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HIGH EXPLOSIVE MOULDING PONDERS FROM RDX AND AQUEOUS
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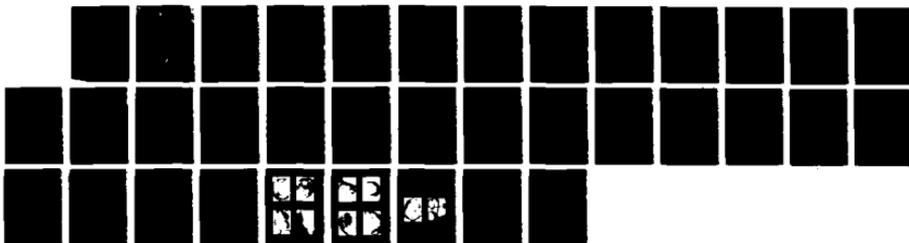
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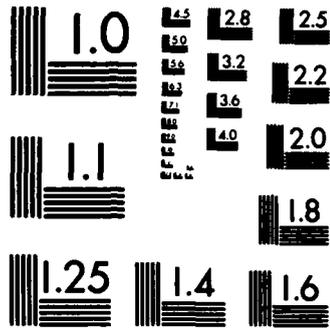
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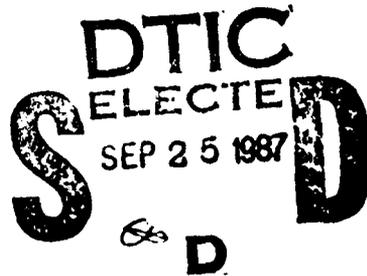
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REPORT

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HIGH EXPLOSIVE MOULDING POWDERS FROM RDX AND AQUEOUS
POLYURETHANE DISPERSIONS

I. Dagley, R.J. Spear and V. Nanut



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ABSTRACT

Eleven commercially available aqueous polyurethane dispersions have been evaluated for use in the preparation of high explosive moulding powders. The polymers were coagulated by addition of electrolytes to aqueous slurries of RDX containing the dispersions. In several cases polymer coating of the explosive crystals has been effected. The impact sensitiveness of these compositions and physical mixtures of the polymer particles with RDX have been determined and the effect of coating efficiency, particle size and polymer properties on sensitiveness is discussed. Formulations suitable for use as high explosive moulding powders have been prepared from two dispersions with additional incorporation of zinc stearate. Impact sensitiveness, shock sensitivity and vacuum thermal stability of these formulations have been assessed.



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HIGH EXPLOSIVE MOULDING POWDERS FROM RDX AND AQUEOUS
POLYURETHANE DISPERSIONS

1. INTRODUCTION

1.1 Use of Polymer Dispersions in the Preparation of High Explosive Moulding Powders

Explosive moulding powders consist of high explosive crystals coated with desensitizing polymers or waxes and are prepared by three main processes.

- (1) The solvent slurry technique [1,2,3] involves stirring a solution of a polymer in organic solvent with an aqueous slurry of the explosive. The solvent is then removed by distillation to give the moulding powder which is collected by filtration. Many chemically different polymers have been employed in this process including thermoplastic polyurethanes [4], fluorocarbons [5] and block copolymers containing styrene [6]. Disadvantages of this technique include flammability and/or toxicity problems associated with the use of organic solvents and the need for processing equipment designed for distillation and often evacuation.
- (2) The "smeared wax" technique involves adding a fine wax to an aqueous slurry of the explosive and heating the stirred mixture to remove the water, melt the wax and coat the explosive [7]. This type of process is not used by some manufacturers for safety reasons [8].
- (3) An aqueous slurry of the explosive is mixed with an aqueous emulsion or dispersion and the emulsion or dispersion is broken to effect coating of the crystals. This approach avoids the problems associated with the other processes and has been used largely for the preparation of explosive moulding powders from natural and synthetic wax emulsions [8-11].

There are comparatively few examples of explosive moulding powders prepared using aqueous polymer dispersions. Examples include the method of Wright [2,3] involving coagulation of aqueous polymer dispersions stirred with aqueous slurries of explosive. In this process a suitable organic solvent is then added to dissolve the precipitate and the solvent then removed by distillation. Recently an unsuccessful attempt to coat RDX crystals with polymer coagulated from an aqueous polyurethane dispersion has been described [12].

Many new commercial polymer dispersions have become available recently as polymer manufacturers have tried to produce safer equivalents to solvent based systems. The potential of these new dispersions to yield polymer desensitized explosive moulding powders by comparatively safe and simple processing was the subject of the investigation described in this report. Aqueous polyurethane dispersions are of particular interest in this regard because polyurethanes have been used extensively in cast-cured PBX formulations [13,14] and in moulding powders prepared by the solvent slurry technique [4]. Dispersions of this type have only been commercially available since 1967 [15] and have not been systematically evaluated for the preparation of high explosive moulding powders. These dispersions possess special properties which make them particularly suitable for this application; they are emulsifier free, contain either low levels of or no organic solvent and typically have good film formation properties at room temperature [15]. They are used in industrial surface coating applications where coagulation of the dispersion effects deposition of a film of polymer onto the substrate [16] and might be expected to similarly coat explosive crystals. Accordingly a study of a large number of commercially available polyurethane dispersions was undertaken to determine their suitability for the preparation of polymer desensitized, high explosive moulding powders.

1.2 Aqueous Polyurethane Dispersions

Polyurethane polymers prepared for aqueous dispersion have chemical structures which differ significantly from the polyurethane polymers which are currently used in high explosive formulations.

Polyurethanes have been used most widely in the preparation of cast-cured polymer bonded explosives where the rubbery nature of the cured formulations bestow low shock sensitivity and explosiveness [13]. These compositions are typically prepared from mixtures of high explosive with liquid low molecular weight (1500-3000) diols of the polyester, polyether or hydroxyl terminated polybutadiene [17] type by reaction with low molecular weight diisocyanates in the absence of water.

Explosive moulding powders incorporating thermoplastic polyurethane resins are prepared by the solvent slurry technique. Resins of this type possess most of the properties of the crosslinked elastomers but are easier to use since no curing is required. In general these solid resins are prepared from polyester or polyether prepolymers with molecular weights in the range 1000-3000 [18]. The main diisocyanate used is diphenylmethane diisocyanate and the only common chain extender is 1,4-butanediol. The resulting polymers are essentially linear with molecular weights greater than 15,000. The structure is segmented with the hard segments melting reversibly without

degradation. The polymers possess elastomeric properties and have glass transition temperatures less than 0°C [19]. The explosive moulding powders LX-14 [4] and PEX-9011 [1,4] are prepared using different thermoplastic polyurethane resins called Estanes which are proprietary materials manufactured by B.F. Goodrich. A general structure for the Estanes [1] is shown in Figure 1.

