851—9001, Austin, Texas, 1979.

APPENDIX D

PROPAGATION OF DETONATION TESTING OF ENERGETIC-FUEL MIXTURES

INTRODUCTION

Burning of energetic materials such as TNT and Comp. B in an oil-fired combustor requires the handling of solvent and slurry mixtures of TNT and/or RDX (from Comp. B — 60% RDX, 40% TNT) and/or fuel oils. To cover a range of operating conditions, the propagation of detonation characteristics was investigated for three solvents (acetone, toluene, and an acetone and water mixture that contained 90 wt % acetone), three fuel oils (Nos. 2, 5, and 6), and two energetics (TNT and RDX) at the Los Alamos National Laboratory.*

Two modes of operating conditions were simulated. The first was a dynamic flow system condition in which the particulate energetics were in a homogeneous suspension; the second, a static condition to simulate a flow stoppage that would allow the energetic particles to settle.

PROCEDURES

The experimental arrangements used for the dynamic and static propagation of detonation testing in schedule 40 stainless steel pipes are shown in Figs. D.1 and D.2, respectively. The pipe size and booster diameter shown as 2 in. in Figs. D.1 and D.2 can be varied for the determination of the effects of diameter on the propagation.

The test samples were mixed thoroughly and carefully poured into the open end of an upright test pipe. A Teflon diaphragm secured to the bottom end provided a leakproof seal. After the pipe was filled, a second Teflon diaphragm was secured over the open end. After the pipe was loaded and sealed, it was taken to the firing pad, armed with the appropriate boosters and detonator, and fired. Variations in the procedure for the dynamic and static testing were as follow.

For the dynamic testing (see Fig. D.1) the energetic-fuel oil or energetic-solvent mixtures were allowed to cool with agitation to a temperature that approximates system operating conditions, that is,

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*Los Alamos National Laboratory, subcontract 41X-90448V, May 15, 1984.

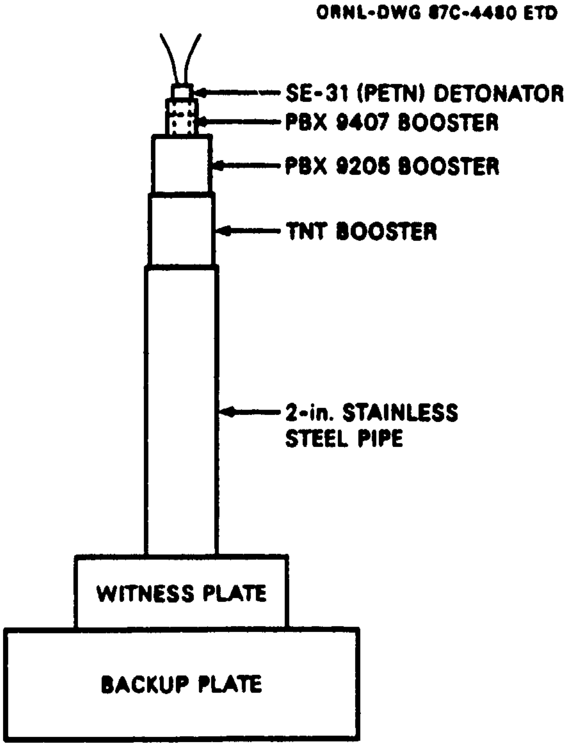

Fig. D.1. Setup for dynamic mode.

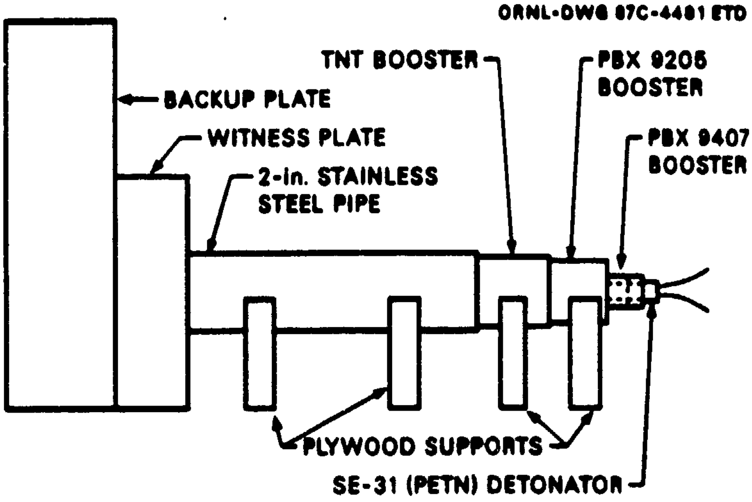

Fig. D.2. Setup for static mode.

