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PLASTICIZED ETHYLENE - VINYL ACETATE
BINDERS FOR INSENSITIVE BOOSTER
COMPOSITIONS

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I.J. DAGLEY, R.P. PARKER, L. MONTELLI AND C.N. LOUEY

MRL-TR-91-34

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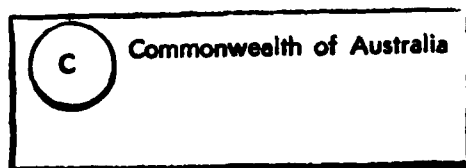
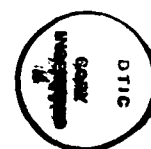
Plasticized Ethylene-Vinyl Acetate Binders for Insensitive Booster Compositions

I.J. Dagley, R.P. Parker, L. Montelli
and C.N. Louey

MRL Technical Report
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Abstract

Plasticizers have been incorporated into RDX/ethylene-vinyl acetate (EVA) compositions prepared by the solvent slurry coating technique. The effect of plasticizers on impact sensitiveness, shock sensitivity, cookoff behaviour and integrity of pressed pellets has been examined for compositions with EVA binders of varying vinyl acetate content. A compatible EVA/plasticizer combination gave an appreciable reduction in cookoff response and impact sensitiveness. Plasticizer migration is likely to affect the long-term stability of these compositions.



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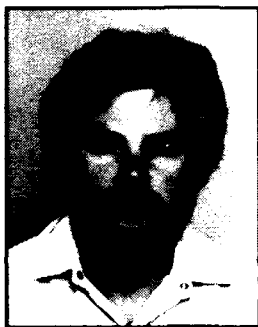
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Plasticized Ethylene-Vinyl Acetate Binders for Insensitive Booster Compositions

1. Introduction

For decades Australian ordnance has typically contained tetryl as the fuze booster composition and TNT-based main charge fillings. These compositions are vulnerable to unplanned hazardous stimuli (e.g. bullet/fragment impact, fuel fire, detonation of adjacent charges) and must be replaced in new Australian ordnance currently being designed to meet insensitive munitions criteria. Less sensitive polymer bonded explosive (PBX) compositions developed overseas are being evaluated at MRL as alternative main charge compositions [1]. Because overseas technology for insensitive booster compositions was less advanced, a project to develop a suitable pressed composition for use in Australian production and filling was commenced at MRL.

Ideally the new booster composition should:

- be no more sensitive than tetryl to initiation by impact for compliance with current fuze explosive train guidelines [2],
- be no more sensitive than tetryl to shock initiation,
- have pickup and output properties similar to those of tetryl,
- respond mildly (deflagration or less) under both fast and slow cookoff conditions, and
- be processible on a small batch scale in existing or easily acquired plant.

Insensitive PBX booster compositions developed in the US and UK contain nitramines (RDX or HMX), to confer shock sensitivity and performance, in blends with more thermally stable and less sensitive high explosives (e.g. TATB) [3, 4]. Most overseas research has concentrated on optimizing these blends to achieve the desired level of sensitivity and vulnerability. In contrast the early stages of the Australian study have focused more closely on binder selection and coating techniques since sensitivity and vulnerability are

also influenced by the properties of the binder and the extent to which it coats the explosive crystals [5, 6]. By adopting this approach it should be possible to place less reliance on blending explosives to obtain the correct balance of properties.

Initial studies examined the sensitivity and cookoff response of numerous RDX/polymer (95:5) compositions prepared by the slurry and dispersion coating processes [7, 8]. Certain ethylene-vinyl acetate (EVA) copolymers moderated the sensitivity and vulnerability of RDX. The properties of EVA copolymers are largely determined by their vinyl acetate content (quoted as weight percent (% VA) in this report) and their molecular weight. The effects of variations in these parameters on the impact sensitivity and shock sensitivity of these compositions have been largely explained [5, 7]; in contrast no relationship has been established between the structure or properties of these copolymer resins and their ability to moderate cookoff response. In this case minor differences in properties produced marked effects on cookoff response but the experimental data did not reveal any obvious correlations.

