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RECOMMENDED REPLACEMENTS FOR TETRVL IN AUSTRALIAN
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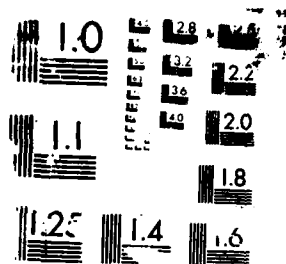
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REPORT

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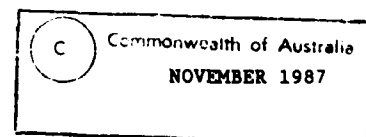
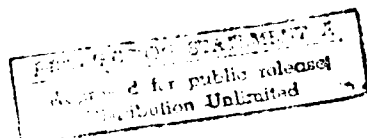
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**RECOMMENDED REPLACEMENTS FOR TETRYL IN
AUSTRALIAN PRODUCTION FUZES AND
RELATED ORDNANCE**

Robert J. Spear, Victor Nanut,
Lance D. Redman and Ian J. Dagley



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ABSTRACT

The final phase of study into RDX-based formulations suitable for replacement of tetryl in Australian ordnance is described. Three formulations using readily available AC629 emulsifiable polyethylene wax and processible on Australian production equipment are identified. It is recommended that pilot batch production and qualification of RDX/AC629 98.75:1.25 for leads and small pellets, RDX/AC629 96.5:3.5 for large boosters and RDX/AC629/zinc stearate/graphite 98.0:1.25:0.5:0.25 for high production rate automatic pelleting be carried out. Performance testing has included propagation in small channels, fuze train function in Fuze Mk 162 and production automatic pelleting. US and UK qualified formulations have been similarly characterised and are compared.

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POSTAL ADDRESS: Director, Materials Research Laboratories
P.O. Box 50, Ascot Vale, Victoria 3032, Australia

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RECOMMENDED REPLACEMENTS FOR TETRYL IN
AUSTRALIAN PRODUCTION FUZES AND
RELATED ORDNANCE

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 now only manufactured at a single German plant because of health and pollution problems associated with production, and future supplies are not assured. Australian stock is sufficient for about 5 years at current usage levels. 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-desensitizer plus additional components.

The US has neither manufactured nor filled with tetryl for quite a number of years. A-5 is used by the US Army as the lead and booster filling for most fuzes. However the US specification for A-5 [1] calls for Type B (Bachmann) RDX [2] and Australia, like the UK, manufactures only Type A (Woolwich) RDX. Indeed the UK recently qualified "UK A-5" [3] which necessitated addition of HMX to Woolwich RDX followed by co-crystallization to prepare pseudo-Bachmann RDX. Australia cannot prepare A-5 using current production facilities. CH-6 has a long history of production difficulties, while 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.

The UK have chosen to replace tetryl with the Debrix formulations listed in Table 1. These formulations use the equivalent of Australian RDX Grade B, Class 1 [4-6] and therefore could be manufactured in Australia. However the Debrixes are produced by the smeared wax technique which is not used by some manufacturers for safety reasons [7]; this must be viewed as significantly more hazardous than the waxing processes in current Australian use.

A-3 and the RDX/wax 8 compositions are principally pressed main charge fillings and are not suitable for most boosting applications.

In the light of the information presented above, we undertook a programme to prepare and assess candidate formulations for their suitability as tetryl replacements. Two parallel investigations were pursued:

- (a) Formulations based on RDX Grade A or B suitable for Australian production and use were prepared and assessed.
- (b) Selected materials from Table 1 were obtained from the US and UK through the auspices of TTCP WTP-1. These materials were characterised and the results compared with formulations originating from our studies.

The work described in this report consists of our studies over the past 12 months. These studies have culminated in recommendations for tetryl replacements based on RDX Grade A, and follow on from two earlier reports [8,9].

2. EXPERIMENTAL APPROACH

For completeness we will restate the key requirements for any material being sought as a replacement for tetryl.

- (a) The material must be no more impact sensitive than tetryl to comply with the current fuze explosive train guidelines [10,11].
- (b) The material should have comparable shock sensitivity to tetryl.
- (c) There should be no lowering of explosive performance relative to tetryl. This includes properties such as critical diameter, detonation pickup and power output.
- (d) The basic materials should be locally available and candidate formulations must be suitable for local production.

RDX was the obvious explosive to replace tetryl because of its ready availability. Guideline (a) above required coating (waxing) of the RDX to decrease impact sensitiveness. The three main processes to achieve coating are described below for RDX, but are applicable to any solid explosive.

- (a) The solvent slurry technique involves stirring a solution of polymer in organic solvent with an aqueous slurry of RDX. The solvent is then removed by distillation to give the product which is collected by filtration. Many chemically different polymers have been employed in this process, which is widely used in the US [12]. The main disadvantages for Australian production are flammability and/or toxicity problems associated with the use of organic solvents, and the need for processing equipment designed for distillation and often evacuation.
- (b) The "smeared wax" technique involves adding a fine wax to an aqueous slurry of RDX and heating the stirred mixture to remove the water, melt the wax and coat the RDX. As discussed in the Introduction, this process is used for the Debrises but is not used by some manufacturers for safety reasons [7]. We would not recommend this process for Australian production.
- (c) The third main process involves mixing an aqueous slurry of RDX with an aqueous emulsion or dispersion of the wax/polymer, then 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 [7,13,14].

We therefore chose to investigate formulations which could be produced via the aqueous emulsion process. This has the added advantage that current Australian production equipment could be used. Two types of formulations were assessed; RDX/polyethylene wax via emulsifiable polyethylene wax and RDX/polyurethane via aqueous polyurethane dispersions.

The results of our preliminary investigations have been published [8,9]. The RDX/polyurethane study [8] yielded two formulations with very favourable properties. However neither offered any significant advantage over the RDX/polyethylene wax formulations, and given their novel nature could prove more difficult to qualify. On the other hand, the RDX/polyethylene wax formulations should be relatively easy to qualify since the HMX/polyethylene wax formulations EDC23 and EDC24 have been interim qualified [15] and the US Navy has qualified A-3 Type 2 which is RDX/polyethylene wax 91:9 [16]*.