In contrast there is a greater diversity in the chemical structures of commercially available aqueous polyurethane dispersions. The synthesis and properties of this class of polymer have been widely reviewed [15,19-22] and are briefly described. Synthesis of these polymers is necessarily more complicated and in all cases includes an isocyanate polyaddition reaction. The final polymers have predominantly hydrophobic long chain segments of the polyether or polyester type and also contain some hydrophilic sites which may be nonionic or charged.

In nonionic dispersions, the hydrophilic component is a lateral or terminal polyether chain (eg polyethylene glycol) with a number average molecular weight in the range 600 to 1500. These groups display reduced solubility in water at higher temperatures and dispersions of this type can be thermally coagulated. This property would make the process of coating explosives by the aqueous slurry technique very simple, however this was not investigated because there are few commercially available dispersions of this type.

Ionomers are formed when the hydrophilic site is charged and these can be either cationic (eg containing quaternized nitrogen sites) or anionic (eg. containing ionized sulphonate or carboxylate groups). Only dispersions of the anionic type were chosen for evaluation because they are the predominant type manufactured and offer the possibility of coagulation by addition of acid which is a procedure compatible with RDX.

The synthesis of anionic dispersions usually involves incorporation of the charged site as part of a low molecular weight diol or diamine. Examples include the incorporation of α,α -dimethylol propionic acid, which is subsequently ionized with a tertiary amine, or the use of a diaminoalkane sulphonate sodium salt. These low molecular weight components are reacted with isocyanate groups in reaction sequences which ultimately yield either polyurethane or polyurethane-urea polymers. Three of the methods for preparing aqueous dispersions of this type are briefly described.

- (1) The acetone process involves formation of a high molecular weight polyurethane or polyurethane-urea in a hydrophilic solvent which is typically acetone. Addition of water gives a polymer dispersion and in a final step the solvent is removed by distillation.
- (11) The prepolymer mixing process requires the dispersion in water of an isocyanate terminated prepolymer containing an anionic site. This process is facilitated by the addition of a solvent, usually N-methylpyrrolidone, which is present in the final dispersion at low levels. The dispersed prepolymer is reacted with a water soluble amine which acts as a chain extender in the formation of a polyurethane-urea.

(111) The melt dispersion process follows the reaction sequence shown in Figure 2. An isocyanate terminated, anionic prepolymer is reacted with ammonia (or urea) to give a urea (or biuret) terminated product. This is dispersed, methylolated with formaldehyde and finally condensed in the presence of acid to give a polyurethane-urea (or biuret).

Some commercial aqueous polyurethane dispersions have polymer chains containing reactive functional groups which participate in crosslinking reactions when a suitable curative is added.

2. EXPERIMENTAL APPROACH

In the study reported here eleven aqueous anionic polyurethane dispersions, giving dried polymer films with a range of properties (Table 1), were selected from four commercial manufacturers. Explosive moulding powders were prepared from each dispersion in an aqueous slurry coating process using RDX Grade A, Class 1. These compositions all had a nominal ratio of RDX to polymer of 100:5 and were all prepared by the addition of an electrolyte to effect coagulation of the polymer. Coating efficiencies, vacuum thermal stabilities and impact sensitiveness were assessed for all compositions to determine which polymers were most suitable for the preparation of desensitized high explosive moulding powders. In a comparative study intimate physical mixtures of the same polymers with RDX (250-300 μm) were prepared and their impact sensitiveness determined to investigate the effects of coating efficiency and particle size of the polymer on this property.

From this initial investigation of the dispersions the polymers which gave the most suitable moulding powders were identified. These were used in formulations with a lower nominal level of polymer and with handling and flow properties modified by the incorporation of zinc stearate. The properties of these compositions were more fully evaluated. Shock sensitivities of the pressed compositions were determined for comparison with other explosive moulding powders containing RDX currently under evaluation as replacements for tetryl.

3. EXPERIMENTAL

3.1 Materials

RDX Grade A Class 1 (recrystallized) used in all experiments was received wet from Albion Explosives Factory, Melbourne and dried at the pump prior to use. For the experiments involving physical mixtures of RDX with polymers the RDX fraction in the range 250-300 μm was obtained by wet sieving.

The polyurethane dispersions examined were all commercially available and were selected from the Witcobond (Baxenden Chemical Company Ltd), Q-thane (Quinn), Helastic (Wilmington Chemical Corporation), Impranil (Bayer) and Bayderm (Bayer) ranges of products.

All inorganic salts used were laboratory reagent or analytical reagent grades. In all the preparations distilled water was used.

3.2 Preparation of RDX/Polyurethane (100:5) Formulations

3.2.1 General Method

These formulations were typically prepared by the following general method. Specific experimental details for the different dispersions are shown in Table 2.

A slurry of RDX (40g) and distilled water (40g) was stirred mechanically and after 15 min the polyurethane dispersion (containing 2g of polymer by weight) was added. The mixture was stirred for a further 15 min and then heated to a temperature in the range of 70-95°C. An aqueous solution of an electrolyte was added slowly, at a uniform rate, to the well agitated slurry until coagulation of the dispersion was completed. The mixture was chilled in ice-water with stirring and the product was then collected by filtration, washed thoroughly with distilled water and dried initially at the pump and then at 50°C in a vacuum oven over silica gel.

3.2.2 RDX/Impranil DLH

A slurry of RDX (30g) and distilled water (30g) was stirred mechanically for 15 min then Impranil DLH (40% w/w, 3.75g) was added. After a further 15 min the stirred slurry was heated to 85-90°C and a solution of sodium chloride (10% w/w, 50 mL) was added gradually over 30 min. A solution of calcium chloride (10% w/w, 5 mL) was then added over 30 min to complete the coagulation and the stirred mixture was chilled in ice-water. The product was collected by filtration, washed thoroughly with water and dried initially at the pump and finally at 60°C under vacuum.

3.2.3 RDX/Witcobond 740

Witcobond 740 (60% w/w, 2.5g) was added to a mechanically stirred slurry of RDX (30g) and distilled water (15g). 15 Min later a solution of aluminium sulphate (20% w/w, 25g) was added gradually over a period of 30 min and the mixture was heated at 55°C to complete coagulation of the polymer. The stirred mixture was cooled in ice-water and the composition was collected and dried at the pump and then at 60°C under vacuum.

3.3 Preparation of RDX/Polyurethane (100:2) Formulations

Compositions of RDX/Impranil DLH and RDX/Helastic WC-6913 with this reduced weight of polyurethane polymer were prepared by the methods described in Section 3.2.

