

# Studies on RDX of Improved Crystal Quality

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## Background

- Data indicates that OSI's RDX (Class 1 Type II) discolors upon accelerated aging
  - Increased shock sensitivity in cast cure PBX formulations also noted
  - Discoloration due to solvent occlusions in crystals
  - Not a new phenomenon: occurs with legacy material
- Discoloration and aging characteristics of OSI's RDX sparked an interest in Improved RDX (Im-RDX)
- Im-RDX previously developed at Holston
  - Higher purity than standard Bachmann RDX
  - Improved crystal quality over standard Bachmann RDX



Holston Army Ammunition Plant

## Background: Elevated Temperature Study of Class 1 RDX

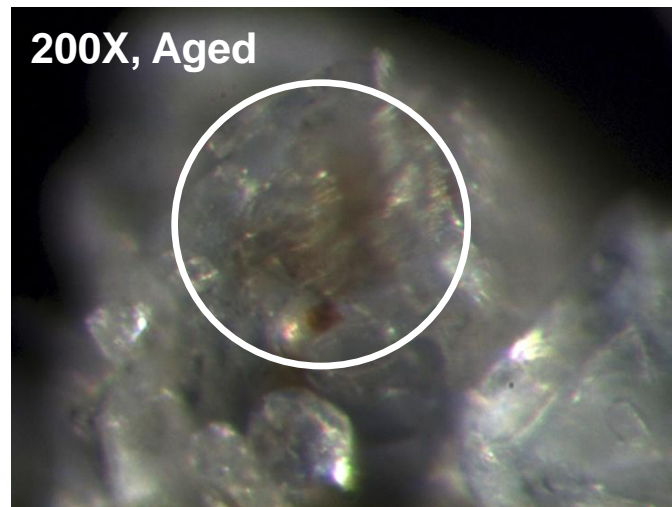
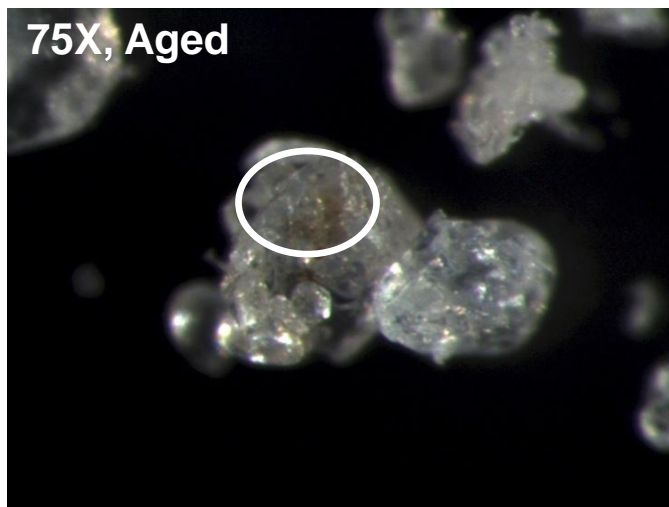
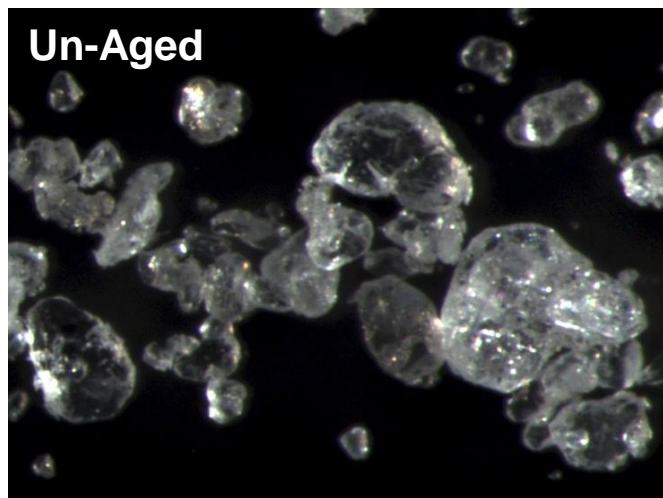
- Class 1 RDX aged 72 h at 100 °C
  - Aged material noticeably discolored



