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RDX-POLYETHYLENE WAX FORMULATIONS AS POTENTIAL REPLACEMENTS FOR TETRYL IN FUZE LEADS, BOOSTERS AND MAGAZINES

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ABSTRACT

Options for materials suitable for replacement of tetryl in Australian fuzes are reviewed, and a study programme is outlined. In this first stage, a series of RDX/AC629 polyethylene wax formulations from 99.60:0.40 to 93.58:6.37 were prepared via the emulsification technique and assessed for powder impact sensitivity, shock sensitivity and vacuum thermal stability. All were less impact sensitive than granular tetryl. Shock sensitivity was comparable with or lower than that of tetryl, and thermal stability was satisfactory. An unusual shock sensitivity/% TMD trend was noted. The use of a higher melting wax AC392 gave no improvement in thermal stability or desensitization to shock. Suggestion for compositions suitable for fuze leads and boosters is made and a programme for further assessment and development outlined.

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RDX-POLYETHYLENE WAX FORMULATIONS AS POTENTIAL REPLACEMENTS FOR TETRYL IN FUZE LEADS, BOOSTERS AND MAGAZINES

1. INTRODUCTION

Tetryl (CE) is used as the filling in leads, boosters and magazines of most Australian produced fuzes. Another major use of tetryl by the services is as an intermediary demolition explosive. Tetryl is no longer manufactured overseas because of health risks associated with production, and future supplies will not be available. Current Australian stock is sufficient for only about 5 years' use. Tetryl is also being phased out because of health hazards associated with its use during filling operations. Investigation of materials suitable to replace tetryl for Australian use was undertaken as a high priority task.

Both the US and the UK have qualified or are in the process of qualifying a number of materials as replacements for tetryl, and these are listed in Table 1. As can be seen, most of these compositions are based on RDX with binder-densensitizer plus additional components. France has also qualified a number of related RDX compositions [1] but accurate technical information is not available to us. Of the compositions listed in Table 1, A-3, A-4, A-5 and the RDX/Wax 8 formulations have been in use for many years. Neither the US nor the UK have chosen to use these compositions as replacements for tetryl, and it must be concluded that their potential to replace tetryl is limited. CH-6 has a long history of production difficulties, while the Debrix process based on hot waxing with evaporation of water must be viewed as significantly more hazardous than the coating processes in current use. Some of the newer US formulations are unattractive for Australian use because they are based on the explosives HMX (PBXN-5) and TATB (PBXW-7), neither of which are produced locally. Similarly there is no local production of either DIPAM or HNS.

In the light of the information presented in the previous paragraph, we undertook a programme to prepare and assess candidate formulations for their suitability as tetryl replacements. Two parallel investigations were pursued; in the first, the initial study of which is reported here, formulations based on RDX suitable for Australian production and use were investigated and assessed. In the second programme, materials listed in

Table 1 were to be obtained from US and UK sources and their explosive properties determined and compared to candidate formulations originating from our studies.

2. EXPERIMENTAL APPROACH

A number of key considerations exist with regard to the choice of replacement materials for tetryl.

1. The material must be no more impact sensitive than tetryl to comply with current fuze explosive train guidelines [2,3].

2. The material should have comparable shock sensitivity to tetryl. This criterion is not as clearcut as the impact sensitivity criterion since the current fuze explosive train guidelines [2,3] could be interpreted to mean that shock sensitivity should be no greater than tetryl. However this could incur a penalty in the explosive train function efficiency, and we accordingly made the decision to pursue materials with equal or greater shock sensitivity than tetryl.

3. There should be no lowering of explosive performance relative to tetryl. This includes properties such as critical diameter, detonation pickup and power output.

4. The basic materials should be locally available and candidate formulations must be suitable for local production.

Clearly RDX is attractive because of its ready availability and relatively low cost. RDX is more impact sensitive than tetryl [4,5]; a compendium of results using different test methods is detailed in Table 2, along with data for shock sensitivity. Waxes and binders are coated onto the RDX crystals primarily to reduce sensitivity, and also to improve processibility. Results for a typical composition produced by the hot wax method, Comp. A-3, and one prepared by the solvent cut method, PBX-9407, are listed for comparison with RDX and Tetryl in Table 2. The substantial decrease in impact sensitivity resulting from wax or polymer coating, as measured by increase in H_{50} [4] and F of I [5], can readily be seen. Shock sensitivity data [4] are also listed in Table 2. They are not so directly comparable as the impact sensitivity data because a complete set of data at the same % TMD is not available. Given this limitation, the substantial decrease in shock sensitivity (decreased 50% point figures) of the coated explosives relative to tetryl and RDX is readily apparent.

Considerable prior experience with RDX-polyethylene wax compositions exists at MRL and this seemed a logical starting point in the search for tetryl replacements. These compositions are produced by the aqueous slurry process using emulsions of AC629 polyethylene wax (Allied Chemicals) and are derived from earlier AWRE Aldermaston Compositions EDC 23 and EDC 24 based on HMX/polyethylene wax/terylene fibre. The earliest MRL studies dealt with RDX grade B/1% polyethylene wax as a potential replacement for beeswax in Comp. B

RDX/TNT/beeswax 60:40:1 [6,7]. Some impact sensitivity data were reported for RDX/1% polyethylene wax [6]. Later studies by Wilson [8-10] were directed to RDX/polyethylene wax compositions as potential pressed fillings for KARINGA. Although formulations ranging from 99:1 to 85:15 were prepared, detailed study centred on the 92:8 formulation [8-10].

It should be stressed that a single composition is unlikely to fill all the perceived applications for tetryl. Indeed, two distinct grades of tetryl are currently used: CE crystalline [11] for leads, and CE granular [11], often mixed with a stearate or graphite for processibility, for boosters and magazines. Examination of the Debrix compositions (Table 1) clearly differentiates the types of compositions. Debrix 11 is for leads, Debrix 12 is for boosters and magazines, while Debrix 18AS is for automatic pelletting. Note that all three compositions contain less than 5% wax.

