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FOX-7 for Insensitive Boosters

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ABSTRACT

Recent focus on Insensitive Munitions has directed research towards ingredients and formulations that can be used to produce insensitive explosives and ordnance, with efforts largely concentrated on main charge fills. Another key component of any munition is the booster system, which is required to reliably initiate the main charge. This requires attention as it is often the most sensitive component and unintentional initiation could lead to a violent reaction from the munition. 1,1-Diamino-2,2-dinitroethylene (FOX-7) is a recently developed high explosive ingredient with significant potential for application in a range of high performance, insensitive explosive compositions. The performance of FOX-7 is close to that of the benchmark nitramine explosive RDX and sensitiveness and hazards testing of FOX-7 have provided favourable results. Previous studies utilising FOX-7 have demonstrated its potential for insensitive booster formulations, easily meeting basic requirements for this application. This report details further testing of the performance and sensitivity of FOX-7 booster formulations. Also included are particle processing techniques using ultrasound, designed to optimise FOX-7 crystal size and morphology to improve booster formulations, and results from these experiments.

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Executive Summary

A key component of any munition is the booster system, which is required to reliably initiate the main charge. The impetus for Insensitive Munitions has directed research towards ingredients and formulations that can be used to produce insensitive boosters. The insensitive explosive TATB has been incorporated into formulations for this reason, but is not ideal as it has lower performance than commonly used explosives such as RDX.

1,1-Diamino-2,2-dinitroethylene (FOX-7) is a recently developed high explosive ingredient with significant potential for application in a range of high performance, insensitive explosive compositions. The performance of FOX-7 is close to that of the benchmark nitramine explosive RDX and sensitiveness and hazards testing of FOX-7 have provided favourable results. Previous studies utilising FOX-7 have demonstrated its potential for insensitive booster formulations, easily meeting basic requirements for this application.

This report details further testing of the performance and sensitivity of FOX-7 booster formulations. Also included are particle processing techniques using ultrasound, designed to optimise FOX-7 crystal size and morphology to improve booster formulations. These experiments indicate that ultrasound recrystallisation will give reproducible results, but traditional techniques can still produce low sensitivity FOX-7 formulations. The prepared FOX-7 boosters had comparable performance to that of similar RDX formulations, with significantly reduced shock sensitivity.

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Merran Daniel joined DSTO in 2006 as part of the Explosives and Pyrotechnics Group after completing a PhD in organic chemistry at Flinders University in 2005. Her work has been focussed on reduced sensitivity explosives, including polymer bonded explosive formulation and FOX-7 for insensitive boosters.

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Phil Davies obtained a PhD in Physical and Inorganic Chemistry from Flinders University of South Australia in 1998. He joined DSTO in 1999 and has been involved in Operations Analysis in support of future land warfare, soldier modernisation, urban operations and counter-terrorism. In 2003 he moved "back into chemistry" and joined Explosives and Pyrotechnics Group (Weapon Systems Division) to undertake research aimed at the development of Insensitive Munitions technologies, including novel formulations and physical mitigation techniques. He is also currently undertaking research aimed at the synthesis and characterisation of improvised explosive materials.

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Ian has been part of the Explosives and Pyrotechnics Group at DSTO Edinburgh since 1998 and became head of the group in 2009. His research has included the synthesis and characterisation of FOX-7, formulation and testing of cast-cured polymer bonded explosives (PBXs) and investigations into Reduced Sensitivity RDX. Current research interests include the characterisation of multiphase blast explosives, and the effect of ageing on properties of PBXs and munitions.

Contents

Abbreviations

DSC	Differential scanning calorimetry
DMF	N,N-Dimethyl formamide
DMSO	Dimethyl sulfoxide
ESD	Electrostatic Discharge
EVA	Poly[ethylene-co-vinyl acetate], 40 wt% vinyl acetate
FofI	Figure of Insensitiveness
FOI	Swedish Defence Research Agency
FOX-7	1,1-Diamino-2,2-dinitroethylene
GBL	γ-Butyrolactone
GC	Gas chromatography
HMX	cyclotetramethylene tetranitramine, octogen
ICT	Institut für Chemische Technologie
IM	Insensitive Munitions
NMP	N-Methyl pyrrolidinone
P	Detonation pressure
PE	polyethylene
PETN	Pentaerythritol tetranitrate
RDX	Cyclotrimethylene trinitramine, hexogen
SEM	Scanning electron microscopy
SSCB	Super Small-Scale Cook-off Bomb
SSGT	Small Scale Gap Test
TATB	1,3,5-Triamino-2,4,6-trinitrobenzene
TGA	Thermogravimetric analysis
TMD	Theoretical maximum density
ToI	Temperature of Ignition
VoD	Velocity of detonation
VTS	Vacuum Thermal Stability

1. Introduction

A key component of any munition is the booster system, which is required to reliably initiate the main charge. In the past tetryl (2,4,6-trinitrophenyl-N-methylnitramine) was used as a filling in leads, boosters and magazines of most Australian produced fuzes, but due to health hazards and pollution problems it has been phased out. As a result of this, DSTO has previously conducted a number of studies related to the development of an Australian booster formulation [1-3], largely based on RDX/polymer formulations [4, 5]. Focus on safer munitions directed the research towards insensitive boosters [6-13], and this requirement was reinforced by the Insensitive Munitions (IM) policy [14], issued in Australia in 2005.

As part of the detonation train, an insensitive booster is a critical component of any future IM compliant system. Dagley and co-workers [13] identified basic elements of an effective insensitive booster composition, and these have been modified to comply with the IM policy:

- it must be no more impact sensitive than tetryl (ie. F of I > 90), in order to comply with fuze train safety guidelines;
- it should be less shock sensitive than tetryl, but must have sufficient shock sensitivity to initiate from fuzing systems; the shock sensitivity of PBXN-7 Type II (an insensitive booster composition developed in the US) was chosen as the minimum acceptable;
- it should have pickup behaviour and output power similar to that of tetryl;
- it should respond mildly (burn or no reaction) under both fast and slow cook-off conditions, since violent (non-detonative) response of the booster may be sufficient to initiate the main charge; and
- it should be processable on a small batch scale in existing or easily acquired plant.