In an extension of this project, the possibility of achieving further reductions in sensitivity and vulnerability of RDX-based compositions by modifying the properties of their EVA binders with low levels of plasticizers has been investigated. This study has focused largely on modifying one EVA binder, Levapren 408, which contains 40% VA and has a comparatively low molecular weight. This copolymer was typical of the majority of EVA resins examined in an earlier study - it did not reduce the impact sensitiveness of RDX or significantly modify its cookoff response. A copolymer containing a lower vinyl acetate content (28% VA) with a low molecular weight (Elvax 210) and one containing a higher vinyl acetate content (50% VA) with a high molecular weight (Levapren 500) were markedly better than other EVA copolymers at moderating fast cookoff response. Both these copolymers were also plasticized, primarily to achieve further moderation of cookoff response.

2. Experimental Approach

RDX/Levapren 408/plasticizer (95:5:0.5) moulding powders containing a range of plasticizers were prepared and their impact sensitiveness was assessed. The shock sensitivity of compacts prepared from promising compositions containing phthalate, flame retardant and polymer plasticizers was then determined. The plasticizer from each class that gave the least shock sensitive compact was used to prepare compositions containing the EVA copolymer with lower vinyl acetate content (Elvax 210). The impact sensitiveness of these compositions was determined and their cookoff response was compared to that of the equivalent compositions containing Levapren 408 and, in one case, Levapren 500.

3. Experimental

3.1 Materials

RDX Grade A, Class 1 (recrystallized) from Albion Explosives Factory was used to prepare all compositions. It was received wet and thoroughly dried at the pump prior to use.

The EVA copolymers examined were Levapren 408 and Levapren 500 from Bayer and Elvax 210 from Du Pont; they have vinyl acetate contents of 40, 50 and 28 percent respectively.

The plasticizers used in these experiments and their commercial manufacturers are listed in Table 1. All the solvents used were laboratory grade. Distilled water was used in all the preparations. Mowiol 4-88 is a partially saponified polyvinyl alcohol manufactured by Hoechst and was used as a protective colloid to stabilize the emulsion formed between the polymer solution and water during the slurry coating process.

Table 1: Chemical Descriptions and Manufacturers of Plasticizers Used in Compositions

Material	Chemical Description	Manufacturer
Adipate and Phthalate Plasticizers		
DOA	Diethyl adipate	CSR Chemicals
DOP	Diethyl phthalate	CSR Chemicals
Edenol DCHP	Dicyclohexyl phthalate	Henkel
Flame Retardant Plasticizers		
Fyrol FR-2	Tris(β,β' -dichloro-isopropyl) phosphate	Stauffer Chemical Company
Reofos 65	Triaryl phosphate ester	Ciba-Geigy
Polymeric Plasticizers		
PEG 12000	Polyethylene glycol (molecular weight 12000)	ICI
Re-plas 1360	High molecular weight long chain polyester	Townsend Chemicals
Repol 720	Linear polyester diol (molecular weight 2200)	Townsend Chemicals

3.2 Mixing Equipment

Batches of these compositions were prepared in an open metal mixing vessel fitted with a heating jacket and containing two internal baffles mounted perpendicular to the walls of the vessel. The slurries were stirred by an overhead air motor driving a rod with an impeller at the base equipped with twelve flat blades.

3.3 Preparation of RDX/EVA/Plasticizer (95: 5: 0.5) Compositions

A slurry of RDX (142.5 g) and water (430 mL) was stirred at 500 r/min for 5 min then an aqueous solution of Mowiol 4-88 (0.01% w/w, 15 mL) was added and the slurry was heated to 65°C. After a further 10 min a solution of the EVA copolymer in toluene (10% w/w, 75 g) containing the dissolved plasticizer (0.75 g) was slowly added. The mixture was stirred vigorously at 700 r/min and the temperature of the slurry was maintained at 65°C until the solvent had evaporated and hard moulding granules had formed. The agitated slurry was cooled to 30°C and the collected granules were washed with water, dried at the pump and then at 60°C under vacuum over silica gel.

3.4 Characterization

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

A Rotter apparatus [9] fitted with a 5 kg weight was used to determine the impact sensitiveness of the compositions. The results were obtained using 25 caps and the tests were carried out in accordance with the Bruceton procedure. The F of I values quoted, derived from the height for 50% initiation probability, are relative to RDX Grade F = 80 and are rounded to the nearest five units. The average gas volumes for positive results are also quoted.

