Im-RDX Production

Dr. Sarah A. Headrick
BAE Systems, Ordnance Systems, Inc.
Holston Army Ammunition Plant
Kingsport, TN
Background

- OSI’s RDX (Class 1 Type II) discolors upon accelerated aging due to solvent occlusions
  - Increased shock sensitivity in cast cure PBX formulations also noted
  - Occurs with legacy material
- Aging characteristics of OSI’s RDX sparked an interest in Improved RDX (Im-RDX)
- Im-RDX developed at Holston
  - Higher purity than standard Bachmann RDX
  - Improved crystal quality over standard Bachmann RDX
- **Pilot-scale completed in 2006**
- **Production-scale completed in late 2012**
Background: Accelerated Aging of Class 1 RDX

Un-Aged

Aged

Brown occlusion

75X, Aged

200X, Aged
Pilot Scale Crystallization Studies: 2006

- 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>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%RDX</td>
<td>%HMX</td>
<td>%RDX</td>
</tr>
<tr>
<td>R1</td>
<td>88.5</td>
<td>11.3</td>
<td>98.4</td>
</tr>
<tr>
<td>R3</td>
<td>88.8</td>
<td>11.2</td>
<td>99.0</td>
</tr>
<tr>
<td>R4</td>
<td>87.1</td>
<td>12.8</td>
<td>98.8</td>
</tr>
<tr>
<td>R7</td>
<td>90.3</td>
<td>9.7</td>
<td>98.8</td>
</tr>
<tr>
<td>R9</td>
<td>90.6</td>
<td>9.6</td>
<td>99.1</td>
</tr>
<tr>
<td>R18</td>
<td>92.0</td>
<td>8.1</td>
<td>99.3</td>
</tr>
<tr>
<td>R25</td>
<td>86.4</td>
<td>13.6</td>
<td>99.8</td>
</tr>
</tbody>
</table>
Im-RDX Aging Study: Pilot Scale Material

- Im-RDX from pilot scale recrystallization stored for five years
  - “True” aging results vs. accelerated aging
- 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>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%RDX</td>
<td>%HMX</td>
<td>%RDX</td>
</tr>
<tr>
<td>R-18</td>
<td>99.3</td>
<td>0.7</td>
<td>99.2</td>
</tr>
<tr>
<td>R-22</td>
<td>99.5</td>
<td>0.5</td>
<td>98.9</td>
</tr>
<tr>
<td>R-25</td>
<td>99.8</td>
<td>0.2</td>
<td>99.0</td>
</tr>
<tr>
<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>
</tbody>
</table>
Pilot Scale Im-RDX Elevated Temperature Study

- Im-RDX was aged for 72 h at 100 °C
  - Accelerated aging 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>
</tr>
</thead>
<tbody>
<tr>
<td>RS-RDX R-25</td>
<td>0.29</td>
</tr>
<tr>
<td>RS-RDX R-26</td>
<td>-0.35</td>
</tr>
<tr>
<td>Class 1 Avg</td>
<td>8.04</td>
</tr>
</tbody>
</table>
Production of Im-RDX

• Two 1500-lb batches produced in November 2012
  – Operating conditions varied slightly

• The two batches were evaluated for defects and purity
  – Batch SH1110-16 deemed to be the optimal batch

• 7RC112-749 is Class I RDX batch used to perform crystallization of SH1110-16

<table>
<thead>
<tr>
<th>Batch Number</th>
<th>Long Impact</th>
<th>% H₂O</th>
<th>% Acidity</th>
<th>% RDX</th>
<th>% HMX</th>
<th>Particle Size d(0.5)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7RC112-749</td>
<td>44.0</td>
<td>8.94</td>
<td>0</td>
<td>88</td>
<td>12</td>
<td>N/A</td>
</tr>
<tr>
<td>SH1110-16</td>
<td>62.5</td>
<td>8.9</td>
<td>0.01</td>
<td>97.26</td>
<td>2.74</td>
<td>118.86</td>
</tr>
</tbody>
</table>
Production Scale Im-RDX Photomicrographs
Production Scale Im-RDX Elevated Temperature Study

- Im-RDX samples placed in a 70 °C oven for 3 days followed by a 100 °C oven for 3 days
  - Class I RDX starting materials also aged
- Batch SH1110-16 exhibits discoloration than Class I RDX starting material

<table>
<thead>
<tr>
<th>Sample</th>
<th>7RC112-749</th>
<th>SH1110-16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Un-aged Impact</td>
<td>44.0</td>
<td>61.6</td>
</tr>
<tr>
<td>Aged Impact</td>
<td>38.8</td>
<td>61.7</td>
</tr>
<tr>
<td>% Change</td>
<td>-11.8%</td>
<td>+0.2%</td>
</tr>
</tbody>
</table>
Elevated Temperature Study Photomicrographs

SH11103-38, aged, 50X

SH11103-38, aged, 100X

SH1110-16, aged, 50X

SH1110-16, aged, 100X
Thermal Screening Unit (TSU)

Thermal Screening Unit

- Computer controlled temperature ramp
- Records several parameters during experiment:
  - Sample & Oven Temperature
  - Sample Pressure
  - Time
- Hastelloy Bombs (ARC type)
- K type thermocouples
- Pressure Transducer

Experimental Setup

- Temperature ramped to 115°C and held for 3 days (4320 minutes)
- Temperature and pressure recorded
TSU Results

RDX Comparisons (Class 1 and Class 5) - 115°C - 3 Days

Class 5/Cyclo
Class 1/Cyclo
TSU Results

RDX Comparisons (Class 1 and Class 5) - 115°C - 3 Days

Class 5/Cyclo

Class 1/Cyclo

Individual Components
TSU Results

RDX Comparisons (Class 1 and Class 5) - 115°C - 3 Days

Pressure, psi

Time, minutes

Cyclohexanone
RDX CL 1 / Cyclo
RDX CL 1
RDX CL 5
RDX CL 5 / Cyclo
IM RDX / Cyclo

IM-RDX
TSU Results

RDX Comparisons (Class 1 and Class 5) - 115°C - 3 Days

- **Class 5/Acetone**
- **Class 1/Acetone**
Conclusions and Future Work

• Im-RDX process has been generated on the production scale
  – Contains less crystal defects than Class I RDX
  – Exhibits improved aging characteristics over Class I RDX
  – Increased purity over Class I RDX

• TSU experiments are ongoing:
  – Stability of RDX classes in recrystallization solvents
  – Stability of IM-RDX to other classes

• Formulation efforts with IM-RDX are ongoing
Acknowledgements

- Tammy Bledsoe, Ben Schreiber, Dusty Cross, Don Darnell and Dave Edwards for Im-RDX production execution
- Ed LeClaire, Thomas Presley and Robyn Wilmoth for Pilot Scale Im-RDX crystallization
- Lisa Hale and Dr. Jeremy Headrick for Im-RDX analysis
- Dr. Neil Tucker, Dr. David Price, Dr. Jacob Morris, Mike Ervin for technical input