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DEPARTMENT OF DEFENCE DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION MATERIALS RESEARCH LABORATORIES **MELBOURNE, VICTORIA**

REPORT

MRL-R-948

A STUDY OF THE FEASIBILITY OF DEVELOPING A LOW SENSITIVITY CC CAP FOR RAUFOSS 20 MM AMMUNITION

> Robert J. Spear and Lance D. Redman

AR-004-196







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A STUDY OF THE FEASIBILITY OF DEVELOPING A LOW SENSITIVITY CC CAP FOR RAUFOSS 20 MM AMMUNITION

> Robert J. Spear and Lance D. Redman

ABSTRACT

The possibility of developing a conducting composition (CC) cap which meets the performance specifications of the M52A3B1 CC cap yet possesses significantly decreased sensitivity to initiation has been examined. Twocomponent mixes consisting of a primary explosive and a conducting component were investigated. Five materials were assessed as alternatives to normal lead styphnate RD1303 but all were found to be unsatisfactory either through excessively high sensitivity to energy delivered by capacitor discharge or excessively long functioning times. RD1303 of smaller crystal size than the normal production type also leads to undesirable sensitivity increases. Five graphites/carbon blacks were assessed in admixture with RD1303. Merck synthetic graphite and Tintacarb 90 and 140, which combine properties of both carbon blacks and graphites, produced mixes which met specification and exhibited substantially decreased sensitivity. Further testing of mixes based on these materials is recommended.

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COSATI GROUPS

ABSTRACT

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SENSITIVITY CC CAP FOR RAUFOSS 20 MM AMMUNITION

INTRODUCTION 1.

Electrical initiation is one of the most common methods employed for ignition of propellant and explosive trains. In this process, electrical energy is degraded to heat which in turn raises the temperature of a small quantity of (usually primary) explosive above its ignition temperature, resulting in ignition. Although various systems could be designed to achieve this, in practice two systems are used almost exclusively: bridgewire (BW) and conducting composition (CC). A BW device consists of a small metallic filament connected across two electrodes and embedded in pressed or plastic bonded explosive. Passage of current heats or explodes the filament and ignites the explosive. CC devices have two electrodes separated by a gap across which is pressed an explosive composition made conductive by admixture of (usually) graphite. Passage of current above a threshold level results in ignition.

The electrical characteristics of both devices depend on a number of variables such as electrode configurations, pressing load (for CC), filament properties (for BW) and the percentage and physical properties of the conducting material (for CC). In practice, it has been much more difficult to control these parameters for CC systems, resulting in production batches having a wide range of resistances and consequently considerable variation in electrical sensitivity. Despite these technical limitations, CC devices are cheap to produce, robust, operate reliably under extremes of climate, have fast functioning times and can be designed to be very sensitive e.g., reliable function from about 10 μ J. As a consequence, the major application for CC devices is in cartridge case primers (CC caps) for high rate of fire small calibre munitions and fast reaction time/limited power supply electronic fuzes. Other applications in the continuum of electrical initiation systems are largely met by BW devices.



Three CC caps are currently used in Australian ordnance. These, detailed in Table 1, are the M52 DEFA cap used in 30 mm cartridge cases, the N8 igniter which is a fuze component and the N43 primer used in the Naval 4.5 in cartridge. All three use lead styphnate/graphite as the conducting increment with, in the case of the M52 DEFA and N43, a second (priming) increment. The difficulty of controlling cap parameters can be seen from the wide resistance range specifications, particularly for the M52 DEFA and N43 primers (Table 1).

The RAAF is currently acquiring the F/A-18 tactical fighter which is fitted, as part of its total armament, with 20 mm cannon. These cannon fire Raufoss 20 mm ammunition which are primed with a CC cap of US design, the M52A3B1, details of which are listed in the far column of Table 1. The M52A3B1 differs in two major ways from current Australian CC caps: the conducting and priming increments are not separate, with the multi-component filling serving both roles, while the resistance specification is high with the range specified being particularly large. Local manufacture of the Raufoss 20 mm round is due to commence within 2 years.

Both CC and BW devices are susceptible to accidental initiation by electromagnetic energy coupling into the firing system. Such energy can be picked up from rf transmitters such as radar, telecommunication, navigation and survey equipment and represents a particular hazard at airfields and on board ships. Limited sensitivity testing on the M52A3B1 cap at MRL [1] and more extensive UK testing [2] has shown sensitivity significantly higher than current in-service CC caps such as the M52 DEFA. As a consequence, a study was undertaken at MRL to examine the feasibility of producing a CC cap of relatively low sensitivity which met the performance requirements of the M52A3B1 cap. This report describes the first stage of the study.

2. HISTORICAL BACKGROUND

The development of lead styphnate based CC primers largely arose out of the need during WWII to replace unsatisfactory graphite - "A" composition (mercury fulminate based) fillings in aircraft 20 mm cannon ammunition. Much of the work extending from this period is still covered by security classification, with the scarcity of data available from US sources probably resulting from this limitation. A coverage of UK work can be obtained from Refs. [3-12] and some additional references cited therein. The only US data found during a routine literature survey was the lead styphnate based composition cited in Ref. [13], which is very similar to that used in the M52A3B1 cap, and some work described in patent [14]. The related field, CC detonators, where fillings are usually lead azide/graphite or lead azide/metal powder based, has also been extensively examined. Descriptions of some UK [10,15,16], US [17-21] and German [22,23] work can be found in the references cited. No attempt has been made to exhaustively search the patent literature on CC detonators or primers and references have presumably been missed. Besides this background literature, two recent Australian studies derived from factory production problems concerning CC primers were also available [24,25].