3.4.2 Shock Sensitivity : Small Scale Gap Test

The MRL small scale gap test (SSGT) [10] was used to obtain the shock sensitivity data. A UK Mk 3 exploding bridge wire detonator was used as the donor and the shock was attenuated by brass shim. The acceptor was two 12.7 mm diameter × 12.7 mm high cold pressed cylinders of the explosive under study. A detonation was confirmed using a mild steel witness block. The results were obtained from 20 to 30 firings using the Bruceton staircase method and are quoted in mm of brass shim for a 50% detonation probability, together with the 95% confidence limits and standard deviation.

3.4.3 Cookoff Test

The cookoff behaviour of the compositions was assessed using the Super Small-scale Cookoff Bomb (SSCB) [11]. Samples were pressed at 90% TMD (theoretical maximum density) into pellets 16 mm diameter $\times$ 16 mm long, and four pellets were used per test. Tests were performed at fast (approximately 1°C/second) and slow (approximately 0.1°C/second) heating rates. Modified SSCBs were used for some of the tests. The original and modified SSCBs are shown in Figures 1a and 1b respectively. The major differences are the elimination of the large air space and the closing plug from the top of the test assembly (giving a symmetrical geometry) and the use of a single full-length inner cylinder instead of two half-length cylinders. Results from a number of tests indicate that the modifications do not affect the rating of the type of response obtained or the temperature at which reaction occurs, however the heating rates obtained with the modified SSCB are slightly lower than with the original test assembly [12].

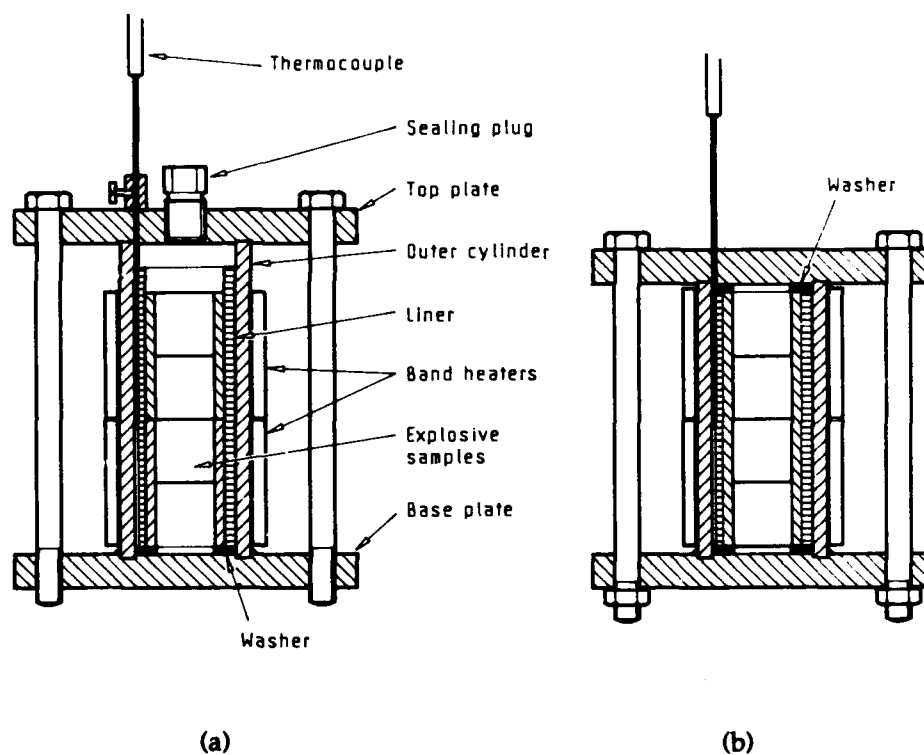


Figure 1: (a) Original SSCB and (b) Modified SSCB.

4. Results and Discussion

4.1 Impact Sensitiveness and Shock Sensitivity

The impact sensitiveness data for the various RDX/Levapren 408/plasticizer (95:5:0.5) moulding powders is listed in Table 2. All the compositions, except the one containing Re-plas 1360, were significantly less sensitive to impact (F of I values of 90 to 105) than the moulding powder without plasticizer (RDX/Levapren 408 95:5, F of I = 75). Two factors that lead to a reduction in impact sensitiveness of RDX/EVA moulding powders are an improvement in the way the binder coats the explosive crystals and greater flow by the polymer during impact. In the latter case greater flow allows the polymer to more efficiently quench hot spots and, as a result of a reduction in viscosity, not undergo heating to such high temperatures [5]. Either or both of these factors may be contributing to the reduced impact sensitiveness of these plasticized moulding powders. Granular tetryl has an F of I of 90 [13], and this figure is the accepted limit for acceptability of materials below the shutter in a fuze train [2].