3.4 Preparation of Moulding Powders Containing Stearate Salts

3.4.1 RDX/Zinc Stearate (100:2)

A slurry of RDX (200g) and distilled water (300g) was stirred mechanically and at the same time heated in a stainless steel jacketed vessel. The temperature was increased from ambient to 84-86°C in 12 min then a mixture of sodium stearate and water (1:13, 55.3g) was added to the slurry. Aqueous zinc chloride (4.8% w/w, 22.7g) was then added drop-wise over a period of 15 min. The mixture was cooled to ambient temperature in about 10 min and the stirring stopped. The product was collected by filtration and washed with distilled water. It was dried by passing air over it and then placed in an oven at 70°C.

3.4.2 RDX/Impranil DLH/Zinc Stearate (100:2:1)

A slurry prepared from RDX (600g) and water (600g) was stirred mechanically for 15 min. Impranil DLH (40% w/w, 30g) was added and after 15 min stirring the mixture was heated to 82°C. A sodium chloride solution (20% w/w, 800 mL) was added at a uniform rate over 1.5 h. During this addition the stirring rate was adjusted to give maximum agitation without allowing the vortex to expose the impellor. The walls of the mixing vessel were occasionally scraped during the coagulation to free deposited solid. The mixture was then cooled with stirring to 20°C and the composition was collected by filtration and washed thoroughly with water (12 L). This material was maintained in a damp state throughout this process to avoid agglomeration. The damp solid and distilled water (100 mL) were then stirred in a mixing vessel and heated to 82°C. A hot solution of sodium stearate (12% w/v, 50 mL) was added and after 15 min addition of a solution of zinc sulphate (2% w/w, 300 mL) was commenced. Half the solution was added at a uniform rate over 1 h and the remainder was added over a further 30 min. The stirred mixture was cooled and the product was collected by filtration and washed thoroughly with water (12 L). The composition was dried for several hours at the pump and then at 45°C under vacuum over silica gel.

3.4.3 RDX/Impranil DLH/Zinc Stearate (100:2:2)

Method A

This formulation was prepared by the method described in Section 3.4.2 except twice the volume of sodium stearate solution was added and the same volume of a more concentrated zinc sulphate solution (4% w/w) was used.

Method B

RDX (300g) and water (300g) were stirred mechanically for 15 min and Impranil DLH (40% w/w, 15g) was added. The slurry was stirred for a further 15 min, heated to 82°C then an addition of a hot solution of sodium stearate (12% w/v, 50 mL) was made. After 15 min a solution of zinc sulphate (4% w/w, 50 mL) was added at a uniform rate over 1.5 h. During the addition, solid collecting at the walls of the mixing vessel was scraped free. The stirring rate was adjusted to maintain the vortex just above the mixing impellor to avoid entrapment of air in the mixture. When the addition was completed the stirred mixture was cooled to 20°C and the collected product was washed with water (6 L), dried at the pump and finally under vacuum at 50°C over silica gel.

3.4.4 RDX/Impranil DLH/Sodium Stearate (100:2:1)

A slurry of RDX (30g) and water (30g) was stirred for 15 min and Impranil DLH (40% w/w, 1.5g) was added. Finely divided sodium stearate (0.3g) was added after a further 15 min stirring and the mixture was then heated to 90°C and maintained at that temperature for 30 min. The temperature was then adjusted to 85°C and a sodium chloride solution (20% w/w, 40 mL) was added slowly over 45 min. The stirred mixture was then cooled in ice-water and the solid material was collected by filtration and washed with water (2 L). The solid was then dried initially at the pump and then at 50°C under vacuum over silica gel.

3.4.5 RDX/Helastic WC-6913/Zinc Stearate (100:2:1)

A slurry of RDX (300g) and water (300g) was stirred mechanically for 15 min. Helastic WC-6913 (60% w/w, 10g) diluted with water (20g) was added and the mixture was stirred for 15 min at room temperature and then a further 15 min at 82°C. A warmed solution of sodium stearate (6% w/v, 50 mL) was added and after 15 min a solution of zinc sulphate (2% w/w, 120 mL) was added at a constant rate over 90 min. The stirred mixture was cooled to 20°C and the product collected and dried at the pump. This composition was then heated in an oven under vacuum at 60°C over silica gel for several hours.

3.4.6 RDX/Helastic WC-6913/zinc stearate (100:2:2)

This formulation was prepared by the method described in Section 3.4.5 except higher concentrations of the solutions of sodium stearate (12% w/v) and zinc sulphate (14% w/w) were used.

3.5 Preparation of RDX/Polymer Physical Mixtures

All polymers were recovered from the aqueous polyurethane dispersions by coagulation caused by electrolyte addition using the methods described in Section 3.2. The polymers were collected by filtration, washed

thoroughly and dried. Polyethylene wax AC629 was emulsified and then recovered from a warmed dilute solution by acidification using methods described by Wilson [9].

The polyurethane samples were reduced to fine particles using a freezer/mill produced by Spex Industries Inc (USA) which was cooled using liquid nitrogen. All polymers were dried over silica gel for 48 h and were generally separated by sieving into two fractions consisting of particles less than 106 μm and particles in the 106-300 μm range. It was difficult to produce fine particles from the softer polymers and no fractions less than 106 μm were obtained for Estane 5702-F2, Helastic WC-6913 and Witcobond 740. Finally RDX (250-300 μm) was thoroughly mixed with the various polymer fractions to give physical mixtures of RDX and polymers in the ratio 100:5.

3.6 Characterization

3.6.1 Scanning Electron Microscopy

A Cambridge Instruments Model S250 MkII scanning electron microscope with a tungsten electron gun was used. The instrument was operated at 15-21kV in the secondary electron mode. The samples were prepared by mounting crystals of the formulation with PVA adhesive on to a stub coated with PVA adhesive. The sample was then splutter coated with a conducting film of gold. Micrographs were generally obtained for tilt angles of 30 degrees.

3.6.2 Rotter Impact Sensitivity: Figure of Insensitiveness (F of I)

Impact sensitiveness was determined on a Rotter Apparatus [23] using a 5 kg weight. Results from 50 caps tested by the Bruceton procedure were used to determine the F of I values relative to RDX = 80. The values quoted are derived from the height for 50% initiation probability and are rounded to the nearest 5 units. Average gas volumes for positive results are also quoted.

3.6.3 Shock Sensitivity: Small Scale Gap Test

The small scale gap test (SSGT) used at MRL has been described by Wolfson [24]. The donor is a UK Mk3 exploding bridgewire detonator attenuated by laminated brass shim. The acceptor is two 12.7 mm diameter x 12.7 mm height pressed cylinders of the explosive under study and detonation is confirmed using a mild steel witness block. The results were obtained from 25-30 firings using the Bruceton staircase method and are quoted as mm of brass shim for 50% detonation probability, 95% confidence limits and standard deviation. The acceptor pellets were pressed to the specified density using an Instron Universal Testing Machine adjusted to operate as a press [9].