Most of the work on CC primers described above has been directed to caps of moderate to high sensitivity. This was a consequence of their historical requirement in on-board aircraft armaments, where power supply was limited and sensitivity had to be high for reliable functioning, and applications in fuzing where power supply limitations are/were important. It should also be noted that most of the patent literature, both on CC primers and detonators, is similarly targeted to applications where low firing energies and fast functioning times were required. Although investigations into less sensitive CC devices have been undertaken in the past, they have largely not been carried through to the development stage. Exceptions include the N43 primer, which has a no-fire level greater than 100 μ J and is thus comparable with the more sensitive BW devices such as the T30 detonator, while the British have developed a range of lower sensitivity N8 igniters [7,10,12]. A review of UK work in the area of low sensitivity CC devices can be found in Ref. [10].

3. EXPERIMENTAL APPROACH

The concept of an insensitive CC cap is thus not a new one. It has been examined previously at MRL in a limited study [26], where N8 igniter bodies were filled with two component mixes of monobasic lead styphnate RD1349 and two types of graphite as the conducting component. The aim was to investigate conducting composition igniters which would primarily be insensitive to dc current*, with the possibility of meeting the 1A/1W no-fire requirement contained in (for example) the US design requirement MIL-I-23659C for electric initiators. The initial study [26] was successful in meeting this criterion, but sensitivity of the devices to energy delivered by capacitor discharge** was still high with 50% fire probability below 100 µJ.

The aim of the present programme was to examine the feasibility of developing a low sensitivity CC cap as replacement for the M52A3B1 in 20 mm Raufoss ammunition. The first stage, reported here, was directed solely to initiation properties of a range of CC fillings. It was recognised that a filling possessing the desired low sensitivity to electrical initiation might not necessarily possess the required priming properties. Although the filling in the M52A3B1 (Table 1) serves the dual function of initiation and priming, it was envisaged that the final choice of cap filling would possibly be two increments, cf. M52 DEFA (Table 1). A suitable primer filling was not seen as a major problem. Possibilities include the actual M52A3B1 filling, the M52 DEFA priming increment or a modified alternative composition. This would be examined at a later stage of the programme.

* This will be referred to as power sensitivity throughout this report.

** This will be referred to as energy sensitivity throughout this report.

The initial investigation into the conducting increment was divided into two sections: an investigation of possible alternatives to normal lead styphnate RD1303, the basic component of most current CC cap fillings (Table 1), and an alternative conducting component. The overall goal was a cap with low sensitivity to energy (delivered by capacitor discharge), power, and rf. Typical rf energy sources can be either continuous wave (CW) or pulsed. Sensitivity to CW input is directly related to power sensitivity while sensitivity to pulsed energy sources is related to energy sensitivity. Since CC caps are satisfactorily insensitive to power (even when quite sensitive to energy, see for example ref. [26]) our investigation concentrated on energy sensitivity with power sensitivity being much less rigorously pursued. Note that this is the opposite of the earlier study [26]. The specific targeting of the project imposed one particular constraint i.e., the specification (MIL P-1394E) of the M52A3B1 that "the cap should function in less than 0.3 ms when fired by a 10 µs energy pulse from a 2 µF capacitor charged to 160 V." All experimental caps had therefore to meet this criteria. Unfortunately no unfilled M52A3B1 caps were available within Australia during this first stage of the project and all investigations utilized M52 DEFA caps. Although both caps are similar in size and design, (Fig. 1), critical parameters such as electrode gap width and length are different. Results from a general feasibility study should be broadly applicable to both caps. However later stages of the project where a narrowing down process should result in selection of one or two candidate compositions for production and service qualification must be carried out on M52A3B1 caps.

4. RESULTS AND DISCUSSION

4.1 Possible Alternative Materials to Normal Lead Styphnate RD1303

Five materials were chosen for assessment as alternatives to RD1303: monobasic lead styphnate RD1346 and RD1349, barium styphnate RD1320(B), lead azotetrazole RD1355 and potassium picrate. The choice was limited to these materials because they are in current service use and hence readily available, while as a "series" their total range of initiation and explosive properties is sufficiently wide to encompass a number of other potential materials. If any of these five materials exhibited promising properties, further related materials could subsequently be examined.

The five explosives were each mixed with foliac graphite in the ratio 93:7 by weight. The choice of foliac graphite and at that percentage arose from previous experience [26] and limited exploratory testing; a number of other graphites or carbon blacks could equally have been chosen to provide a comparison between the various primary explosive materials. The mixes were pressed into M52 DEFA caps, then lead nitrate was added and pressed on for consolidation. Lead nitrate was used rather than a primer mix for two reasons; since only the conducting mix was being examined in this study, the total explosive content was minimised, while the consolidation that the conducting mix would experience from the pressed primer increment was effectively reproduced. The resistance of the caps was measured after pressing and again 1 week later, then energy sensitivity was assessed by standard Bruceton staircase testing and functioning times were determined using the 2 μ F/160 V/10 μ s pulse as specified in MIL-P-1394E for the M52A3B1. Results are detailed in Table 2; the corresponding RD1303/foliac graphite mix is included for comparison.