* The requirement for a replacement for the microcrystalline petroleum wax used in A-3 arose both from supply difficulties and the expectation that an improved product could be found [16]. However, the US Navy has now phased out use of A-3 and did not carry out service introduction of A-3 Type 2 [17].

Production of A-3 Type 2 was said to have presented no problems [17]. It is interesting to note that breakdown of Soviet surface-to-air missiles SAM-3, SAM-6 and SAM-7 revealed booster pellets of RDX/polyethylene 97:3 [18].

3. US VERSUS UK: A DIFFERENCE IN APPROACH

As stated in the Introduction, the US Army uses A-5 for most fuze fillings, ie both leads and boosters. A-5 is nominally RDX/stearic acid 98.75:1.25 but the specification permits ratios as high as 99.0:1.0 [1]. The other widely used RDX-based formulation is CH-6 which contains RDX/desensitizers (see Table 1) 97.5:2.5 [19]. Although PBXN-5, PBXN-6 and PBXW-7 Type II (Table 1) contain 5% Viton A as binder, Viton is a poor desensitizer (see later) and can sensitize under some conditions [20].

In contrast the UK have elected to use Debrix 11, RDX/wax 99:1, only for leads and small pellets [4]. Larger booster (exploder and magazine) pellets are pressed from the more highly waxed Debrix 12 and 18AS [5,6], RDX/wax 95.8:4.2 and RDX/desensitizers 95.3:4.7 respectively (see Table 1).

Faced with the decision on whether to adopt UK or US philosophy, we decided to compromise by developing one composition comparable in performance to both A-5 and Debrix 11, a second composition comparable to Debrix 12, and a third composition suitable for automatic pelletting.

4. RESULTS AND DISCUSSION

At the end of the first phase of the study, we recommended that an RDX Grade A or B/AC629 polyethylene wax 99.25:0.75 formulation be further assessed for fuze leads, and a 96.5:3.5 formulation be further assessed for pressed boosters [9]. No attempt had been made to address the problem of a formulation suitable for automatic pelletting.

The only change in production method for this second phase of the study was substitution of stearic acid for oleic acid in the AC629 emulsion. This was carried out because the presence of oleic acid meant that zinc oleate could be precipitated in competition with zinc stearate.

4.1 Fuze Leads

The key properties required for leads are high shock sensitivity, ie good pickup with short run distance to detonation, and propagation at small (2-4 mm) diameter.

The RDX/AC629* 99.6:0.4 formulation, ie only 0.4% wax, had F of I 100 and thus this level of wax was sufficient to give impact sensitiveness less than granular tetryl [9]. RDX/AC629, nominally 99:1**, was assessed using both RDX Grade A and Grade B. The Grade B formulation was expected to give significantly higher shock sensitivity. Results for both formulations are listed in Table 2 together with earlier data [9] for a 98.69:1.31 formulation, ie wax level slightly greater than A-5.

In comparison with the Grade A formulation, the Grade B formulation exhibited marginally higher shock sensitivity while impact sensitiveness was appreciably greater (Table 2). However flow and general handling properties were markedly inferior. This, coupled with the relatively poor impact sensitiveness, led to discontinuation of studies on a Grade B formulation.

The formulation on which extended studies were carried out was RDX Grade A/AC629 98.94:1.06, ie a wax level intermediate between that for Debrix 11 and A-5 and within the A-5 specification [1]. (See also footnote ** below).

Estimation of failure diameter was carried out on unconfined pellets of RDX/AC629 97.8:2.2 at 6, 4 and 2 mm diameter pressed to 1.69 Mg/m^3 (95 ITMD). 2 mm diameter pellets were also studied at 1.64 Mg/m^3 (91 ITMD). Velocity of detonation results are detailed in Table 3.

Stable detonation was observed for the 2 mm charges at both densities; total charge length was > 15 charge diameters and a fading detonation was not occurring. We can feel confident on the basis of these results that a nominally 98.75:1.25 formulation would propagate normally in a lead of internal diameter less than 2 mm since both the lower wax content and the confinement from the lead would decrease failure diameter.

* AC629 will be used throughout the following text, but it should be remembered that the wax coating is polyethylene wax AC629 - stearic acid in the approximate ratio 92:8.

** Although the recommendation from the first phase was a 99.25:0.75 formulation [9], the decision to produce a material comparable in performance with Debrix 11 and A-5, rather than optimised for shock sensitivity, led to choice of higher wax levels.

Confirmation of satisfactory fuze train performance was carried out in Fuze Mk 162. Full experimental details are described in Section 7.5.2. Leads were filled with RDX/AC629 98.94:1.06 at three densities; 1.24, 1.42 and 1.60 Mg/m³. Booster pellets were also prepared from three materials; RDX/AC629 96.4:3.6 at 1.57 Mg/m³, RDX/AC629/zinc stearate 96.25:1.75:2.0 at 1.55 Mg/m³ and granular tetryl at 1.54 Mg/m³. Fuze trains were made up using detonators prepared at MRL with the same explosive content as the standard LZ 2.6 Gr. stab detonator but fired electrically using an ICI match-head igniter.

All fuzes gave dents in the witness plate confirming booster detonation. Full details of dent depths and volumes are listed in Table 4.

The RDX/AC629 99:1 (lead) - 96.4:3.6 (booster) fuzes gave higher output as measured by witness plate dent volume/depth than did the standard all-tetryl fuze. Replacement of the booster by RDX/AC629/zinc stearate 96.25:1.75:2.0 similarly gave better performance than the all-tetryl fuze. In these firings there is only minimal differences between output from fuzes with RDX/AC629 99:1 leads at 1.24, 1.42 and 1.60 Mg/m³ density; derivation of an optimum lead density would require a much larger number of firings than the few carried out here. The firings using the RDX/AC629 99:1 (lead) and granular tetryl (booster) suggest that performance has decreased at lead density 1.6 Mg/m³ but further firings would be needed to substantiate this trend. Direct comparison between crystalline tetryl and RDX/AC629 as lead filling confirms comparable or better performance by RDX/AC629 on either of the RDX-based boosters.