Table 2: Impact Sensitiveness of Various RDX/EVA/Plasticizer (95:5:0.5) Compositions

EVA Polymer and Plasticizer	F of I ^a	Gas Evolution (mL)
Levapren 408		
-	75 ^b	15 ^b
Re-plas 1360	70	11
Reofos 65	90	10
Fyrol FR-2	90	9
PEG 12000	95	9
Repol 720	95	11
DOA	95	11
DOP	100	10
Edenol DCHP	105	11
Elvax 210		
-	130 ^b , 90	15 ^b , 16
Repol 720	80	13
Edenol DCHP	85	13
Reofos 65	100	17
Tetryl		
Granular	90 ^c , 110 ^d	na, 18 ^d
Crystalline	105 ^d , 110 ^c	16 ^d , na

a Relative to RDX Grade F = 80.

b Data from reference 7.

c Data from reference 13.

d Data from reference 14.

Typical production batches of both granular and crystalline tetryl have F of I values of 105 to 110 [14]. Most of the plasticized RDX/Levapren 408 compositions have F of I values within the range of those of tetryl and would be acceptable compositions in this respect.

Some RDX/Levapren 408 compositions containing different phthalate and polymeric plasticizers and one flame retardant plasticizer (see Table 1) were pressed to 90% TMD and their shock sensitivity was determined; results are presented in Table 3. All these compositions were less shock sensitive than RDX (250 to 300 μ m) and granular tetryl, and more shock sensitive than the unplasticized RDX/Levapren 408 and PBXW-7 Type II (an insensitive booster composition developed in the US). The shock sensitivity of pressed RDX/EVA moulding powders has been examined and the nature of the voids in the compact (determined by the binder plasticity), relative coating efficiencies of the polymers on the surface of the crystals in the moulding powders, the high strain-rate mechanical properties of the pressed pellet and susceptibility for cracking under shock loading may all influence sensitivity in this test [5]. The relative shock sensitivities of the pressed plasticized compositions may also be influenced by the extent of plasticizer migration in the pressed pellet which may have occurred between pressing and testing these compositions (see section 4.2).

Table 3: Shock Sensitivity (SSGT) of RDX/Levapren 408/Plasticizer (95:5:0.5) Compositions

Plasticizer	Relative Density (% TMD)	Shock Sensitivity (mm) ^a		
		M _{50%}	Range L _{95%}	Standard Deviation
_ b	90.05	1.85	1.89 - 1.81	0.018
PEG 12000	90.00	2.41	2.51 - 2.31	0.048
DOP	89.97	2.18	2.19 - 2.12	0.018
Repol 720	89.99	2.09	2.14 - 2.02	0.022
Reofos 65	90.00	2.02	2.07 - 1.97	0.023
Edenol DCHP	90.03	1.98	2.07 - 1.89	0.041
Other Explosives				
RDX, Grade A (250-300 μ m sieve cut) ^c	90.00	3.360	3.622-3.100	0.12
Tetryl, granular ^d	90.0	3.259	3.315-3.203	0.021
PBXW-7 Type II ^d	90.0	1.415	1.448-1.382	0.015

a All figures are in mm of brass shim.

b Data from reference 7.

c Data from reference 15.

d Data from reference 16.

The phthalate, polymeric and flame retardant plasticizers (Edenol DCHP, Repol 720, Reofos 65) that gave the least shock sensitive compositions were used to prepare RDX/EVA/plasticizer 95:5:0.5 compositions containing an EVA resin with a lower vinyl acetate content, Elvax 210, for comparative cookoff studies. The impact sensitiveness of these moulding powders was also

measured (see Table 2). The compositions were as sensitive or more sensitive to impact than the moulding powder without plasticizer (RDX/Elvax 210, 95:5, F of I = 90 to 130).

