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Assessment of Hazard Properties of Tetryl Replacement Formulations

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MRL Technical Report
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Abstract

Pilot production batches of three RDX/wax compositions proposed as replacements for tetryl in fuze systems have been assessed for hazard properties and sensitivity. All were found to be less sensitive to impact and electrostatic spark than tetryl, and to have similar sensitiveness to friction. Their response to thermal stimuli, including cookoff, was similar to RDX. Shock sensitivity, as assessed by small scale gap test, was in the required range for their proposed applications.

The pilot production materials retained their characteristics after twelve months accelerated environmental exposure.

Material from one full scale production batch was also tested, and found to be similar to the equivalent pilot production material.

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Contents

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1. Introduction

Tetryl (CE) has been used for many years as the filling in leads, boosters and magazines of most Australian produced fuzes, and as an intermediate demolition explosive. Because of health and pollution problems associated with production, manufacture in western nations has ceased, and supplies are no longer available.

Both the US and UK have qualified, or are in the process of qualifying replacements for tetryl, but for various reasons the replacements chosen are considered unsuitable for Australian production [1].

In previous work [2] a number of RDX-polyethylene wax compositions were prepared on a laboratory scale, and characterised for impact sensitiveness and shock sensitivity. Further batches of selected formulations were then prepared and assessed more comprehensively; this assessment included suitability for automatic pelleting at a production facility [1]. This latter work resulted in the identification of three formulations as suitable for replacing tetryl. These were a 98.75/1.25% RDX/wax (designated TR1) for fuze leads and small pellets, a 96.5/3.5% RDX/wax (designated TR2) for large boosters, and TR1 with added zinc stearate and graphite (designated TR1SG) for automatic pelleting.

Before introduction into service, the proposed formulations are required to be assessed for safety and suitability [3]. This report describes the tests carried out, principally on pilot production material, to characterise and provide data on the three formulations. The purpose of these tests is to provide information to permit the qualification of the explosive materials for service use. Type qualification, to permit their use in specific munitions, requires that the explosives be assessed as part of the design of each munition and be shown to be safe and suitable for service in conjunction with that munition.

The test program is based on test requirements set out by the Australian Ordnance Council [3].

2. Experimental

2.1 Materials

Pilot scale batches of RDX-polyethylene wax formulations were produced at Explosives Factory Maribyrnong [4] and full scale production batches at Albion Explosives Factory.

RDX Grade A Class 1 (recrystallised) [5] was used for all batches.

AC629 emulsifiable polyethylene wax was supplied by Allied Chemicals. The AC629 emulsion was prepared as described in [4].

The RDX-wax formulations TR1 and TR2 were prepared by the aqueous slurry process [4]. TR1SG was prepared by blending 0.5% zinc stearate and 0.25% graphite with selected batches of formulation TR1.

For each of the formulations recommended in [1], ranges of wax content were proposed. These were:

TR1 1.25 ± 0.25%
TR2 3.5 ± 0.5%.

Samples to be assessed in this study were selected from pilot scale production batches representing, as far as availability permitted, the extremes of the proposed specification range of wax contents. This approach is in accord with US policy on qualification of explosive materials for military use [6].

A limited series of tests was also performed on samples selected from two full scale (nominal 900 kg) production batches of formulation TR1 manufactured at Albion Explosives Factory in February 1989.

The wax contents of all samples are shown in Table 1.

Table 1: *Materials Tested*

Designation	Wax Content	
	Nominal	Actual
TR1 Pilot Batch 2	1.25%	1.07%
TR1 Pilot Batch 5	1.25%	1.26%
TR1 Pilot Batch 3	1.25%	1.37%
TR1 Production Batch 1	1.25%	1.18%
TR1 Production Batch 2	1.25%	1.17%
TR1SG Pilot Batch 2SG	1.25%	1.07%
TR1SG Pilot Batch 5SG	1.25%	1.26%
TR1SG Pilot Batch 3SG	1.25%	1.37%
TR2 Pilot Batch 9	3.5%	3.23%
TR2 Pilot Batch 7	3.5%	3.48%

2.2 *Test Methods*

2.2.1 *Composition*

The composition of each sample of formulation TR1 and TR2 was determined by sequential extractions with cold petroleum ether, cold acetone and hot toluene by the procedure described in [1]. All wax contents quoted refer to the sum of the AC629 wax plus stearic acid. As TR1SG was prepared by addition of zinc stearate and graphite to selected batches of formulation TR1, the determination of wax was not repeated on the TR1SG samples.