A-5 leads in Fuze M739 are pressed to a density of 1.62 ± 0.07 Mg/m³ [21] and it can be concluded that a comparable RDX/AC629 formulation would function satisfactorily as a lead filling over this density range.

4.2 Fuze Boosters

For applications involving low production rate pressing of large booster pellets or where the booster is pressed directly into the fuze body or component, a higher wax content may be required to give an increased margin of safety (see comments later in Conclusions/Recommendations). Provided automatic pelletting at high production rates is not required, an RDX Grade A/AC629 formulation is also suitable. All formulations containing more than 2% AC629 had F of I > 120 and clearly were sufficiently desensitized. Adequate performance required that shock sensitivity be kept as high as possible. Data for a 96.4:3.6 formulation, and two which might represent extreme production variation, are listed in Table 2.

Impact sensitiveness data for all three formulations indicate excellent desensitization while shock sensitivity still remains high. Confirmation of satisfactory fuze train performance was carried out in Fuze Mk 162 using RDX/AC629 96.4:3.6 as booster, as described in the previous section. All firings resulted in detonation (Table 4) either from RDX/AC629 or tetryl leads. Power output, as measured by witness plate dent volume, was up to 10% greater than for the standard tetryl booster.

4.3 Automatic Pelletting Operations

Normally RDX/wax formulations are difficult to pellet at high production rates unless wax levels are low; both A-5 and Debrix 11 are suitable for automatic pelletting. Our aim was to develop a formulation which could be pelleted at high rates yet contained increased wax/desensitizer levels for enhanced production safety.

Consultation with production staff at MFF St Marys identified three key problems which have to be overcome for an RDX/wax formulation to be suitable for automatic pelletting.

- (a) Inadequate powder flow in the hopper, leading to variability in pellet weight. Low bulk density often is the cause of this problem.
- (b) Buildup of wax deposits on the pressing tools, resulting in progressive shortening of the pellets until they are out of specification range.
- (c) Poor mechanical strength of the pellets which could result in plucking upon ejection from the press or crumbling upon subsequent handling.

Typically, a metal stearate and/or graphite are added to overcome or minimize these problems. In Australian production this has been carried out at MFF St Marys by blending in zinc stearate or graphite prior to addition to press feed hoppers.

Our initial approach was to alter the method of breaking the AC629 emulsion such that a metal stearate could be co-precipitated. We had readily prepared RDX/polyurethane powders from polyurethane dispersions by breaking the dispersion with metal salts of divalent cations [8] and polyethylene wax emulsions had also been successfully broken using divalent cation salts [14,16]. We therefore made two changes to the basic procedure described earlier [9].

- (a) The stirred aqueous RDX/emulsion slurry was neutralised (not taken to pH 4) with acid, then an aqueous solution of a zinc or calcium salt was added to break the emulsion and precipitate zinc/calcium stearate.
- (b) In order that zinc/calcium oleate not be formed in a competing reaction, the oleic acid in the AC629 emulsion was replaced by stearic acid.

This process was carried out using both zinc and calcium chloride and while both processes tended to cause some agglomeration, the problem was much more severe with calcium stearate. As a result work on the calcium

stearate modified formulations was terminated and we concentrated on co-precipitation of zinc stearate; this had the added advantage for production that factory personnel are familiar with zinc stearate which is widely used as a lubricant. The obvious strategy for minimising problems with wax was to minimise the wax content. We accordingly set an upper limit of about 2% AC629, and prepared a range of formulations with differing levels of zinc stearate. Data for these formulations, which are the first five in the table plus two prepared earlier using calcium stearate, are listed in Table 5.

Impact sensitiveness data for these five formulations (plus the two with calcium stearate) demonstrate satisfactory desensitization while shock sensitivity data indicates adequate shock sensitivity. However it was noted in pressing the pellets for gap testing that the three formulations containing higher % zinc stearate than % AC629 (indicated by * in Table 5) had a tendency to pluck and thus were unsuitable for automatic pelleting. It was concluded that adequate mechanical strength for the pellets could only be achieved if there was less zinc stearate than AC629 in the formulations.

Therefore in order to maintain desensitization, AC629 content of the moulding powders was increased and five formulations were shipped to MFF St Marys for trial pelleting. The five formulations were as follows:

RDx/AC629/zinc stearate 95.6:2.75:1.65, 96.15:1.9:1.95 and 96.1:3.25:0.65

RDx/AC629/zinc stearate/graphite 95.9:2.5:1.15:0.45

RDx/AC629 95.95:4.05

All were prepared in 3 kg batches. The graphited composition was prepared by addition of graphite to the RDx/AC629/zinc stearate formulation in the mixer, stirring, then filtration of the product. The trial was conducted on a Manesty E2 automatic press to produce pellets of 0.119 in (3.02 mm) and 0.38 in (9.65 mm) diameter. This was considered a "worst case" by production personnel since these two types of pellets are difficult to press and a composition which performed well here would be suitable for most other applications.

Results of the trial are described in detail in Table 6. The overall conclusion was that none of the formulations performed satisfactorily. Composition 1 (95.6:2.75:1.65) and Composition 4 (graphited) were the best; both exhibited reasonable flow but had a tendency to pluck. The other three compositions had inconsistent to very poor flow, which was not improved by blending in 0.5% graphite prior to addition to the hopper, and pellets were weak. The differences observed between morning (am) and afternoon (pm) processing could have been due to two causes.

- (a) Increased temperature: the pressing area is air conditioned but this is turned off at night. Presumably all the equipment warms up during the day, and the weather was quite hot during the trial.

- (b) Humidity: this might have risen during the day and absorption of water by the composition could cause problems. It was said to be not uncommon for the presses to need resetting in the afternoon.

The recommendation by production personnel at MFF St Marys was that total wax levels needed reducing, bulk density needed to be at least as high and flow improved.

For the second trial, two major changes were made.

- (a) AC629 levels were reduced to about 1%.
- (b) The coating process was changed to depositing the AC629 and zinc stearate in successive operations. In this way the zinc stearate lubricant will be the outermost coating and will minimise flow or sticking problems from the AC629.