~55°C for either No. 2 fuel oil or solvent-energetic mixtures, ~60°C for No. 5 fuel oil—energetic mixtures and a temperature of ~70°C for No. 6 fuel oil-energetic mixtures. (The higher temperatures for the No. 5 and 6 mixtures are required to give lower viscosities.) After filling and before the booster attachment, the test pipe was rotated end over end to ensure particulate suspension. After rotation, the booster and detonator are attached, armed, and fired. To ensure a minimum of settling of the energetic material, a maximum elasped time of 10 min was used between the filling and the firing.

For the static testing (see Fig. D.2) the test pipe was filled with the energetic mixture as described for the dynamic case. After filling, the test pipe was allowed to soak in a horizontal position for a minimum of 4 h for slurries and 8 h for solvent solutions. The soaking temperatures were 55°C for No. 2 fuel oil mixtures, 60°C for No. 5 fuel oil mixtures, 70°C for No. 6 fuel oil mixtures, and 20°C for solvent mixtures. These temperatures were chosen to allow settling from the energetic-oil mixtures and potential recrystallization from solvent solutions. After the soaking period, the boosters and detonator were attached, armed, and fired.

For each test condition, an energetic-free test was performed to establish a basis for comparison in the determination of propagation of detonation. The concentration (wt %) of energetic was varied in ~5% increments until propagation of detonation occurred as evidenced by the witness plate condition. Upper limits of concentration were determined for some cases in which very high concentrations of energetics did not produce propagation of detonation. A test is considered positive if the witness plate shows a definite indenture. The condition of the pipe varies, depending on the material tested. The most common posttest pipe conditions are total fragmentation, several long strips, and "banana-peeling" at the initiation end of the pipe.

RESULTS AND DISCUSSION

TNT AND ACETONE

The propagation of detonation characteristics for an acetone solution of TNT were determined in a 2-in. schedule 40 stainless steel pipe for both dynamic and static operational modes.

Six attempts in the static mode were unsuccessful in attaining propagation of detonation for 75 wt % TNT and 25 wt % acetone. Higher concentrations of TNT did give propagation of detonation in the static mode but not in the dynamic mode. All test data results are shown in Table D.1.

Table D.1. Test results for acetone[a]

wt %		Static	Dynamic
TNT	Acetone		
25	75	N	N
35	65	N	N
45	55	N	N
65	35	N	N
80	20	PROP	N
80	20	PROP	N
70	30	N	
70	30	N	
70	30	N	
70	30	N	
70	30	N	
70	30	N	
75	25	N	
75	25	N	
75	25	N	
75	25	N	
75	25	N	
75	25	N	

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

TNT AND TOLUENE

The propagation of detonation characteristics for a toluene solution of TNT were determined in a 2-in. schedule 40 stainless pipe for both dynamic and static operational modes.

Six attempts in the static mode were unsuccessful in attaining propagation of detonation for 65 wt % TNT and 35 wt % toluene. Higher concentrations of TNT did give propagation of detonation in the static mode but not in the dynamic mode. All test data results are shown in Table D.2.

Table D.2. Test results
for toluene[a]

wt %		Static	Dynamic
TNT	Toulene		
35	65	N	N
55	45	N	N
70	30	PROP	N
75	25	PROP	N
70	30	N	
70	30	N	
70	30	N	
73	27	N	
73	27	N	
73	27	N	
65	35	N	
65	35	N	
65	35	N	
65	35	N	
65	35	N	
65	35	N	

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

TNT IN ACETONE AND WATER (90 wt % ACETONE)

The propagation of detonation characteristics for TNT dissolved in a solution of acetone and water (90 wt % acetone) were determined in a 2-in. schedule 40 stainless steel pipe for both dynamic and static operational modes. Propagation of detonation was not attained in these tests and upper limits as indicated by the tests were established.