Most of the original replacements for tetryl were RDX-based, with a binder/desensitiser, plus additional components, such as pentaerythritol tetranitrate (PETN) or 1,3,5-triamino-2,4,6-trinitrobenzene (TATB)[13] . The recent focus on insensitive munitions has seen the inclusion of the insensitive explosive TATB into many new booster formulations. TATB has lower performance than other high explosives and so it must be combined with RDX or HMX to meet performance requirements. PBXN-7 (RDX 35%; TATB 60%; Viton 5%) is an example of a TATB-based formulation which has been qualified for use in boosters in the US.

Another recently developed explosive with low sensitivity is 1,1-diamino-2,2-dinitroethylene (FOX-7) [15]. The performance of FOX-7 is close to that of the benchmark nitramine explosive RDX and sensitiveness and hazards testing of FOX-7 have provided favourable results, showing significant potential for application in a range of high performance, insensitive explosive compositions. These properties have identified FOX-7 as a potential candidate to meet booster requirements without the need to combine it with other explosive materials.

2. Preliminary Evaluation of FOX-7 Booster Properties

Sensitiveness characteristics of various batches of FOX-7 were assessed (Table 1) and were found to be comparable or better than RDX, but additional tests were required to further evaluate the suitability of FOX-7 in booster formulations in terms of both safety and performance.

Table 1: Sensitiveness test data

Test	FOX-7 (Bofors 20017011)[3]	FOX-7 (Bofors 20017002)[4]	FOX-7 (DSTO)[5][16]	RDX
Rotter Impact (F of I)	100	100	110-140	80
BAM Friction (N)	240	240	168-288	~120
ESD[1] . Ignition (J)	4.5	4.5	4.5	4.5
ESD[1] . No Ignition (J)	0.45	0.45	0.45	0.45
VTS[2] (mL/g)	0.28	-	≤ 0.1	≤ 0.1
Temp. of Ignition (°C)	217	-	226	223
Bickford Fuse	Ignition	-	Fails to ignite	Fails to ignite
Train Test	Ignition	-	Ignition	Ignition

[1] Electrostatic Discharge
[2] Vacuum Thermal Stability (performed at 100°C for 48 h)
[3] Bofors, not recrystallised
[4] Bofors, recrystallised
[5] Synthesised at DSTO, not recrystallised

2.1 Formulation and Preparation

For preliminary assessment in booster formulations, FOX-7 was coated with 5%[1] EVA polymer (poly[ethylene-co-vinyl acetate], 40% vinyl acetate) [16] and the resultant material pressed into pellets. Pellets for shock sensitivity testing were 12.7 mm diameter x 12.0 mm, pressed to a constant load of 20 kN, with average density 1.666 g/cm^3 (93.1% theoretical maximum density (TMD)). Pellets for cook-off tests were 15.9 mm diameter x 16 mm, pressed to a constant load of 40 kN, with average density 1.664 g/cm^3 (93% TMD). Pellets for velocity of detonation (VoD) and detonation pressure were 25.1 mm diameter x 25 mm and pressed to 100 kN, with average density 1.686 g/cm^3 (94.3% TMD). The actual EVA content was measured by removing the polymer with dichloromethane and weighing the remaining FOX-7, giving an average of 4.9% EVA. RDX was coated using the same technique and pressed into pellets of the same size and approximate %TMD.

2.2 Cook-off Response

A key concern regarding booster sensitivity is cook-off response. Violent response of the booster to fast and slow cook-off could provide sufficient shock stimulus to the main charge for a shock to detonation transition to occur. Alternatively, a violent response leading to hot, high velocity fragments from the booster assembly could also lead to main charge initiation [6, 17, 18]. The

[1] Unless otherwise stated percentages are weight/weight.

response of FOX-7/EVA (95:5) and RDX/EVA (95:5) to fast and slow cook-off was assessed using the MRL super small-scale cook-off bomb (SSCB) [19]. Tests were performed in triplicate for each material, and the results are detailed in Table 2. These results clearly demonstrated that for both fast and slow cook-off, FOX-7 reacts at higher temperatures with notably milder responses than an equivalent RDX formulation.

Table 2: Cook-off data from SSCB

Explosive	% TMD	Rate	Temp. (°C)	Reaction Type
FOX-7/EVA(95:5)	93	Fast	235	Mild burn × 3
FOX-7/EVA(95:5)	93	Slow	240	Mild burn × 2, burn × 1
RDX/EVA(95:5)	96	Fast	220	Detonation × 2, deflagration × 1
RDX/EVA(95:5)	96	Slow	209	Deflagration × 3

2.3 Shock Sensitivity

The shock sensitivity of FOX-7/EVA (95:5), together with analogous RDX/EVA (95:5) pressed formulations, was determined using the MRL small-scale gap test (SSGT) [20], which uses the formation of a dent in the witness plate as confirmation of detonation. Results are presented in Table 3 with some comparative data, obtained via the same test, for PBXW-7 and tetryl [21]. The shock sensitivity of the FOX-7/EVA is low, but lies between that of PBXW-7 and tetryl, meeting the stated requirements. The relatively low sensitivity of FOX-7 to shock stimuli is consistent with earlier results obtained by FOI [22].

Table 3: Shock sensitivity data from SSGT

Explosive	Density (g/cm^3)	% TMD	$M_{50\%}$ mm brass shim (thou. in.)
FOX-7 / EVA (95:5)	1.666	93.1	1.575 (62)
RDX / EVA (Thales) (95:5)	1.621	94.0	1.956 (77)
RDX / EVA (SNPE) (95:5)	1.627	94.4	1.676 (66)
PBXW-7 Type II (PBXN-7)[21]	-	90	1.415 (56)
Tetryl (Crystalline)[21]	-	90	2.814 (111)
Tetryl (Granular)[21]	-	90	3.259 (128)

2.4 Performance

Preliminary velocity of detonation (VoD) and Chapman-Jouget pressure (P_{CJ}, detonation pressure) results for FOX-7/EVA formulations had been determined at DSTO [16] (12.7 mm pellets, Table 4). More recently, charges of 25 mm diameter × 225 mm, made from 10 stacked pellets, underwent assessment. The charges were initiated using an EBW detonator with a 25 mm × 20 mm RDX/EVA pellet as a booster. VoD was measured using ionisation pins spaced 20 mm apart (Fig. 1). Detonation pressure was estimated from the dent depth in a 150 × 150 × 50 mm steel witness plate relative to that created by a 25 mm diameter Composition B charge (P_{CJ} = 26.4 GPa). Duplicate experiments were performed, and the results of these tests (Table 4), indicate that FOX-7 has comparable performance to RDX.