Eight formulations were forwarded to MFF St Marys:

RDX/AC629 99:1 and RDX/AC629/zinc stearate 98.5:1.0:0.5
RDX/AC392 99:1 and RDX/AC392/zinc stearate 98.5:1.0:0.5
A-5 and Debrix 18AS
CH-6 and RDX/stearic acid 98.75:1.25 prepared at MRL

The strategy behind the follow up trial was straightforward; check the suitability of formulations containing about 1% AC629, check whether the higher melting wax AC392 [9] offers any improvement, and make a direct comparison with US and UK formulations. The trial was carried out on the E2 Manesty press to produce both 0.161 in (4.09 mm) and 0.38 in (9.65 mm) pellets. The trial duration was extended due to building shutdowns, and in general the weather was cool to cold. Results are detailed in Table 6 as second and third trial.

Production staff rated US A-5 at the best composition. The RDX/AC629 was satisfactory and blending in 0.25% graphite gave performance only slightly inferior to A-5. The RDX/AC629/zinc stearate 98.5:1.0:0.5 formulation performed marginally with some flow problems and pellet mass variability. Blending in 0.25% graphite gave good results. Substitution of AC629 by AC392 gave no improvement for any of the formulations.

Composition CH-6 and "A-5" prepared at MRL were rated as very good. However Debrix 18AS was rated unsatisfactory by production personnel due to very poor pellet reproducibility, even after blending in graphite.

5. COMPARATIVE ASSESSMENT OF US AND UK QUALIFIED FORMULATIONS

Production formulations supplied from both US and UK sources were characterised for powder impact sensitiveness (Rotter F of I) and shock sensitivity (MRL SSGT) on charges pressed nominally to 90 ITMD. Full results from these tests are listed in Table 7.

5.1 Powder Impact Sensitiveness

The UK Debrixxes and US A-5 and CH-6 all have high RDX contents and impact sensitivenesses (F of I) which span the narrow range of 100-120 (Table 7). This can be compared with the minimum accepted figure for tetryl of 90 [22,23] upon which UK fuze safety guidelines [11] are based. A-3, with its much higher wax content, shows the expected increase in F of I to 140. Mean values of evolved gas for ignitions decrease with increased wax content from Debrix 11 (18 mL) to A-3 (1 mL) due to suppression of propagation by increased wax [9,13,22].

Both the US formulations PBXN-5 and PBXW-7 Type II contain 5% Viton A as binder. The F of I values (Table 7) confirm the poor desensitization by fluoropolymers noted previously [20]; PBXN-5 is as sensitive as binderless HMX [22] while PBXW-7, which contains 60% of the very insensitive explosive TATB and 35% RDX, has ignitability only slightly reduced from binderless RDX. PBXN-5 would thus not meet strict UK fuze-safety guidelines [11] while PBXW-7 would do so only marginally. The moderating effect of TATB can be seen in the low mean gas evolution for PBXW-7, indicating suppression of propagation. The poor desensitizing ability of Viton A suggests that PBXN-6 (RDX/Viton A 95:5), which was not supplied for this study, would have impact sensitiveness comparable with binderless RDX. We would consequently not recommend fluorocarbon binders for Australian booster formulations, and preliminary unpublished studies at MRL have confirmed this.

The F of I for both HNS samples was 90. This was unexpected since published US results [12] for HNS (type not specified) for 50% impact heights are double those for RDX on both Type 12 and 12B tooling. Thus HNS would be expected to have an F of I of at least 120.

5.2 Shock Sensitivity (SSGT)

Shock sensitivity determined using the MRL SSGT [24] on unconfined pellets pressed nominally to 90.0 ITMD (Table 7) can be compared with tetryl granular and crystalline by cross-referencing to Table 2. Results for M_{50x}.

L_{95} and std. dev. are quoted in mm of brass shim; the higher the M_{50} , the higher the shock sensitivity.

Debrix 11, A-5, CH-6 and tetryl crystalline are all similar in shock sensitivity. Increase in wax/desensitizer level decreases shock sensitivity; compare Debrix 11, 12 and 18AS. Debrix 18AS displays relatively low sensitivity although substantially increased over the highly waxed A-3. Note also that PBXW-7 is well down on sensitivity. Use of this formulation as a cook-off resistant booster in Australian ordnance would be accompanied by a performance penalty of decreased shock sensitivity.

The two HNS samples exhibit excellent shock sensitivity, particularly HNS Type IIB.

6. CONCLUSION/RECOMMENDATIONS

The production of a range of RDX/AC629 formulations by an aqueous emulsion process has been achieved. AC629 is readily available in Australia, easy to emulsify with morpholine/stearic acid, and the emulsion has a shelf life of at least 3-6 months depending on storage conditions. Existing Australian production equipment is suitable for the coating operation, which is considered to be inherently safer than the hot-waxing process used for the Debrixes. Adoption of processes based on solvent slurry techniques would require modified equipment and increased attention to health and safety to deal with large volumes of toxic/flammable solvents. RDX Grade A is used as feedstock, and no "special" grades of RDX are necessary to give the desired properties. Qualification of these formulations is expected to be straightforward since related HMX (EDC 23 and 24) and RDX (A-3 Type 2) formulations have been qualified in the UK and US respectively, and because of their similarity with existing qualified formulations such as the Debrixes, A-5 and CH-6.

Three formulations have been identified as suitable for replacing tetryl crystalline and granular. These are listed in Table 8 and compared with their US/UK equivalents. Vacuum thermal stability for all formulations (5 g/120°C/40 h) was excellent (see Table 2), as had been noted previously for other RDX/AC629 formulations [9].

6.1 Fuze Leads and Small Pellets

An RDX/AC629 98.75±0.25:1.25±0.25 is recommended. Powder impact sensitiveness is better (lower) than A-5 or Debrix 11, and shock sensitivity is similar to these two formulations and tetryl crystalline (Table 8).

Pellets can be produced automatically at high rates following blending of 0.25% graphite prior to addition to the pressing hopper.