Eight attempts (six in the static mode and two in the dynamic mode) were unsuccessful in attaining propagation of detonation for 75 wt % TNT and 25 wt % acetone and water solution (90 wt % acetone). Higher concentrations of TNT were not attempted in either the static or dynamic operational mode. All test data results are shown in Table D.3.

Table D.3. Test results for acetone and water solution (90 wt % acetone)[a]

wt %		Static	Dynamic
TNT	Acetone and water		
35	65	N	N
55	45	N	N
65	35	N	N
75	25	N	N
75	25	N	N
75	25	N	
75	25	N	
75	25	N	
75	25	N	

[a] N = no propagation occurred, and a blank space indicates no test was conducted.

TNT AND ACETONE SATURATED WITH RDX

The propagation of detonation characteristics for TNT dissolved in acetone that was saturated with RDX at a temperature of 50°C was determined in 2-in. schedule 40 stainless steel pipe for both the dynamic and static operational modes.

Six attempts in the static mode were unsuccessful in attaining propagation of detonation for 70 wt % TNT and 30 wt % acetone saturated with RDX. Higher concentrations of TNT did give propagation of detonation in the static mode but not in the dynamic mode. All test data results are shown in Table D.4.

Table D.4. Test results for acetone saturated with RDX[a]

wt %		Static	Dynamic
TNT	Acetone/RDX		
40	60	N	N
60	40	N	N
77	23	N	N
77	23	N	N
77	23	N	
77	23	N	
77	23	N	
77	23	PROP	
70	30	N	
70	30	N	
70	30	N	
70	30	N	
70	30	N	
70	30	N	

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

In addition to the tests with acetone saturated with RDX, two concentrations of TNT (60 and 70 wt %) in a 90 wt % acetone and water solvent that was saturated with RDX at a temperature of 50°C were fired in both the dynamic and static operational modes. No propagation of detonation was observed in any of the four tests. The test results are given in Table D.5.

Table D.5. Test results for acetone and water solution (90 wt % acetone) saturated with RDX[a]

wt %		Static	Dynamic
TNT	Acetone/water/RDX		
60	40	N	N
70	30	N	N

[a] N = no propagation occurred.

TNT, ACETONE, AND NO. 2 FUEL OIL

The propagation of detonation characteristics of single-phase mixtures of TNT, acetone, and No. 2 fuel oil were determined in a 2-in. schedule 40 stainless steel pipe for the static operational mode. In addition, one two-phase test was performed on a single-phase mixture of TNT, acetone, and No. 2 fuel oil and solid TNT. Propagation of detonation was not attained in these tests, and upper limits as indicated by the tests were established.

Six attempts in the static mode were unsuccessful in attaining propagation of detonation for a mixture of 78 wt % TNT, 4.4 wt % No. 2 fuel oil, and 17.6 wt % acetone. No higher concentrations of TNT were attempted. One static mode test was conducted with a mixture of 77.3 wt % TNT, 5.9 wt % No. 2 fuel oil, and 16.8 wt % acetone prepared by adding TNT flakes to a single-phase mixture of 73 wt % TNT, 7 wt % No. 2 fuel oil, and 20 wt % acetone. Again, no propagation of detonation occurred. The test results are given in Table D.6.

Table D.6. Test results for single-phase mixtures of No. 2 fuel oil, acetone, and TNT[a]

wt %			Static	Dynamic
TNT	No. 2 fuel oil	Acetone		
39.3	29.5	31.2	N	
65.0	10.0	25.0	N	
73.0	7.0	20.0	N	
78.0	4.4	17.6	N	
78.0	4.4	17.6	N	
78.0	4.4	17.6	N	
78.0	4.4	17.6	N	
78.0	4.4	17.6	N	
78.0	4.4	17.6	N	
77.3[b]	5.9	16.8	N	

[a] N = no propagation occurred, and a blank space indicates no test was conducted.

[b] Prepared by adding TNT flakes to a single-phase mixture of 73 wt % TNT, 7.0 wt % No. 2 fuel oil, and 20 wt % acetone.