We accordingly commenced a study of RDX/polyethylene wax, concentrating on the 0.5-5% wax range for which only minimal data exists. The first stage of the study, which we report here, deals only with RDX/polyethylene wax compositions. The compositions were characterized and assessed with respect to their potential to replace tetryl in any of the perceived applications. A following study, which has already commenced, is examining more complex formulations where materials such as stearates and graphite are added to improve processibility. This is particularly important for automatic pelletting where the wax content is critical.

After commencement of the RDX/polyethylene wax studies, parallel investigations of compositions based on RDX/polyurethane and RDX/fluoropolymers were initiated. These studies, which are in a much less advanced stage, will form the basis of further publications.

3. EXPERIMENTAL

3.1 Materials

RDX was grade A Class 1 (recrystallised) from Albion Explosives Factory, Melbourne. The material, transported wet, was dried by suction filtration and air drying.

AC629 and AC392 emulsifiable polyethylene wax were purchased from Allied Chemicals and used as received. Morpholine and Oleic acid were commercial materials. Morpholine was distilled, bp 127-9°C, prior to use. Oleic acid was used as received.

3.2 Emulsions

3.2.1 AC629

A 25% polyethylene wax emulsion was prepared from polyethylene wax AC629 (250 g), oleic acid (42 mL) and morpholine (37.5 mL), made up to 1 L by addition of water, exactly as described in ref. [8].

3.2.2 AC392

Emulsifiable polyethylene wax AC392 (100 g), oleic acid (15.8 g), morpholine (36.6 g) and water (200 g) were placed in a pressure vessel equipped with an external pressure gauge and internal stirring blades. The sealed vessel was heated in a silicone oil bath and stirred slowly using an overhead mechanical stirrer until the reaction vessel reached a temperature of 160°C and the internal pressure was 85 psi. Vigorous stirring was commenced and was continued for 45 min at this temperature. The oil bath was removed and the stirring was continued for 1 h and then for a further 30 min while the vessel was cooled to 40°C in a cold water bath. This gave a consistent, clear, highly viscous emulsion (316 g) with a calculated wax content of 31.7%. This was higher than expected and resulted from slight steam losses from the vessel.

3.3 Preparation of RDX/Polyethylene Wax Formulations

3.3.1 AC629

Coating of the RDX grade A (500 g or 1 kg) was carried out on a 40% w/w slurry with distilled water using the appropriate volume of emulsion basically as described in ref. [8]. The product was recovered by suction filtration, washed with 0.05% aqueous methyl p-hydroxybenzoate solution, dried thoroughly at the pump and was finally dried to constant weight at 70°C. Two modifications on the basic method [8] were investigated. Acidification was carried out to pH of about 4 rather than 4-5 in [8]; this was beneficial in preparing a consistent product. The reaction temperature of 85°C recommended in [8] was raised to 95°C for a limited number of preparations but there appeared to be no advantage in this change.

3.3.2 AC392

The method employed was basically as described for AC629 at 85°C. Although a number of small exploratory batches were prepared, only one large batch, nominal composition 2% wax, was prepared. Quantities used were RDX (200 g) and the 31.7% emulsion described in section 3.2.2 (12.6 g).

3.4 Characterisation

3.4.1 Chemical Analysis

The analysis was carried out in three stages:

a. Extraction with cold petroleum ether, bp 40-60°C fraction. This removes oleic acid and low molecular weight fractions in the polyethylene wax.

b. Extraction with cold acetone. This removes the RDX.

c. Extraction with hot toluene. This removes the remainder of the polyethylene wax. In all the AC629 compositions studied, there was no residue after this stage. This extraction was not practicable for the AC392 formulation due to the low solubility of the wax.

Analysis was carried out in triplicate. The oleic acid constitutes approximately 12% of the total wax. Unfortunately the oleic acid and polyethylene wax cannot be determined separately and wax contents quoted in the text refer to the sum of these materials.

3.4.2 Rotter Impact Sensitivity (F of I)

Impact sensitivity was determined on a Rotter Apparatus [5]. Samples of approximately 27 mg were tested using a 5 kg weight by the standard Bruceton staircase procedure [12]. A total of 50 caps were tested, the F of I being the figure relative to RDX=80 at which probability for ignition was 50%. Experimentally determined F of Is were rounded to the nearest 5 units. Gas volume was measured for each positive result and the figures quoted in the text represent the mean.

3.4.3 Shock Sensitivity: Small Scale Gap Test [13]

The MRL small scale gap test (SSGT) has been described previously in detail [13] and is very similar to the experimental procedure devised by Cachia and Whitbread [14]. The system consists of a donor of a PETN filled exploding bridgewire (EBW) detonator, an acceptor of two 12.7 mm diameter x 12.7 mm height pressed cylinders of the explosive under study, with the gap being of laminated brass shim. Detonation of the acceptor is confirmed by a sharply defined dent in a 25 mm square x 12.7 mm thick mild steel witness block. A typical run consists of 25-30 firings conducted using the Bruceton staircase method [12], the result being expressed as the gap in mm at which detonation probability is 50%. Gap testing commenced with the MRL EBW detonator [13] but as stocks became depleted a change was made to UK Mk 3 EBWs supplied by AWRE Aldermaston. The tests were duplicated on two experimental formulations using both MRL and UK EBWs. The results (gap thickness) were similar but not identical (see Text).

Explosive samples were prepared to the required density by pressing on an Instron Universal Testing Machine operated as a press. Complete

experimental details of this procedure are given in Ref. [8]. Pellets were pressed at the required load for two successive 1 min periods.

3.4.4 Vacuum Stability Testing

The test procedure consisted of placing duplicate 5 g samples in sample tubes, attaching the tubes to a mercury filled manometer and evacuating. The sample tube was then placed in the heated bath, the first 1.5 h was neglected, then readings of gas evolved were conducted for the next 40 h. The samples were tested at both 100°C and 120°C. The quoted result is gas evolved in mL/g over 40 h at the stipulated temperature.

3.4.5 Particle Size Measurements

Particle sizes were determined using a Malvern Particle Size Analyser Model 2600/3600. The large batches were split down to a representative sample. The samples as aqueous slurries were dispersed using energy from an ultrasonic bath for 1 minute. Measurement was then performed in triplicate.