A pilot lot of 100-200 kg should be produced and the formulation (interim) qualified. It should be noted, as discussed in the Introduction, that we cannot produce A-5 to specification within Australia. An Australian RDX/stearic acid 98.75:1.25 is not A-5; qualification for filling US designed fuzes which call for A-5 would still be necessary and no easier than qualifying an RDX/AC629 formulation. In addition Australian production equipment would need substantial modification to produce "Aus A-5".

6.2 Low Production Rate/Large Boosters

As discussed extensively in Section 3, the UK strategy is to use a more highly waxed/desensitized formulation for larger booster pellets. While not endorsing this decision (see also Section 6.3), we have identified the formulation RDX/AC629 95.5±0.5:3.5±0.5 as suitable for replacing Debrix 12 in UK designed fuzes. This formulation could naturally be used for large low production rate boosters in Australian ordnance should the need arise. Comparison of the data in Table 8 reveals that the recommended formulation is comparable or lower in impact sensitiveness than Debrix 12 while possessing enhanced shock sensitivity; both are desirable features.

It is recommended that a pilot production of 100 kg of this formulation be prepared and interim qualified.

6.3 Automatic Pelletting

As discussed in Section 6.1, RDX/AC629 98.75:1.25 can be pelleted at high production rates after blending with 0.25% graphite. Should higher levels of desensitizers be required, a formulation which can be pelleted at high production rates is RDX/AC629/zinc stearate/graphite approximately 98:1.25:0.5:0.25. This has the production advantage that it could be prepared from the lead filling described in 6.1.

It is recommended that an RDX/AC629/zinc stearate/graphite 98.0±0.25:1.25±0.25:0.5±0.25:0.25±0.25 be prepared on a pilot scale and qualified. A suitable procedure would be to take the 98.75:1.25 formulation and blend in the zinc stearate and graphite; zinc stearate and aerosil are blended into Debrix 18 (RDX/wax 97.25:2.75) at the factory to prepare Debrix 18AS.

Comparison with Debrix 18AS can be made from the data in Table 8. Impact sensitiveness is comparable while shock sensitivity for the recommended composition is considerably enhanced. It should be stressed that production personnel at MFF St Marys rated Debrix 18AS as unsuitable for automatic pelletting. While the trial was conducted on small diameter pellets, and

Debrix 18AS is recommended for larger pellets which may be easier to produce, the result is strongly indicative of production difficulties.

The use of the higher melting wax AC392 did not result in better processibility. Given the greater difficulty of emulsifying AC392 [9], further consideration of this wax is not recommended. The AC629 and zinc stearate levels could be increased to some extent to still give a formulation with acceptable pelletting capabilities for larger pellets. However at this stage it is not clear that this is necessary.

7. EXPERIMENTAL

7.1 Materials

The RDX Grade A Class 1 (recrystallized) and Grade B Class 1 (milled and boiled) used in all preparations was received wet from Albion Explosives Factory, Melbourne and dried at the pump prior to use. All other materials were commercially available and used without further purification except morpholine which was redistilled prior to use.

7.2 Emulsions

The AC629 emulsion was prepared exactly as described previously [9] except that stearic acid was substituted for oleic acid. The AC392 emulsion used was remaining from our earlier study [9].

7.3 Moulding Powders

7.3.1 RDX/AC629 (or AC392)

Preparation was at 85°C exactly as described previously [9] except in the case of AC629 where the emulsion was based on stearic acid (see above).

7.3.2 RDX/AC629/Zinc Stearate/(graphite)

(a) One Step Process

The required amount of emulsion is added to a stirred slurry of RDX in water (40% w/v) and then heated to 85°C. H_2SO_4 (approximately 0.75 N) is added dropwise till pH 7, then the required amount (note allowance must be taken of the stearic acid in the emulsion) of a slurry of sodium stearate in water (1:13 w/v) is added. The emulsion is then broken and zinc stearate precipitated by dropwise addition of ZnCl_2 in water (1:20 w/v). About 10 mole percent excess of the stoichiometric quantity of ZnCl_2 is required. Following this procedure solid graphite can be added with stirring. The final product is isolated by cooling, filtering, washing and drying.

(b) Two Step Process

The desired RDX/AC629 formulation is prepared as described in 7.3.1 and washed with water. The still wet material is then transferred back to the reaction vessel to make an approximate 40% w/v slurry with water and heated to 85°C with stirring. An aqueous slurry of sodium stearate (1:13 w/v) is then added followed by a 10% molar excess of ZnCl_2 in water (1:20 w/v). After addition is completed the product is filtered, washed and dried. Graphite powder could be added after the addition of ZnCl_2 solution.

(c) Blending

An RDX/AC629/zinc stearate/graphite formulation can also be made from the required RDX/AC629 powder by first dry blending with zinc stearate then graphite. Alternatively graphite could be dry blended with an RDX/AC629/zinc stearate formulation prepared as above.

7.4 Characterisation of Moulding Powders

7.4.1 Chemical Analysis

Analysis of RDX/AC629/zinc stearate/graphite was carried out in four stages (a-d) described below. Analysis of RDX/AC629 formulations requires only processes a, b and d [9].

- (a) Extraction with cold petroleum ether, bp 40-60°C fraction. This removes stearic acid and low molecular weight fractions in the AC629.
- (b) Extraction with cold acetone. This removes the RDX.

- (c) Extraction with hot (110°C) glacial acetic acid. This removes the zinc stearate.
- (d) Extraction with hot toluene, which removes the remainder of the AC629. There is no residue at this stage except graphite (if present).

Analysis was carried out in triplicate. The stearic acid constitutes about 12% of total wax, but cannot be determined separately from the AC629; wax contents quoted in the text refer to the sum.

7.4.2 Rotter Impact Sensitiveness: Figure of Insensitiveness (F of I)

Impact sensitiveness was determined on a Rotter Apparatus [22] 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 of 50% initiation probability and are rounded to the nearest 5 units. Average gas volumes for positive results are also quoted.

7.4.3 Shock Sensitivity: Small Scale Gap Test

The small scale gap test (SSGT) used at MRL has been described in detail by Wolfson [24]. The donor is a UK Mk 3 exploding bridgewire detonator (EBW) 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 [13].