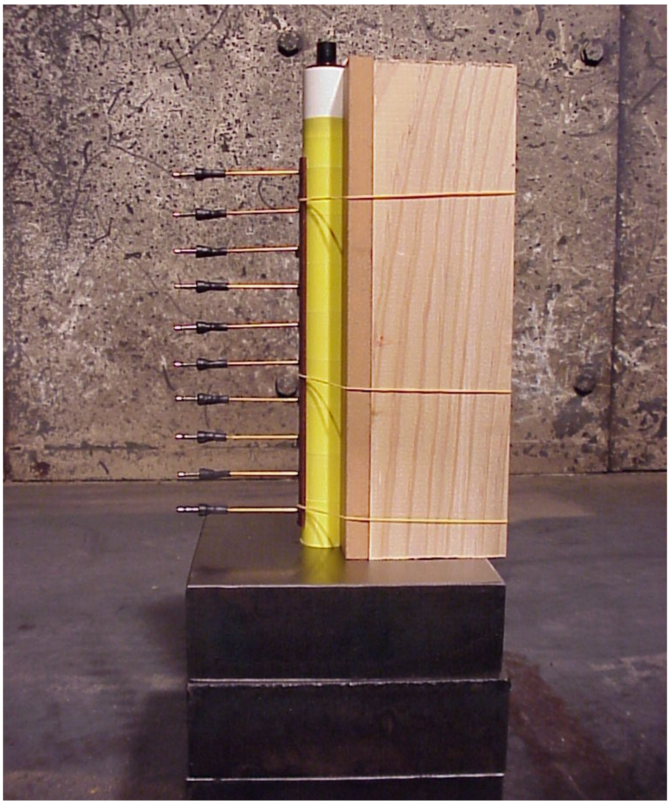

Figure 1: Velocity of detonation/detonation pressure experiment on FOX-7/EVA

Table 4: Measured performance parameters

Explosive	Diameter (mm)	%TMD	Experimental		Theoretical[1]	
			VoD (m/s)	P_{CJ} (GPa)	VoD (m/s)	P_{CJ} (GPa)
FOX-7/EVA	12.7	92	7730[16]	24.1[16]	7691	22.9
RDX/EVA	12.7	92	7630[16]		7731	23.0
FOX-7/EVA	25	94.3[2]	8110	25.1	7845	24.4
RDX/EVA	25	94.4[2]	8248	26.9	8008	25.7

[1] Cheetah v2.0
[2] Actual densities: FOX-7/EVA = 1.686 g/cm^3 RDX/EVA = 1.628 g/cm^3

Comparison of calculated FOX-7 performance parameters with other insensitive explosives is shown in Table 5. These calculations indicate that while comparable to RDX, FOX-7 has significantly higher performance than both NTO and TATB.

Table 5: Calculated performance parameters for selected explosives

Explosive	Calculated[1] Performance Parameters	
	Velocity of Detonation (m/s)	Detonation Pressure (GPa)
FOX-7	8849	33.7
RDX	8940	34.7
NTO	8564	31.2
TATB	8108	31.1

[1] Cheetah v2.0

These studies utilising FOX-7 demonstrated its potential for insensitive booster formulations, easily meeting basic requirements for this application. The shock sensitivity of FOX-7/EVA (95:5) booster pellets was within the suggested range [13] while the velocity of detonation, both experimental and calculated, was within 2% of an equivalent RDX formulation.

Recently, Orzechowski and coworkers [23] tested a similar formulation containing FOX-7 phlegmatised with 5% PTFE. When pressed into charges 22 mm in diameter by 18 mm at a density of 1.82 g/cm^3, the explosive was found to have a detonation velocity of 8200 m/s. The formulation possessed impact, friction and shock sensitivity significantly lower than an equivalent RDX formulation, which gave a detonation velocity of 8200 m/s at a density of 1.72 g/cm^3.

3. Particle Processing

Given the results indicating that FOX-7 had suitable characteristics for insensitive boosters, methods to optimise both sensitivity and performance were investigated. The sensitivity of an explosive can be affected by crystal quality, size and morphology, and by the packing of the crystals during pressing. Altering the particle size and shape of the explosive can also improve performance, by maximising the packing density. It was found that the original batch of recrystallised FOX-7 supplied to DSTO by Eurenco Bofors was generally unsuitable for pressing by virtue of its large mean particle size (typically ~350 µm), leading to pellets with poor physical properties. Crystallisation techniques were therefore aimed at developing a reliable method for producing crystals of a suitable size (~100 µm) and morphology for boosters, whilst attempting to minimise crystal defects. The ideal crystal morphology for maximum packing efficiency is spherical crystals; plates and needles are undesirable as they fracture during pressing. A suitable morphology would be rhombic or similar blocky crystals.

3.1 Recrystallisation of FOX-7

In recent years considerable effort has been expended overseas to develop methods for recrystallising FOX-7 on an industrial scale [24]. Recrystallisation studies have thus been dominated by typical cooling methods utilising solvent or solvent/anti-solvent approaches. This requirement has been driven by the concept of mass production of FOX-7 for the eventual replacement of more sensitive explosives (including RDX) in a variety of ordnance. The DSTO concept of developing FOX-7 based insensitive boosters leads to a different set of requirements, which includes smaller scale batch-process crystallisation options. We could therefore explore other recrystallisation techniques which may not be suitable for commercial production, such as sonocrystallisation.

3.1.1 Overseas Developments in FOX-7 Recrystallisation

A collaborative study [24] conducted by FOI and ICT (Germany) identified a number of potential solvents and solvent/anti-solvent mixtures for the recrystallisation of FOX-7. The main aim of these studies was to investigate the effects of solvent on particle shape, surface texture, size and yield.

In the FOI/ICT study [24] the solubility of FOX-7 at various temperatures for a number of solvent systems, including N-methyl pyrrolidinone (NMP), NMP/water mixtures and N,N-dimethyl formamide (DMF), was explored. Additional recrystallisations were attempted using γ–butyrolactone (GBL) and acetonitrile. There was considerable variation in mean particle size when experiments were repeated, emphasising the difficulties in the reproducibility of standard cooling-type recrystallisation techniques. Morphologies of recrystallised FOX-7 ranged from rhombic to flat or rectangular, and were determined by SEM imagery.