4.2 Pellet Integrity and Plasticizer Migration

The pressed pellets prepared for cookoff testing were stored for several months under ambient conditions prior to use. It was found that the pellets of RDX/Elvax 210 with Edenol DCHP and Reofos 65 plasticizers became crumbly on storage, and the surfaces roughened and became dull; in some cases slight tackiness was noticed at the lower end of the pellet. These pellets also "grew" appreciably on storage, from the original diameter of 15.9 mm to about 16.1 mm. The pellets of RDX/Levapren 408 with Edenol DCHP and Reofos 65 plasticizers aged better, retaining their smooth finish and showing little or no tendency to crumble or chip at the edges. They also grew less, having diameters of 15.95 to 16.00 mm when tested. The RDX/Levapren 500/Reofos 65 pellets were also noted as being smooth and non-crumbly, and had diameters less than 16.00 mm; however they were not stored for as long as the other pellets, being used within a few weeks of pressing. Pellets of RDX/Elvax 210/Repol 720 and RDX/Levapren 408/Repol 720 aged well, showing only slight roughening of the end faces and maintaining their diameters between 15.95 and 16.00 mm. The RDX/Levapren 408/Repol 720 pellets were slightly crumbly at the edges.

Pressed pellets of all the plasticized compositions, both for shock sensitivity and cookoff tests, were found to produce "grease spots" under the pellets in the cardboard containers in which they were stored, even after relatively short periods at ambient conditions. This is considered to be due to migration of plasticizer from the pellets. The tackiness noticed with some of the RDX/Elvax 210/plasticizer SSCB pellets is believed to be due to the same reason. For the limited range of plasticizers used with different EVA binders, this migration problem appeared to be more severe with the binder with the lower vinyl acetate content (Elvax 210, 28% VA) than with binders with higher vinyl acetate contents (Levapren 408, 40% VA and Levapren 500, 50% VA). This is probably due to differences in physical compatibility between the plasticizers and the various EVAs; it is recognized that compatibility of polymer/plasticizer combinations is dependent upon the chemical structures of both materials [17]. Experiments to quantify the extent of plasticizer migration have not been undertaken.

4.3 Cookoff Behaviour

The results of SSCB tests on plasticized RDX/EVA compositions are shown in Table 4, together with previously reported results for the equivalent unplasticized compositions [7].

Table 4: Cookoff Test (SSCB) Results for Plasticized RDX/EVA Compositions

Composition	Heating Rate	Temperature (°C)	Time (s)	Response
RDX/Elvax 210/Edenol DCHP 95:5:0.5	Fast	227	260	Detonation
	Fast	237	371	Mild explosion ^a
	Slow	223	1588	Detonation
	Slow	206	1779	Explosion ^a
RDX/Elvax 210/Reofos 65 95:5:0.5	Fast	235	282	Detonation
	Slow	213	1688	Detonation
	Slow	211	1976	Explosion ^a
RDX/Elvax 210/Repol 720 95:5:0.5	Fast	246	233	Deflagration ^a
	Fast	239	358	Detonation
	Slow	220	1602	Deflagration ^a
	Slow	219	1774	Explosion ^a
RDX/Elvax 210 ^b 95:5	Fast	245	235	Burning ^c
	Fast	234	238	Deflagration ^a
	Fast	237	246	Mild explosion
	Fast	242	264	Mild explosion
	Slow	217	1628	Detonation
	Slow	220	1681	Detonation
RDX/Levapren 408/Edenol DCHP 95:5:0.5	Fast	231	260	Detonation
	Fast	249	365	Explosion
	Slow	218	1473	Detonation
	Slow	223	1808	Detonation
RDX/Levapren 408/Reofos 65 95:5:0.5	Fast	228	275	Mild explosion
	Fast	251	350	Burn
	Slow	218	1692	Explosion
	Slow	211	1838	Explosion
RDX/Levapren 408/Repol 720 95:5:0.5	Fast	235	275	Explosion
	Fast	242	326	Detonation
	Slow	219	1662	Explosion
	Slow	218	1929	Explosion
RDX/Levapren 408 ^b 95:5	Fast	266	284	Detonation
	Fast	230	250	Detonation
RDX/Levapren 500/Reofos 65 95:5:0.5	Fast	241	293	Deflagration
	Slow	221	1852	Explosion
RDX/Levapren 500 ^b 95:5	Fast	274	293	Deflagration ^c
	Fast	240	255	Deflagration ^c
	Slow	217	1704	Detonation
	Slow	217	1592	Mild explosion ^{a,d}

a. Traces of explosive on parts after test.

b. Data from reference 7.

c. Appreciable amounts (> 2 g) of unconsumed explosive recovered after test.

d. Composition prepared by alternative (confidential) method.