4. RESULTS

Full chemical analysis data together with mean particle size and impact sensitivity (F of I, gas volume evolved) for the moulding powders prepared from AC629 polyethylene wax, and the single moulding powder from AC392 polyethylene wax, are listed in Table 3. Data for RDX grade A and tetryl crystalline and granular are listed for comparison.

Shock sensitivity for the moulding powders pressed to nominal 96% and 91% theoretical maximum density (TMD) are listed in Table 4. Data for tetryl crystalline at 90% TMD and tetryl granular at 90% and 83% TMD are listed for comparison. The two densities for tetryl granular cover the range of booster pellets pressed at MFF St Mary's: 1.45 Mg/m^3 - 1.55 Mg/m^3. The relationship between shock sensitivity and TMD was determined for the 97.94:2.06 formulation over the TMD range 96-80%, and these results are listed in Table 5.

Vacuum stability for selected compositions was determined at 100°C and 120°C and these results are detailed in Table 6.

5. DISCUSSION

5.1 *Moulding Powders from AC629 Polyethylene Wax*

5.1.1 *Composition and Particle Size*

Exact compositions for the moulding powders, determined by chemical analysis, are compared to the nominal composition in Table 3. In the exploratory stage of the programme difficulty was experienced when determining the end point for acidification to break the emulsion. This often resulted in lower wax content than nominal eg the first 99:1 formulation. When this experimental difficulty had been overcome (see Experimental), total wax contents typically were within 10% of the nominal content, as can be seen for the 98:2 nominal compositions (Table 3). The 96:4 and 95:5 moulding powders have significantly higher wax contents than the nominal values (Table 3) because they were prepared to a specified polyethylene wax level rather than total wax as reflected in the nominal composition.

Median particle sizes for the moulding powders increase from RDX grade A (225 μm) with increasing wax content (see Table 3 and Fig. 1). This process can be readily understood as occurring in two stages. Initially, the small particle size component of the RDX (the "fines") is incorporated into the waxed particles. Indeed the most noticeable difference between RDX grade A and the moulding powders incorporating 2% or less wax, when viewed by optical microscopy, is the absence of fine particles in the latter. The second process is subsequent agglomeration of the waxed particles into larger agglomerates, being on average over twice the size of RDX grade A at 6.37% wax (Table 3). Considerably larger agglomerates are observed at higher wax contents [8,9].

5.1.2 *Impact Sensitivity*

The F of I of all RDX/AC629 moulding powders prepared during this study was 100 or greater (Table 3). The F of I for granular tetryl is quoted at 85 [5] or 90 [15]*, and is somewhat higher for the smaller particle size crystalline tetryl at 110 [15]. We determined F of I on Australian production materials and obtained comparable figures (Table 3). The conclusion that can immediately be drawn is that the incorporation of only 0.4% polyethylene wax results in desensitization sufficient to meet fuze safety guidelines [2,3].

With the exception of the 99.6:0.4 moulding powder all the other formulations have F of I in the range 120–140**, with no obvious dependence on wax content. In contrast, there is a strong dependence between the gas volume produced upon ignition and wax content (Table 3); a steady decrease in gas

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* Significant variation between samples is noted [15].

** F of I is not a quantitative measure, hence the normal procedure of rounding to the nearest 5 or 10 units.

volume is observed as wax content increases above 2%. Wilson [9] had earlier observed both these trends for RDX grade A/AC629 moulding powders up to 15% wax. The 85:15 formulation had an F of I of 149 and gas evolution of only 1 mL [9].

It is generally agreed that F of I correlates with ignition and mean gas volume with propagation [5]. For the range of compositions studied here, ignition probability is unaffected above 1% wax content but propagation success drops off rapidly below 2% wax content. These results are consistent with published data on impact and friction sensitivity of HMX and PETN coated with less than 5% wax [16], and also support conclusions reached earlier by Eadie [17] that good dispersion of wax is more important than coating efficiency.

5.1.3 Shock Sensitivity

Shock sensitivity was determined using the MRL SSGT [13] on two sets of samples for each moulding powder: about 96% and 91% TMD. The single exception was the 99.60:0.40 moulding powder which was only studied at 90.8% TMD owing to the excessively high load required to press to 96% TMD. Australian production booster pellets of granular tetryl are pressed to 1.55-1.45 Mg/m^3 (90.0-83.5% TMD) hence the 91% TMD results are more relevant for direct comparison.

Experimentally determined gap widths for 50% detonation probability ($M_{50\%}$), 95% probability limits ($L_{95\%}$), standard deviations (σ) and accurate % TMD values are listed in Table 4. During the course of this work supply of MRL EBW donors [13] became exhausted and replacement by UK Mk 3 EBW donors was made (see note in Experimental). Duplicate determinations on the 97.94:2.06 moulding powder pressed to 96.0% TMD and the 94.61:5.39 moulding powder pressed to 90.7% TMD were carried out, and the results for both donors are similar (Table 4).*

Shock sensitivity decreases with increasing wax content for pellets pressed to 96% or 91% TMD (Table 4). The complete set of data for both % TMD series are plotted in Fig. 2, showing the clear trend. Comparative figures for tetryl crystalline (90.0% TMD) and granular (90.0 and 83.5% TMD) are listed in Table 4. The 99.60:0.40 moulding powder is similar to tetryl granular at 90.0% TMD and significantly more shock sensitive than tetryl crystalline at 90.0% TMD and tetryl granular at 83.5% TMD. The latter two are only slightly more shock sensitive than the 98.69:1.31 moulding powder. All other formulations studied here are less shock sensitive than either tetryl type.

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* This should not be taken to mean that results will be similar outside the relatively small range of shock sensitivity observed here. A comparison on much less sensitive formulations such as cast TNT based materials is currently being undertaken.

An unusual feature of the data is the decrease in shock sensitivity as % TMD decreases. The 97.94:2.06 moulding powder was subsequently studied over an extended range to 80.8% TMD (Table 5 and Fig. 3), confirming the trend observed over the more limited range. Wilson had previously noted the same trend for the 92.3:7.7 moulding powder over the range 89.0–96.0% TMD [9].

