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Im-RDX Production

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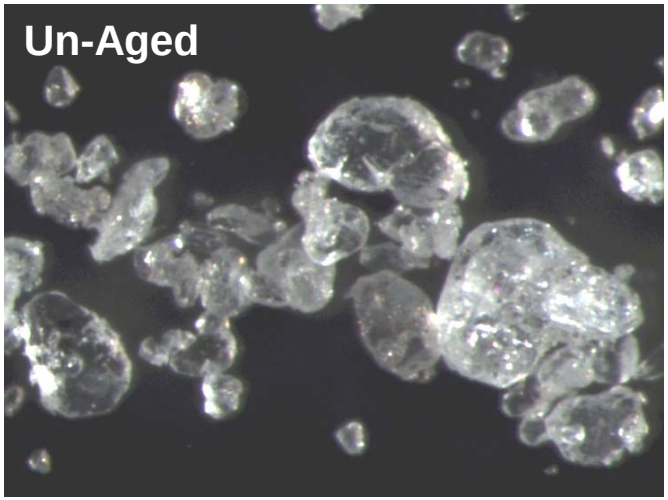
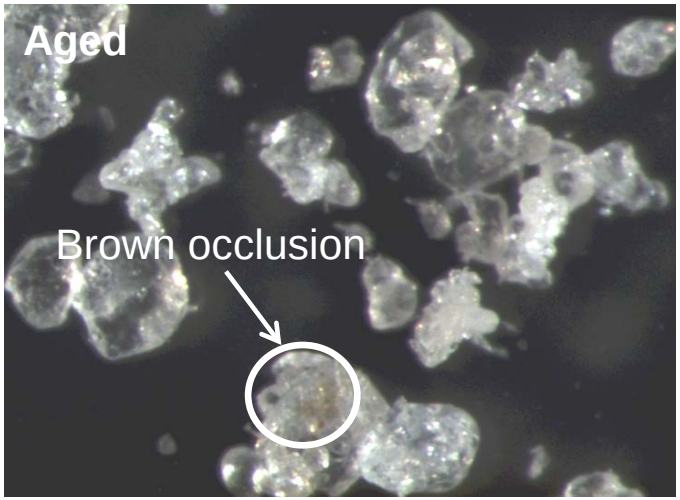
Background

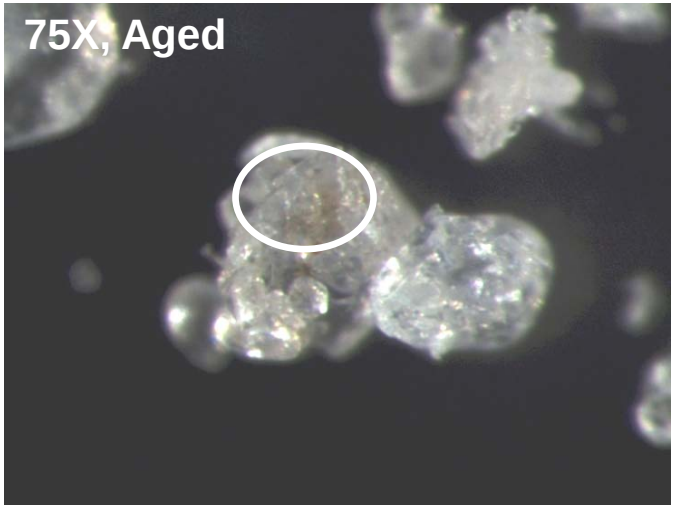
- OSI's RDX (Class 1 Type II) discolors upon accelerated aging due to solvent occlusions
  - Increased shock sensitivity in cast cure PBX formulations also noted
  - Occurs with legacy material
- Aging characteristics of OSI's RDX sparked an interest in Improved RDX (Im-RDX)
- Im-RDX developed at Holston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