The RDX/Elvax 210/plasticizer compositions all showed more violent responses than the unplasticized composition when tested at the fast heating rate; all three plasticized compositions gave responses as severe as detonations, in marked contrast to the unplasticized composition which gave no reaction more severe than a mild explosion. The plasticized compositions all showed slight reduction in the response violence at the slow heating rate, in two cases (Edenol DCHP and Reofos 65) giving detonation and explosion responses, and in one case (Repol 720) giving deflagration and explosion responses.

RDX/Levapren 408 exhibits violent cookoff behaviour. Incorporation of Edenol DCHP plasticizer had little effect on the type of response - detonations were obtained at both heating rates, although in one test an explosion was obtained at the fast heating rate. Repol 720 plasticizer gave slight reductions in cookoff violence, with explosions being observed at both fast and slow heating rates, although one test at the fast heating rate gave a detonation. Reofos 65 plasticizer, however, had a pronounced effect, giving responses of burn and mild explosion at the fast heating rate, and explosions at the slow heating rate. The effect of this plasticizer on RDX/Levapren 408 is in marked contrast to its effect on RDX/Elvax 210 (see above); this difference may be due to improved compatibility between this plasticizer and the binder with a higher vinyl acetate content (40% VA for Levapren 408 cf. 28% VA for Elvax 210).

RDX/Levapren 500 exhibits milder cookoff responses than RDX/Levapren 408 but also has a high vinyl acetate content binder (50% VA); however, the addition of Reofos 65 to this composition did not result in a similar reduction of response violence - the plasticized and unplasticized compositions behaved similarly. The different effects of plasticizers on the cookoff response of the various RDX/EVA compositions is the subject of further research at this laboratory.

5. Conclusions

Plasticizers can be incorporated into RDX/EVA binder compositions prepared by the solvent slurry coating technique. Pressed pellets of the plasticized compositions were found to vary in their mechanical integrity after storage under ambient conditions, and evidence of plasticizer migration from the pressed pellets was observed for all compositions. These effects are believed to be due to the physical compatibility of the EVAs and the plasticizers, and vary with plasticizer composition and vinyl acetate content of the EVA. EVAs with higher vinyl acetate contents appeared to be more compatible with the plasticizers examined.

Incorporation of a range of plasticizers into RDX/Levapren 408 generally led to a decrease in impact sensitiveness and a slight increase in shock sensitivity. With RDX/Elvax 210 (with a lower vinyl acetate content EVA), the incorporation of plasticizers generally led to increased impact sensitiveness. RDX/Levapren 408/Reofos 65 (a flame retardant plasticizer) showed a substantial reduction in the violence of the cookoff response; however, this same plasticizer had little effect on the cookoff response of RDX/Levapren 500, and led to more violent responses with RDX/Elvax 210. Other plasticizers also

increased the cookoff violence of RDX/Elvax 210 at a fast heating rate, although some moderation was obtained at a slower rate.

Overall, it appears that compatible EVA/plasticizer combinations can give appreciable reductions in cookoff response and impact sensitiveness of RDX-based booster compositions, but less compatible EVA/plasticizer combinations will have the opposite effect.

Plasticizer migration, observed with all compositions under laboratory ambient conditions within several months, indicates that long-term stability of the compositions under service conditions would be a major problem. Accordingly, development of plasticized booster compositions will not be pursued further. However, a compatible binder/plasticizer combination may be a possible future option if other current approaches to development of an insensitive booster composition (e.g. use of other explosives, particle size modification) prove to be unsuccessful.

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ABSTRACT

Plasticizers have been incorporated into RDX/ethylene-vinyl acetate (EVA) compositions prepared by the solvent slurry coating technique. The effect of plasticizers on impact sensitiveness, shock sensitivity, cookoff behaviour and integrity of pressed pellets has been examined for compositions with EVA binders of varying vinyl acetate content. A compatible EVA/plasticizer combination gave an appreciable reduction in cookoff response and impact sensitiveness. Plasticizer migration is likely to affect the long-term stability of these compositions.