The relationship between shock sensitivity and % TMD is often presented as being straightforward. For example, in Ref. [18] it is stated that "Without exception the shock sensitivity of any explosive increases as its (packing) density is decreased", while Price [19] states that "The trends in critical initiation pressure (P_g) versus % TMD are the same for all porous explosives. The higher the % TMD, the higher P_g (hence smaller the gap in a gap test), which means the less sensitive the explosive. The more porous the explosive, the more sensitive it is". However careful scrutiny of the literature reveals a number of exceptions, with one key example being tetryl. Seely [20] noted a reversal in the SSGT shock sensitivity/density relationship for coarse tetryl over the density range 1.4–1.6 Mg/m^3, exactly as we observed for granular (ie coarse) tetryl (Table 4). At lower and higher density, or for fine tetryl over the entire density range 1.1–1.7 Mg/m^3, the normal relationship described above was observed [20]. Dinegar and Millican also observed that higher density tetryl charges were more sensitive than lower density [21].

For pressed PBX compositions, there is limited published data indicative of a shock sensitivity/% TMD reversal as observed here for the RDX/AC629 moulding powders. For example, the data for PBX-9407 (Table 2) indicate slightly lower shock sensitivity at the lower density and further perusal of the data in Ref. [4] identified other examples. Wilson [8] has suggested a number of reasons for the observed behaviour. This phenomenon warrants further study both to determine its cause, for assessment of how widespread it is in pressed PBX formulations, and over what density range it occurs. We are currently conducting further investigations into this subject.

5.1.4 Vacuum Thermal Stability

Vacuum stability was determined at both 100°C and 120°C for two compositions, nominally 2 and 5% wax (Table 6). The thermal stability of either moulding powder, as measured by evolved gas volume, is only slightly lower than the RDX grade A that the samples were prepared from and thus satisfactory.

5.2 Moulding Powders from AC392, and Comparison with AC629

A number of material properties for AC629 and AC392 emulsifiable polyethylene waxes are compared in Table 7. The key differences are the higher melting point and significantly greater hardness (the figures quoted are penetrations) of AC392. It was the higher melting point of AC392 which initially led to the choice of this material in anticipation that a moulding powder of enhanced thermal stability may result.

AC392 is more difficult to emulsify and it was necessary to emulsify under pressure (see Section 3.2.2). The coating process with RDX can be carried out in a similar manner to that described for the AC629 emulsion. Only a single moulding powder, nominal composition 98:2, was prepared in quantity and thoroughly assessed.

Impact sensitivity of this moulding powder is similar to the 98:2 RDX/AC629 compositions (Table 3) but it is significantly more shock sensitive. At 90% TMD the relative figures are $M_{50\%}$ 2.718 mm compared with approximately 2.4 mm (Table 4); both nominal 98:2 RDX/AC629 compositions have slightly higher wax contents than the RDX/AC392, hence estimate for $M_{50\%}$ is slightly higher than any of the figures listed in Table 4. The $M_{50\%}$ for RDX/AC392 (98.10:1.90) is in fact higher than the RDX/AC629 98.69:1.31 composition (Table 4), indicating that at least 0.5% additional AC392 is required to give comparable desensitization to AC629.

The impact sensitivity results imply that the AC392 is well dispersed (see comments in section 5.1.2). The higher shock sensitivity of the AC392 formulation probably results from two main causes. Firstly, shock sensitivity decreases as the coating efficiency increases [22], and coating efficiency decreases if the emulsion is broken below the melting point of the wax (ie AC392) compared with breaking at or above the wax melting point (ie AC629) [16]. Secondly, waxes of higher plasticity fill voids more effectively during pressing, again favouring AC629 over AC392.

Thermal stability of the AC392 moulding powder is slightly less than the AC629 moulding powder (Table 6), despite the higher melting point of the wax. Since the AC392 moulding powder offers no increase in thermal stability, while the emulsion is more difficult to prepare, it was decided to discontinue further studies on AC392.

5.3 The Effect of Reaction Temperature During Wax Coating

The AC629 emulsion was broken by acidification at 80°C, allowed to cool, and the polyethylene/oleic acid filtered off. Examination of the product by hot stage microscopy revealed that melting commenced at 85°C and was not complete till 96°C. The AC392 emulsion, treated in a similar manner, produced a polyethylene/oleic acid product which melted at 127.5–129°C.

Both these materials melt about 10°C lower than the pure polyethylene waxes, no doubt due to the presence of the oleic acid. However the important point to note is that the AC629/oleic acid product only <u>starts</u> to melt at 85°C, the temperature recommended for the coating procedure [8], and is not completely liquid till 96°C. Since the quality of coating could depend crucially on whether the precipitated wax is largely liquid or solid

(see comments in section 5.2) it was decided to reprepare some of the moulding powders at 95-6°C where it would be certain that all the wax was liquid.*

Only a single moulding powder was studied in detail, nominal composition RDX/AC629 98:2, and handling properties, particle size, and impact and shock sensitivity were unchanged from the other 98:2 (nominal) formulations prepared at 85°C. A number of other moulding powders were prepared on a smaller scale at 95°C and were qualitatively the same as their analogues prepared at 85°C. It was decided therefore to continue the coating process at 85°C.

5.4 Suggestions for Compositions Suitable for Replacing Tetryl and Directions for Future Developmental Studies

This study was primarily aimed at determining the suitability of RDX/polyethylene wax formulations as replacements for tetryl, and to identify compositions which have desirable properties for leads, boosters and magazines. With respect to the latter two applications, a composition suitable for automatic pelletting was required but was not addressed in this initial stage of the study.

5.4.1 Fuze Leads

As discussed in previous sections, the 99.60:0.40 moulding powder was less impact sensitive but similar in shock sensitivity to granular tetryl, while the 98.69:1.31 moulding powder was significantly less impact sensitive but marginally lower in shock sensitivity at 91% TMD. A moulding powder of composition RDX/AC629 99.25±0.25:0.75±0.25 should meet fuze safety guidelines while not sacrificing shock sensitivity.