7.5 Test Firings

7.5.1 Velocity of Detonation by Streak Photography

Explosive pellets were pressed to the required diameter and density on an Eltor press. Firings were conducted by forming a column of pellets of length at least 15 times the charge diameter. An EBW detonator (UK Mk 3) was used to initiate the column and the event was photographed using a Cordin Model 330 Continuous Access Streak and Framing Camera operating in the streak

mode. Photographic traces were analysed by two independent operators using a measuring microscope and an "Opto-scale" manufactured by Bishop Graphics.

7.5.2 Fuze Mk 162 Firings

Components for the firings were prepared as follows:

Detonator: Fuze Mk 162 uses a 2.6 GR, LZ, stab detonator. For ease of firing and alignment, an "identical" electrically fired detonator was prepared by pressing lead azide (0.110 g) and L-mix (0.58 g) into a 4 mm id, 25 mm od, 7 mm high perspex disc at 227 kg dead load. An ICI electric fuzehead was taped to the detonator prior to firing.

Lead: A lead disc identical with that in Fuze Mk 162 was prepared except the hole was centrally placed (not offset) and there was no locking notch. Material filled into the leads was tetryl crystalline at 1.2 Mg/m³ and RDX/AC629 98.94:1.06 at 1.24, 1.42 and 1.60 Mg/m³ (70.7, 80 and 90 ITMD). Pressings were achieved by adding the appropriate mass and using a hand press fitted with a stop so that the given mass just filled the lead.

Booster Pellets: Booster pellets were pressed on a Malco 20 ton press from tetryl granular, RDX/AC629 96.4:3.6 and RDX/AC629/zinc stearate 96.25:1.75:2.00. The respective line pressures and pellet densities were 100 psi/1.54 Mg/m³, 65 psi/1.57 Mg/m³ and 75 psi/1.55 Mg/m³. The pellets weighed approximately 6.5 g and were 26 mm diameter x 7.5 mm high.

Witness Blocks: Cylindrical mild steel blocks of diameter at least 75 mm and height at least 25 mm were used.

The detonator, lead and booster were taped together with masking tape for ease of handling. Firings were conducted by taping the above assembly onto the witness block, taping on the lead, connecting to the firing circuit and initiating remotely.

Dent depths were determined by micrometer. Dent volumes were determined as mass of plasticine to fill the cavity, and are quoted in the text as such.

7.6 Automatic Pelletting

The formulation under test was initially screened by production staff at MFF St Marys for flowability. If flow was considered inadequate graphite (0.25%) or zinc stearate (0.5-1.0%) was added and blended by shaking

in a plastic container. The formulation was then added to the flow hopper of the E2 Manesty press which was operated remotely.

Pellets were measured for height (micrometer) and mass, and mechanical strength was assessed qualitatively by squeezing between the fingers. Visual examination for defects such as plucking, capping and chipping was also made.

8. ACKNOWLEDGEMENTS

A large number of people provided invaluable technical assistance to this programme. Mr E. Wanat and Ms H. Fairweather (Rotter F of I), Mrs S. Spencer (chemical analysis), Mr R. Porteous and Mr M. Maksacheff (pressing of pellets and SSGT), Ms J. Gray (pressing of pellets), Mr M. Wolfson (SSGT and test firings) and Mr K. Lee (test firings) all made important contributions. Mr P. Knight and Mr R. Desmond conducted all the automatic pelletting trials and their extensive experience and expertise made assessment of the formulations extremely straightforward.

Many discussions were held with Dr J. Eadie who gave freely of his extensive knowledge of these types of formulations. Other colleagues who gave valuable advice were Mr M.A. Parry and Mr J. Bentley, while Mr G. Goodman and Mr K. Rowles from EDE provided a number of very useful discussions.

The contributions of all these people is very greatly appreciated.

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TABLE 1

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

Composition	Country of Origin	Formulation
A-3 ^a	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 (Type I) (Type II)	US	RDx (35%), TATB (60%), PTFE (5%) RDx (35%), TATB (60%), Viton A (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 ^a	UK	RDx, wax No. 8; ratio 88:12, 91:9, 93:7

^a Principally pressed main charge fillings

TABLE 2

Sensitivity Data for RDX/Polyethylene Wax Formulations
Being Considered as Tetryl Replacements

Formulation	Impact Sensitiveness F of I (mL gas) <u>a</u>	Thermal Stability T of I (°C) or vac. stab. (mL/5 g at 120°C/40 h)	Shock Sensitivity (mm)			
			ZTMD	M _{50Z}	L _{95Z}	σ
RDX/AC629 Polyethylene Wax						
FUZE LEADS						
98.94 Grade A/ 1.06	115,120 (19)	224°C	90.9	2.769	2.855–2.682	0.040
98.92 Grade B/ 1.08	95 (16)		90.7	2.896	2.939–2.852	0.020
98.69 Grade A/ 1.31	130(19)		91.0	2.593	2.642–2.548	0.022
FUZE BOOSTERS						
96.4 Grade A/ 3.6	125(11)	224°C, 0.69 mL	90.0	1.844	1.897–1.788	0.026
97.15 Grade A/ 2.85	130(14)		90.8	2.276	2.377–2.174	0.048
95.3 Grade A/ 4.7	140(9)	1.78 mL	90.8	1.681	1.737–1.628	0.026
TETRYL						
crystalline	105(16),110 ^b		90.0	2.814	2.858–2.771	0.021
granular	110(16),90 ^b	2.61 mL	90.0	3.259	3.315–3.203	0.026

^a F of I relative to RDX Grade G = 80, evolved gas is average of all positive (go) tests.

^b Data from UK Safety Certificates [23].