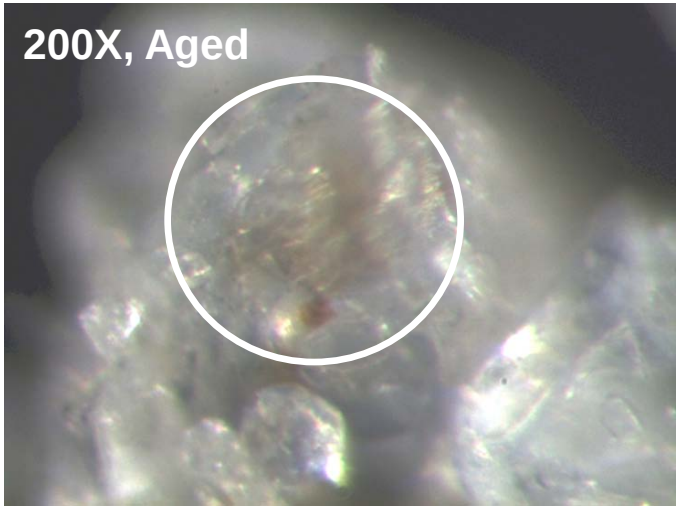
Holston Army Ammunition Plant

Background: Accelerated Aging of Class 1 RDX



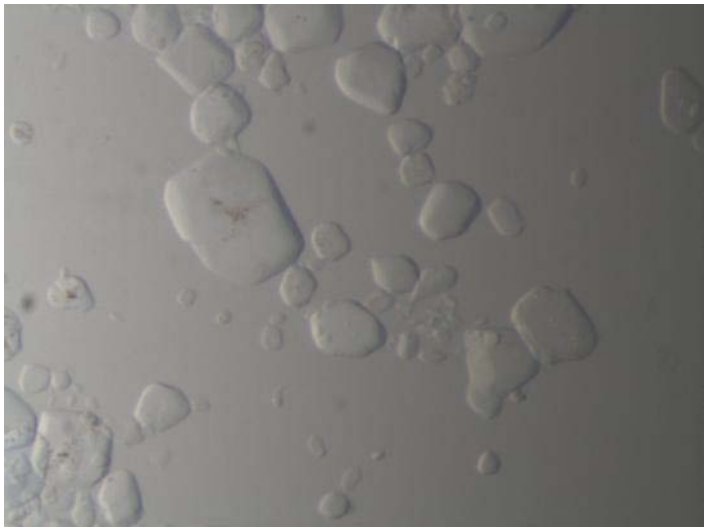






Pilot Scale Crystallization Studies: 2006

- 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



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

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

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

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

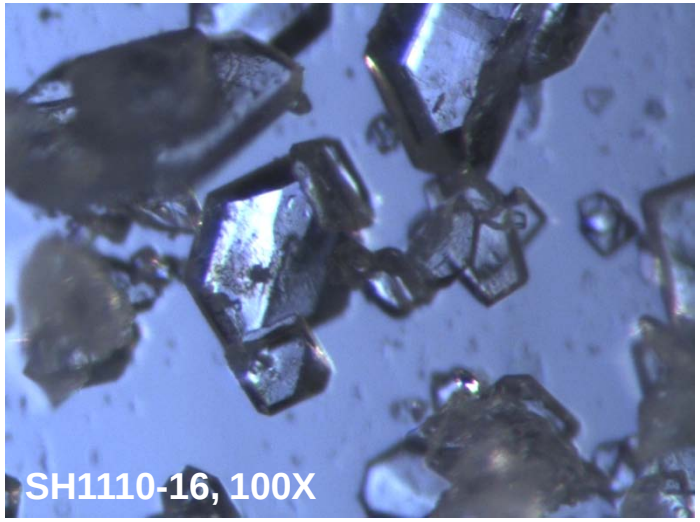
Sample #	Impact Sensitivity Change (%)
RS-RDX R-25	0.29
RS-RDX R-26	-0.35
Class 1 Avg	8.04

