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SENSITIVITY
OF
SOME EXPLOSIVE/BRINE MIXTURES

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Threshold levels were determined for the dried explosive- and propellant-contaminated salts derived from the decontamination brine solution used for the demilitarization of chemical munitions at the Chemical Agent Munition Disposal System Directorate demilitarization facility. Impact, friction, and thermal sensitivity tests revealed that the thermal hazard was the controlling factor in determining the safe explosive concentrations. The test method used shows there is no thermal hazard if the total concentrations of the energetic materials are not greater than 10% by weight.

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INTRODUCTION

A Chemical Agent Munition Disposal System (CAMDS), prototype facility for the demilitarization of various chemical munitions has been built by the US Army at Tooele Army Depot, Utah. The plant is operated by the personnel from the CAMDS Directorate.

The munitions are processed in the explosive containment cubicle (ECC) which contains a demilitarization machine, fluid stream filters, and a 600 gallon recirculation tank. In the demilitarization machine, the munitions are drained of chemical agent and then submerged in a saw tank filled with 250 gallons of decontamination solution. The aqueous solution contains approximately 10% by weight sodium carbonate, 3% CIMCOOL (organic lubricant), and 0.2% diammonium phosphate (a scavenger for hydrogen).

The munitions are then cut into seven sections and sent to the deactivation furnace, where the explosives and any remaining agents are destroyed. After processing two rounds, 50 gallons of decontamination solution are drawn from the saw tank and filtered successively through 800 and 100 micron fluid stream filters. The filtered solution is recirculated via a recirculation tank. After 9 hours, approximately 130 munitions are processed and the sump and washdown liquids are added to the saw tank solution. The total quantity of saw tank and washdown solutions, approximately 600 gallons, is then processed through 25-micron fluid stream filters. The filter media and the sludge collected in them are burned in the deactivation furnace. After this polishing operation, the solution is sent to the explosive treatment system, where 18% sodium hydroxide is added to provide a minimum of 5% excess caustic. The decontamination solution is then transferred to the agent destruction system where it is gradually fed to the rotary drum dryers operated at 171°C (340°F). At equilibrium, approximately 25% of the solution fed to the dryers is evaporated and about 75% recirculated back to the holding tank. The dried salts are scraped from the drum dryers by knife edges with an applied force of ten pounds per linear inch and are then stored in corrugated drums.

A qualitative study was carried out by personnel from the Energetic Materials Division (EMD), LCWSL, ARRADCOM, Dover, NJ at Tooele Army Depot to determine if an explosive hazard exists from residual explosives during the drying operation (ref 1). Salt samples obtained by evaporation of decontamination solution taken during various stages of the demilitarization process were analyzed. Differential thermal analysis (DTA) and thin layer chromatography (TLC) revealed that only an insignificant amount of explosives was present in the residual salts after processing 131 munitions. It was concluded that an explosive hazard does not exist during the drying operation.

Although the above indicated that the demilitarization process was free of explosive hazard, it was deemed desirable to determine the maximum concentration of explosives in the dried brine salts that does not constitute a hazard. The following describes the results of such a study conducted by the EMD at ARRADCOM, Dover, NJ, at the request of the US Army Toxic and Hazardous Materials Agency and supported by Project C25-CAMDS.

EXPERIMENTAL

Sample Preparation

The decontamination solution contains seven possible explosives: RDX, TNT, Comp. B, nitrocellulose (NC), nitroglycerine (NG), M28 propellant, and a 4.4 to 1 ratio of M28 and Comp. B (table 1). Explosive- and propellant-brine mixtures were prepared by the suspension of the desired amount of explosive or propellant in the brine solution and subsequent evaporation at ambient temperature (22°C to 25°C) utilizing a flash evaporation apparatus. Pressures of about 0.5 to 1.0 mm were obtained with a vacuum pump which was protected by a dry ice-acetone trap. A McLeod gage and a one liter 3-necked flask were situated between the pump and the trap. After most of the water was evaporated, absolute ethanol was added in three portions (50 mL, 50 mL, and 30 mL) to the flask in order to eliminate almost all traces of water via azeotrope formation. The solids were subsequently removed from the flask, powdered, dried overnight over concentrated sulfuric acid, and analyzed for explosive content. The use of ethanol was omitted with M28 because of the solubility of nitroglycerine in ethanol. In that case, the powdered mixtures were additionally dried in a vacuum oven at 60°C/3 mm.

The nitroglycerine-brine mixtures were prepared by adding the desired amount of nitroglycerine dissolved in acetone, to dry sodium carbonate, mixing, and then evaporating the acetone.