TNT AND NO. 6 FUEL OIL

The propagation of detonation characteristics were determined for TNT and No. 6 fuel oil mixtures in a 2-in. schedule 40 stainless steel pipe for both the dynamic and the static operational modes.

Six attempts in the static mode were unsuccessful in attaining propagation of detonation for 55 wt % TNT and 45 wt % No. 6 fuel oil. One of two attempts in the static mode did propagate flagrantly for 65 wt % TNT and 35 wt % No. 6 fuel oil (the other test did not propagate). Six attempts in the dynamic mode were unsuccessful in attaining propagation of detonation: three attempts with 55 wt % TNT and 45 wt % No. 6 fuel oil, two with 65 wt % TNT and 35 wt % No. 6 fuel oil, and one with 75 wt % TNT and 25 wt % No. 6 fuel oil. The test results are given in Table D.7.

Table D.7. Test results for No. 6 fuel oil and TNT[a]

wt %		Static	Dynamic
TNT	No. 6 fuel oil		
55.0[b]	45.0	N	
65.0	35.0	N	N
75.0	25.0	PROP	N
65.0	35.0	PROP	N
55.0	45.0	N	N
55.0	45.0	N	N
55.0	45.0	N	N
55.0	45.0	N	
55.0	45.0	N	
55.0	45.0	N	

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

[b] Mixing problems; the actual TNT concentration was probably lower than the 55 wt % indicated.

RDX AND NO. 2 FUEL OIL

The propagation of detonation characteristics were determined for RDX and No. 2 fuel oil mixtures in a 2-in. schedule 40 stainless steel pipe for both dynamic and static operational modes. In addition, the characteristics for the dynamic operation were established for a 1-in. schedule 40 stainless steel pipe.

No concentration was tested that did not result in propagation of detonation as evidenced by witness plate damage. The lowest concentration tested was 5.3 wt % RDX and 94.7 wt % No. 2 fuel oil. The RDX that settled in the pipe appeared to form a fuselike trail along the bottom of the pipe as evidenced by localized damage to the witness plate. When this trail of RDX was ignited, it propagated a detonation. These test data results are shown in Table D.8. To prevent the rapid settling of

Table D.8. Results from tests (in 2-in. pipe) for No. 2 fuel oil and RDX[a]

wt %		Static	Dynamic
RDX	No. 2 fuel oil		
46.7	53.3	PROP	
39.0	61.0	PROP	
23.6	76.4	PROP	
18.2	81.8	PROP	
12.6	87.4	PROP	
9.7	90.3	PROP	
5.3	94.7	PROP	

[a] PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

the RDX in the test pipes, a thickening agent (Cabosil) was added to the No. 2 fuel oil to allow both dynamic and static operational mode testing.

Several tests were conducted with both the dynamic and static operational modes. There appeared to be no difference between the two modes in the sensitivity to propagate a detonation. Six attempts in the dynamic mode were unsuccessful in attaining propagation of detonation for 22 wt % RDX, 71.7 wt % No. 2 fuel oil, and 6.3 wt % Cabosil in a 2-in. pipe. Higher concentrations of RDX did give propagation of detonation in both the dynamic and static operational modes. The test results are given in Table D.9.

Six tests conducted in a 1-in. schedule 40 stainless steel pipe for the dynamic operational mode did not give propagation of detonation for a mixture of 25 wt % RDX, 70 wt % No. 2 fuel oil, and 5 wt % Cabosil. Higher concentrations of RDX did give propagation of detonation. These data are given in Table D.10.