Attempts to optimise FOX-7 recrystallisation have since been undertaken by other groups [25-27], using primarily NMP, DMF and dimethyl sulfoxide (DMSO) as solvents in both cooling and anti-solvent recrystallisation, with acetone or cyclohexane as common anti-solvents. In each case the method selected as producing the best crystals used different solvents, stirring rate and cooling rate, while some used anti-solvent. Each method gave 'optimised' crystals that varied considerably from those selected by other groups. All selected methods were chosen for crystals that were most spherical or gave the highest bulk density. The size range was then limited by what could be achieved with that solvent, with the stirring and cooling rates adjusted to give a final size distribution that was dependent upon how the FOX-7 was to be used.

3.1.2 Sonocrystallisation

In recent years researchers have explored the use of ultrasound for the recrystallisation of energetic materials [28, 29], and have found that improvements in the morphology can be achieved together with greater control of crystal size. Ultrasonic cavitation induces nucleation providing a dominant mechanism of crystal inception. The statistical relevance of fluctuations in homogeneous or heterogeneous seed particles (e.g. foreign particles, scratches on glassware etc) is eliminated leading to improved reproducibility.

It is generally understood that the application of ultrasound can have several effects. If ultrasound is applied only during the nucleation phase, larger crystal sizes result, than in analogous non-insonated recrystallisations. On the other hand if continuous ultrasound is applied smaller crystals result over a generally reduced span of particle sizes (Figure 2) [30]. It is claimed that similar results can be expected for essentially any recrystallisation product.

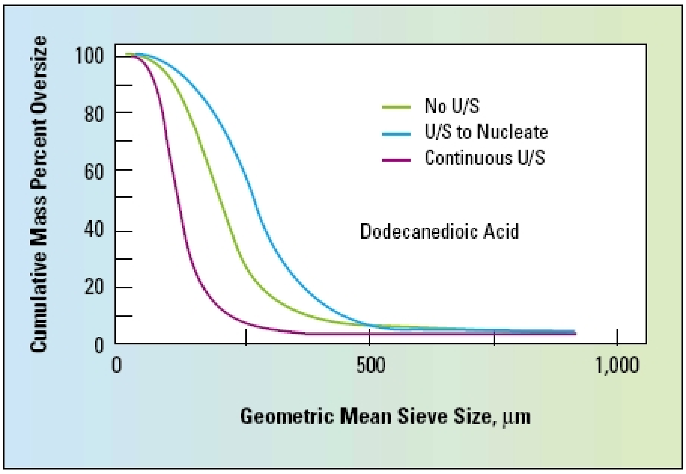

Figure 2: Typical results for application of ultrasound to the recrystallisation of dodecanedioic acid [30]

3.2 Recrystallisation of FOX-7 at DSTO

In an attempt to achieve improved morphology and reproducibility over slow cooling techniques, a relatively new sonocrystallisation technique and a simple anti-solvent (crashing) technique were investigated. It was anticipated that both of these methods would have characteristics (e.g. dominant secondary nucleation environments) that would minimise the opportunity for more random recrystallisation pathways and provide reproducible results. Anti-solvent experiments yielded quite small crystals (<50 um) with irregular or multi-layered morphologies, and so further experiments focused on sonication techniques.

3.2.1 Particle Size and Morphology

Development of a recrystallisation method required selection of a solvent that gave suitable morphology and size range under ultrasonic conditions, as well as determining appropriate frequency, amplitude and duration of ultrasound to give good quality crystals. Initial experiments in NMP/H_2O (50:50), using a 20 kHz Ultrasonics VCX750 ultrasound probe, evaluated the effect of amplitude and pulsing patterns. It was found that continuous ultrasound with an amplitude of 20% gave suitable results. As it was not feasible to trial every possible combination with all solvents, these conditions were employed for subsequent experiments.

3.2.2 Solvent Effects

The effect of solvent on particle size and morphology was investigated. All recrystallisations were conducted on a 3 g scale, with the exception of acetonitrile for which 1 g was used due to low solubility of the FOX-7. The temperature was controlled using a programmable water bath, set to cool from 90°C to 10°C at 2°C/min. The size and distribution of particles was obtained by particle sizing using a Malvern Mastersizer 2000 instrument, and scanning electron microscope (SEM) images were used to assess morphology (Table 6). The application of ultrasound in these experiments appeared to improve crystal quality in comparison to traditional recrystallisation, giving single crystals with limited agglomeration or twinning, and evidence of rounding.

Table 6: Effect of solvent on FOX-7 recrystallisation with 20 kHz ultrasound – 3 g scale

Solvent	**Particle Size (µm)**			**Morphology**
	d (0.1)	**d (0.5)**	**d (0.9)**	
NMP/H_2O (50:50)	10	29	66	Sharp blocks
GBL	7	16	30	Rounded plates
DMF	3	11	25	Rounded oblongs
DMF/H_2O (75:25)	12	35	78	Sharp blocks
Acetonitrile	30	59	103	Rounded blocks

After initial experiments, acetonitrile was deemed not viable as a solvent for large scale recrystallisation due to the low solubility of FOX-7 (1 g/160 mL). FOX-7 recrystallised from GBL was observed by DSC and TGA to decompose at a lower temperature (onset 190°C) than other samples (onset 220°C). The lowered decomposition was confirmed using temperature of ignition tests (Table 7), and is being investigated further. As this is an undesirable quality, GBL was not used further as a solvent for FOX-7 recrystallisation.

Table 7: Temperature of Ignition of FOX-7

	FOX-7 Bofors (20037002)	FOX-7 from NMP/H_2O	FOX-7 from GBL
Temperature of Ignition (°C)	215	226	200

The remaining three solvent combinations were evaluated further in recrystallisations on a 20 g scale. Once again, continuous 20 kHz ultrasound at 20% intensity was applied, and the solution was cooled from 90°C to 10°C at 2°C/min. Results are shown in Table 8.