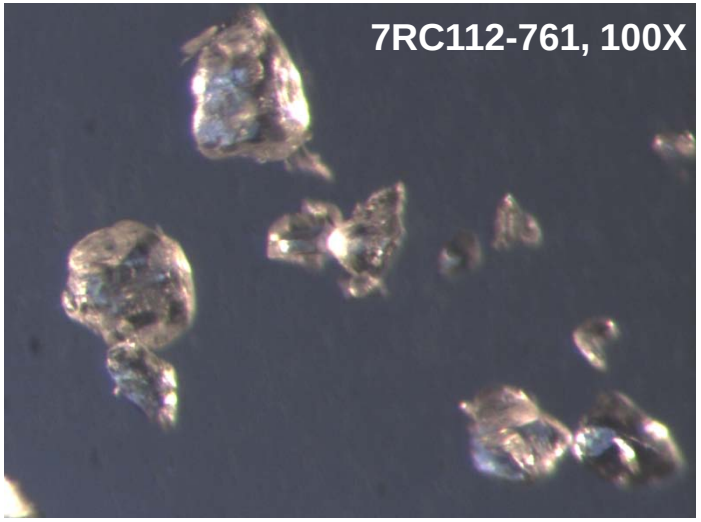
Production of Im-RDX

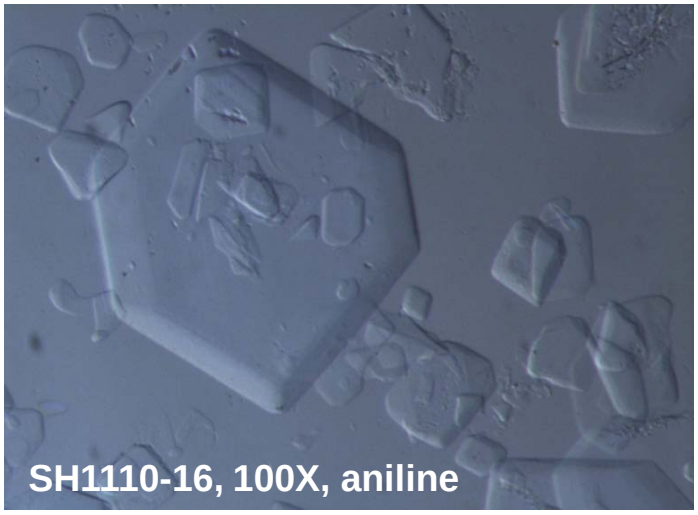
- Two 1500-lb batches produced in November 2012
  - Operating conditions varied slightly
- The two batches were evaluated for defects and purity
  - Batch SH1110-16 deemed to be the optimal batch
- 7RC112-749 is Class I RDX batch used to perform crystallization of SH1110-16

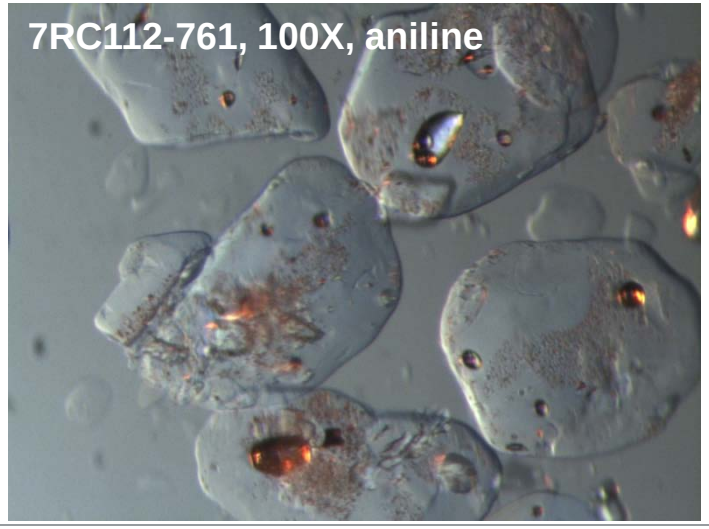
Batch Number	Long Impact	% H_2O	% Acidity	% RDX	% HMX	Particle Size d(0.5)
7RC112-749	44.0	8.94	0	88	12	N/A
SH1110-16	62.5	8.9	0.01	97.26	2.74	118.86