2.2.2 *Thermal Stability*

Vacuum thermal stability was determined with the standard evacuated mercury manometer apparatus [7] using duplicate 5 g samples. Samples were tested at 120°C. The gas evolved in the first 1.5 hours heating was ignored, then readings were taken at intervals over the next 40 hours. Results are expressed as cm^3 per 5 g sample over the 40 hour period.

2.2.3 *Powder Sensitiveness Tests*

The standard powder sensitiveness tests required for preparation of a safety certificate were used to assess the response of the materials in unpressed form to mechanical, thermal and electrical stimuli.

These tests are the Rotter Impact, Mallet Friction, Temperature of Ignition, Ease of Ignition (Bickford Fuze), Train Test, and Electric Spark Test, performed in accord with [8], and the MRL Glancing Blow Test [9].

In the Rotter Impact test a sample of approximately 30 mg placed between a hardened steel anvil and a brass cap is impacted by a falling 5 kg weight. The median drop height for 50% response is expressed as a "Figure of Insensitiveness" (F of I), and is the ratio of the median drop height of the sample to that of a standard RDX, to which is assigned an F of I value of 80. The mean volume of gas evolved by all positive responses is also reported, and is usually interpreted as an indication of the degree of propagation.

The effect of the presence of grit on sensitiveness to impact is assessed by performing the Rotter test on samples to which set proportions of a standard alumina grit have been added.

In the Mallet Friction test, a small sample of the explosive is spread on anvils of wood, metal and stone, and struck a glancing blow with hand held wood and mild steel mallets. Responses, either audible or visible, are expressed as a percentage out of ten tests, and because of the non-quantitative nature of the test, are rounded to 0, 50 or 100% response.

The Temperature of Ignition test consists of heating 200 mg samples of the explosive in open borosilicate glass tubes at a controlled rate of 5°C per minute until an event occurs.

The Ease of Ignition (Bickford Fuze) test assesses the susceptibility of the explosive in open glass tubes to ignition by incendive sparks from a gunpowder filled fuze. The nature of the response is observed.

In the Train Test (Behaviour on Inflammation) an unconfined trough of explosive is ignited by a naked flame. Observations are made of the liability of the explosive to burn, and how vigorously it does so.

In the Electric Spark test, samples, lightly confined between copper electrodes, are subjected to electrical discharges of 4.5, 0.45 and 0.045 joules. The lowest energy at which ignition occurs is recorded.

MRL Glancing Blow test. In this test a frictional impact is applied to a layer of explosive, spread on a flat anvil, by a spherical ended cylindrical weight sliding freely down an inclined guide. Materials normally used for anvils are steel and bakelised cloth, and for weights steel and brass. The energy of impact is calculated from the mass of the weight and the height of fall, and the lowest energy at which ignitions are obtained is reported.

2.2.4 *Shock Sensitivity*

Shock sensitivity of pressed pellets was determined by the MRL Small Scale Gap Test (SSGT) [10]. This test uses a UK Mk 3 exploding bridgewire detonator as donor, with brass shim attenuators. The acceptor is two unconfined 12.7 mm diameter by 12.7 mm high pressed cylinders, with a mild steel witness block. Results are given in mm of laminated brass shim for 50% detonation probability, determined by the Bruceton staircase technique from 20 to 30 firings.

2.2.5 *Cookoff Test*

Response of the pressed materials to thermal initiation was assessed by the Super Small-Scale Cookoff Bomb (SSCB) [11]. In this test a sample of about 20 g of explosive, in the form of cylindrical pellets 15.9 mm diameter and totalling 63.6 mm in length, is heated at a controlled rate under moderate confinement. Response is assessed by the nature of the damage or fragmentation of the bomb assembly and is usually classed as mild burning, deflagration, explosion or detonation. All samples were pressed to nominal 90% TMD. Samples were tested at both the fast heating rate, at which a temperature of 300°C is reached in about 5 min, and the slow heating rate, at which 230°C is reached in about 30 min.

2.2.6 *Mould Growth*

Pressed pellets were subjected to the mould growth test described in [12], in which the material under test is exposed to a mixed culture of mould spores for 28 days at 24 to 28°C and 88 to 98% relative humidity.