Table 8: Effect of solvent on FOX-7 recrystallisation with 20 kHz ultrasound – 20 g

Solvent	**Particle Size (μm)**			**Morphology**
	d (0.1)	**d (0.5)**	**d (0.9)**	
NMP/H_2O (50:50)	33	71	163	Sharp blocks
DMF	4	14	33	Rounded oblongs/plates
DMF/H_2O (53:47)	22	56	117	Sharp plates

Recrystallisation from NMP/H_2O produced FOX-7 crystals of the best size, with reasonable morphology (Fig. 3), and also yielded better returns than the DMF systems, due to the lower solubility of the explosive. In large scale production this would not be an issue as material would remain in the system and be reused for subsequent recrystallisations.

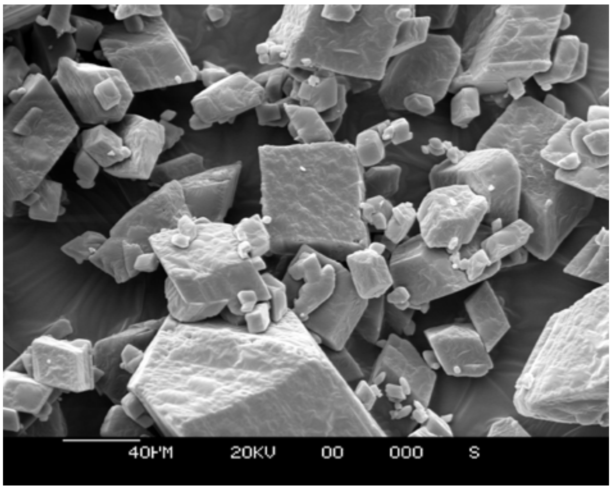

Figure 3: FOX-7 recrystallised from NMP/H_2O with 20 kHz ultrasound

3.2.3 Ultrasound Effects

It is not only the intensity and duration of the applied ultrasound that can affect particle formation, the frequency of the ultrasound has also been observed to influence the morphology and size of particles during recrystallisation [31]. Lower frequency ultrasound is expected to give smaller particles while higher frequencies give particles that are larger in size and smoother. This is believed to be due to the differing time available for cavitation, and therefore nucleation, to occur at each frequency. As well as producing ultrasound at different frequencies, the method of delivery of the ultrasound, and consequently the power density, differs in each technique as well. A probe was used to deliver the 20 kHz ultrasound, resulting in a focussed output that only affects particles in the pathway of the ultrasound, close to the tip. The 45 kHz ultrasound was delivered in a bath, with several point sources of ultrasound, producing a lower power density that is effective throughout the bath. The crystals formed using 20 kHz ultrasound in NMP/H_2O were compared to those using higher frequency ultrasound, and with no ultrasound.

Cooling recrystallisation in the absence of ultrasound was conducted in 50:50 NMP/H_2O on a 10 g scale initially, and then increased to 100 g, where a faster cooling rate was applied in an attempt to decrease particle size. In all experiments FOX-7 was dissolved at 90°C then cooled to 5°C with stirring at 500 rpm. The resultant crystals in each case were blocky and jagged with a large mean particle size (>200 µm), while the rapid stirring appeared to reduce the incidence of twinning and agglomeration compared to a non-stirred recrystallisation, although there were obvious defects (Fig. 4).

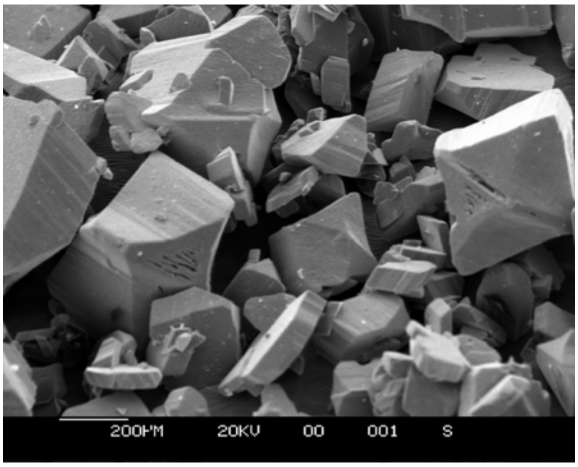

Figure 4: FOX-7 from traditional cooling recrystallisation

Recrystallisations using the 45±1 kHz (Decon FS200) ultrasound bath were conducted under the same cooling conditions as above, on a 10 g scale initially, and then increased to 100 g after a promising method was identified.

Application of continuous 45 kHz ultrasound produced crystals considerably different in size and morphology compared to that of the non-sonicated crystals, with rounding of the rhombic (in 3D) crystals evident (Fig. 5). Rounding appeared to occur primarily in the larger crystals and may be due to either Ostwald ripening (i.e. growing out at the expense of smaller crystals which are consumed), or perhaps grinding, since the larger crystals possess greater mass and momentum. Rounding of this nature is highly desirable in terms of both compaction and flow properties.

Application of continuous ultrasound with more rapid cooling regimes failed to yield similar rounding results. Use of sodium stearate (~0.1%) as a surfactant in the experiment increased the size to ~95 µm and produced good quality rhombic crystals indicating that sodium stearate may have some limited effect on crystal growth.

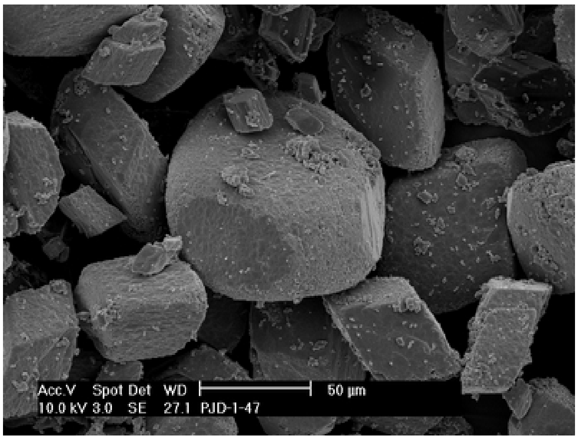

Figure 5: FOX-7 recrystallised using 45 kHz ultrasound

A number of other experiments were conducted in order to tailor the crystal sizes to approximately 100 µm while maintaining good crystal quality. After several small scale attempts it was found that pulsing the ultrasound at fixed durations could achieve the desired result. Increasing the scale of these experiments proved successful and highly reproducible. Good single crystals resulted and rounding of some of the larger rhombic particles was evident. Note that no attempt was made to create larger particles by applying ultrasound only during nucleation because the aim was to obtain smaller crystal sizes.