**Unaged**

**Aged**

# Background: Elevated Temperature Study of Class 1 RDX



## Objective

- Determine aging properties of Im-RDX in a cast cure PBX formulation
  - Im-RDX alone (not in a formulation) does not discolor upon aging
  - Theory: shock sensitivity of the formulation containing Im-RDX will be consistent before and after aging



## Approach

- Task 1: Im-RDX development
  - Laboratory scale development
  - Pilot scale development
  - Initial small scale aging study
- Task 2: Preparation and delivery of Im-RDX to China Lake
  - Im-RDX blending
- Task 3: PBXC-139 Formulation & Initial Testing
- Task 4: Accelerated Aging tests of Im-RDX in PBXC-139



# Task 1: Small Scale Crystal Modification Studies

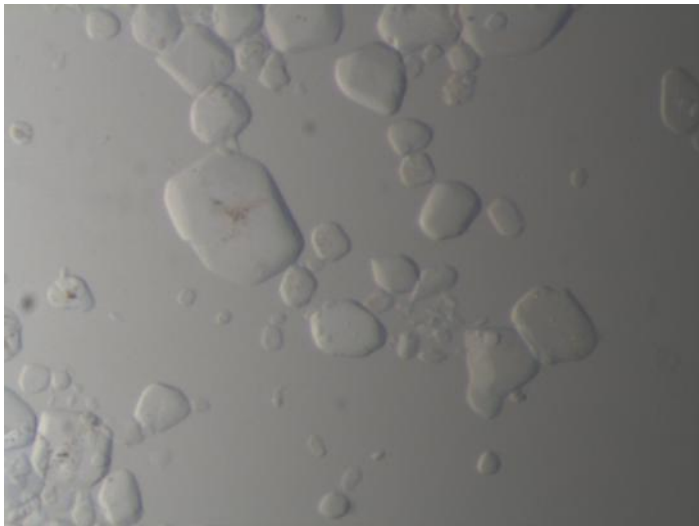
- Modified crystallization process explored to produce Im-RDX
  - Experiments focused on minimizing HMX content, voids, and occlusions
  - Process crystals with “smooth” edges
  - Yields >65% obtained with HMX content less than 0.5%



Lab Scale Im-Studies					
Sample	Before		After		%Yield
	%RDX	%HMX	%RDX	%HMX	
<b>138</b>	93.8	6.2	99.7	0.3	68
<b>142</b>	97.5	2.5	99.8	0.2	75
<b>143</b>	95.0	5.0	99.8	0.2	70
<b>144</b>	97.1	2.9	99.9	0.1	70
<b>149</b>	92.2	7.8	99.6	0.4	69
<b>150</b>	96.0	4.0	99.7	0.3	68
<b>128</b>	88.2	11.8	99.5	0.5	66

# Task 1: Pilot Scale Crystallization Studies

- Twenty six pilot scale recrystallizations performed
  - 350 lbs produced from OSI's class 1 RDX
  - Optimized dissolving temperature and cooling parameters
  - Material less sensitive to impact than OSI's class 1 RDX



Pilot Scale Im-Recrystallization					
Sample	Before		After		%Yield
	%RDX	%HMX	%RDX	%HMX	
<b>R1</b>	88.5	11.3	98.4	1.7	51
<b>R3</b>	88.8	11.2	99.0	1.1	73
<b>R4</b>	87.1	12.8	98.8	1.2	60
<b>R7</b>	90.3	9.7	98.8	0.9	53
<b>R9</b>	90.6	9.6	99.1	0.8	49
<b>R18</b>	92.0	8.1	99.3	0.7	39
<b>R25</b>	86.4	13.6	99.8	0.2	33



# Task 1: Im-RDX Aging Study

- Im-RDX from pilot scale recrystallization stored for five years
  - “True” aging results vs. accelerated aging
  - Material stored dry, ambient temperature
- Evaluated purity, impact, thermal properties (DSC) and also analyzed by optical microscopy
  - No changes in appearance under 50x magnification
  - No significant change in purity
  - DSC trace revealed sharp melting transitions
- Material less sensitive to impact than OSI’s Class 1 RDX