2.2.7 *Effects of Accelerated Environmental Exposure*

Formulations in both powder and pressed pellet form were subjected to ISAT "A" conditions [13] in a climatic chamber for up to 12 months. Samples of powder were withdrawn at intervals of 3 and 6 months and assessed for changes in impact sensitiveness. After 12 months powder samples were tested for impact and friction sensitiveness and thermal stability, and pellets were assessed for shock sensitivity and physical properties.

3. Results

3.1 Composition

The wax content of each material as determined by analysis is given in Table 1. Batches from pilot scale production were deliberately selected to represent a range of wax contents for each formulation, to provide some assurance that variation in wax content did not adversely affect hazard properties or sensitivity.

In order to check uniformity of wax distribution in the full scale production material, six drums of production batch 1, chosen at random, were sampled. Each sample was analysed in duplicate. All results (Table 2) were within the proposed 1.25 ± 0.25% range proposed for the TR1 formulation. There was no evidence, within the limits of uncertainty of the analytical method, of uneven wax distribution in the samples examined.

Table 2: Variation in Wax Content in TR1 Production Batch 1

Sample	Wax Content (%)
A	1.27, 1.03
B	1.25, 1.25
C	1.24, 1.25
D	1.21, 1.21
E	1.09, 1.11
F	1.15, 1.20

3.2 Stability

The results of vacuum stability tests are given in Table 3. All samples gave gas volumes well within the range considered acceptable for compatibility of materials to be used in intimate mixture with RDX, and are therefore considered to have acceptable thermal stability.

Table 3: Vacuum Stability

Material	Evolved Gas (cm^3/5 g)
TR1 Pilot Batch 2	0.2, 0.2
TR1 Pilot Batch 5	0.2, 0.3
TR1 Pilot Batch 3	0.3, 0.2
TR1 Production Batch 1	0.3, 0.4
TR1 Production Batch 2	0.4, 0.3
TR1SG Pilot Batch 2SG	0.2, 0.2
TR1SG Pilot Batch 5SG	0.2, 0.1
TR1SG Pilot Batch 3SG	0.1, 0.1
TR2 Pilot Batch 9	0.8, 0.8
TR2 Pilot Batch 7	0.8, 0.9

3.3 Powder Sensitiveness

Powder sensitiveness (Safety Certificate) test results are given in Table 4. The pilot batches given full safety certificate tests (batches 5, 5SG and 7) represented wax contents very close to the specified median wax content. Full safety certificate tests were also performed on Production batch 1. Included for comparison are results for tetryl [14].

Rotter F of I for each formulation was consistent with that obtained previously on laboratory prepared materials [1] and was independent of wax content within the range of formulations examined. The volumes of gas evolved were, as previously observed [1, 2], lower with higher wax content.

The effect of added grit was to increase the sensitiveness to impact to approximately the same level as that resulting from addition of grit to uncoated RDX [8]. This would be expected of formulations containing relatively small amounts of desensitising material such as wax.

The response to the mallet friction test and MRL glancing blow indicate that all the materials have similar sensitiveness to friction, and are not significantly different to tetryl.

The responses to thermal stimuli (temperature of ignition, ease of ignition and behaviour on inflammation) were, as expected, controlled by the thermal decomposition of RDX, and are no different to the responses reported for RDX [15].

All formulations failed to respond to the electric spark test, and are thus regarded as insensitive to ignition by electrostatic spark.

3.4 Shock Sensitivity

Shock sensitivity results as assessed by the SSGT are given in Table 5. All materials were pressed to a nominal 90% TMD. Comparative results for the recommended formulations, taken from Table 8 of [1], are included. Although TR1 pilot scale

Test	Material TR1 Pilot Batch 5	TR1 Production Batch 1	TR1SG Pilot Batch 5SG	TR2 Pilot Batch 7	Tetryl (3) Crystalline	Tetryl (3) Granular
Rotter Figure of Insensitiveness	150 ($16\ cm^3$)	120 ($18\ cm^3$)	140 ($13\ cm^3$)	150 ($7\ cm^3$)	110	90
F of I with 0.1% added grit	-	25	-	-	-	-
F of I with 1% added grit	-	21	-	-	-	-
Mallet Friction (1)	0,0,0:50,0,0	0,0,0:0,0,0	0,0,0:50,50,0	0,0,0:0,0,0	0,0,0:50,0,0	
Glancing Blow (2)	> 11 J	> 11 J	> 11 J	> 11 J	-	-
Temperature of Ignition	222°C	211°C	214°C	214°C	173°C	171°C
Ease of Ignition	Fails to ignite	Fails to ignite	Fails to ignite	Fails to ignite	Partial ignition	
Behaviour on Inflammation	Ignites and supports train steadily	Ignites and supports train steadily	Ignites and supports train steadily	Ignites and supports train steadily	Ignites but supports train for one inch only	
Electric Spark	No ignitions at 4.5 J	No ignitions at 4.5 J	No ignitions at 4.5 J	No ignitions at 4.5 J	Ignitions at 4.5 J but not at 0.45 J	No ignitions at 4.5 J
Safety Certificate Reference	MRL No 97	MRL No 113	MRL No 98	MRL No 103	UK No 1124(A)	