3.6.4 Vacuum Stability Test

The average volume (mL) of gas evolved at 100°C and 120°C over 40 h was determined for two duplicate 5g samples of all RDX/polyurethane binder (100:5) compositions. These tests were carried out in accordance with standard Australian procedures [25].

4. RESULTS AND DISCUSSION

4.1 Preparation of the Formulations

Two alternative methods for coagulating the anionic dispersions to effect coating of RDX crystals were evaluated. Initial experiments using Witcobond 240 showed superior coating was achieved when coagulation was effected by electrolyte rather than acid addition. Accordingly this method has been used in all the procedures described. The suitability of the dispersions was determined by preparation of explosive moulding powders from RDX Grade A with a nominal ratio of RDX to polymer of 100:5 and comparison of their properties. The method of preparation involved heating a stirred aqueous slurry of RDX and the aqueous dispersion followed by the slow addition of sufficient electrolyte to completely coagulate the dispersion over a period of approximately 30 min. Coagulation is enhanced at higher temperatures but in some cases it was necessary to operate at reduced temperatures to prevent agglomeration. Occasionally coagulation could be effected by the addition of sodium chloride solutions but in difficult cases ions of higher valency (eg Ca^{2+}) were used (Schulze-Hardy rule [26]).

Compositions containing Bayderm Finish 80 UD and Witcobond 231-240 are free flowing powders with good handling properties. The other dispersions contain softer polymers and the resulting moulding powders often clumped together during the drying process. The two most promising polymers for desensitizing RDX were Helastic WC-6913 and Impranil DLH and most investigations centred on these formulations. From these two polymers moulding powders with good flow properties were obtained when zinc stearate was also included in the formulation.

Impranil DLH formulations with the best flow properties were obtained by first coating the RDX crystals with the polyurethane polymer and then applying a coating of zinc stearate by precipitation in a separate second operation. It was necessary to wash the polymer coated RDX with water to remove electrolyte prior to the second coating operation to achieve a good coating of zinc stearate: during this washing process the polymer coated explosive was kept damp to avoid agglomeration. In an alternative one step process the zinc salt was added to a stirred, hot slurry of RDX, Impranil DLH and sodium stearate to effect the deposition of both coatings on the RDX crystals. The product obtained was less homogeneous and had inferior flow properties. An attempt to coat RDX with Impranil DLH in the presence of sodium stearate and then deposit sodium stearate on the surface of the polymer coated crystals by cooling the slurry gave similar results. Moulding powders containing Helastic WC-6913 and zinc stearate were most conveniently prepared

by the one step process involving addition of a zinc salt to a slurry containing RDX, the dispersion and sodium stearate. Moulding powders of identical composition prepared by the two step coating process appeared less homogeneous but had very similar flow properties and impact sensitiveness. The moulding powders prepared from both polymers had a RDX/polymer/zinc stearate ratio of 100:2:1 or 2. A moulding powder containing only RDX and zinc stearate (ratio 100:2) was prepared for comparison.

4.2 Coating Efficiency

Initially all the compositions prepared from RDX with the different dispersions were examined by optical microscopy and relative coating efficiencies of the different polymers were estimated (Table 3). The results ranged from apparent mixtures of polymer and explosive to examples of efficient coating of the crystals. The affinities of the coagulated polyurethane polymers for the surface of the RDX crystals obviously varied markedly for the range of dispersions studied. Interactions of colloidal particles and surfaces under similar conditions have been considered in numerous studies of heterocoagulation [27]. Presumably studies of this type, involving measurement of zeta potentials, would explain why some polymers attach at the crystal surface and others fail to coat. The adhesion of polymer particles to the explosive crystal surface is determined by London-van der Waals forces, contact areas and other factors [28]. In cases where good coating was achieved the adhesion of the polymer seemed adequate for further processing.

The compositions where the crystals appeared from optical microscopy to be well coated were examined more closely using scanning electron microscopy. This technique has been frequently used to compare different coating processes qualitatively by visual assessment [8,12,29]. Micrographs of these compositions and RDX Grade A crystals (Micrograph A) are compared in Figure 3. The polymer in the RDX/Witcobond 240 (100:5) composition is clearly deposited unevenly over the surface of the crystals in small clumps and large areas of uncoated crystal are clearly visible (Micrograph B). In contrast the micrographs (C and D) for the RDX/Bayderm Finish 80 UD (100:5) composition show the RDX crystals are very well coated by the polymer. The polymer coating is readily distinguished because it is considerably more textured than the surface of the RDX crystals.

Compositions prepared from Helastic WC-6913 and Impranil DLH with a RDX/polymer ratio of 100:2 were compared by scanning electron microscopy. The Helastic WC-6913 polymer was distributed unevenly on the crystal surface with large clumps of the polymer and exposed crystal faces clearly evident (Micrograph E). Some of the exposed RDX surfaces evident on some crystals probably results from disruption of the crystal coating caused by the clumping of the moulding powders during the drying process. This effect was minimized for the Impranil DLH formulation by drying the composition in a finely divided state. Examination of this composition showed the crystals were well coated with a film of the polymer containing fine polymer agglomerates (Micrograph F). This reduced but did not eliminate the problem. A disrupted film of the polymer is visible at the leading edge of the coated crystal shown.

Three formulations containing zinc stearate were examined by scanning electron microscopy. A micrograph of an RDX crystal coated with zinc stearate alone (micrograph G) shows the coating is well distributed over the crystal surfaces. The deposited zinc stearate is easily identified because of the characteristic coarse texture of this coating. A micrograph of the RDX/Impranil DLH/zinc stearate (100:2:1) moulding powder prepared by the two step process shows the zinc stearate is clearly well deposited over the surface of most of the polymer coated crystal (Micrograph H). A micrograph of the RDX/Helastoc WC-6913/zinc stearate (100:2:2) prepared by the one step process (micrograph I) shows large areas of the surface of the coated RDX crystals are additionally coated with zinc stearate.

4.3 Impact Sensitiveness

Rotter impact sensitiveness data for all the RDX/polyurethane (100:5) compositions prepared by polymer coagulation are shown in Table 3. These results are compared with those obtained for simple physical mixtures of fine (less than 106 μm) and coarse (106-300 μm) particles of the polymers with RDX (250-300 μm) to distinguish the effect of the different coating efficiencies and the different polymer properties on impact sensitiveness.