Production Scale Im-RDX Photomicrographs

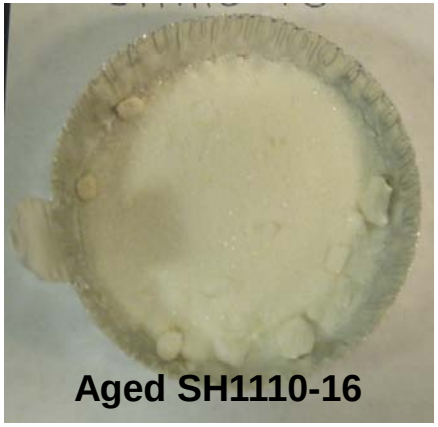


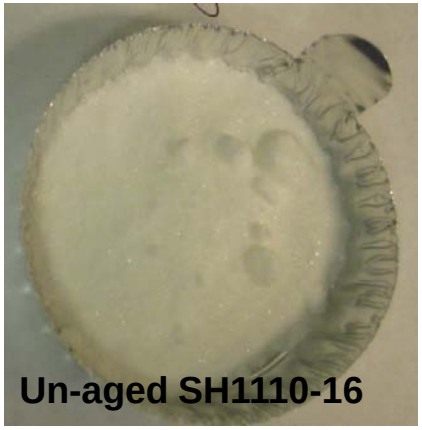


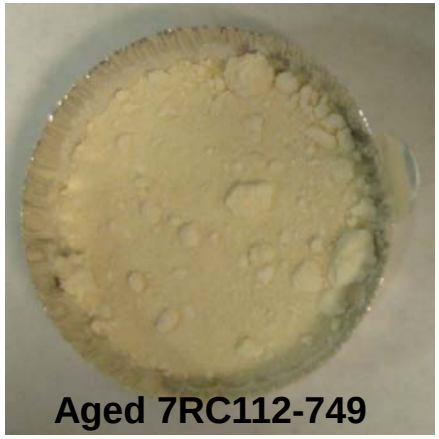


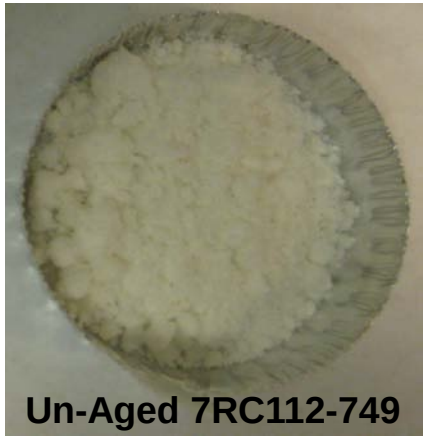


Production Scale Im-RDX Elevated Temperature Study





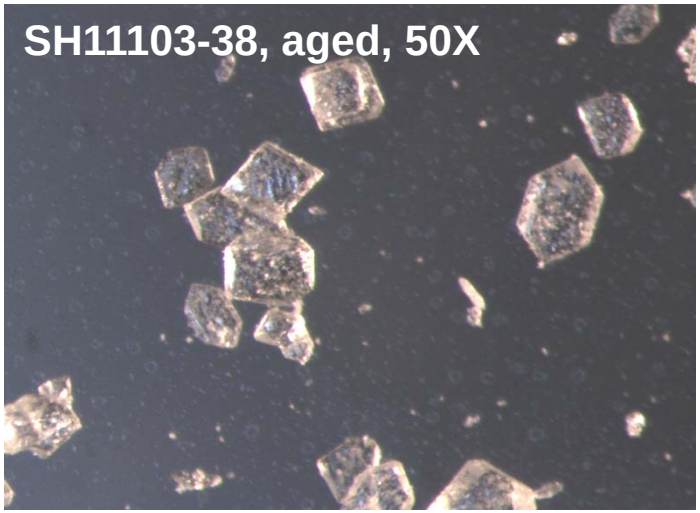


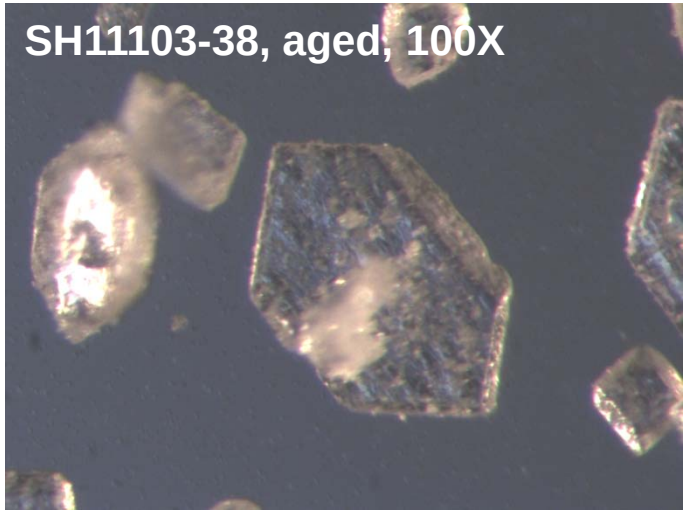


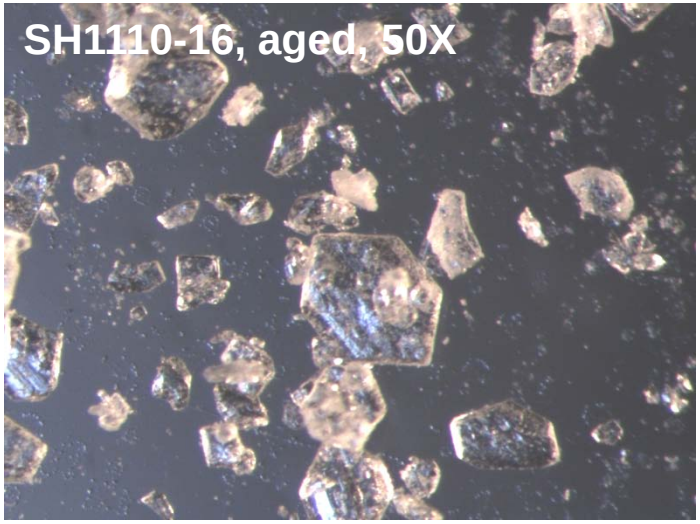
- Im-RDX samples placed in a 70 °C oven for 3 days followed by a 100 °C oven for 3 days
  - Class I RDX starting materials also aged
- Batch SH1110-16 exhibits discoloration than Class I RDX starting material