Table D-9. Results from tests (in 2-in. pipe) for No. 2 fuel oil, Cabosil, and RDX[a]

wt %			Static	Dynamic
RDX	No. 2 fuel oil	Cabosil		
44.9	51.0	4.1	PROP	
35.0	59.8	5.2	PROP	
29.0	65.3	5.7	PROP	
26.0	68.0	6.0	PROP	
15.0	78.2	6.8	N	
20.0	73.6	6.4		N
26.0	68.0	6.0		PROP
22.0	71.7	6.3		N
22.0	71.7	6.3		N
22.0	71.7	6.3		N
22.0	71.7	6.3		N
22.0	71.7	6.3		N
22.0	71.7	6.3		N

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

Table D.10. Results from tests (in 1-in. pipe) for No. 2 fuel oil, RDX, and Cabosil[a]

wt %			Static	Dynamic
RDX	No. 2 fuel oil	Cabosil		
26.0	68.0	6.0		N
26.0	68.0	6.0		N
45.0	51.0	4.0		PROP
45.0	51.0	4.0		PROP
35.0	60.0	5.0		PROP
30.0[b]	65.0	5.0		N
30.0	65.0	5.0		PROP
25.0	70.0	5.0		N
25.0	70.0	5.0		N
25.0	70.0	5.0		N
25.0	70.0	5.0		N
25.0	70.0	5.0		N
25.0	70.0	5.0		N

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

[b] Mixing problems: not enough Cabosil added for the 5% indicated.

RDX AND NO. 5 FUEL OIL

The propagation of detonation characteristics was determined for RDX and No. 5 fuel oil mixtures in a 2-in. schedule 40 stainless steel pipe for both the dynamic and static operational modes. The test results are given in Table D.11.

The RDX concentration limit was not determined for the static operational mode. The lowest concentration tested in the static operational mode was 5 wt % RDX and 95 wt % No. 5 fuel oil. Six attempts in the

Table D.11. Test results for No. 5 fuel oil and RDX[a]

wt %		Static[b]	Dynamic
RDX	No. 5 fuel oil		
30	70	PROP	
20	80	PROP	
10	90	PROP	
7.5	92.5	N	
7.5	92.5	PROP	N
7.5	92.5		N
7.5	92.5		N
7.5	92.5		N
7.5	92.5		N
7.5	92.5		N
5	95	PROP	
15	85		N
40	60		PROP
25	75		PROP
20	80		PROP
15	85		N
15	85		N
15	85		N
15	85		N
15	85		N

[a] N = no propagation occurred, PROP = propagation of detonation occurred, and a blank space indicates no test was conducted.

[b] Pipe fired in the static pipe position within 10 min of pipe loading.

dynamic mode were unsuccessful in attaining propagation of detonation for 15 wt % RDX and 85 wt % No. 5 fuel oil. Higher concentrations of RDX did give violent propagations.

CONCLUSIONS

TNT-acetone solutions will not produce propagation of detonations for either the dynamic or static operational modes at TNT concentrations of <75 wt %.

TNT-toluene solutions will not produce propagation of detonation for either the dynamic or static operational modes at TNT concentrations of <65 wt %.

TNT-acetone and water (90 wt % acetone) will not produce propagation of detonation for either the dynamic or static operational modes at TNT concentrations of <75 wt %.

TNT-acetone (saturated with RDX at 50°C) or TNT-acetone and water (90 wt % acetone) (saturated with RDX at 50°C) will not produce propagation of detonation for the dynamic or static operational modes at TNT concentrations of <70 wt %.

Single-phase mixtures of TNT, acetone, and No. 2 fuel oil will not produce propagation of detonation for the static operational mode for TNT concentrations of <78 wt %. The addition of solid TNT to a single-phase mixture of 73 wt % TNT, 7 wt % No. 2 fuel oil, and 20 wt % acetone to give a total TNT concentration of 77.3 wt % TNT did not result in a propagation of detonation for one static operational mode test.

TNT-No. 6 fuel oil mixtures will not produce propagation of detonation for either the dynamic or static operational modes at TNT concentrations of <55 wt %.

RDX—No. 2 fuel oil mixtures will produce propagation of detonation at concentrations as low as 5.3 wt % for the static operational conditions when allowed to settle freely. Hindered settling by the use of Cabosil indicated that RDX concentrations of <22 wt % for a 2-in. pipe and 25 wt % for a 1-in. pipe will not propagate detonations for the dynamic operational mode.

RDX—No. 5 fuel oil mixtures will produce propagation of detonations of concentrations as low as 5 wt % for static operational conditions. Concentrations of RDX of <15 wt % will not propagate detonations for the dynamic operational mode in a 2-in. pipe.

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