Sensitivity Tests

Impact

Apparatus

The ERL type 12 impact machine was used to determine impact sensitivity of the dried brine mixtures. The apparatus, developed by the Explosives Research Laboratory at Bruceton, PA, consists of a free-falling 2.5 kg hardened steel weight, a test sample holder, and a supporting frame. The sample holder is a 3.18 cm (1.25 in.)

diameter hardened steel anvil mounted on a massive steel plate situated on a concrete foundation. The drop weight impacts on a 3.18 cm (1.25 in.) diameter hardened steel, stationary cylinder (called a drift or a striker), which rests on the test sample. The drift slides freely within a guide and transmits the energy to the test sample. The drop weight is raised pneumatically to the desired height. A detailed description of the apparatus is contained in reference 2.

Procedure

Impact sensitivity tests were conducted at 20°C with 55% relative humidity. A 2.54 cm (1 in.) square piece of fine, flint sandpaper (Norton Co., Troy, NY) was placed on the anvil and approximately 35 mg of the test powder was placed in a loose pile in the center of the sandpaper. The drift was then lowered gently onto the top of the test sample, the drop weight released from a preselected height, and the impact reaction determined. After each run, the sandpaper and test sample were discarded and the drift and anvil faces were cleaned.

The drop height corresponding to the 50% and 10% probability of initiation was used as a measure of impact sensitivity of the explosive- or propellant-brine samples. The maximum explosive or propellant concentration level which would not pose an explosive hazard from impact was also determined. The 50% initiation point was determined by means of the Bruceton up-and-down method (ref 3). The 10% value was the minimum height which resulted in initiation of the sample in at least one of ten trials, after failure of initiation in 10 trials from a height one increment lower. The lower limit of explosive hazard was arbitrarily considered as the explosive or propellant concentration which resulted in no explosive reaction in 20 trials, after reaction had occurred with a 5% higher concentration, using a 2.5 kg weight at 240 cm.

The amount of the 35 mg test sample consumed during a run varied from low levels, evidenced by the emission of a very small amount of smoke, sound or a slight burn mark, to complete burning or detonation. The criterion for initiation in this study was any evidence of burning or detonation observed during impact or in the post-test examination of the sample. The degree of the impact reaction was not determined although most of the reactions were low level.

Friction

Apparatus

The Picatinny Arsenal large-scale friction pendulum apparatus used in this study consists of a fixed steel anvil and a weighted pendulum with a steel shoe. The sample is placed on the anvil and subjected to a series of glancing blows by the shoe which is automatically released from a height of one meter. The upper face of the anvil, which is mounted on a massive concrete base, is a smooth, polished surface. Three parallel grooves are cut across the central portion to prevent the test sample from being brushed off the anvil by movement of the shoe. A detailed description of the apparatus is given in reference 2.

Procedure

Each explosive- or propellant-brine mixture, which was determined to be nonexplosive by the impact test, was tested for friction sensitivity. Before each trial, the pendulum was aligned, without a sample on the anvil, so that it would swing across the face of the anvil 18±1 times before coming to rest. After alignment, 7±0.1 grams of the test powder were spread in an even layer in and about the grooves on the anvil face. The pendulum was released and reactions recorded. A test consisted of ten trials. Unreacted powder was brushed from the anvil and shoe, and the latter was cleaned thoroughly with a suitable solvent after each trial.

Thermal

Each explosive- or propellant-brine mixture, which was determined to be nonexplosive by the impact test, was also tested for thermal reactivity. Thermal hazard was determined by means of an open tube explosion temperature test (ref 4) with minor modifications.

Approximately 40 mg of each sample were loosely loaded into an empty No. 8 blasting cap, tapped to settle the powder to the bottom, and immersed in a Wood's metal bath at 177°C (350°F) for 30 minutes or until reaction occurred.

RESULTS AND DISCUSSION

Impact initiation probability as a function of concentration was determined for the seven explosive- or propellant-brine mixtures listed in table 2. The study shows that the maximum dry explosive or propellant concentration by weight that can be present in the decontaminated salts, without posing an explosive impact hazard, is 30% RDX, 25% TNT, 20% Comp. B., 25% nitrocellulose, 30% nitroglycerine, 30% M28, and 15% of a 4.4 to 1 mixture of M28 and Comp. B. It should be noted that the M28-Comp. B mixture is more sensitive to impact than either material alone and is also the most sensitive of the mixtures tested.

Table 2 shows that mixtures of 60% RDX and 100% RDX were equally impact sensitive. In general, the data indicate that mixtures containing 50-60% of the energetic materials are almost as impact sensitive as the pure materials. This increased sensitivity may be due to the formation of Meisenheimer complexes (ref 5) in some of the compositions. Deep purple coloration developed in the TNT compositions, typical of this type complex, during the evaporation process. Very slight coloration was also noted in compositions containing RDX.

The Picatinny Arsenal large-scale friction pendulum test (steel shoe) was conducted on the explosive mixtures considered impact insensitive except for NG which was reduced to a concentration of 20% (table 3). None exhibited friction sensitivity in ten trials.