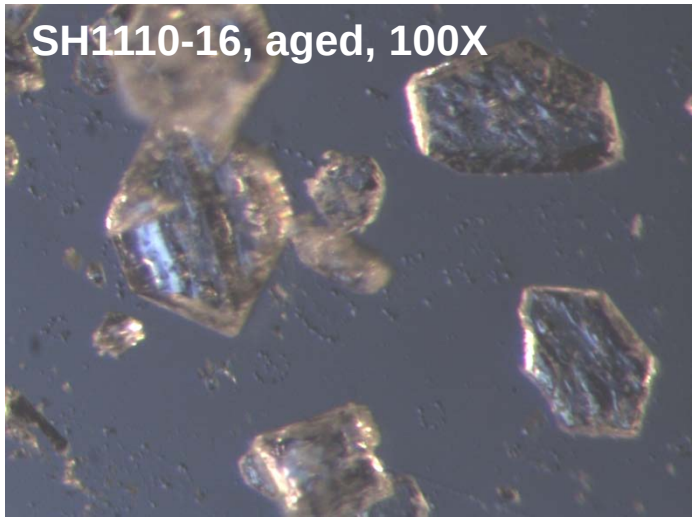
Sample	7RC112-749	SH1110-16
Un-aged Impact (cm)	44.0	61.6
Aged Impact (cm)	38.8	61.7
% Change	-11.8%	+0.2%

Elevated Temperature Study Photomicrographs









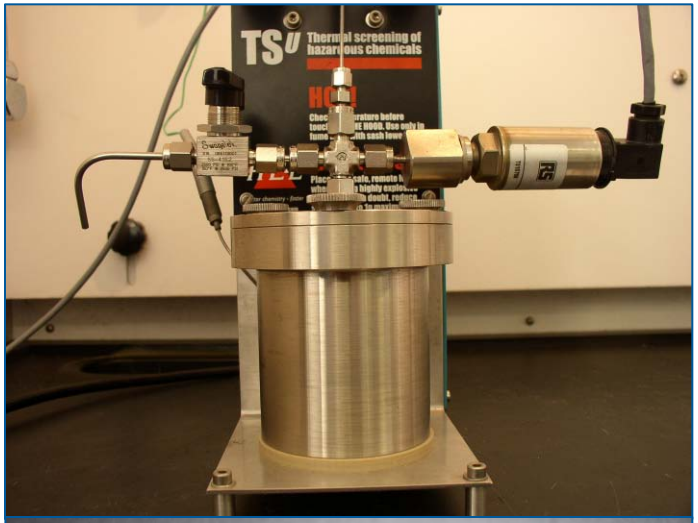
Thermal Screening Unit (TSU)