A number of points need clarifying. Leads in Australia are normally hand pressed crystalline tetryl, density 1.1-1.3 Mg/m^3. The detonation pick-up and critical diameter of the 99.25:0.75 moulding powder needs to be determined and compared to tetryl at the above densities. Should shock performance be inadequate, RDX grade B could be substituted since these compositions are similar to or lower in impact sensitivity than the corresponding RDX grade A moulding powder, but shock sensitivity is higher [8,9]. Reproducibility of the formulation process also needs to be checked.

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* The wax/oleic acid would initially be present as micelles which will differ from the filtered product studied by hot-stage microscopy. The material may in fact be liquid in the micelles at 85°C and subsequently coat the RDX as a liquid.

5.4.2 Fuze Boosters and Magazines

Two applications are covered under this broad heading; material pressed directly into the fuze body or component thereof, and material used for manufacturing pellets which are subsequently fitted into the required fuze or ordnance component. For both these applications a lower shock sensitivity than leads is desirable.

Australian fuzes which utilize the first option, ie material pressed directly into the fuze body or part thereof, are produced exclusively using granular tetryl as a physical admixture with 1% or less of a metal stearate and/or graphite. These additives aid pelletting. All the RDX/AC629 moulding powders studied here with 2% or more wax have acceptable impact insensitivity. In order that the pellet have desirable mechanical strength, wax levels should be high, but this reduces shock sensitivity. An acceptable compromise could be an RDX/AC629 formulation around 96.5±0.5/3.5±0.5. Study of shock performance, as outlined in the previous section on leads, as well as processibility and mechanical properties needs to be carried out to further define an optimum formulation.

A composition for automatic pelletting has somewhat different requirements. The principal problem occurs through buildup of wax deposits on the pressing drifts, and this increases with increased wax content. At present we are working with an RDX/AC629 98:2 formulation co-precipitated with zinc stearate and graphite. Studies are now at an early stage and will be reported fully in a subsequent report.

6. CONCLUSIONS

A series of RDX grade A/AC629 emulsifiable polyethylene wax compositions from 99.60:0.40 to 95.58:6.37 have been prepared in 500 g to 1 kg batches. The production process uses aqueous conditions with no evolution of solvent or toxic vapours, and all major ingredients are of low toxicity. The polyethylene wax is readily available through local suppliers and the process should be very suitable for local production.

The RDX/AC629 compositions were characterised by powder impact sensitivity and shock sensitivity (SSGT) at 91 and 96% TMD. Australian production grade tetryl, both crystalline and granular, has been similarly characterised for comparison. All AC629 compositions were less impact sensitive than granular tetryl. Compared with granular tetryl, the 99.60:0.40 formulation was similar in shock sensitivity at 90% TMD, while the 98.69:1.31 formulation was marginally less shock sensitive. All other RDX/AC629 formulations were less shock sensitive than pure granular tetryl. The RDX/AC629 formulations display an unusual shock sensitivity behaviour in that they become less shock sensitive at lower density. This tread was confirmed down to 80.8% TMD for the 97.94:2.06 formulation. All moulding powders exhibited adequate thermal stability at 100°C and 120°C, being only slightly lower than the RDX grade A from which they were prepared.

It is recommended that a 99.25:0.75 formulation be further assessed for use in fuze leads. It is also recommended that a 96.5:3.5 formulation be further assessed for applications where direct pressing into fuze bodies is required. Further study of shock performance, including detonation pickup and critical diameter, as well as processibility and mechanical properties needs to be carried out. A composition suitable for automatic pelletting needs to be investigated: initially a 98:2 formulation co-precipitated with a metal stearate and graphite looks attractive based on the data from this study.

The use of a higher melting emulsifiable polyethylene wax, AC392, was also investigated. This wax was more difficult to emulsify than AC629. While the 98:2 moulding powder was similar in impact sensitivity, it had higher shock sensitivity at 90% TMD, and slightly lower thermal stability than the corresponding AC629 formulation. On the basis of these results, particularly the difficulty in emulsification and lower shock desensitization efficiency, further study of AC392 was discontinued.

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TABLE 1

Compositions Qualified or Undergoing Qualification in the US and UK as Replacements for Tetryl

Composition	Country of Origin	Formulation
A-3	US	RDX (91.0%), wax (9%)
A-4	US	RDX (97.0%), wax (3%)
A-5	US	RDX (98.75%), stearic acid (1.25%)
CH-6	US	RDX (97.5%), polyisobutylene (0.5%), graphite (0.5%), calcium stearate (1.5%)
PBXN-5	US	HMX (95%), Viton A (5%)
PBXN-6	US	RDX (95%), Viton A (5%)
PBXW-7	US	RDX (35%), TATB (60%), PTFE (5%)
DIPAM	US	DIPAM
HNS Type I or II, Grade A	US	HNS
Debrix 11	UK	RDX 1B (99.0%), wax No. 10 (1.0%)
Debrix 12	UK	RDX 1B (95.8%), wax No. 10 (4.2%)
Debrix 18AS	UK	RDX 1B (95.3%), wax No. 10 (2.7%), zinc stearate (1.5%), aerosil (0.5%)
RDX/Wax 8	UK	RDX, wax No. 8; ratio 88:12, 91:9, 93:7

TABLE 2

Impact and Shock Sensitivity Data for Tetryl, RDX and Two RDX/Polymer Formulations: Composition A-3 and PBX-9407[a]

Test	Composition			
	Tetryl	RDX	Composition A-3 RDX-WAX 91:9	PBX-9407 RDX-FPC 461 94:6
IMPACT SENSITIVITY				
2.5 kg, Type 12 Tooling, H_{50} in m	0.37	0.28	0.81	0.46
2.5 kg, Type 12B Tooling, H_{50} in m	0.41	0.32	2.45	0.46
Rotter Impact, F of I[b]	86	80	98,140	
SHOCK SENSITIVITY				
Density Mg/m^3 (% TMD), 50% point in mm				
LANL SSGT	1.676 (96.9) 4.04 1.684 (97.3) 3.84	1.735 (95.9) 5.18	1.635 (96.9) 0.89	1.770 (98.2) 2.57 1.696 (94.1) 3.91
LANL LSGT	1.690 (97.7) 59.82 1.666 (96.3) 60.60	1.750 (96.7) 61.82	1.638 (97.1) 54.51	1.772 (98.3) 54.75

a Data from Ref. [4] unless indicated otherwise.

b Data from Ref. [5]. The value for A-3 is actually for RDX/wax 8 91:9; 98 from [5] and 140 from UK Explosives Safety Certificate 1142.