TABLE 3

Velocity of Detonation as a Function of Charge Diameter
on Pressed Charges of RDX/AC629 97.8:2.2

Density (Mg/m ³)	Velocity of Detonation (m/s)		
	2 mm diameter	4 mm diameter	6 mm diameter
1.69	8290	8455	8465, 8480
1.64	7970		

TABLE 4

Experimental Results for Firings of Simulated Fuze Mk 162
with Tetryl and RDX/Polyethylene Wax AC629 Fillings

Booster Composition	Witness Plate Dent Volume and Depths ^a for Fuzes Filled with Lead Formulations as Below			
	Tetryl Crystalline ^b 1.2 Mg/m ³	RDX Grade A/AC629 99:94:1.06		
		1.24 Mg/m ³	1.42 Mg/m ³	1.60 Mg/m ³
Tetryl Granular, ^c 1.54 Mg/m ³	1.55(2.20) ^a 1.79(2.36)	1.58(2.00)	1.52(2.05)	1.45(1.80)
RDX Grade A/AC629 96.4:3.6, 1.57 Mg/m ³	1.85(2.51) 1.75(2.66)	1.70(2.50) 1.86(3.01)	1.97(2.80) 2.00(2.80)	1.87(2.75) 1.93(2.81)
RDX Grade A/AC629/ Zinc Stearate 1.55 Mg/m ³	1.55(2.30)	1.82(3.23) 2.04(2.98)	1.57(2.80) 1.82(2.46)	1.79(2.60) 1.79(2.80)

^a All fuzes functioned high order. Dent volumes are mass of plasticine (g) to fill witness plate crater. Numbers in parentheses are dent depths in mm.

^b Specified lead in Fuze Mk 162.

^c Specified booster in Fuze Mk 162.

TABLE 5

Sensitivity Data for Compositions Prepared to Assess their
Potential for Automatic Pelletting

Formulation	Impact Sensitiveness		Shock Sensitivity (SSGT, mm)			
	Rotter F of I ^a	Evolved Gas (mL)	Density ITMD	M _{50%}	L _{95%}	σ
RDX/AC629/Zinc Stearate						
95.15:2.20:2.57*	160	10.7	90.9	1.689	1.758-1.618	0.033
96.13:2.19:1.69	130	11.9	90.8	1.753	1.781-1.725	0.013
97.00:2.12:0.99	150	16.8	90.8	1.806	1.844-1.768	0.018
96.25:1.74:2.01*	150	12.3	91.3	1.915	1.976-1.854	0.028
97.02:1.14:1.85*	125	19.1	91.1	2.141	2.210-2.073	0.033
98.5:1.0:0.5	130	17.5				
98.22: - :1.78	120	17.1	91.0	2.685	2.746-2.621	0.029
RDX/AC629/Calcium Stearate						
95.83:2.01:2.07	130	8.2				
95.99:2.08:2.02	130	12.5				
RDX/AC629/Graphite						
96.66:2.41:0.94	110	17	91.0	2.324	2.426-2.225	0.046
97.16:1.87:0.96	150	16.8				
98.16:1.51:0.33	100	16.5	91.0	2.558	2.604-2.512	0.021

^a Relative to RDX Grade G = 80.

^b Mean of gas evolved for all "go" results, ie ignitions.

* Formulations which gave pellets with poor mechanical strength
(see text).

TABLE 6

Results for Automatic Pelletting Trials Carried
out at MFF St Marys

Composition	Result
<u>First Trial</u>	16/12/86
RDX/AC629/zinc stearate	AM. Composition flowed well in hopper. 0.119 in diam. pellets were strong but had a tendency to pluck on being knocked out. 0.38 in diam. pellets were strong but tended to pluck at top and bottom and were unsuitable.
1. 95.6:2.75:1.65	PM. Flow had deteriorated relative to AM despite resieving.
2. 96.15:1.9:1.95	PM. Inconsistent flow and 0.119 in pellets were often weak. Blending in graphite (0.5%) was a failure since the composition formed soft balls in the hopper.
3. 96.1:3.25:0.65	PM. Flow very poor and could not be tested.
4. RDX/AC629/zinc stearate/graphite	AM. Flow was good. 0.119 in pellets were strong but like Composition 1 stuck to the top of the drift. 0.38 in pellets stuck to the drifts at both top and bottom and was definitely worse than composition 1 due to excessive plucking. Unsuitable.
95.9:2.5:1.15:0.45	PM. Inconsistent flow even after resieving.
5. RDX/AC629	PM. Much lower bulk density than compositions 1 and 4 and flow much poorer. Machine was adjusted to try and make the powder run but 0.119 in pellets were all weak and crumbled. Considered not possible to pellet on this machine. 0.38 in pellets had poor strength and stuck to the pressing plungers. Flow poor. Unsuitable. Blending 0.5% graphite did not improve flow sufficiently.
95.95:4.05	

TABLE 6

(continued)

Composition	Result
<u>Second Trial</u>	26/5/87 to 26/6/87 on various days. All pellets 0.161 in (4.08 mm) diameter.
6. RDX/AC629 99:1	Flow satisfactory but tendency to stick to the sides of the hopper. Pellet quality good, weight range 0.110-0.116 g, length 5.33 mm, density 1.56 Mg/m ³ . Blending 0.25% graphite gave very good flow, good pellets with excellent reproducibility; 0.120-0.121 g, length 4.95 mm, density 1.72 Mg/m ³ . Blended with 0.5% zinc stearate gave a similar result to the graphite blend but bulk density was lower, giving a slight problem.
7. RDX/AC629/zinc stearate 98.5:1.0:0.5	Initial flow problems which settled down after a while. Good pellet quality but pellet mass was wide; 0.140-0.145 g, rerun 0.136-0.142 g. Blended with 0.25% graphite had good flow and pelleted well.
8. RDX/AC392 99:1	Comparable with composition 6. Some variation with pellet mass, 0.137-0.146 g, but rerun more consistent; 0.144-0.146 g, length 5.35 mm, density 1.755 Mg/m ³ . Blending 0.25% graphite did not make much difference; pellet mass 0.139-0.144 g, rerun 0.144-0.146 g, length 6.32 mm, density 1.735 Mg/m ³ .
9. RDX/AC392/zinc stearate 98.5:1.0:0.5	Comparable with composition 7; pellet mass 0.147-0.149 g, length 6.50 mm, density 1.745 Mg/m ³ .
10. Comp A-5 ex US	Excellent flow and pellet quality; pellet mass 0.138-0.140 g, length 6.20 mm. Blending 0.25% graphite gave a slight improvement.
11. Debrix 18AS	Flow satisfactory in hopper but very poor reproducibility of pellets; mass 0.114-0.137 g. Pellet quality good. Production staff conclude unsatisfactory. Blending with 0.25% graphite gave no improvement; 0.108-0.135 g mass range.
12. Comp CH-6 ex US	Good flow and pellet quality, excellent reproducibility; mass 0.152-0.153 g, length 6.50 mm, density 1.75 Mg/m ³ .