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



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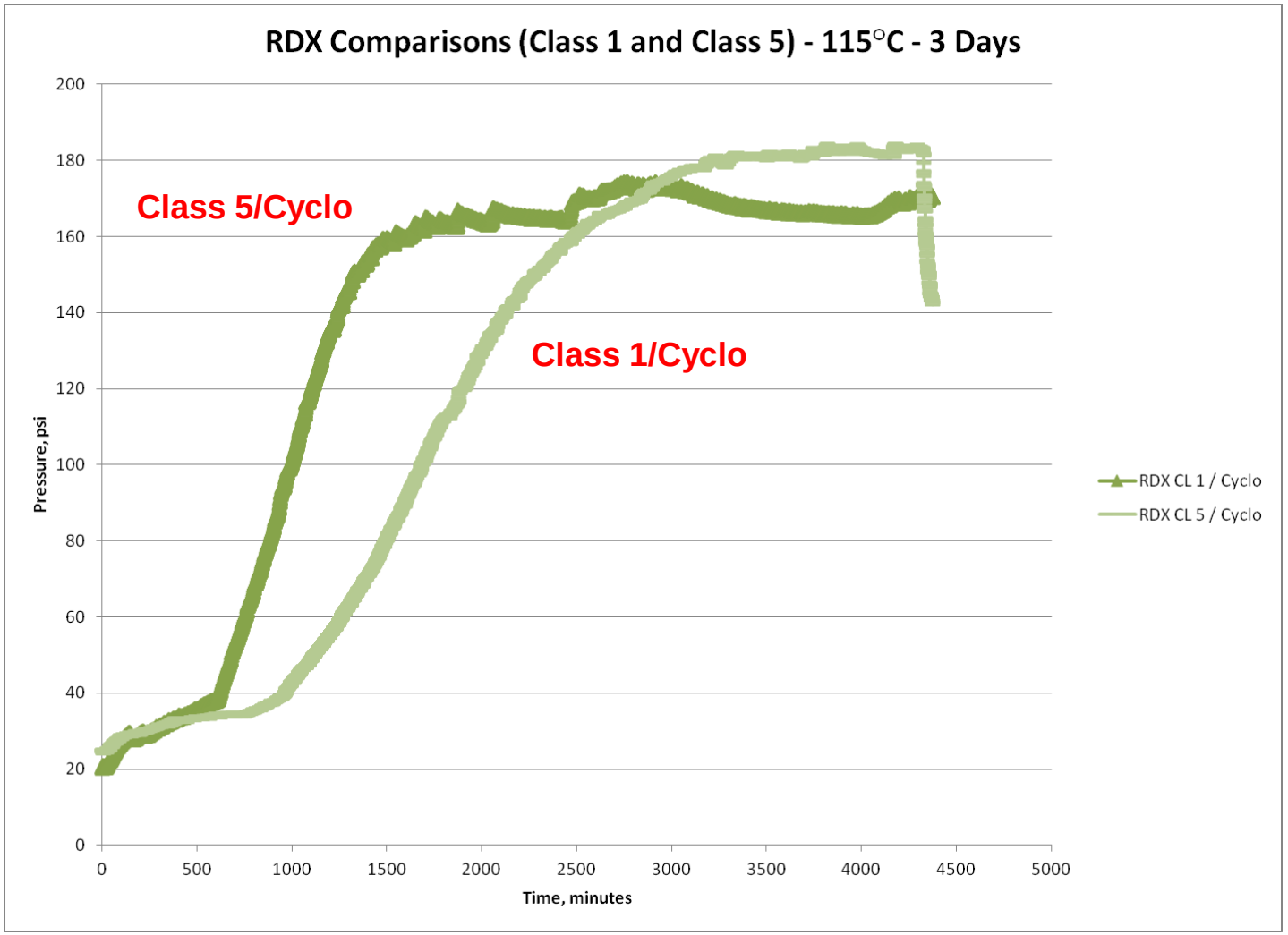
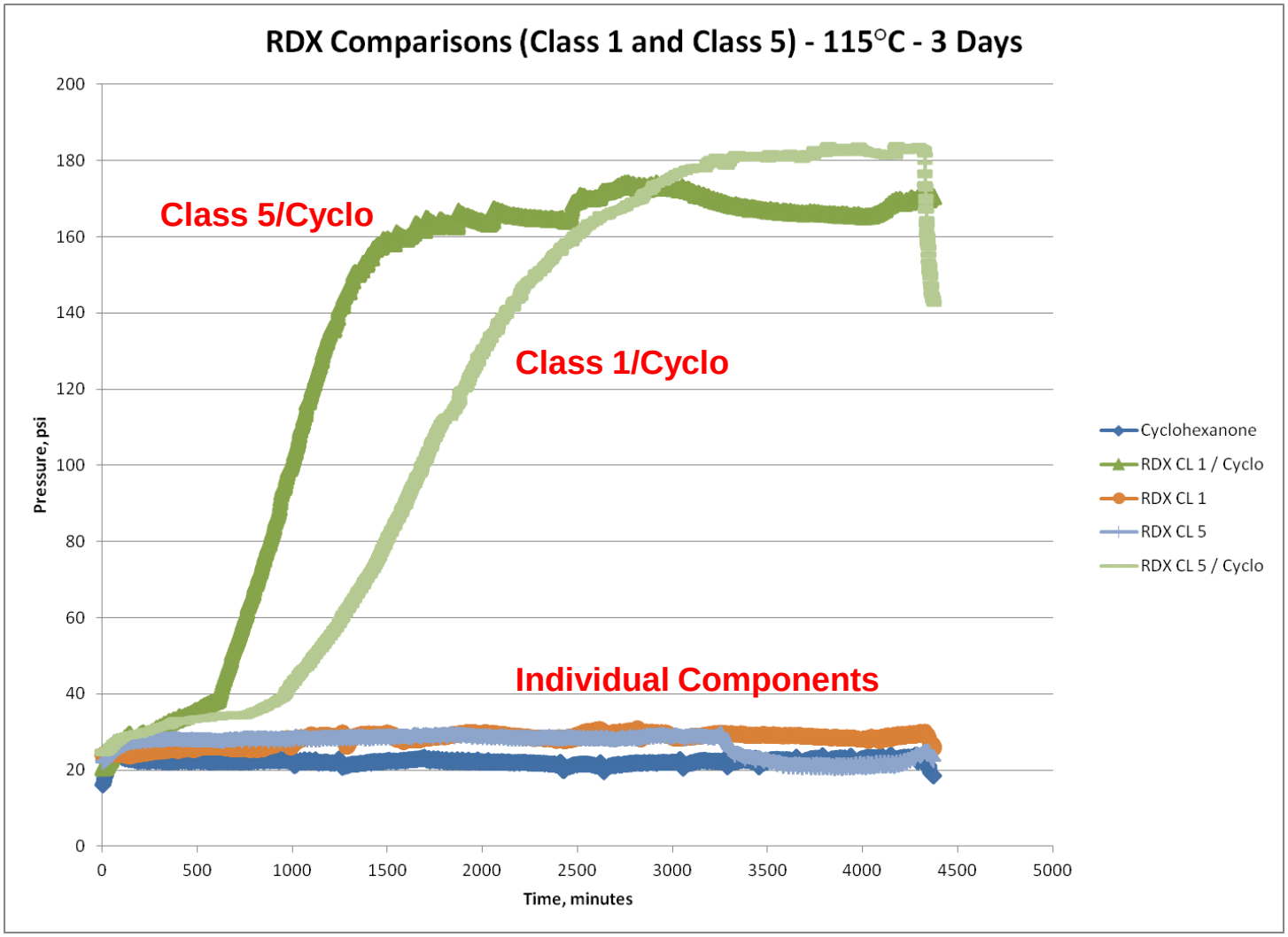
TSU Results