The replacement of RD1303 by the other styphnates leads to quite marked changes. The monobasic RD1346 and RD1349 both exhibit increased sensitivity over RD1303 (lower 50% firing energies) and longer functioning times; both are undesirable features. It should be noted that substitution of the "inert" lead nitrate increment by a priming increment leads to reduction of functioning times by up to 50%. However, the functioning times of both the RD1346 and RD1349 mixes are sufficiently longer than the RD1303 mix to rule out further testing of these materials. This is not a completely unexpected result since ignition of monobasic lead styphnates is characterised by good flash but comparatively low propagation speed. As a consequence none of the LDNR types were tested since functioning times would similarly be expected to be long. The barium styphnate mix (Table 1) possesses the desired sensitivity decrease (50% energy >2000 µJ) but again functioning times are long. The lead nitrate had to be replaced by a priming increment (DEFA primer) since the low flash output from the barium styphnate was insufficient to trigger the photocell, yet the functioning time so obtained was still outside specification. Note the wide resistance ranges and high resistance of caps filled with the barium styphnate mix (Table 1). This is symptomatic of poor mixing, a fact easily confirmed by optical microscopy.

The remaining two materials were chosen with the knowledge that they would probably not be suitable for use in CC primers. The lead azotetrazole RD1355/foliac graphite mix (Table 1) exhibited diminished energy sensitivity relative to the RD1303 mix but flash output was so poor that the photocell would not trigger. The problem was overcome by incorporation of a primer increment; the primer was subsequently reliably ignited and functioning times were satisfactory. RD1355 is used primarily in stab and CC detonators [16]. Its violent output together with the possibility of transition to detonation would suggest that RD1355 is unsuitable for use in primers, although careful design could overcome these problems. The potassium picrate mix (Table 2) displays very substantially diminished energy sensitivity but the long functioning times would preclude its use in the projected application.

Normal lead styphnate would thus appear to be the only readily available material suitable for the intended application, but some advantages might conceivably be gained by variation in particle size distribution. The RD1303 used here was the "normal" production type with an average particle size of 115 μ m, which is significantly larger than most graphite/carbon black particles. Specifically, the use of a normal lead styphnate with particle size more similar to that of the graphites may promote better mixing and ultimately better performance. This particular question has been addressed by Sheridan [25] in a study of three types of normal lead styphnate:- average particle size 110 μ m (cf the 115 μ m RD1303 used here), 55 μ m and 30 μ m. Sheridan studied mixes of these three lead styphnates with a 1:1 mixture of Merck synthetic and NK natural graphite. It was found that as the lead styphnate particle size decreased, the resistance of the pressed conducting mixes increased appreciably. For example, the resistance of the pressed mixes ntaining 3.4% graphite changed from average 2.6 Ω for 110 µm lead styphnate, average 20.0 Ω for 55 µm lead styphnate, to very high (> 1 M Ω) for µm lead styphnate. Action times and their standard deviations also creased in the same sequence. Similar data can be seen for the two nobasic lead styphnates, ie RD1346 and the smaller particle size RD1349 able 1). Although Sheridan [25] gives no data for energy sensitivity of his xes, the pronounced increase in sensitivity of the RD1349 mix (Table 2) uld strongly suggest that the smaller particle size lead styphnates would hibit similar, undesirable, increases in sensitivity.

An experimental three component mix of RD1303/barium nitrate/foliac aphite 73:20:7 was briefly investigated to assess what effect the presence i an oxidant would have. The results obtained on this single mix (Table 2) dicate minimal effects with a tendency to desensitization. No further ivestigation was attempted although it could be examined in greater detail at later stage.

.2 Choice of the Conducting Component

The conducting component in CC primers is invariably graphite or arbon black, although metal powders and flakes have been used in CC detonator ixes [19-22]. The possibility of using metal powders/flakes as the onducting material in CC primers was examined in a preliminary study, not eported here. It was found that although very fast functioning times could e obtained, the mixes were invariably very sensitive (50% functioning nergies <70 μ J) and accordingly not suitable for the projected application. uch mixes could have useful properties for other applications but further ork in this area was left for the future.

A number of graphites and carbon blacks are currently used in ustralian ordnance production or manufactured locally. For example, a 1:1 ixture of Merck synthetic and NK natural graphite is used in the M52 DEFA hile Dohm's air-floated graphite is used in the N43 primer. The conducting omponent in the M52A3B1 is acetylene black. Preliminary testing of a range f such graphites admixed with RD1303 indicated a wide range of explosive erformance, as had been noted in earlier studies e.g. Ref. [24]. Much of hese differences could be overcome by adjusting percentages, e.g. 8% of raphite A may produce a mix with similar performance to one with 5% of raphite B. As a consequence, it was decided to severely limit the number of aterials tested in the first instance, and only extend the survey if none of hese proved satisfactory.

Previous studies have shown that the performance of lead styphnate ased CC mixes is strongly influenced by the particle size distribution of the raphite/carbon black. In addition, graphites and carbon blacks display ntrinsic differences in their conducting properties in these mixes. The onducting materials to be tested were chosen to cover these broad properties nd yet be limited to a manageable number. The final five selected were: Foliac graphite No. 1371, sieved sub 38 µm Merck synthetic graphite (ex MFF St. Marys, M52 DEFA) Carbon black (ex EFM) Tintacarb 140) Tintacarb 90) manufactured locally, Australian Carbon Black

The two graphites were chosen as materials at the extremities of particle size range which would be both useful, i.e. not too big, and obtainable, i.e. not too small. One carbon black was chosen as representative of this class of material, while two locally manufactured materials, Tintacarb 140 and 90, were selected. The choice of the latter two largely resulted from the expectation that they might exhibit properties of both graphites and carbon blacks. Both these materials are of small particle size and are used in printing inks.