<b>Aged Im-RDX Purity and Impact</b>					
<b>Sample</b>	<b>Original</b>		<b>5 Years</b>		<b>Impact</b>
	<b>%RDX</b>	<b>%HMX</b>	<b>%RDX</b>	<b>%HMX</b>	<b>cm</b>
<b>R-18</b>	99.3	0.7	99.2	0.9	51
<b>R-22</b>	99.5	0.5	98.9	1.1	47
<b>R-25</b>	99.8	0.2	99.0	1.0	52
<b>R-26</b>	98.8	1.0	98.4	1.6	53
<b>Class 1 Std</b>			-	-	37

# Task 1: Im-RDX Elevated Temperature Study

- Im-RDX aged for 72 h at 100 °C
  - Elevated temperature study completed on 5 year aged material
  - Im-RDX does not discolor
  - No appreciable change in impact sensitivity

Sample #	Impact Sensitivity Change (%)
RS-RDX R-25	0.29
RS-RDX R-26	-0.35
Class 1 Avg	8.04

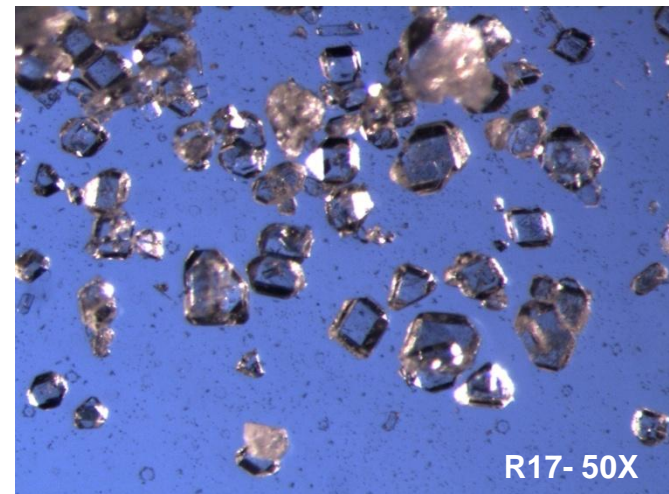
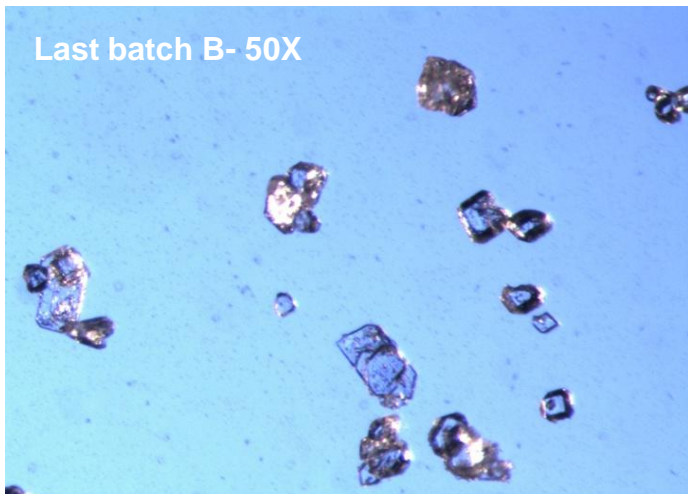
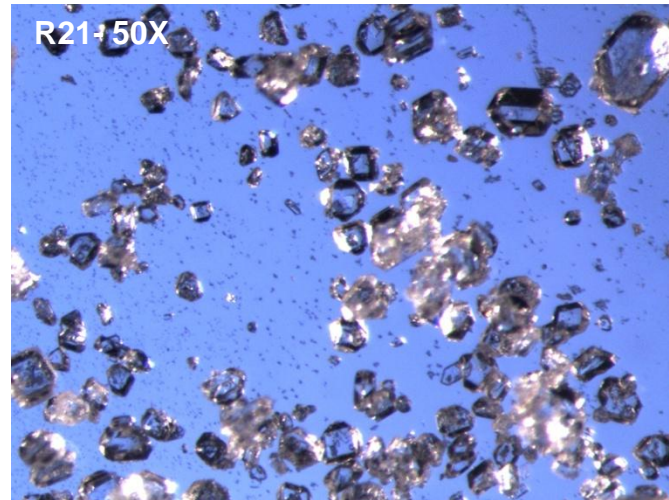
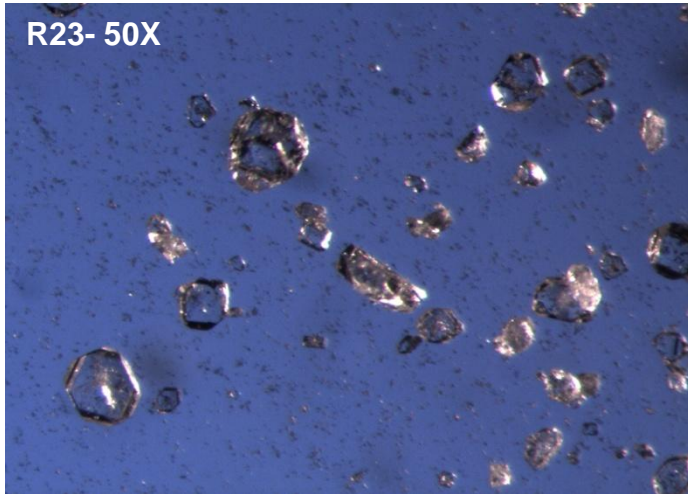