NOTE 1: Reported as % ignitions with: Wood mallet on stone, hardwood, softwood anvils. Steel mallet on steel, naval brass, aluminiur ›ronze anvils.

NOTE 2: Surfaces tested: Steel on steel; brass on steel; steel on bakelised cloth; brass on bakelised cloth.

NOTE 3: Data from Reference [14]. Separate results are given for crystalline and granulated tetryl where applicable. Where a single result is given it is applicable to both types.

material had a slightly reduced sensitivity the full scale production material was almost exactly as predicted. Pilot scale composition TR2 was slightly more sensitive than the laboratory material. All shock sensitivities were well within the ranges required for the proposed applications.

Table 5: Shock Sensitivity

Material	Shock Sensitivity (mm brass gap)			
	%TMD	$M_{50\%}$	$L_{95\%}$	σ
TR1 Pilot Batch 5	90.0	2.48	2.41-2.55	0.033
TR1 Production Batch 1	90.0	2.68	2.59-2.77	0.043
Laboratory Scale, 1.25% Wax (1)	90	2.64	-	-
TR1SG Pilot Batch 5SG	89.9	2.41	2.35-2.46	0.026
Laboratory Scale, 1.25% Wax with Zinc Stearate and Graphite (1)	90	Approx. 2.5	-	-
TR2 Pilot Batch 7	90.0	2.14	2.09-2.19	0.024
Laboratory Scale 3.5% Wax (1)	90	1.84	-	-
Tetryl: (1)				
Crystalline	90.0	2.81	2.77-2.86	0.021
Granular	90.0	3.26	3.20-3.32	0.026

NOTE 1: Data from Reference [1].

3.5 *Cookoff*

Responses to the SSCB test are summarised in Table 6, with results for tetryl from [11] for comparison. As the objective of the tetryl replacement task was to develop materials no more hazardous than tetryl, no attempt was made to obtain a less violent response to cookoff. Development of a booster material to meet "insensitive" munitions requirements is a longer term objective which is being addressed separately. It was expected that in the SSCB test these materials would give the responses characteristic of RDX and the test was therefore performed only on two materials. TR2 batch 7 was selected as the material with highest wax content, and the test was also performed on full scale production batch 2.

The response to all cookoff tests was with one exception detonation. The temperatures of reaction of both materials at the slow heating rate were close to that of thermal decomposition of RDX. Reaction temperatures of TR1 production material at the fast heating rate were similar to tetryl, and slightly lower for TR2 pilot scale material. In comparing the type of response to the responses obtained for tetryl, it must be remembered that, because of loss of sample, the SSCB is not considered suitable for evaluating the response of materials which melt at temperatures considerably lower than that at which reaction occurs. The reported responses for tetryl at the slow heating rate are not considered to represent a real situation where loss of molten material could be prevented by confinement [11].

Table 6: Response to Cookoff Test

Composition	Heat Rate	Temperature (°C) (1)	Cookoff Reaction
TR2 Pilot Batch 7	Fast	226	Explosion
		231	Detonation
	Slow	218	Detonation
TR1 Production Batch 2	Fast	239	Detonation
		245	Detonation
	Slow	221	Detonation
Tetryl (2)	Fast	257	Detonation
		238	Detonation
		239	Detonation
	Slow	205	Burning
		196	Deflagration
		197	Deflagration

NOTES 1. Temperature is the explosive surface temperature
2. Results from Reference [11]

3.6 Mould Growth

Pellets of TR1 (pilot batch 5), TR1SG (pilot batch 5SG) and TR2 (pilot batch 7) were subjected to the mould growth test. There was no evidence of mould growth on any samples.

3.7 Effects of Aging

The effects of exposure to ISAT "A" conditions were assessed on both powder and pressed pellets. Although it is recognised that the ISAT "A" cycle may be unduly severe, it is useful in providing an early indication of potential degradation problems. Items which survive six to twelve months of this cycle are unlikely to suffer from unacceptably short service lives. Predictive life trials, if required, would need to be performed on material in the planned weapon system configuration, under test conditions derived from the predicted service environment.