The determination of thermal hazard was also carried out on these impact insensitive mixtures. Of the seven mixtures only the 15% M28-Comp. B sample passed the thermal test, since smoking occurred in the other six within 2 minutes (table 4). This indicates that the six mixtures that failed would be susceptible to thermal reaction (burning) when heated to 177°C during the drying operation. These laboratory tests indicate that all thermal hazard is completely eliminated when the explosive or propellant content is reduced to 10% since, with such samples, reaction was not observed in 30 minutes. To ascertain the effect of longer heating periods on thermally stable compositions, the 15% M28-Comp. B-brine mixture was subjected to a DTA study using a Perkin-Elmer differential scanning calorimeter (DSC). A sealed 10 mg sample was heated from room temperature to 177°C at a heating rate of 10°C/min and maintained at that temperature for 20 hours. Thermal reactions were not observed in the thermogram and it appears that a thermal hazard does not exist at the drum dryer for this particular brine mixture.

CONCLUSIONS

1. The impact test method used in this study indicates that safe concentrations of explosives or propellants in the brine mixtures are 30% RDX, 25% TNT, 20% Comp. B, 25% NC, 30% NG, 30% M28, and 15% of a 4.4 to 1 mixture of M28 and Comp. B.

2. The above mentioned mixtures did not exhibit friction sensitivity.

3. The above mentioned mixtures, except the last, were thermal reactive, which indicates that these mixtures would be susceptible to thermal reaction (burning) when heated to 177°C during the drying operation. The test method used shows that the thermal hazard is eliminated upon reduction of the concentration of these six energetic materials to 10%.

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Table 1. Explosives and propellants.

Explosives	Lot No.
TNT	VOL 77A-014407
RDX	HOL 21-18
Comp. B	HOL 053-97
Nitroglycerine	FP-1
Propellants	
Nitrocellulose (12.59% N)	RADFORD C3164L-7P013
M28 Propellant*	RADFORD 63 002-087

* Nominal composition: 60% NC, 23.8% NG, 9.9% triacetin, 2.6% dimethyl phthalate, 2.0% lead stearate, and 1.7% 2- nitrodiphenylamine

Table 2. Impact initiation probability as a function of explosive or propellant concentration in brine mixtures.

Explosive or propellant	Concentration (% by wt)	Height (cm)	Fired (%)	Height for 10% fire point (cm)
RDX	10	240	0	-
	20	240	0	-
	30	240	0	-
	35	240	25	200
	40	240	5	240
	45	117	50	80
	50	72	50	< 53
	60	39	50	33
	100	39	50	31
TNT	10	240	0	-
	20	240	0	-
	25	240	0	-
	30	240	30	210
	40	240	10	190
	50	98	50	75
	100	70	50	49
Comp. B	10	240	0	-
	20	240	0	-
	25	240	40	<140
	30	240	55	<120
	35	240	15	160
	40	135	15	70
	40	140	75	-
	100	42	50	35
NG	5	240	0	-
	10	240	0	-
	20	240	0	-
	30	240	0	-
	35	240	55	120
	40	45	50	38
	50	67	50	30

Table 2. (Cont'd)

Explosive or propellant	Concentration (% by wt)	Height (cm)	Fired (%)	Height for 10% fire point (cm)
NC	10	240	0	–
	25	240	0	–
	30	240	20	140
	50	60	50	25
	100	30	50	25
M28 Propellant	10	240	0	–
	30	240	0	–
	35	240	30	140
	40	240	30	210
	50	240	65	120
	100	35	50	28
M28-Comp. B Mixture (4.4 to 1)	10	240	0	–
	15	240	0	–
	20	240	5	240
	50	111	50	60
	100	21	50	20

Table 3. Friction test results of brine mixtures.

Explosive or propellant	Concentration (% by wt)	Results
RDX	30	No Reaction
TNT	25	No Reaction
Comp. B	20	No Reaction
NG	20	No Reaction
NC	25	No Reaction
M28 Propellant	30	No Reaction
M28–Comp. B (4.4 to 1)	15	No Reaction

Table 4. Thermal test results of brine mixtures.

Explosive or propellant	Concentration (% by wt)	Time (min)	Temp (°F)	Observations
RDX	30	2	350	Smoke
RDX	10	30	350	No visual effects
TNT	25	1½	350	Smoke
TNT	10	30	350	No visual effects
Comp. B	20	2	341	Smoke
Comp. B	10	30	350	No visual effects
NG	30	1½	350	Smoke
NG	20	30	350	No visual effects
NC	25	1½	341	Smoke
NC	10	30	350	No visual effects
M28 Propellant	30	1½	350	Smoke
M28 Propellant	10	30	350	No visual effects
M28-Comp. B (4.4 to 1)	15	30	350	No visual effects

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