The results clearly show the importance of achieving good coating of the explosive crystals as the compositions with good coating efficiencies were less sensitive than the corresponding physical mixtures. This desensitizing effect was most pronounced for the Impranil DLH compositions where the physical mixtures were slightly more sensitive (F of I = 70) and the coated crystals considerably less sensitive (F of I = 130) than RDX. In cases where poor coating efficiencies were observed the impact sensitiveness values were very similar to those of physical mixtures with fine particles of the same polymer. In one experiment coagulation of the Witcobond 240 polymer gave a composition with an unusually high F of I value (100). This variability appears to be peculiar to this dispersion and may be related to coating efficiencies. This polymer is the only self cross-linking dispersion [30] examined; presumably cross-linking is occurring during the coagulation and processing, resulting in changes to the degree of coating or the strength of the coating to physical disruption during handling.

The polymers which give physical mixtures with RDX that are extremely impact sensitive fall into two broad classes. The Witcobond 231, 232 and 240 polymers have comparatively high tensile strengths and elastic moduli and low figures for maximum elongation at break (Table 1). These stronger, harder polymers might be expected to sensitize the RDX by mechanisms involving catastrophic failure of the polymer particles under impact. It has been shown that impact energies can be concentrated in polymers to generate high temperatures [31] which may exceed the polymer softening point [32,33]. Studies at the Cavendish Laboratory on the effect of polymers on the impact sensitiveness of explosives [32-38] have demonstrated the importance of this mechanism in sensitizing explosives. The marked increase in sensitiveness measured for the physical mixtures containing coarse particles of these and two other polymers is a trend not previously observed. Swallowe and Field [33] performed drop weight experiments on compacts prepared from physical mixtures of explosives with polymer additives differing in particle size and observed no significant particle size effects.

Physical mixtures of Witcobond 740 or Estane 5702-F2 with RDX also gave mixtures which are extremely sensitive to impact. Estane 5702-F2 is mixed in a polymer blend to form the polyurethane Estane 5702-F1 used in the solvent slurry preparation of the explosive moulding powder LX-14 [4]. The Witcobond 740 polymer is soft and easily deformed and has a very high value for elongation at break and comparatively low elastic modulus (Table 1). Like Estane 5702-F2 this polymer would not be expected to sensitize the mixtures by the mechanism involving catastrophic failure of the polymer [34]. These polymers and Helastic X-961 and WC-6913, which have similar properties, were extremely difficult to reduce in particle size in the freezer mill. Presumably these polymers deform rather than fracture even under these extreme conditions. Examination of the physical mixtures revealed that the soft Witcobond 740, Estane 5702-F2 and Helastic X-961 polymer particles had adhered to particles of RDX to form agglomerates. A scanning electron micrograph of an agglomerate consisting of a particle of Witcobond 740 surrounded by RDX crystals is shown in Figure 3, Micrograph J. It seems likely air entrapment, possibly arising from the nature of the agglomerates formed between Witcobond 740 or Estane 5702-F2 and the RDX crystals, causes sensitizing of these mixtures as a result of adiabatic compression under impact. Air entrapment between a binder and RDX crystals has been associated with sensitizing effects proposed for some compositions containing Viton [12] or wax [8].

Four RDX/polyurethane compositions prepared by coagulation were less sensitive to impact than RDX. The desensitizing effects were most pronounced for the Impranil DLH and Helastic WC-6913 compositions and therefore these compositions were selected for further formulation to give moulding powders with more acceptable handling properties. This involved the preparation of compositions with lower levels of polyurethane polymers, metal stearates and combinations of these materials prepared under different experimental conditions. The impact sensitiveness values measured for these compositions are shown in Table 4. The results show that reducing the RDX/polyurethane ratio from 100:5 to 100:2 results in only a minor increase in impact sensitiveness (see also results in Table 3) and that zinc stearate alone at this level gives desensitizing effects similar to Impranil DLH. Results for the formulations with Helastic WC-6913 show the expected trend of decreasing impact sensitiveness with increasing levels of zinc stearate. Differences between the impact sensitiveness of the Impranil DLH compositions containing low levels of zinc stearate and sodium stearate probably result from poorer coating and inhomogenities in the latter composition.

The impact sensitiveness results for RDX/Impranil DLH/zinc stearate (100:2:2) compositions prepared by different methods shows similar sensitiveness for compositions prepared by the one step (Method A) or two step (Method B) process. Reducing the mixture temperature to 72°C (Method A) gives a composition which is substantially less impact sensitive.

4.4 Shock Sensitivity (SSGT)

The shock sensitivities of pressed moulding powders containing Impranil DLH and Helastic WC-6913 with zinc stearate have been determined by small scale gap test (SSGT) [24] and are listed in Table 5. Most formulations were pressed to approximately 90% TMD although one of the RDX/Impranil

DLH/zinc stearate formulations was studied over an extended range of % TMD. Data for related RDX/polyethylene wax AC629/zinc stearate formulations and tetryl are included for comparison.

The $M_{50\%}$ values indicate that Impranil DLH is significantly better than Helastic WC-6913 at desensitizing to shock for formulations with the same zinc stearate level. Increase in zinc stearate from 1 to 2% results in shock sensitivity for the Helastic WC-6913 formulation being indistinguishable from Impranil DLH with 1% zinc stearate. The trend in shock sensitivity, ie Impranil DLH desensitizes more than Helastic WC-6913, is the same as observed for impact sensitiveness (Table 3) although there was no significant difference in the zinc stearate formulations (Table 4). Polyethylene wax AC629 desensitizes to shock to a greater extent than Impranil DLH. All the moulding powders prepared during the course of this study had considerably lower shock sensitivity than either granular or crystalline tetryl pressed to the same % TMD.

The shock sensitivity results for RDX/Impranil DLH/zinc stearate (100:2:2) compositions prepared by three different methods indicate that preparation at 72°C by the two step process (Method A) is the most effective way of applying the coatings. Presumably the low shock sensitivity and reduced impact sensitiveness (Table 4) of this composition results from more efficient coating of the RDX crystals or less physical disruption of the deposited soft polymer coating during mixing at the lower temperature. The composition prepared by the one step process (Method B) was the most shock sensitive. This composition was the least homogeneous and probably had the poorest coating of the RDX crystals.

The RDX/Impranil DLH/zinc stearate (100:2:1) composition shows a clear trend of increasing shock sensitivity with increasing density of the compacts in the range 85-95% TMD. Similar trends have been noted for RDX/polyethylene wax formulations [9,39] and are the subject of further investigations in these laboratories.

4.5 Vacuum Thermal Stability

Vacuum thermal stability was determined on 5g samples of the moulding powders prepared during the course of this study. Results at both 100°C and 120°C are detailed in Table 6. The normally accepted criterion for pass/fail is that gas evolution should be less than 5 mL over 40 h [40].

The results fall into three main categories.

- (i) Most of the Witcobond polymers show poor compatibility with RDX as evidenced by high evolved gas volumes. This probably arises because the polymer dispersions contain amine functions which are incorporated to permit crosslinking reactions with epoxy resin emulsions. Amines characteristically show poor compatibility with RDX.