The results given in Table 7 indicate that there was no evidence of increase in hazard properties over a period of twelve months exposure. Changes observed in F of I are not regarded as significant, as all remained well above the figure for tetryl. Mallet friction results indicate a trend towards decreased friction sensitiveness, but in view of the subjective nature of this test, this should not be regarded as more than assurance that friction sensitiveness has not increased with aging. Compositions TR1 and TR1SG showed small increases in gas evolved in the vacuum stability test, but the gas volumes were well within the range regarded as acceptable for unaged materials.

Table 7: *Effect of ISAT "A" Cycling*

Material	Test	Duration (months)			
		0	3	6	12
TR1 Batch 2	Figure of Insensitiveness	130	120	120	120
TR1 Batch 5	Vacuum Stability (cm^3/5 g)	0.2,0.3	-	-	0.7,0.8
	Figure of Insensitiveness	150	130	150	110
	Mallet Friction	0,0,0:0,0,50	-	-	0,0,0:0,0,0
	Small Scale Gap Test (mm)	2.48 ± 0.033	-	-	2.57 ± 0.018
	Density (% TMD)	90.0	-	-	87.7
TR1 Batch 3	Figure of Insensitiveness	150	120	130	140
TR1SG Batch 2SG	Figure of Insensitiveness	140	120	140	140
TR1SG Batch 5SG	Vacuum Stability (cm^3/5 g)	0.2,0.1	-	-	0.6,0.5
	Figure of Insensitiveness	140	130	130	130
	Mallet Friction	0,0,0:0,50,50	-	-	0,0,0:0,0,0
TR1SG Batch 3SG	Figure of Insensitiveness	130	150	120	130
TR2 Batch 9	Figure of Insensitiveness	150	150	140	180
TR2 Batch 7	Vacuum Stability (cm^3/5 g)	0.8,0.9	-	-	0.8,0.8
	Figure of Insensitiveness	150	150	150	140
	Mallet Friction	0,0,0:0,0,0	-	-	0,0,0:0,0,0
	Small Scale Gap Test (mm)	2.14 ± 0.024	-	-	2.02 ± 0.029
	Density (% TMD)	90.0	-	-	88.6

Shock sensitivity of TR1 increased slightly, while that of TR2 decreased. However, both remained within the expected range for materials of these wax contents.

Pressed pellets of nominal dimensions 12.7 mm × 12.7 mm were inspected before and after environmental exposure for evidence of deterioration in cohesiveness, and checked for dimensional changes. Cohesiveness was assessed by tendency to crumble on handling, and under finger pressure. There was no observable difference in cohesiveness between aged and newly pressed pellets. Aged pellets showed a slight discolouration. A decrease in density was observed, and was found to be almost entirely due to increase in dimensions of the pressed pellets. In the case of composition TR1, which showed the greater decrease in TMD, there was an average increase of approximately 1% in both length and diameter. Whether this would constitute a problem in a weapon system would have to be assessed as part of the qualification of the composition in that system.

Powders showed some caking after twelve months exposure, but the material could easily be broken up by sieving.

4. *Conclusions*

Pilot scale production batches of proposed tetryl replacement compositions TR1, TR1SG and TR2 have been assessed for powder sensitiveness, shock sensitivity and thermal stability. All are significantly less sensitive to drop weight impact than tetryl, have similar friction sensitiveness, and are insensitive to electrostatic discharge. Their responses to thermal stimuli are similar to that of RDX. Shock sensitivity, although slightly lower than that of tetryl, is in the required range for their proposed applications, and is similar to that of earlier laboratory scale material. Thermal stability is considered satisfactory.

After twelve months exposure to ISAT "A" conditions, there was no evidence of significant change in any of the above characteristics of pilot scale material, from which it is considered that the materials are likely to possess reasonable service lives, and are not likely either to become more hazardous or suffer a loss in shock sensitivity on aging.

The full scale production material was found to possess similar thermal stability, sensitiveness and sensitivity characteristics to pilot scale TR1. Although it was not subjected to accelerated aging, it is considered that its behaviour on aging is not likely to be inferior to that of the pilot scale material.

It is considered that the materials are safe and suitable for general service use. Subject to their successful qualification in specific weapon systems, they are considered to be satisfactory as replacements for tetryl.

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