The various experiments show that a significant difference in particle size and morphology occurs between the case where no ultrasound was applied, to each case where ultrasound was used (Table 9). Figure 6 shows the crystal morphology produced from each technique – note that the magnification is different in each image to allow single crystal comparison.

Table 9: Effect of ultrasound on FOX-7 recrystallisation

Expt.	**Method**			**Particle size (µm)**			**Morphology**
	Application of Ultrasound	**Scale (g)**	**Cooling Rate (°C/min)**	**d(0.1)**	**d(0.5)**	**d(0.9)**	
#1	None	10	0.5	113	278	543	Blocky, jagged
#2	None	100	2.0	86	213	445	Blocky
#3	45 kHz continuous	10	0.5	30	59	105	Rounded, rhombic
#4	45 kHz continuous	10	1.0	-	<60[1]	-	Blocky
#5	45 kHz continuous, with surfactant	10	0.5	45	94	183	Rhombic
#6	45 kHz pulsed at 15 min intervals	100	0.5	48	98	186	Smooth, rhombic
#7	20 kHz continuous	100	0.5	65	105	170	Blocky
#8	20 kHz continuous	100	2.0	27	59	121	Blocky

[1] This is an estimate of the single crystal size, as particle sizing revealed a bimodal distribution, indicating agglomeration of particles, which began to break up when ultrasound was applied during measurement.

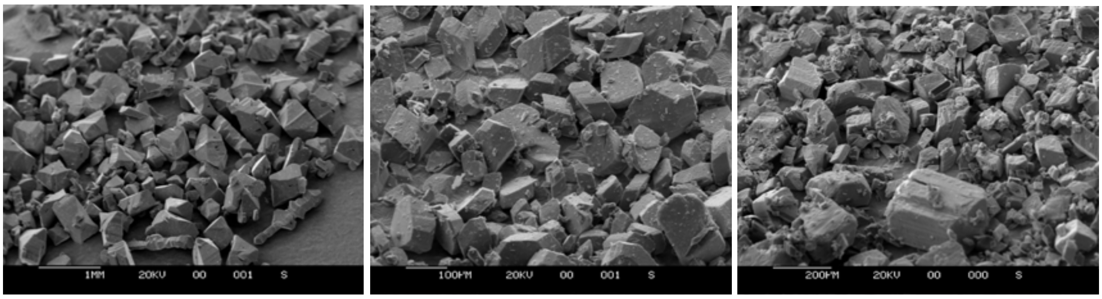

Figure 6: SEM images of FOX-7 from Experiments #2 (no ultrasound, left), #6 (45 kHz ultrasound, centre) and #8 (20 kHz ultrasound, right)

In summary, sonocrystallisation appears to offer considerable benefits in terms of both control of particle size and reproducibility, and attempts to produce a method for obtaining FOX-7 crystals likely to be of suitable size and shape for pressing have proven highly successful. To determine whether the optimised particle size and morphology of these crystals correlated to improved shock sensitivity, coating of the crystals and pressing into pellets, followed by testing, was conducted (section 4).

3.3 Solvent Inclusion

Apart from crystal size and morphology, crystal quality is also an important property for explosive sensitiveness and performance. While the above methods produced the desired single crystals, there are other possible defects. Solvent inclusion is often a concern during recrystallisation, as it leads to lower crystal quality and may affect sensitivity. One of the findings of the recent FOI/ICT work [24, 32] is that considerable solvent inclusion occurs in many recrystallised FOX-7 samples, particularly those samples recrystallised from NMP/water which show 2% solvent inclusion. Because loss of the included solvent occurred at around 160°C and the boiling point of NMP is high (202°C), the FOI/ICT report concluded that water was the likely inclusion. We note that boiling point is a bulk property, and since included solvent molecules within the lattice are likely to be separated from one another,[2] bulk properties are probably irrelevant and NMP molecules may desorp/vapourise at alternate temperatures. Indeed, the inclusion of NMP in the FOX-7 lattice has been observed by Bellamy and Brzoska [33] using GC/MS. In a separate study, Kjellestrom and co-workers determined that the solvent trapped in FOX-7 crystals formed from NMP/H_2O is principally NMP, with <0.1% H_2O (determined by Karl Fischer titration) [34].

The amount of solvent included in the FOX-7 crystals recrystallised in this study was estimated using thermogravimetric analysis (TGA), from the mass loss during the second phase transition (~160–180°C). This loss is identified in the differential scanning calorimetry (DSC) trace in the broadening and disfiguration of the peak during this transition (Fig. 7). The solvent inclusion in all samples appeared to be less than 1%.

2 For example, residing between the 3Å spaces formed between the planar arrangements of FOX-7 molecules within the crystal structure.

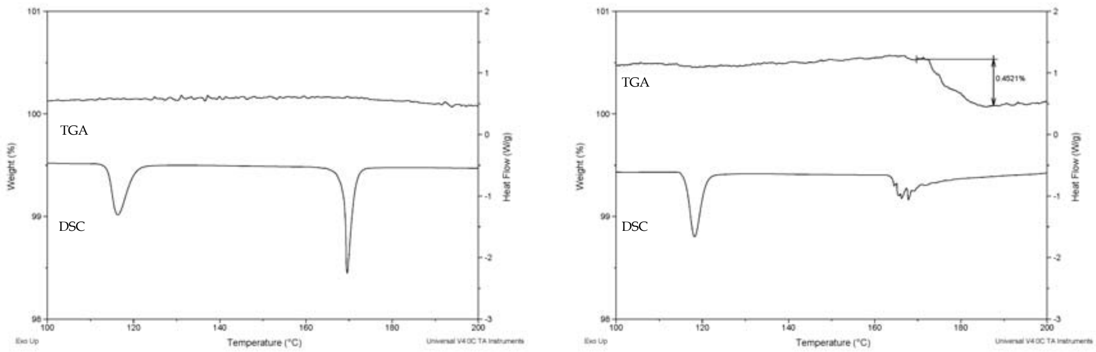

Figure 7: TGA and DSC traces of FOX-7: with no solvent (left) and with <1% solvent (right)

The concentration of NMP in the relevant samples was determined using gas chromatography (GC) (Varian CP-3800) with a flame ionisation detector (FID). A 25 m × 0.3 mm internal diameter BP5 column was used. The temperature of the injector was 200ºC, the column was heated from 100°C to 250°C at 15°C/min, and the detector was at 220ºC. The NMP standards and samples of FOX-7 were dissolved in ethyl acetate, and evaluated by integration. In the sample recrystallised with no ultrasound (#2) 0.4% NMP was found, while in the ultrasound-assisted recrystallisations the NMP was not detectable (<0.1%). The small amount of solvent did not appear to have a detrimental effect on the shock sensitivity of the crystals (see Table 11).