## Task 2: Im-RDX Blending for Evaluation in PBXC-139

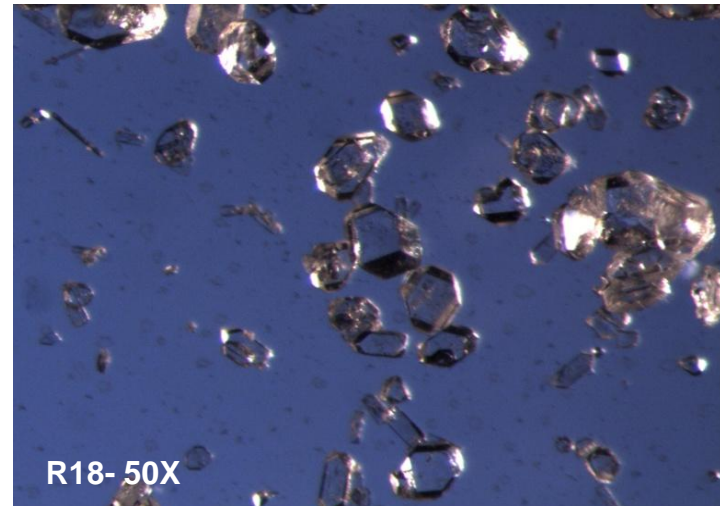
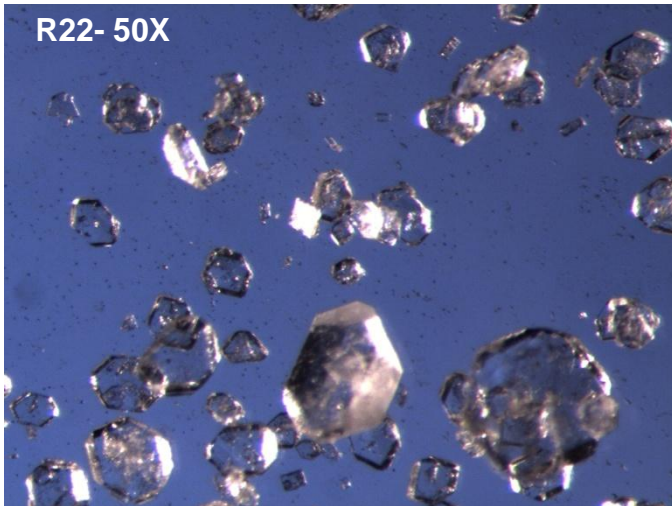
- Various pilot scale production lots evaluated
  - Analyzed morphology and particle size
  - Materials with best morphology and particle size combination chosen for blending
- 70 lbs of material blended water-wet
- Average particle size: 128 microns
  - Class 1 average approximately 228 microns
- Measured HMX content: 0.75%