- (ii) The Q-thane QW-4020 polymer shows very poor compatibility. However there is insufficient data on its formulation to permit an explanation but again amine functions may be the problem.
- (iii) The Helastic WC-6913 and Impranil DLH polymers show good compatibility with RDX as do the derived moulding powders containing added zinc stearate.

5. CONCLUSIONS

It has been demonstrated that high explosive moulding powders can be prepared from certain polyurethane dispersions by a comparatively simple and safe process involving the use of the aqueous slurry technique and coagulation of the dispersion by electrolyte additions. Several examples of good polymer coating of the RDX crystals have been demonstrated using this technique. The importance of coating efficiencies and polymer properties in determining the impact sensitiveness of these compositions has been shown by the results of comparative tests on these compositions and physical mixtures of the polymers and explosives. It has been shown that two different mechanisms might explain the strong sensitization to impact initiation seen in some of these experiments. Certain polymers have shown a sensitization dependent on particle size and this has not previously been observed for physical mixtures of polymer particles and explosives.

Moulding powders prepared from two of the polymers under study, Impranil DLH and Helastic WC-6913, exhibited significantly reduced impact sensitiveness relative to RDX as well as good compatibility and thermal stability. However both moulding powders had unacceptable flow and handling properties and it was necessary to incorporate zinc stearate to alleviate this problem. This has lost some of the advantages of the simple coating process, particularly in the case of Impranil DLH where the zinc stearate had to be applied in a second coating operation.

The range of moulding powders which were formulated display properties and sensitivities similar to RDX/polyethylene wax formulations currently under evaluation as replacements for tetryl [39]. At this stage they appear to offer no distinct advantages and, given the novel nature of these formulations, would prove considerably more difficult to qualify.

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TABLE 1

SOME PROPERTIES OF THE ANIONIC POLYURETHANE DISPERSIONS AND DRIED FILMS OF THE POLYMERS

Dispersion Properties	Baydeem Finish 80 UD	Helastic WC-6534	Helastic WC-6913	Helastic X-961	Impranil DLH	Q/thane QW-4020	W'bond 231	W'bond 232	W'bond 234	W'bond 240	W'bar 1 740
DISPERSION											
Non-Volatile (%)	30	40	60	40	40	40	30	30	30	30	60
Particle Size (μm)	NA	0.4	0.4	0.7	0.1-0.4	NA	COLLOIDAL	COLLOIDAL	COLLOIDAL	COLLOIDAL	5
DRIED POLYMER											
Density ^a (g/mL)	1.344	1.194	1.166	1.222	1.142	1.306	1.440	1.262	ND	1.153	1.178
Tensile Strength (MPa)	23	8	8	13	42	52	55	48	55	41	24
Maximum Elongation at Break (%)	35	320	530	870	670	325	100	150	270	70	1000
Elastic Modulus at 100% (MPa)	-	4	3	4	5	4	34	41	22	-	2

^a Densities were determined using a gas density pycnometer at $20 \pm 2^\circ\text{C}$.

NA Not available.

ND Not determined.

TABLE 2

EXPERIMENTAL CONDITIONS FOR THE GENERAL PREPARATION OF RDX/POLYURETHANE
(100:5) COMPOSITIONS

	Electrolyte added (conc. w/w)	Volume added (mL)	Time taken for addition (min)	Temperature during coagulation (°C)
Bayderm Finish 80 UD	NaCl (10%)	7	35	80-85
Helastic WC-6534	CaCl ₂ (1%)	12	17	75-80
Helastic WC-6913	CaCl ₂ (0.1%)	15	20	80
Helastic X-961	CaCl ₂ (0.5%)	17	30	75
Q-thane QW-4020	CaCl ₂ (0.1%)	15	20	80
Witcobond 231	CaCl ₂ (5%)	15	15	70
Witcobond 232	NaCl (10%)	40	60	70-75
Witcobond 234	NaCl (10%)	12	20	90-95
Witcobond 240	CaCl ₂ (1%)	7	25	90-95

TABLE 3

IMPACT SENSITIVENESS OF RDX-POLYURETHANE (100:5) MOULDING POWDERS AND PHYSICAL MIXTURES OF RDX AND POLYURETHANES OF DEFINED PARTICLE SIZE

Polyurethane Polymer	Baydorm Finish 80 UD	Helastic WC-6534	Helastic WC-6913	Helastic X-961	Impranil DLH	Q-thane QM-4020	W'bond 231	W'bond 232	W'bond 234	W'bond 240	W'bond 740	Estane 5702-F2
MOULDING POWDERS												
F of I ^a	80	75	95	75	130	75	60	65	90	70 ^c	85	-
Gas Evolution (mL)	16.9	16.0	15.9	19.4	14.7	14.8	16.4	17.1	14.2	14.0	18.3	-
Coating Efficiency ^d	Good	Fair	Good	Fair	Good	Poor	Poor	Poor	Fair	Good	Poor	-
PHYSICAL MIXTURES												
Polyurethane <106 μm												
F of I ^{a,b}	65	75	-	-	70	80	60	60	80	60	-	-
Gas Evolution (mL)	12	15	-	-	12.0	14	13	12.4	15	13	-	-
Polyurethane 106-300 μm												
F of I ^{a,b}	70	65 ^f	65	70 ^e	70	60 ^f	40	45	80	40	35 ^e	35 ^e
Gas Evolution (mL)	12.7	12.5	13.0	14.2	12.9	13.0	15	14.6	14.8	15.4	12.6	12.1

^a Relative to RDX Grade G (F of I = 80).

^b RDX used in the physical mixtures was a 250-300 μm sieve fraction of RDX Grade A with F of I = 75.

^c This is the usual F of I value obtained, however one batch of this formulation has an F of I value of 100.

^d Determined qualitatively by optical microscopy.

^e Not a simple physical mixture by optical microscopy. The polymer particles and RDX crystals have formed agglomerates.

^f By optical microscopy some polymer particles are attached to RDX crystals.

TABLE 4

IMPACT SENSITIVENESS OF RDX/POLYURETHANE/ZINC STEARATE FORMULATIONS

	F of I ^a	Gas Evolution (mL)
RDX/zinc stearate (100:2)	120	
RDX/Impranil DLH (100:2)	130	17
RDX/Impranil DLH/zinc stearate (100:2:1)	125	15
RDX/Impranil DLH/zinc stearate (100:2:2)	115 135 ^b 115 ^c	11 15 16
RDX/Impranil DLH/sodium stearate (100:2:1)	95	14
RDX/Helastic WC-6913 (100:2)	90	10
RDX/Helastic WC-6913/zinc stearate (100:2:1)	110	15
RDX/Helastic WC-6912/zinc stearate (100:2:2)	120	16

^a Relative to RDX Grade G = 80

^b The coating process was carried out at 72°C.