TABLE 3

Chemical Analysis and Impact Sensitivity of RDX-Polyethylene Wax Moulding Powders and Reference Materials

Composition			Median Particle size (μm)	Impact Sensitivity	
Nominal	% RDX [std. dev.]	% Polyethylene wax [std. dev.]		F of I[a]	Gas Evolution (mL)
RDX-AC629					
99:1	99.60 [0.005]	0.40 [0.009]	235	100	19.7
99:1	98.74 [0.005]	1.31 [0.008]	300	130	19.3
98:2	98.25 [0.053]	1.75 [0.009]	265	140	20.0
98:2	98.14 [0.021]	1.80 [0.243]	320	140	20.0
98:2	97.97 [0.016]	2.06 [0.025]	280	135	19.9
98:2	97.82 [0.008]	2.20 [0.014]	265	140	16.0
97:3	97.15 [0.017]	2.86 [0.014]	325	130	13.7
96:4	95.33 [0.024]	4.69 [0.019]	395	140	8.5
95:5	94.59 [0.095]	5.39 [0.093]	375	130	3.6
95:5	93.58 [0.148]	6.37 [0.105]	455	120	3.0
RDX-AC392					
98:2	98.10 [0.054]	1.90[b]	275	135	20.0
RDX Grade A			225	80	19.3
Tetryl (CE)					
Crystalline [c]				105,110[d]	15.5
Granular[c]				110, 90[d]	17.9

a Relative to RDX Grade G = 80
b By difference.
c Production material ex MFF St. Marys, conforms to specification [11].
d Data from Ref. 15.

TABLE 4

Small Scale Gap Test Shock Sensitivity Data for Pressed RDX-Polyethylene Wax Formulations, with Tetryl Data for Comparison

Composition	Shock Sensitivity Data[a]							
	Nominal 96% TMD				Nominal 91% TMD			
	$M_{50\%}$	$L_{95\%}$	σ	Exact % TMD [std. dev.]	$M_{50\%}$	$L_{95\%}$	σ	Exact % TMD [std. dev.]
RDX:WAX (%)								
AC629 WAX								
99.60:0.40		nd			3.175	3.218–3.132	0.020	90.8 [0.3]
98.69:1.31	2.651	2.692–2.611	0.019	95.9 [0.2]	2.593	2.642–2.548	0.022	91.0 [0.4]
97.94:2.06	2.583	2.639–2.527	0.027	96.0 [0.1]				
	2.614[b]	2.705–2.522	0.042	96.1 [0.1]	2.431[b]	2.507–2.352	0.036	91.0 [0.1]
97.80:2.20	2.647	2.728–2.568	0.038	96.1 [0.4]	2.466	2.515–2.421	0.022	90.9 [0.2]
97.14:2.86	2.474	2.586–2.365	0.052	96.1 [0.1]	2.276	2.377–2.174	0.048	90.9 [0.1]
95.31:4.69	2.126	2.174–2.078	0.022	95.9 [0.1]	1.681	1.737–1.628	0.026	90.8 [0.3]
94.61:5.39	1.831	1.877–1.786	0.022	95.7 [0.1]	1.288	1.331–1.246	0.019	90.8 [0.3]
					1.234[b]	1.273–1.199	0.017	90.6 [0.1]
AC392 WAX								
98.10:1.90		nd			2.718	2.858–2.771	0.089	90.0 [0.1]
TETRYL (CE)								
Crystalline		nd			2.814[b]	2.858–2.771	0.021	90.0 [0.1]
Granular		nd			3.259[b]	3.315–3.203	0.026	90.0 [0.1]
					2.814[b]	2.934–2.692	0.056	83.5 [0.1]

a Data for $M_{50\%}$, $L_{95\%}$ and σ are in mm, determined using MRL EBW donors [13] unless superscripted b.
b Obtained using UK Mk 3 EBW donors.
c Production of CE granular booster pellets is in the density range 1.55 Mg/m^3 (90.0% TMD) – 1.45 Mg/m^3 (83.5% TMD) as studied here.

TABLE 5

The Variation of Small Scale Gap Test Shock Sensitivity with Theoretical Maximum Density for a Nominal 98:2 RDX/AC629 Formulation[a]

Theoretical Maximum density (%) [std. dev.]	Shock Sensitivity[b]		
	$M_{50\%}$	$L_{95\%}$	σ
96.1 [0.1]	2.614	2.705–2.522	0.042
91.0 [0.1]	2.431	2.507–2.352	0.036
85.8 [0.1]	1.880	1.938–1.821	0.027
80.8 [0.1]	1.656	1.725–1.588	0.032

a Exact composition RDX/AC629 97.94:2.06.

b All data in mm, using UK Mk 3 EBW donors, partially repeated from Table 4.

TABLE 6

Vacuum Thermal Stability Test Results for
Selected RDX/Polyethylene Wax Samples

Composition	Gas Evolved (mL/g) After 40 h	
	100°C	120°C
RDX/AC629 97.80:2.20	0.03	0.22
RDX/AC629 95.33:4.69	0.01	0.36
RDX/AC392 98.10:1.90	0.03	0.46
RDX Grade A[a]	0.04	0.13

a Material from same batch as used for formulation preparations.