4. Booster Evaluation

4.1 Preliminary Coating

A number of binder coating options exist to facilitate the production of good quality pressed booster pellets. EVA was used in preliminary studies, however focus was moved to the use of a polyethylene wax (PE-wax) binder system currently employed in the production of the in-service RDX booster compositions (TR1 and TR2). The coating process is more straightforward than the EVA coating previously used at DSTO. Initial results on PE-wax coated FOX-7 on a laboratory scale (10 g) proved to be highly promising, and were commensurate with those found by Wilson [4] in which PE-wax appears to coat the crystals and produces clusters with sizes broadly congruent with the percentage of PE-wax present (i.e. larger clusters for higher percentages), as shown in Figure 8.

Initial results for the thermal characterisation of PE-wax coated FOX-7 were also promising (Table 10). PE-wax appears to provide a degree of buffering from heat leading to marginal increases in temperature of ignition. This has also been observed in the results of DSC testing.

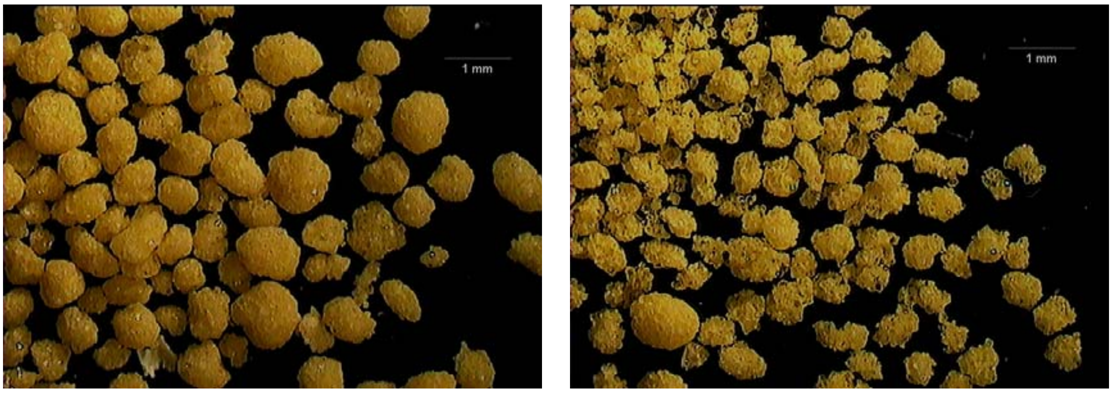

Figure 8: Micrographs of polyethylene wax coated FOX-7 (based on ~ 100 μm size) showing 10% PE-wax (left) and 1.5% PE-wax (right)

Table 10: Preliminary thermal characterisation of PE-wax coated FOX-7

%PE-wax Coating	T of I (°C)
0%	217
1.25%	236
1.5%	226
5%	231
7.5% PE-wax	232
10% PE-wax	241
15% PE-wax	243

Evaluation of the actual binder content was achieved using a method adapted from that used for PE-coated RDX [4]. A sample of the PE-coated FOX-7 was stirred in hot toluene and filtered. The filtrate was evaporated and the mass of the recovered wax was determined. Microscopy was used to confirm that all the wax had been removed from the FOX-7. A sample coated with a nominal wax content of 5% actually had 5.7% wax content, while a 1.25% sample contained 1.8% wax (slightly outside the TR1 specification of 1.25% + 0.25% wax). The 25% wax emulsion was analysed and found to have approximately 28% wax content, with further variation attributed to uneven mixing and/or settling of the solution.

4.2 Shock Sensitivity and Thermal Properties

The selected FOX-7 crystals from each of the sonication methods (#2, #6, #8 from Table 9) were coated using a polyethylene (PE) wax emulsion (Hoechst PED 521 wax) [4] with a nominal wax content of 5%. The coated material was pressed into pellets approximately 12.7 mm in diameter × 12.7 mm at the load indicated in Table 11, to approximately 97.5% TMD. RDX (Type 1) coated with 5% PE wax was used as a comparison. A mixture of Thales 70% grade A and 30% class 5, pressed at 25 kN was used to achieve the desired % TMD.

The shock sensitivity of each formulation was determined using the MRL Small Scale Gap Test (SSGT) [20], using 15 pellets due to limited material instead of the recommended 25–30. Details of the samples used in coating and the results of the tests are given in Table 11. FOX-7 coated with 5% EVA is included for comparison.

Table 11: Shock sensitivity data

Sample	Recrystallisation Technique	Pressing Force (kN)	%TMD	$M_{50\%}$ mm brass shim (thou. in.)
FOX-7 #2/PE	no ultrasound	15	97.5	<0.102 (<4)
FOX-7 #6/PE	45 kHz u/s	21	97.5	0.381 (15)
FOX-7 #8/PE	20 kHz u/s	25	97.3	0.544 (21)
FOX-7/EVA	Non-recryst. (DSTO)	20	93.1	1.575 (62)
RDX/PE	n/a	25	97.3	2.134 (84)

It can be seen that all of the PE-coated FOX-7 samples demonstrate low shock sensitivity, significantly below that of the equivalent RDX formulation and also the EVA-coated FOX-7.

During the testing of FOX-7 prepared using no ultrasound (experiment #2), the explosive required the removal of all gap cards to initiate, but even under these conditions the results varied between full detonation, partial detonation (very small dent in witness plate) and no detonation. This formulation appeared to be close to its critical diameter, which was confirmed by the reliable detonation of larger diameter (1″) pellets. As the formulation was close to the critical diameter for the initial experiments, the SSGT result could not be directly compared to the results of the other formulations, but the shock sensitivity appeared to be quite low. Potentially all of the FOX-7 formulations are close to their critical diameter, so this must be taken into consideration when viewing the results. It is also possible that the higher pressing forces used for some formulations may have damaged the crystals and affected the sensitivity.