TABLE 6

(continued)

Composition	Result
13. RDX/stearic acid 98.75:1.25 ex MRL	Good flow with good pellet quality but a little inferior to A-5. Pellet mass 0.137-0.141 g, rerun 0.133-0.135 g, length 6.05 mm.
<u>Third Trial</u>	25/6/87-29/6/87. All pellets 9.68 mm, pressing load 350 lb.
14. Comp A-5 ex US	Blended 0.25% graphite. Flow good, pellets 0.896-0.907 g, length 7.60-7.70 mm.
15. Comp CH-6 ex US	Blended 0.25% graphite. Flow good, pellets 0.965-0.977 g, length 7.99-8.11 mm.
16. See entry 7 (Trial 2)	Blended 0.25% graphite. Flow good, pellets 0.897-0.901 g, length 7.59-7.65 mm.
17. See entry 9 (Trial 2)	Blended 0.25% graphite. Flow good, pellets 0.947-0.953 g, length 7.91-7.95 mm.
18. See entry 13 (Trial 2)	Blended 0.25% graphite. Flow good, pellets 0.870-0.881 g, length 7.31-7.39 mm.
19. RDX/AC629 99:1	Blended 0.25% graphite. Flow good, pellets 0.705-0.714 g, length 5.88-5.95 mm.
20. RDX/AC392 99:1	Blended 0.25% graphite. Flow good, pellets 0.933-0.940 g, length 7.73-7.83 mm.
21. See entry 1 (Trial 1)	Flow very poor in hopper, unsatisfactory for pelletting.

TABLE 7

Powder Impact Sensitiveness and Shock Sensitivity Data for
Production UK and US Formulations Qualified as
Tetryl Replacements

Formulation	Powder Impact Sensitiveness		Shock Sensitivity (SSGT, mm)			
	Rotter F of I ^a	Evolved Gas (mL) ^b	ZTMD	M _{50%}	L _{95%}	o
Debrix 11	110	13	91.0	2.715	2.807-2.624	0.043
Debrix 12	120	7	90.6	1.730	1.801-1.661	0.033
Debrix 18AS	120	10	90.9	1.496	1.544-1.450	0.022
A-5	110	16	90.0	2.642	2.786-2.497	0.067
CH-6	100	15	90.0	2.600	2.654-2.548	0.025
PBXN-5	60	12	90.0	2.383	2.548-2.220	0.076
PBXW-7 Type II	90	4	90.0	1.415	1.448-1.382	0.015
HNS Type IB	90	7	90.1	2.438	2.525-2.352	0.040
HNS Type IIB	90	12	90.0	2.822	2.873-2.769	0.024
A-3 ^c	140	1	90.9	0.498	0.513-0.480	0.007

^a Relative to RDX Grade G = 80.

^b Mean of gas evolved for all "go" results, ie ignitions.

^c Shock sensitivity is for Australian production A-3.
The US sample was only tested for impact sensitiveness.

TABLE 8

Recommended Formulations for Pilot Lot Production and
Qualification and Comparison with US/UK Equivalents

Formulation/Use	Powder Impact Sensitiveness (F of I)	SSGT (mm) at 90 ITMD
1. <u>Fuze Leads/Small Pellets</u>		
Recommended: RDX/AC629 98.75:1.25	120 ^a	2.64 ^a
Comparison: A-5	110	2.642
Debrix 11	110	2.767
2. <u>Low Production Rate Large Boosters</u>		
Recommended: RDX/AC629 96.5:3.5	120-130 ^b	1.844 ^b
Comparison: Debrix 12	120	1.730
3. <u>Automatic Pelletting</u>		
Recommended: RDX/AC629/ zinc stearate/graphite 98.0:1.25:0.5:0.25	130	Approx 2.5 ^c
Comparison: Debrix 18AS	120	1.496

^a Extrapolated from data for 98.94:1.06 and 98.69:1.31 formulations given in Table 2.

^b Data for a 96.4:3.6 formulation which should be very similar.

^c Extrapolated from data in Table 5.

SECURITY CLASSIFICATION OF THIS PAGE

UNCLASSIFIED

DOCUMENT CONTROL DATA SHEET

REPORT NO.
MRL-R-1089AR NO.
AR-005-184REPORT SECURITY CLASSIFICATION
Unclassified

TITLE

Recommended replacements for tetryl in Australian production
fuzes and related ordnance

AUTHOR(S)

Robert J. Spear,
Victor Nanut, Lance D. Redman
and Ian J. Dagley

CORPORATE AUTHOR

Materials Research Laboratories
PO Box 50,
Ascot Vale, Victoria 3032

REPORT DATE

November 1987

TASK NO.

AIR 85/157

SPONSOR

RAAF

FILE NO.

G6/4/8-3407

REFERENCES

24

PAGES

32

CLASSIFICATION/LIMITATION REVIEW DATE

CLASSIFICATION/RELEASE AUTHORITY
Superintendent, MRL
Physical Chemistry Division

SECONDARY DISTRIBUTION

Approved for Public Release

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KEYWORDS

Tetryl
Fuzes (Ordnance)

Pellets
Nitro Compounds

RDX
Explosives

COSATI GROUPS

600

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ABSTRACT

The final phase of study into RDX-based formulations suitable for replacement of tetryl in Australian ordnance is described. Three formulations using readily available AC629 emulsifiable polyethylene wax and processible on Australian production equipment are identified. It is recommended that pilot batch production and qualification of RDX/AC629 98.75:1.25 for leads and small pellets, RDX/AC629 96.5:3.5 for large boosters and RDX/AC629/zinc stearate/graphite 98.0:1.25:0.5:0.25 for high production rate automatic pelletting be carried out. Performance testing has included propagation in small channels, fuze train function in Fuze Mk 162 and production automatic pelletting. US and UK qualified formulations have been similarly characterised and are compared.

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