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
Background: Elevated Temperature Study of Class 1 RDX

- Class 1 RDX aged 72 h at 100 °C
  - Aged material noticeably discolored
Background: Elevated Temperature Study of Class 1 RDX

Un-Aged

Aged

Brown occlusion

75X, Aged

200X, Aged
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%

<table>
<thead>
<tr>
<th>Sample</th>
<th>Lab Scale Im-Studies</th>
<th>%RDX</th>
<th>%HMX</th>
<th>%RDX</th>
<th>%HMX</th>
<th>%Yield</th>
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<tbody>
<tr>
<td>Before</td>
<td>After</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>%RDX</td>
<td>%HMX</td>
<td>%RDX</td>
<td>%HMX</td>
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<tr>
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<td>93.8</td>
<td>6.2</td>
<td>99.7</td>
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<td>149</td>
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<td>128</td>
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<td>11.8</td>
<td>99.5</td>
<td>0.5</td>
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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

<table>
<thead>
<tr>
<th>Sample</th>
<th>Before</th>
<th>After</th>
<th>%Yield</th>
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<tbody>
<tr>
<td></td>
<td>%RDX</td>
<td>%HMX</td>
<td>%RDX</td>
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<tr>
<td>R1</td>
<td>88.5</td>
<td>11.3</td>
<td>98.4</td>
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<td>R3</td>
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<td>R7</td>
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<tr>
<td>R25</td>
<td>86.4</td>
<td>13.6</td>
<td>99.8</td>
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</tbody>
</table>
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

<table>
<thead>
<tr>
<th>Sample</th>
<th>Original</th>
<th>5 Years</th>
<th>Impact</th>
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<tbody>
<tr>
<td></td>
<td>%RDX</td>
<td>%HMX</td>
<td>%RDX</td>
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<tr>
<td>R-18</td>
<td>99.3</td>
<td>0.7</td>
<td>99.2</td>
</tr>
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<td>R-22</td>
<td>99.5</td>
<td>0.5</td>
<td>98.9</td>
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<tr>
<td>R-25</td>
<td>99.8</td>
<td>0.2</td>
<td>99.0</td>
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<td>R-26</td>
<td>98.8</td>
<td>1.0</td>
<td>98.4</td>
</tr>
<tr>
<td>Class 1 Std</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
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</table>
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

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Impact Sensitivity Change (%)</th>
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<tbody>
<tr>
<td>RS-RDX R-25</td>
<td>0.29</td>
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<tr>
<td>RS-RDX R-26</td>
<td>-0.35</td>
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<tr>
<td>Class 1 Avg</td>
<td>8.04</td>
</tr>
</tbody>
</table>
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

R23- 50X

R21+50X

Last batch B- 50X

R17- 50X
Task 2: Im-RDX Samples Chosen for Blending

R22- 50X

R18- 50X
Task 3: PBXC-139 Formulation

- 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

<table>
<thead>
<tr>
<th>Type of RDX</th>
<th>Time (months)</th>
<th>LSGT (50%, Kbars)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Im-RDX</td>
<td>0</td>
<td>36.7</td>
</tr>
<tr>
<td>Im-RDX</td>
<td>3</td>
<td>36.3</td>
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<tr>
<td>Class 1 RDX</td>
<td>0</td>
<td>41.3</td>
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<tr>
<td>Class 1 RDX</td>
<td>3</td>
<td>31</td>
</tr>
<tr>
<td>Class 1 RDX</td>
<td>6</td>
<td>28</td>
</tr>
</tbody>
</table>

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

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

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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

<table>
<thead>
<tr>
<th>Type of RDX</th>
<th>Formulation</th>
<th>Time</th>
<th>% Increase</th>
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<tbody>
<tr>
<td>Class 1 RDX</td>
<td>PBXN-109</td>
<td>13 months</td>
<td>26</td>
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<td>PBXC-139</td>
<td>3 months</td>
<td>25</td>
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<td>Class 1 RDX</td>
<td>PBXC-139</td>
<td>6 months</td>
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<tr>
<td>Im-RDX</td>
<td>PBXC-139</td>
<td>3 months</td>
<td>0</td>
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</table>
Conclusions

- 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
• Lisa Hale for completing analytical work on the Im-RDX
• Dr. Neil Tucker, Dr. David Price, Mike Ervin for technical input