The five materials were characterised by scanning electron microscopy (SEM) and particle size distribution (Malvern particle size analyser). The SEM micrographs (Fig. 2) clearly show both graphites as typical flake particles while the carbon blacks consist of agglomerates of very small spherical particles, often further agglomerated into yet larger particles. Although the foliac graphite was sieved through a 38 µm sieve, the particles are generally larger than this along one axis. This can be seen clearly in Fig. 2a and in the particle size distribution figures (Table 3); in the Malvern determinations, the particles are spun rapidly while in aqueous suspension and particles with a long and short axis will be recorded as having the large axis size. The Tintacarbs, particularly 140, seem from the SEM micrographs to consist of agglomerates of perhaps 1 µm diameter, but are obviously further agglomerated into roughly 5-15 µm units which are difficult to break down further. They are both clearly different from the carbon black (Fig. 2c), which consists of very small particles, with 36% being <2 µm (Table 3). The particle size distribution of the graphites and Tintacarbs appears to be roughly normal with a clear maximum, while the carbon black must have a maximum in the sub micron range with a long tail up to 20 µm.

The experimental approach chosen was to study each of the five materials as 10, 7, 4 and 1% mixes with RD1303, then extend the range to fill in gaps depending upon the results obtained. The mixes were pressed into M52 DEFA caps and consolidated with a lead nitrate increment as in Section 4.1 full details are given in the Experimental Section. Cap resistances were measured after pressing and 1 week later, then functioning energies and times were measured. The complete set of results is detailed in Table 4.

Foliac graphite does not mix well with RD1303, producing a nonuniform coating with a definite tendency towards segregation. Resistance ranges and standard deviations of the pressed caps are accordingly high (Table 4). The foliac mixes are of unacceptably high sensitivity until 7% foliac is present. The sensitivity of the 10% foliac mix is quite low (50% energy >2000 μ J) yet the functioning time requirement is easily met - it must be kept in mind that replacement of the lead nitrate by a primer increment would substantially diminish this time, as discussed in Section 4.1. Increase to 15% foliac again substantially diminishes energy sensitivity, but to the point where the energy delivered in the functioning time test is marginal for cap

tion; 4/6 failed to fire, and those that did had substantially extended :tioning times. At the other extremity of the test mixes studied, 1% lac graphite, failure in the functioning time test also occurred. These used 1% foliac caps all had very high resistance (> 2 M Ω) and exploratory ting for energy sensitivity failed to achieve ignition at the highest rgy level, 90,000 μ J = 2 μ F/300 V. The remaining caps were subsequently ted at 300 V using successively 330 pF, 1000 pF, 0.1 µF and 2 µF acitors. Although most caps failed completely, ignitions occurred right n to 14.9 µJ (330 pF/300 V, Table 4). A likely explanation for this aviour is that the resistance of most caps is too high to achieve a current w sufficient to cause ignition. Some caps, however, have sufficiently er resistance (but still > 2 M Ω) that current flow above the required eshold can occur. These caps are then very sensitive, as expected from the nd to increased sensitivity observed for the series 10+7+6+4% foliac. hough foliac graphite can produce mixes of adequately low energy sitivity which still meet the functioning time requirements, its relatively or mixing properties would preclude further consideration.

Merck graphite mixes well with RD1303, coats it effectively and regation does not appear to be a problem. The resistances of the pressed apositions are significantly lower than the corresponding foliac apositions, with the much better mixing properties reflected in the Latively narrow ranges/low standard deviations. The resistances rise ostantially over 1 week, a typical property of many CC caps eq. M52 DEFA. B 50% functioning energies are acceptably high for all mixes containing 4% more Merck; they roughly follow the trends for the foliac mixes with 3% ss Merck being required to obtain similar values for energy sensitivity. e mix containing 10% Merck has too low a sensitivity for reliable nctioning in the function time test, and those that did fire had times only rginally under the .3 ms specification. The 1% mix is characterised by very gh resistance; the caps were fired as described above for the 1% foliac mix 1 a similar result was obtained. Merck graphite, as representative of mall" particle size graphites, exhibits promising characteristics. The per and lower levels of usefulness have been broadly identified.

The carbon black mixed very poorly with the RD1303 even at only 4% weight and higher incorporations were not attempted. Segregation was so d that much of the carbon black had "floated" to the surface of the mix ter only a few minutes, and needed to be constantly remixed during weighing rior to pressing). This is not altogether unexpected since the difficulty incorporating particles of <5 µm had been noted in a number of previous udies. The pressed mixes consequently displayed moderately large resistance nges/standard deviations (Table 4). The energy sensitivity of the 4% mix is ite accept ble. and the 3% mix marginally so, and both easily meet the nctioning tim virements. Two differences in comparison with the aphite mixes are table: much less carbon black is needed to produce mixes comparable resis ce and energy sensitivity, while there is no pronounced me drift of the ca, :esistances. The carbon black still produces a mix of derately low resi __nce but high energy sensitivity even at only 1% by ss. This carbon lack was not considered further due to its very poor xing properties.