TSU Results


RDX Comparisons (Class 1 and Class 5) - 115°C - 3 Days
Class 5/Cyclo
Class 1/Cyclo
Individual Components
Pressure, psi
Time, minutes
0
20
40
60
80
100
120
140
160
180
200
0
500
1000
1500
2000
2500
3000
3500
4000
4500
5000
Cyclohexanone
RDX CL 1 / Cyclo
RDX CL 1
RDX CL 5
RDX CL 5 / Cyclo

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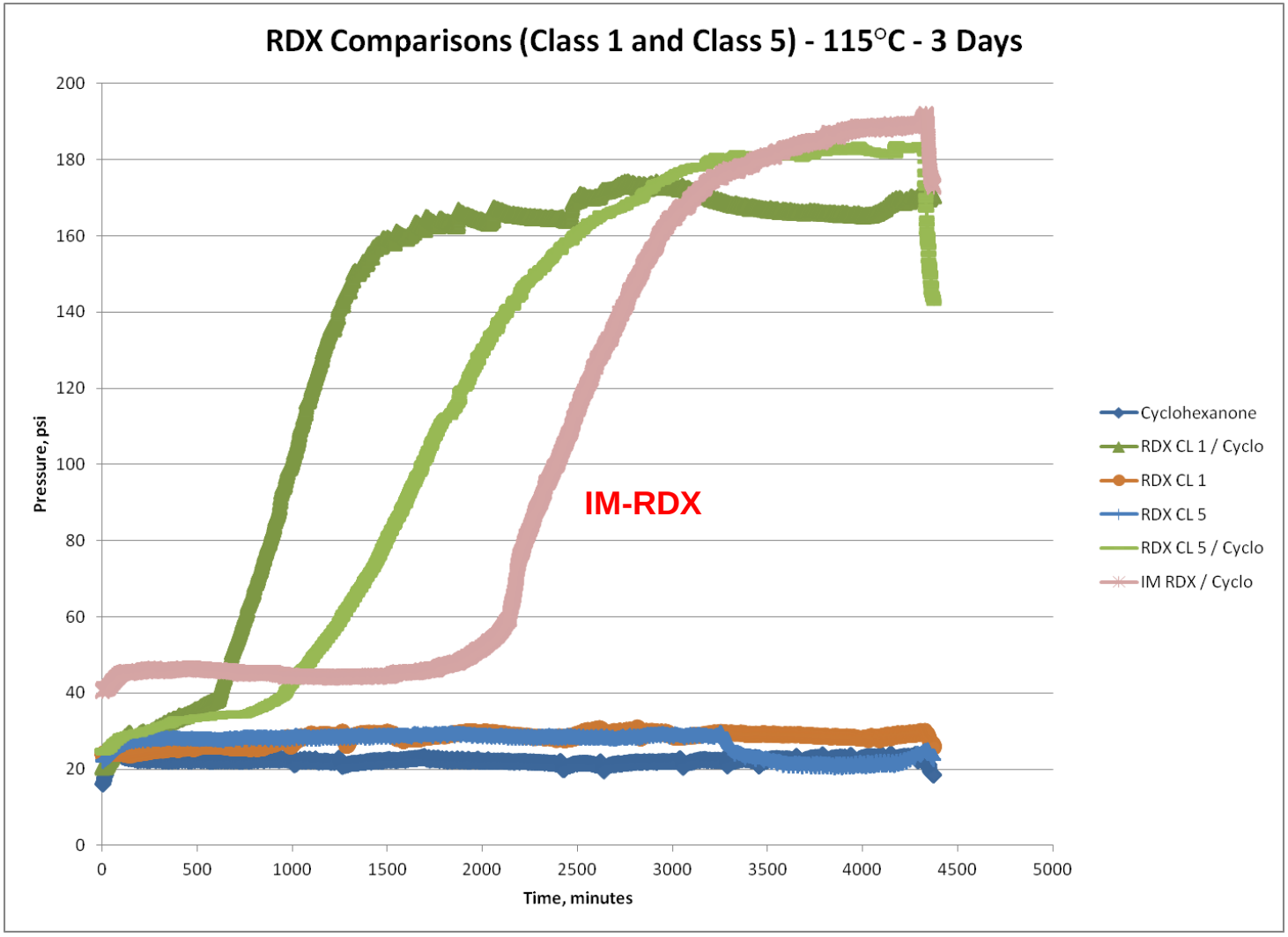
TSU Results