## Task 2: Im-RDX Samples Chosen for Blending



## Task 2: Im-RDX Samples Chosen for Blending



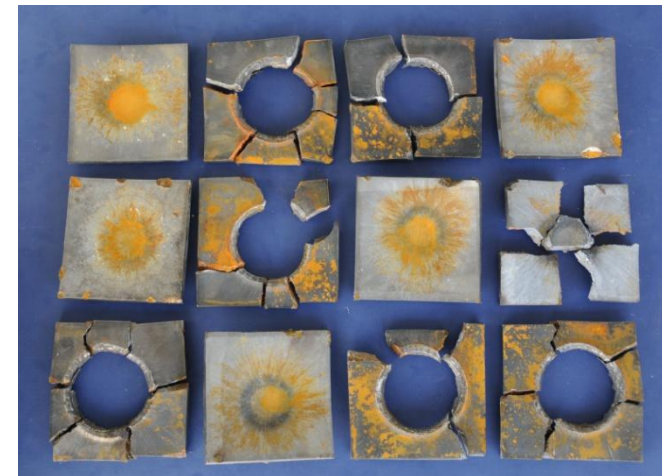
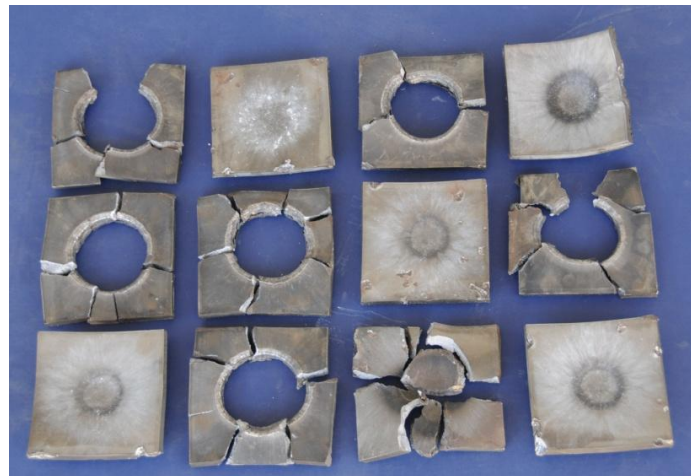
## Task 3: PBXC-139 Formulation

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- Im-RDX samples shipped to China Lake and formulated in PBXC-139
  - PBXC-139 samples subjected to a 1 year accelerated aging study: 70 °C
    - Samples taken every 3 months and evaluated for shock sensitivity
    - Time zero and first 3-month sample evaluated at this point
- Processing not significantly different than baseline formulation
  - End of mix viscosity less than 1 kpoise
  - Formulation flows nicely with low vibration level
- Formulation density no different than baseline formulation

# Task 4: Im-RDX Aging in PBXC-139

Type of RDX	Time (months)	LSGT (50%, Kbars)
Im-RDX	0	36.7
Im-RDX	3	36.3
Class 1 RDX	0	41.3
Class 1 RDX	3	31
Class 1 RDX	6	28



**Im-RDX Witness Plates at t = 0 Months**

**Im-RDX Witness Plates at t = 3 Months**

## Task 3: OSI's Im-RDX vs. Class 1 RDX

- Shock sensitivity of PBXN-109 formulated with standard Holston Class 1 RDX was previously tested by the US Navy (Indian Head)
  - Shock sensitivity determined via IMADGT: Insensitive Munitions Advanced Development Gap Test
    - IMADGT employs shorter, larger diameter than is used in LSGT
  - Different test methods for Im-RDX vs Class 1 RDX requires comparison using percent change in sensitivity
  - Class 1 baseline formulation aged for 1 year at 70 °C
- Table below compares Im-RDX to Class 1 RDX in both PBXN-109 and PBXC-139 studies

Type of RDX	Formulation	Time	% Increase
Class 1 RDX	PBXN-109	13 months	26
Class 1 RDX	PBXC-139	3 months	25
Class 1 RDX	PBXC-139	6 months	32
Im-RDX	PBXC-139	3 months	0



## Conclusions

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- Im-RDX has improved purity over Holston Class 1 RDX
- Im-RDX has improved crystal quality over Holston Class 1 RDX
- Unlike standard Holston Class 1 RDX, Im-RDX does not discolor upon accelerated aging
- Shock sensitivity of PBXC-139 formulation containing Im-RDX does not change upon accelerated aging for 3 months
  - Time zero shock of sensitivity of PBXC-139 containing Im-RDX greater than that of Class 1 RDX lots
  - Material will be sampled and tested again at 6 months

## Acknowledgements

- Ed LeClaire, Thomas Presley and Robyn Wilmoth for executing the Im-RDX crystallization work
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