Scanning electron micrographs of graphites/carbon blacks used in conducting composition mixes. Note magnification scale shown below each illustration. (a) Foliac graphite (b) Merck synthetic graphite Tintacarb 90 (f) Lead styphnate RD1303 (for Tintacarb 140 (e) (P) (c) Carbon black ex EFM comparison). Figure 2.

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POWER SENSITIVITY TEST RESULTS FOR SELECTED LEAD STYPHNATE

RD1303 - GRAPHITE/CARBON BLACK CC MIXES

COMPACT LONG		RESISTANCE	(Ω) <u>b</u>	POWER SENSIT	IVITY (w)	
		møan (std.	dev.)	50% fire level	all-fire (ramp)	
RD1303/Foliac graphite	94: 6	63,9 (50	L 0)	0, 74	14/14 2.0	
RD1303/Merck graphite	95:5	31.2 (13	.6)	0,63	15/15 2.0	
RD1303/Carbon black	97: 3	12,8 (6	.4)	0, 92	15/15 1.5	
RD1303/Tintacarb 140	95:5	8.3 (0	.6)	2.10	15/15 4.0	
RD1303/TIntacarb 90	93: 7	4.8 (0	.3)	2.16	15/15 3.0	

<u>a</u> Percentages by mass

- <u>b</u> Resistances after fitting into test firing cartridge case.
- 50% level obtained by Bruceton staircase testing. All-fire level was obtained by re-testing no-fires from Bruceton at successively higher power levels till the cap fired.

				Cap Resist	ance (Ω)		Function	ng Energies	Funct ioni	ng Times
Composition ^a		Caps	When Pres	Ised	Mter 1	woe k	5	ų _	(m)	
		D 91 591	meen (std. dev.	,) range	meen (std. dev	v.) range	50% fire level	Al I-fire (ramp) ^C	s u Gew	td. dev.
RD1303¢ Tintacarb 140) 90±10	21	2.8 (0.2)	2, 5-3, 1	3.3 (0.25)	2, 9-3, 8	259 04	15/15 5625	0.21년	0, 053
	93:7	21	4.5 (0.5)	3.6-5.3	5.4 (0.5)	4.6-6.4	1565	15/15 3781	0.106	0,022
	95: 5	21	8.7 (0.9)	7.3-10.0	9.3 (0.9)	7.9-10.5	860	16/16 2000	0, 098	0, 020
	95.5:4.5	21	10.3 (1.3)	7.8-12.0	11.0 (1.3)	8,5-12,8	760	13/13 2000	0.110	0,027
	96: 4	21	13.3 (2.4)	8-16	14.9 (2.3)	9-18	6 66	14/14 1531	0. 078	0, 020
	98:2	2	77.3 (6.2)	65 . 7-90 . 7	82.0 (6.6)	69°0-97°1	215	15/15 500	0.088	0.010
	99: 1	21	2430 (461)	1310-3120	2567 (460)	1410-3260	130	14/14 281	0,079	0. 016
RD1303: Tlatacarb 90	90:10	21	2.6 (0.2)	2.1-3.0	3.2 (0.3)	2,5-3,6	4250 <u>d</u>	15/15 7656	0.1831	0-053
	93: 7	35	4.3 (0.3)	3.8-4.8	4.7 (0.3)	4.2-5.2	2290	15/15 3781	0. 129	0.018
	96:4	21	11.3 (3.3)	5.9-17.4	12.9 (3.4)	7.2-19.7	565	15/15 1531	0.075	0.027
	1:66	21	3172 (807)	66-4-4270	3451 (865)	123, 1-4700	95	15/15 195	0, 087	0-021
<u>B</u> Percentages	by weigh · dischar	.t. 0. 0. 1	f raparltor	inlace stata	d otherwise.	50% energies	hv Aruce	ton stalress	bottom e	
C Obtained by	successi	vely step	ping up discha	irge energie	s till cap fli	red (ramp met)	hod).			
4 2 µF capacit	tor used.			•						
4/6 failed 1	to fire.									
- Remaining ca	aps which	failed t	o fire up to .	4500 µJ were	not ramped fi	urther but fl	red off a	t 90,000 μJ.		
북 Data from ea	sriier ex	ploratory	testing, pres	ssed and tes	ted same day.					
	sistance	7.5 KN, e	xcluded from	lata.						
INO CAPS, FE	esistance	- 15 KQ,	excluded from	n data.		1	•			
Le Caps rested	by TIFIN to fire	1/6 fired	using success	sivery sou p	r, tuuu pr, u		apacitors	•		
- 1/6 needed 1	two disch	arges to	fire.	•••						
		•								

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VARIATION OF PERFORMANCE CHARACTERISTICS WITH GRAPHITE/CARBON BLACK CONTENT