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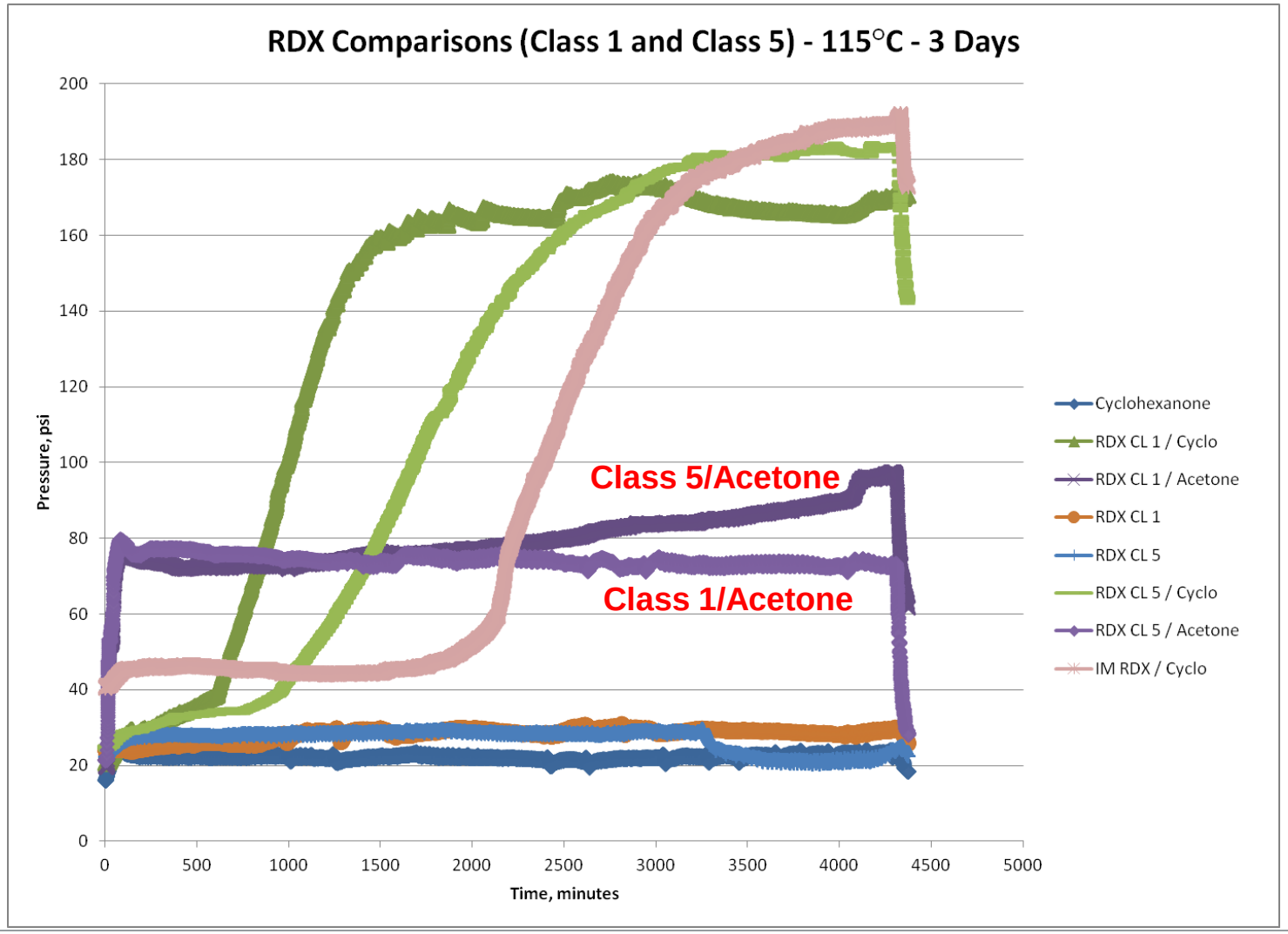

Conclusions and Future Work

- 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
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