The FOX-7 with the best packing and low shock sensitivity was the sample recrystallised without the application of ultrasound (#2). This indicates that the use of ultrasound, while giving rounded, more reproducible crystals within a narrow particle size distribution, is not required to produce crystals with low sensitivity for booster pellets. The improved packing of this sample is thought to be due to the broader particle size distribution giving a range of particle sizes which interact favourably. Optimised packing could also be achieved by blending batches of crystals formed from ultrasound recrystallisation, potentially leading to even lower sensitivity.

To accurately test the shock sensitivity of the crystals used in formulation #2, the possible critical diameter influence had to be removed. A procedure for the small scale gap test of 1″ pellets has not been qualified at DSTO, and limited material was also an issue, so the problem was addressed by increasing the sensitivity of the formulation to ensure full detonation. This was achieved by reducing the amount of binder from 5% to 1.25%, which then also made the formulation comparable to the RDX-based TR1 formulation (RDX 98.75%, PE wax 1.25%), used in Australian in-service boosters.

FOX-7 crystals coated with 1.25% PE wax were pressed as described previously into pellets for SSGT (15 kN load, ~95% TMD), and also into 16 mm diameter × 16 mm pellets for Super Small-scale Cook-off Bombs (SSCB) (23 kN load) to test the thermal properties of the formulation. Pellets of the TR1 formulation (Thales Australia) were also prepared for testing (at 25 kN for SSGT, 40 kN for SSCB). It was noted that the FOX-7 formulation had improved processing properties over TR1, as it was less prone to the breaking of pellet edges during and after pressing. The SSGT and cook-off results are given in Table 12.

Table 12: Sensitivity results for selected formulations

Sample	**%TMD**	**$M_{50\%}$ mm brass shim (thou. in.)**	**SSCB**			
			Fast		**Slow**	
			T(°C)	**Reaction**	**T(°C)**	**Reaction**
FOX-7/PE wax (98.75:1.25)	95.2	0.483(19)	240	Burn x 2 Mild burn x 1	233	Mild burn x 3
RDX (TR1) (98.75:1.25)	95.2	3.023(119)	185	Detonation x 2 Explosion x 1	206	Detonation x 1 Explosion x 2

The SSGT gave full detonation for the 1.25% wax FOX-7 formulation, and again it was seen that the shock sensitivity of the FOX-7 formulation was much lower than that of the comparable RDX formulation.

Earlier SSCB results demonstrated that the violence of reaction of FOX-7/EVA (95:5) formulations (mild burn/burn) was much lower than that of the equivalent RDX formulation, and an acceptable reaction for an insensitive munition which has the criteria of a response no higher than Type V (burning). The FOX-7/PE wax formulation also gave burn/mild burn responses, meeting IM criteria, reacting much less violently that the equivalent RDX formulation, which is currently used in service.

The higher proportion of explosive and higher %TMD of the PE formulation as compared to the EVA formulation leads to the prediction that the performance would be increased, giving VoD greater than the observed 8110 m/s of the FOX-7/EVA (95:5) formulation. FOX-7/PE wax (98.75:1.25) with a density of 1.77 g/cm^3 (95.2% TMD) was calculated to have a theoretical VoD of 8330 m/s and a detonation pressure of 28.5 GPa (Cheetah v2.0).

Other sensitiveness testing confirmed that the FOX-7/PE wax (98.75:1.25) formulation retained the low sensitivity properties of FOX-7 (Table 13).

Table 13: Sensitiveness test data

Sample	**Rotter Impact (F of I)**	**BAM friction (N)**	**ESD (J)**	**T of I (°C)**
FOX-7	100	240	4.5	226
FOX-7/PE wax 1.25%	110	216	>4.5	236

5. Conclusions

The processing of FOX-7 can alter the physical properties of the crystals such as size, morphology and crystal quality. Optimisation of recrystallisation techniques to produce improved crystals, with focus on the use of ultrasound, and the use of PE wax has led to a decrease in the sensitivity of FOX-7/wax formulations in pressed booster pellets. While sonocrystallisation gave the most reproducible results, yielding crystals of the morphology and size expected to produce boosters with enhanced performance and lowered sensitivity, it was traditional cooling techniques that led to crystals which gave the formulation with the best processing and lowest shock sensitivity. This does not imply that further optimisation of sonocrystallisation techniques could not lead to crystals with superior properties, as the current study focused on the potential of FOX-7 in boosters, rather than being a comprehensive investigation of recrystallisation methods. The lower solvent content obtained using ultrasound may also be important in other areas that have not been examined, such as ageing characteristics.

The identification of a suitable recrystallisation method allowed manipulation of the formulation to further improve the properties of the boosters. The result is a formulation comparable to that of the in-service composition TR1 booster. This and previous research indicate that the sensitiveness and thermal properties of FOX-7 formulations are much improved over similar RDX based formulations, and that their performance is not likely to be significantly different. While the shock sensitivity of the formulations was below the shock sensitivity previously recommended for insensitive boosters [13], it can be increased by blending FOX-7 with high-performance explosives such as HMX, which would be expected to further increase the performance, whilst maintaining lowered sensitivity, or the sensitivity can be tailored to a specific requirement by altering the binder type or amount.

With the current emphasis on producing insensitive munitions, FOX-7 appears to be a viable alternative to the more sensitive explosives in use and should be considered as an ingredient in insensitive boosters.

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Insensitive Munitions, Booster formulations, Booster system, FOX-7, High explosive ingredients, Polymer bonded explosives, Ultrasonics

19. ABSTRACT
Recent focus on Insensitive Munitions has directed research towards ingredients and formulations that can be used to produce insensitive explosives and ordnance, with efforts largely concentrated on main charge fills. Another key component of any munition is the booster system, which is required to reliably initiate the main charge. This requires attention as it is often the most sensitive component and unintentional initiation could lead to a violent reaction from the munition. 1,1-Diamino-2,2-dinitroethylene (FOX-7) is a recently developed high explosive ingredient with significant potential for application in a range of high performance, insensitive explosive compositions. The performance of FOX-7 is close to that of the benchmark nitramine explosive RDX and sensitiveness and hazards testing of FOX-7 have provided favourable results. Previous studies utilising FOX-7 have demonstrated its potential for insensitive booster formulations, easily meeting basic requirements for this application. This report details further testing of the performance and sensitivity of FOX-7 booster formulations. Also included are particle processing techniques using ultrasound, designed to optimise FOX-7 crystal size and morphology to improve booster formulations, and results from these experiments.