FOR ADMIXTURES WITH LEAD STYPHNATE RD1303

				Cap Resist	tance (Ω)		Function	lng Energies	Function	Ing Times
Composition <mark>a</mark>		Caps Tested	When Press	þ	Ater 1 w	96 K	म.)	ঀ	(mi	2
			maan (std. dev.)	range	mean (std. dev.	egner (50% fire level	Al I-fire (ramp) ^C	u đđi	std. døv.
RD1303: Follac Graphite	85:15	20	3.4 (0.9)	2.2-5.3	13, 3 (6, 6)	6.5-29.2	7655 <mark>4</mark>	11/11 22.500	0.23 1 0	
•	90:10	21	11.9 (15.9)	2-72	74.6 (247.5)	3-1150	2335	10/13 4500-	0.128	0,025
	92:8	2 3 9	7.5	2-19			1490		0, 113	0.018
	93:7	21	30.9 (44.6)	6-167	67,6 (95,9)	11-350	945	11/12 153년	0.123	0,012
		249	34.3	5-100			1015		0.103	0.012
	94:6	35	55.6 (44.0) ⁿ	10.8-207	96,3 (82,5)	17.2-388	360	16/16 1125	0,092	0,020
		2 3 9	218	21-900			235		0.112	0, 030
	96:4	20	210.4 (229.9)	40-1060	416.9 (551.9)	56-2440	160	12/12 383	0.074	0.018
	99:1	21	aii > 2M	alt > 24			1/16 14.9	, 2/16 45,	all fa	
							4/16 4500	, 0/16 90,000		
							no fire 9	716L		
RD1303: Marck Graphite	90:10	21	2.9 (0.5)	2.1-3.9	11.5 (9.3)	4.4-47.7	3165 <u>4</u>	15/15 10,000	0.224 ⁰	0,008
	93: 7	21	2.8 (1.1)	2-6	7.1 (2.3)	4-13	2370 ^d	10/10 3906	0.111	0.010
6	4.5:5.5	21	6.9 (1.2)	5.5-10.6	11.0 (2.5)	8.4-17.5	1625	16/16 3781	0.115	0.025
	95:5	20	11.9 (2.0)	8.1-15.6	21.6 (5.8)	12, 1-32, 6	1438	14/15 2531	0.108	0, 031
	96:4	21	15.8 (5.4)	9-29	25.8 (11.2)	13-55	945	13/13 2000	0.088	0.013
	99: 1	21	all > 2M		al I > 2M		1/16 14.9	, 1/16 45,	all fa	8
							5/16 4500 8/16 no f	, 1/16 90,000 ir ol		
RD1303: Carbon Black	96:4	21	5.0 (2.4)	2,6-10,6	5.2 (2.5)	2.7-10.6	1100	15/15 3125	0.109	0.034
	97:3	35	7.8 (2.9)	3.8-14.9	8.0 (2.8)	4.1-14.5	565	15/15 2531	0. 088	0,013
	1:66	21	270.7 (128.9)	6.5-497	289.6 (134.6)	12.1-525	55	14/14 281	0.074	0.019

PARTICLE SIZE DISTRIBUTION[®] FOR GRAPHITES/CARBON BLACKS

USED IN EXPERIMENTAL CC PRIMER MIXES

PARTICLE SIZE		PER	CENT BY WEIGHT		
RANGE (jum)	FOLIAC GRAPHITE	MEROK GRAPHITE	CARBON BLACK	TINTACARB 140	TINTACARB 90
>100	0.4				
80-100	3.5				
70-80	5.2				
60-70	9.4				
50-60	14,4				
45-50	8.8				
40-45	9,6				
35-40	9,9				
30-35	9,6				
25-30	8.9				0.2
20-25	7.6	0,7	0.2	0.4	1.5
15-20	6.0	6, 8	1.2	5.7	7.2
13-15	<	7.6	1.2	7.1	6.5
12-13	Total 4.1	5, 3	1.0	5, 2	4, 4
11-12		6,5	1.3	6.4	5.2
10-11	\mathbf{i}	7.5	1.8	7.6	6.1
9-10	$\mathbf{\lambda}$	8.4	2.4	8.6	6.9
8-9	Total 2.2	9. 1	3, 1	9.4	7.7
7-8	1	9.3	4.1	9.7	8.3
6-7		9,2	5.4	9.6	8.7
5-6	, ,	8,6	7.1	8.9	8.7
4-5	\mathbf{X}	7.5	9.1	7.7	8.4
3-4	Total 0.6	6.0	11.6	6,2	7.6
2-3	/	4.3	14.4	4, 3	6, 3
1-2	/	2.4	17.4	2.4	4.4
<1	/	0.7	18.4	0, 7	1.9
TOTAL (\$)	100.0	100.0	100.0	100,0	100.0
MEAN (µm)	47.6	9.7	4• 1	9, 5	9.2

A For comparison, lead styphnate RD1303, mean particle size 115.2 μm, 98\$ < 240 μm, 90\$ < 180 μm, 70\$ < 140 μm, 10\$ < 60 μm.



RESULTS OF EXPERIMENTAL FIRINGS OF CC COMPOSITIONS USING 7% FOLIAC GRAPHITE AS CONDUCTING COMPONENT

			Cap Resis	tance (Ω)		Function	ng Energies	Function	Ing Times
Component (93%) mixed with Follac Graphite (7%) ⁸	Caps Tested	When Pres Moan (std. dev.)	ised range	After 1 wo mean (std. dev.)	ek range	() 50% flre Level	u)= Al 1-f1re (remp) ^C	a is	s) td. dev.
Normal lead styphnate RD1303	21 240	30.9 (44.6) 34.3	6-167 5-100	67.6 (95.9)	11-350	945 1015	11/12 153년	0. 123 0.103	0, 007 0,012
Monobasic lead styphnate RD1346	21	16.1 (8.2)	4, 5-35, 4	29.6 (18.6)	8.4-90.4	760	13/13 2531	0.209	0, 060
Monobasic lead styphnate RD1349	20	43.5 (24.2)	20-110	66 . 8 (44 . 7)	24-186	220	12/12 500	0.264	0.054
Barium styphnate RD1320(B)	21	290.2 (196.4)	6-3500	420 (843)	14-3050	2050	15/17 3781 <u>t</u>	0. 3869	0, 093
Lead azotetrazole RO1355	21	8.0 (2.9)	4.0-13.4	15.2 (6.4)	6,1-29,7	1630	15/15 4500	0.088 ^g	0.029
Potassium picrate	21	43.0 (21.4)	17-101	74.4 (41.3)	34-211	3365	18/18 3781	48, 1	5.0
RD1303/barium nitrate 73:20	21	35.4 (82.4)	3-378	94.0 (262.1)	5-1120	1150	13/13 3781	0.134	0.012

Percentages by weight.