TABLE 7

A Comparison of Material Properties for the Emulsifiable Polyethylene Waxes AC629 and AC392 (Allied Chemicals)

Parameter	Polyethylene Wax	
	AC629	AC392
Melting Point, ^{o}C (ASTM E-2-8)	104	138
Hardness, dnn (ASTM D-5)	5.5	<0.5
Density, Mg/m^3 (ASTM D-1505)	0.93	0.99
Viscosity, cps at $140^{o}C$ (Brookfield)	200	9000
Acid No., mg KOH/g	16	28

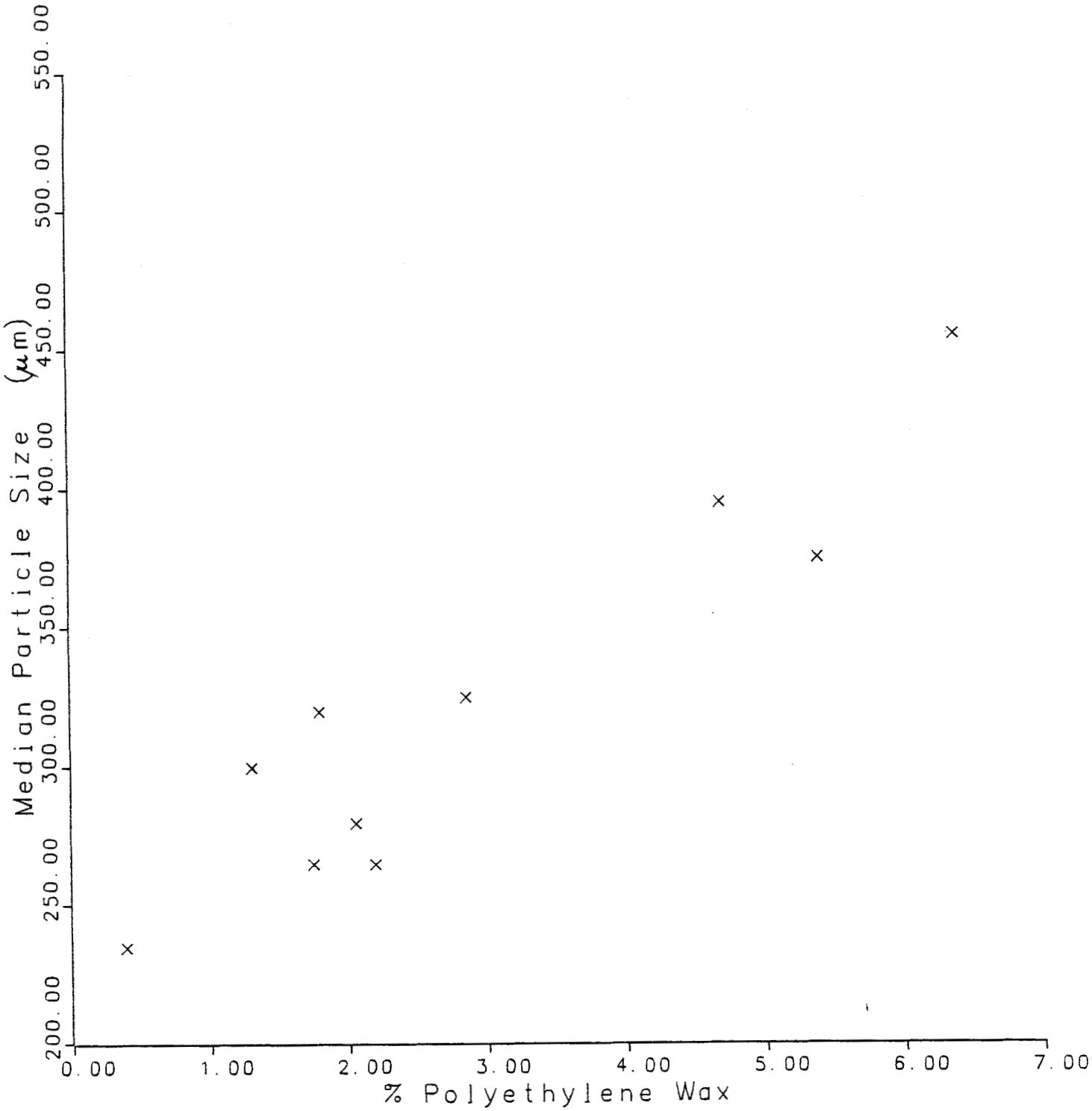

FIGURE 1 A plot of median particle size (μm) versus wax content (%) for RDX/AC629 polyethylene wax moulding powders.