^c The coating process was carried out by the one step process described as Method B in Section 3.4.3.

TABLE 5

SHOCK SENSITIVITY (SSGT) OF RDX/POLYURETHANE/ZINC STEARATE FORMULATIONS

Shock Sensitivity (mm)				
RDX/Polyurethane/ Zinc Stearate	Relative Density % TMD	M _{50%}	Range L _{95%}	Standard Deviation
<u>Impranil DLH formulations</u>				
100.00:2.00:1.03	85.03	1.814	1.875-1.753	0.028
	90.04	2.268	2.314-2.223	0.021
	95.11	2.616	2.692-2.540	0.036
100.00:2.00:2.06	90.07	2.093	2.157-2.030	0.030
	90.06 ^a	1.908	1.938-1.875	0.015
	90.08 ^b	2.268	2.332-2.202	0.030
<u>Helastic WC-6913 formulations</u>				
100.00:2.00:1.03	90.09	2.591	2.634-2.548	0.020
100.00:2.00:2.06	90.04	2.200	2.266-2.131	0.031
FOR COMPARISON				
<u>RDX/Polyethylene wax AC629/zinc stearate</u>				
100.0:2.2:1.0	90.8	1.806	1.844-1.768	0.018
100.0:1.8:2.1	91.3	1.915	1.976-1.854	0.028
<u>Tetryl (CE)</u>				
Crystalline	90.0	2.814	2.858-2.771	0.021
Granular	90.0	3.259	3.315-3.203	0.026

^a This coating process was carried out at 72°C

^b This coating process was carried out by the one step process described as method B in section 3.4.3.

TABLE 6

VACUUM THERMAL STABILITY TEST RESULTS FOR RDX AND MOULDING POWDERS
FROM POLYURETHANE DISPERSIONS

Composition		Average Gas Evolved after 40h for 5g of the composition	
		100°C	120°C
RDX Grade A		0.2	0.6
RDX/Bayderm 80 UD	100:5	0.06	2.59
RDX/Helastic WC-6534	100:5	0.52	7.40
RDX/Helastic WC-6913	100:5	- 0.04	1.27
RDX/Helastic X-961	100:5	0.38	7.00
RDX/Impranil DLH	100:5	0.41	0.89
RDX/Q-thane QW-4020	100:5	4.19	12.49
RDX/Witcobond 231	100:5	1.75	10.68
RDX/Witcobond 232	100:5	2.27	9.08
RDX/Witcobond 234	100:5	1.1 ^a	10.10
RDX/Witcobond 240	100:5	2.20	10.39
RDX/Witcobond 740	100:5	0.13	1.82
RDX/Helastic WC-6913/zinc stearate	100:2:2	0.01	0.63
RDX/Impranil DLH/zinc stearate	100:2:2	- 0.03	1.13

^a This figure was obtained for a composition with an RDX/polymer ratio of 100:10.

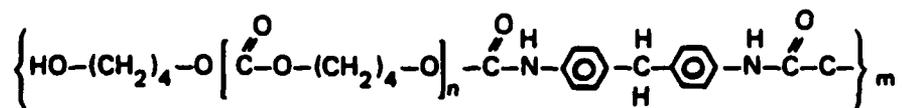


FIGURE 1 A general structure for Estane 5703-F1 which is used in PBX-9011 [1] and PBX-9501 [1].