By capacitor discharge, 0.1 µF capacitor.

Obtainied by successively stepping up discharge energies till cap fired (ramp method). e = 0 = = 0 = = 0

1/12 failed to fire up to 4500 µJ, subsequently fired off at 90,000 µJ.

Data from an earlier exploratory testing, pressed and tested same day.

2/17 failed to fire up to 4500 µJ, subsequently fired off at 90,000 µJ.

Increment alone produced insufficient flash to trigger photoceil, obtained from cap with

RD1303 priming increment.



EXPLOSIVE FILLINGS IN CONDUCTING COMPOSITION DEVICES

USED IN AUSTRALIAN ORDNANCE

			DEVIC	C E	
COMPONENT [®]	N52 Primer (Conducting increment	30mm DEFA) Priming increment	N8 (gniter	N43 Primer ^b (Naval 4,5 in)	M52A381 Projected 20 mm Primer F/A~18
Lead Styphnate RD1303	95, 0-95, 5	48	97	95, 5	40 ± 2,5
Graphite	4.5-5.0	2	3	4,5	
Acetylene Black					0.75 ± 0.25
Barium Nitrate		12			44.25 ± 2.5
Potassium Perchiorate		28			
Calcium Silicide		10			13.0 ± 2.5
Gum Arabic					1.0 ± 0.25
Styphnic Acid					1.0 ± 0.25
Resistance Specifications (Ω)	20-250		500-2000	20-60	1K - 1,2M

<u>A</u> Numbers in each column refer to percentagees of each component.

b Second (priming) increment of SR 227A: Potassium nitrate 72 \pm 1, sulfur 7 \pm 0.5, charcoal 21 \pm 0.5%.



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(Bruceton) test were subsequently retested at a voltage increased successively by the test increment till firing occurred - this is referred to in the text as "ramping". The compositions containing only 1% graphite were, as described in the text, tested successively using the 300 pF, 1000 pF, 0.1 μ F then 2 μ F capacitors all charged to 300 V, the sequence being terminated when a fire was obtained. The least sensitive compositions were tested using the 2 μ F capacitor (50% energies >2500 μ J).

(iii) Power Sensitivity

Power sensitivity was assessed using a firing box which consisted of a stabilized power supply on which voltage could be varied from 0-30 V. The experimental CC caps were fitted into the test firing rig described above and the cap resistance was measured. The voltage corresponding to the selected power level (from $P = V^2/R$) was accurately set, then the test rig was connected and current flow commenced. The current flow was terminated after 10 s if the cap had not fired. The 50% fire level was determined by the Bruceton staircase method [27], commencing at a level of 1.5 watt and using 0.25 watt increments. Caps which failed the initial (Bruceton) testing were subsequently retested for 10 s at higher power levels till firing occurred, giving a rough estimate of the all-fire level (referred to as ramping in the text). A total of 15 caps were tested for each composition.

It should be noted that initial application of current to a CC cap usually results in a rapid dramatic drop in cap resistance [2, 28]. In our tests, this can be seen as a rise in current flow. Thus the power delivered to the cap during testing will rise considerably above the initial power setting, and the results for power sensitivity obtained here must be regarded as very conservative; values for 50% firing power are probably up to double those reported here.

7.2 Preparation of Conducting Composition Mixes and Experimental Caps

The two components (or three in the case of the composition containing barium nitrate) were weighed separately in correct proportions to give a total of 1 g. The materials were placed on a sheet of paper and foldmixed until the mixture appeared to be homogeneous.

Experimental caps were prepared by pressing the conducting mix (46.5 \pm 0.5 mg) into an empty M52 DEFA cap using an Eltor press at a pressure of 123 MPa (400 Kg dead load). Lead nitrate (185 \pm 15 mg) was then added on top and the cap repressed at the same pressure. Due to the difficulties of measuring functioning times of some compositions pressed as above, a few caps were prepared by replacing the lead nitrate with a priming increment (145 \pm 5 mg) of DEFA primer (see Table 1). Resistances were measured using a Hioki 3208 Calcu Hi Tester.

7.3 Determination of Functioning Parameters

(i) Functioning Times

A firing box designed to meet the test specifications of the M52A3B1 was constructed at MRL. It is shown schematically in Fig. 3. The basic components of the system are a 160V power supply to charge the 2 μ F capacitor and a specially constructed timer to terminate the output pulse from the capacitor after 10 μ s. Fast application of energy was achieved by use of a mercury wetted relay switch. The experimental caps were fitted into an empty M52 DEFA cartridge case which was then fitted into a test firing rig. Functioning times were measured using a microsecond timer triggered by a synchronised pulse from the firing box for start and the output from a BPX 25 photocell for stop.