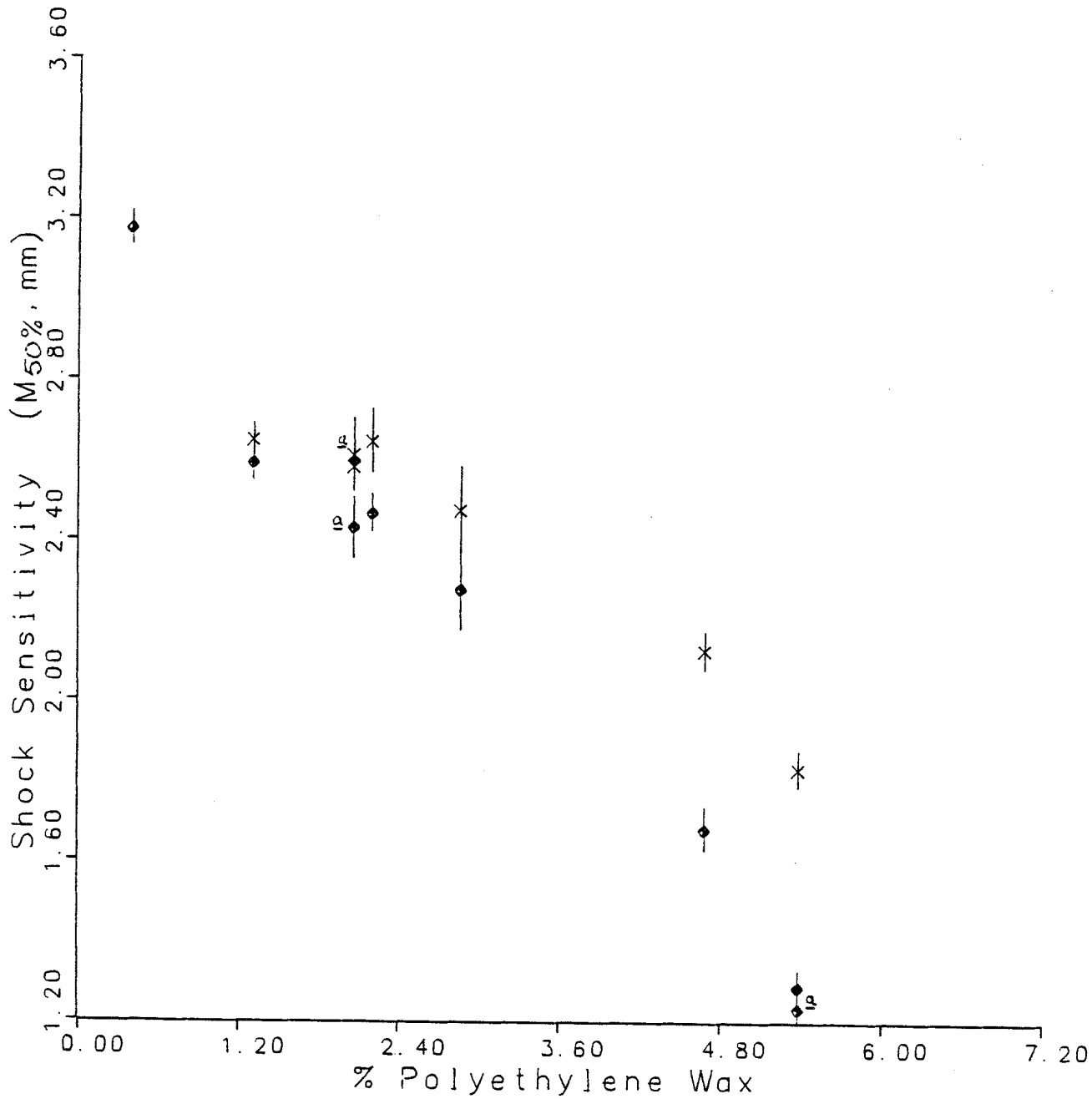

FIGURE 2 A plot of shock sensitivity ($M_{50\%}$), measured as mm of brass shim attenuator in the MRL SSGT, versus wax content (%) for RDX/AC629 polyethylene wax moulding powders.
× 95% TMD 90% TMD a UK Mk 3 EBW donors. Limit bars represent $L_{95\%}$ probabilities.

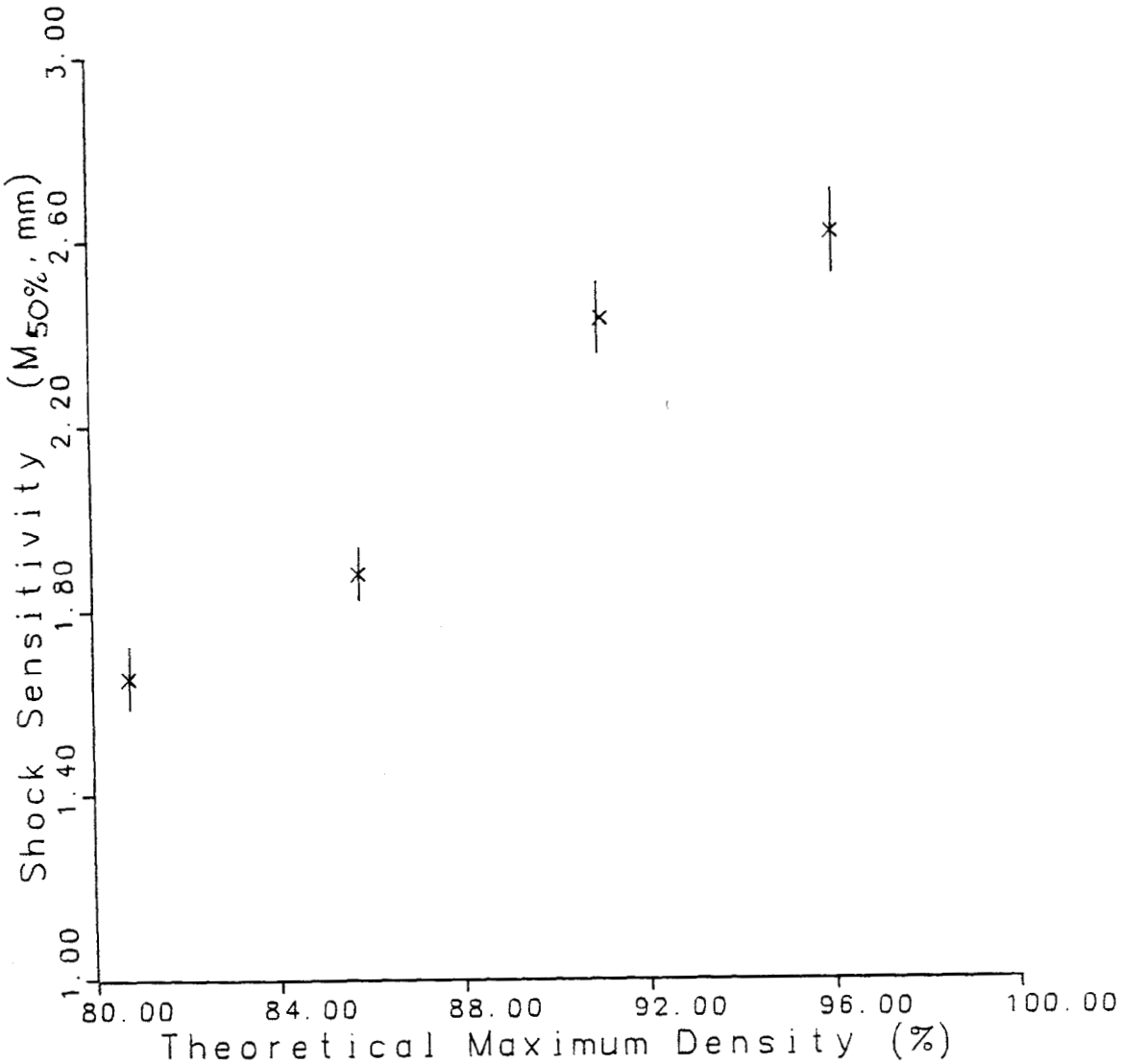

FIGURE 3 A plot of shock sensitivity ($M_{50\%}$) from the MRL SSGT versus % TMD for the RDX/AC629 97.94:2.06 moulding powder. Limit bars represent $L_{95\%}$ probabilities.