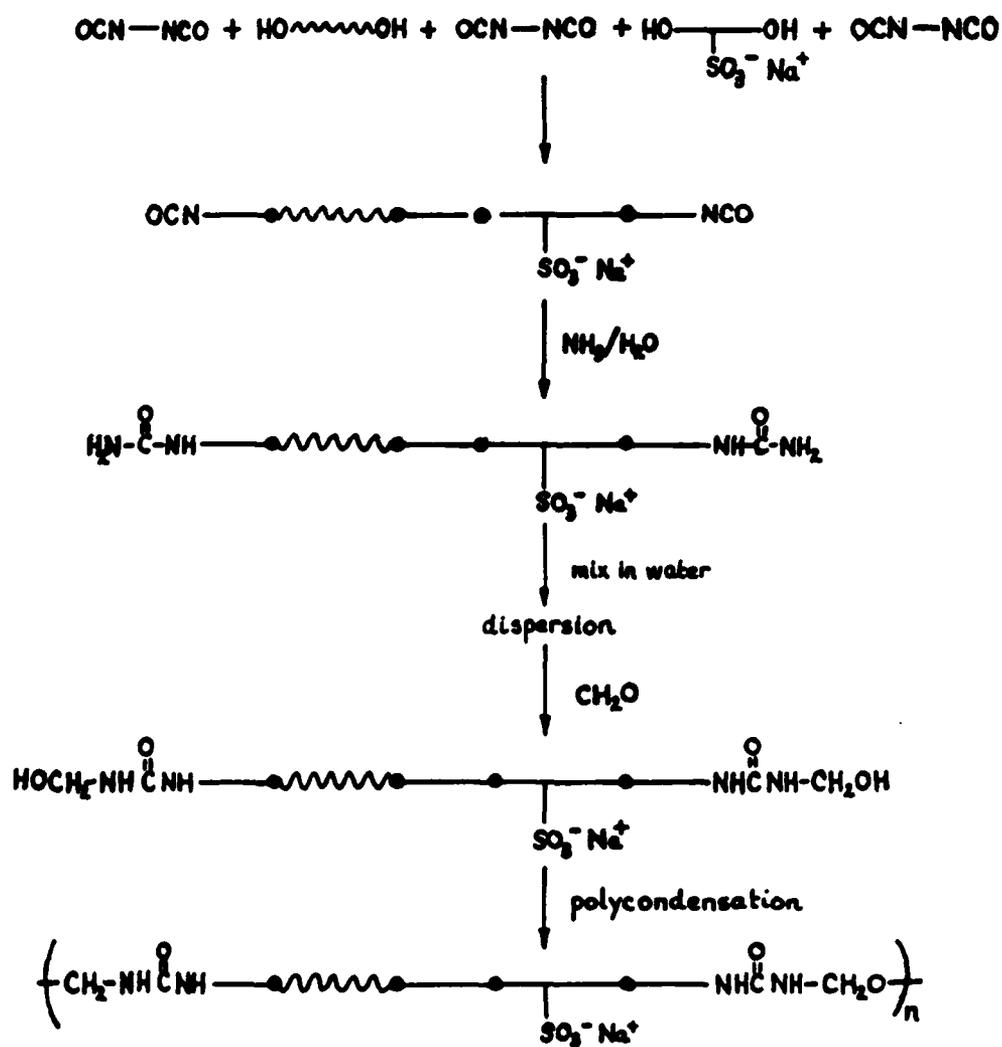


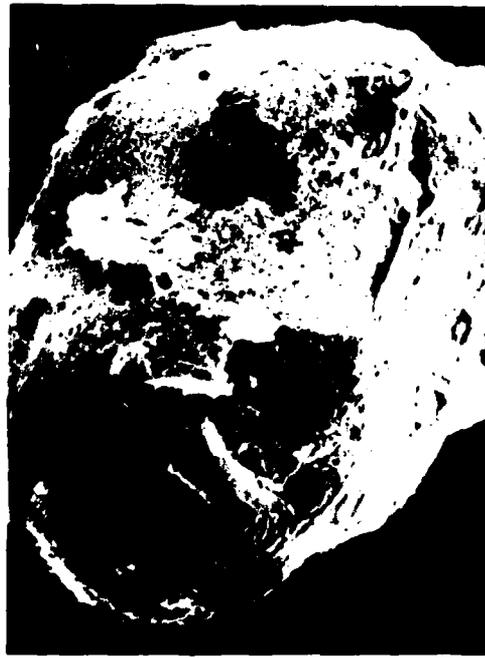
FIGURE 2 Synthetic route to an anionic polyurethane-urea using the melt dispersion process.

FIGURE 3 Various photomicrographs of RDX compositions obtained using scanning electron microscopy.

- Micrograph A. RDX Grade A Class 1 (recrystallized) - X 500.
- Micrograph B. RDX/Witcobond 240 (100:5) - X 350. Large areas of uncoated crystal are clearly visible.
- Micrograph C. RDX/Bayderm Finish 80 UD (100:5) - X 200. Crystals are well coated with polymer.
- Micrograph D. RDX/Bayderm Finish 80 UD (100:5) - X 500. Texture of the polymer coating can be clearly seen.
- Micrograph E. RDX/Helastic WC-6913 (100:2) - X 450. Exposed crystal faces are clearly evident.
- Micrograph F. RDX/Impranil DLH (100:2) - X 500. Crystal coated with a polymer film containing fine agglomerates.
- Micrograph G. RDX/zinc stearate (100:2) - X 500. Zinc stearate coating is well distributed over the crystal surface.
- Micrograph H. RDX/Impranil DLH/zinc stearate (100:2:1) - X 500. The zinc stearate applied in a second step is well distributed on the polymer coated crystal.
- Micrograph I. RDX/Helastic WC-6913/zinc stearate (100:2:2) - X 500. Most of the surface is coated with zinc stearate.
- Micrograph J. RDX/Witcobond 740 (100:5) Physical mixture - X 200. A particle of the polymer has adhered to RDX crystals to form agglomerates.



Micrograph A



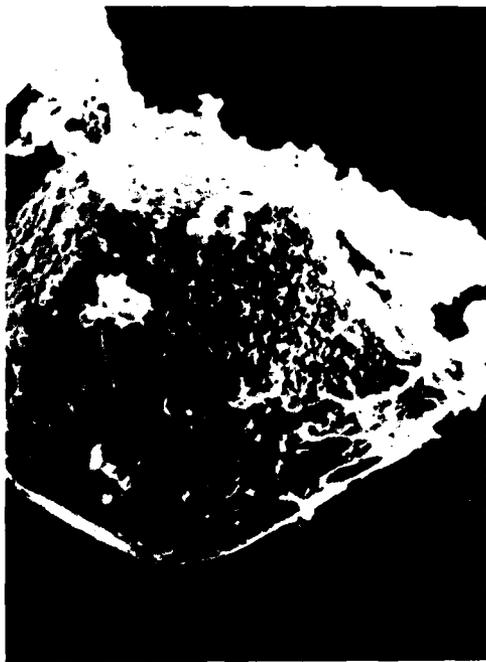
Micrograph B



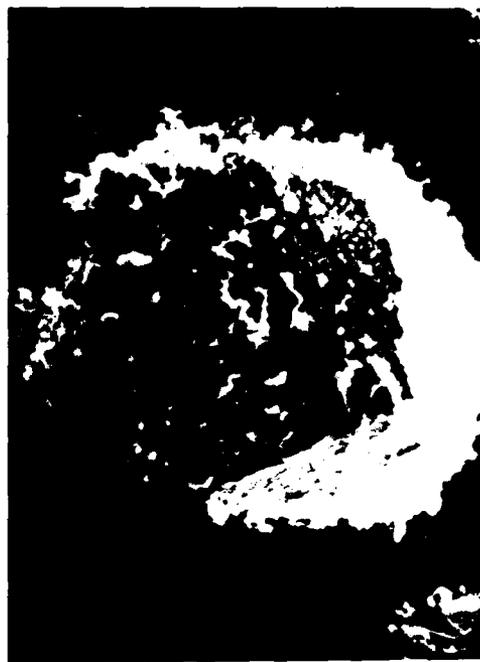
Micrograph C



Micrograph D



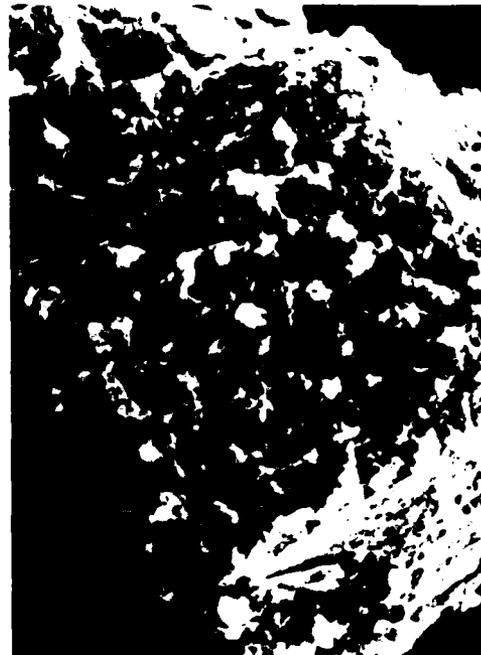
Micrograph E



Micrograph F



Micrograph G



Micrograph H



Micrograph I



Micrograph J

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Plastic bonded explosives
Polyurethane resins

Explosive moulding powders
RDX

SUBJECT GROUPS 0079A 0071E

ABSTRACT

Eleven commercially available aqueous polyurethane dispersions have been evaluated for use in the preparation of high explosive moulding powders. The polymers were coagulated by addition of electrolytes to aqueous slurries of RDX containing the dispersions. In several cases polymer coating of the explosive crystals has been effected. The impact sensitiveness of these compositions and physical mixtures of the polymer particles with RDX have been determined and the effect of coating efficiency, particle size and polymer properties on sensitiveness is discussed. Formulations suitable for use as high explosive moulding powders have been prepared from two dispersions with additional incorporation of zinc stearate. Impact sensitiveness, shock sensitivity and vacuum thermal stability of these formulations have been assessed.

END

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