(ii) <u>Energy Sensitivity</u>

A variable energy firing box was constructed at MRL. The firing pulse could be varied by selection of four capacitors, 330 pF, 1000 pF, 0.1 μ F and 2 μ F, which could be charged from 0-300 V. Fast application of energy was achieved by use of a mercury wetted relay switch. The experimental caps were fitted into the experimental test rig described above and tested using the Bruceton staircase method [27]. A total of fifteen caps were tested using constant voltage increments. The quantity under test, sensitivity to energy, is probably log-normally rather than normally distributed and the Bruceton testing procedure should therefore be based on constant lorgarithmic increments in energy. Since $E = \frac{1}{2}CV^2$, constant voltage increments closely approximate constant logE increments, and this procedure was followed in this exploratory testing. Testing was usually conducted using the 0.1 µF capacitor, with voltage increments of 25 V where the 50% fire level was >125 V, and 12.5 V for lower 50% voltages. In the case of the very sensitive RD1303/1% carbon black composition, where the 50% firing voltage was below 25 V, increments of 6.25 V were used. Caps which did not fire during the initial

that smaller particle sizes lead to undesirable sensitivity increases. Five graphites/carbon blacks were assessed as admixtures with RD1303. Foliac gaphite and EFM carbon black, representing the extremes of particle size distribution studied, were unsatisfactory mainly due to their poor mixing properties. The Merck graphite and Tintacarb 140 and 90 mixes easily met the performance specifications and energy sensitivities within well-defined upper and lower incorporation levels. It is recommended that more detailed assessments be commenced on selected compositions prepared from these materials with RD1303. Although a material more suitable than RD1303 could potentially be found, this is regarded as a much less cost effective exercise and although a desirable long term strategy, appears much less promising from the viewpoint of the available time scale.

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7. EXPER_MENTAL

7.1 Materials

Lead styphnate RD1303 was a single batch, 14/78, obtained from MFF St. Mary's. Monobasic lead styphnate RD1346 was also ex. MFF ST Mary's, while RD1349, lead azotetrazole RD1355 and potassium picrate had been prepared previously at MRL for other studies using standard preparative procedures for these materials. Barium nitrate was ground and passed through a 75 μ m sieve prior to mixing. Foliac graphite was a commercial sample and was passed through a 38 μ m sieve prior to use. Merck graphite was obtained from MFF St. Mary's, the carbon black was obtained from EFM and the Tintacarbs were donated by Australian Carbon Black as noted above.

Unfilled MF2 DEFA caps were purchased from MFF St. Mary's and came from a standard production lot.

Tintacarb 140 was initially selected from the two Tintacarbs due to its narrower particle size range (Table 3). The material mixed well with RD1303, coated the RD1303 crystals effectively and no tendency to segregation was observed. The Tintacarb 140 was used in the first few mixes "as received", and the functioning energies were somewhat non-uniform in increase with Tintacarb content increase (Table 4). Inspection of the mixes by optical microscopy revealed the presence of small "balled" agglomerates which were not being effectively broken up during mixing. Consequently both Tintacarb 140 and 90 were passed through a 106 µm sieve prior to mixing for the remainder of the mixes. Some measure of the advantage of presieving can be seen from the values for energy sensitivity of the Tintacarb 90, where the results for the 7% and 10% mixes are substantially higher than the corresponding unsieved Tintacarb 140 mixes, while the unsieved 4% Tintacarb 90 mix is lower. All the Tintacarb mixes above 4% have satisfactorily low sensitivity, but Tintacarb 90 is marginal at 4% incorporation. Functioning times are easily met until 10% incorportion where firing failures occur; the sensitivity has diminished to the extent that the energy is not sufficient for reliable initiation. Even at only 1% incorporation both Tintacarbs produce caps of moderate resistance. In all the mixes resistance ranges and standard deviations are very tight and resistances change only marginally with time. It would appear that these materials possess the conducting ability and resistance stability of carbon blacks yet have the good mixing properties of graphites. Both materials stand out as having excellent promise.

As mentioned in Section 3, power sensitivity was not considered to be as important as energy sensitivity and assessment of each batch of caps was not made. Instead, one set of caps from each of the five graphites/carbon blacks was selected for testing. In each case the mix chosen was one which exhibited satisfactorily low energy sensitivity. Fifteen caps were tested using the Bruceton staircase method, with an estimate of all-fire level being obtained by re-testing no-fires at successively higher power levels till the cap fired. Results are listed in Table 5, and should be compared with the corresponding 50% fire levels for the M52A3B1 at 0.232 watt [2], and the M52 DEFA at around 0.3 watt using this test method.

Clearly all mixes are substantially less sensitive than either the M52A3B1 or M52 DEFA, with the two Tintacarb mixes being easily the least sensitive. The results indicate that caps which meet the energy sensitivity requirements will have satisfactorily low power sensitivity.

5. CONCLUSIONS

All of the conducting composition mixes studied here were assessed with the sole objective of developing a CC cap which met the performance specifications of the M52A3B1 while exhibiting substantially diminished initiation sensitivity. Five materials were examined as alternatives to lead styphnate RD1303 and all were unsatisfactory. The results from a previous study of lead styphnate of different particle size distribution [25], coupled with the results obtained here for the monobasic RD1346 and RD1349, indicate



not to scale.

1. Control (Control 4.5.4) (Control 4.5.5)



Figure 3. Schematic diagram for firing box used for determination of CC cap functioning times.

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Schematic diagram for firing box used for determination of CC cap Figure 3. functioning times.

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