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Improved Energetic Materials as Fuze Ingredients:

TATB

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TATB Applications

- Besides the two biggest users of PBXN-7 (FMU-139 and FMU-152 bomb fuze programs) there are also other users of PBXN-7 which include FMU-143 (BLU-116, BLU-109), FMU-148A/B (Tomahawk), FMU-155/B (SLAM ER), MK436 fuze (MK146 warhead 2.75) , M734A1, M934, and JSOW
- PBXW-14
- LX-17
- PBX-9502
- And many others

A Brief History of TATB

- 1888: TATB first described (no mention of use as explosive)
- 1950s: TATB evaluated as high-temperature resistant explosive for space applications
- 1960s:
  - TATB evaluated for use in nuclear weapons
  - Benziger process initially developed
  - Initial formulations developed with TATB and polymeric binders
- 2003-2005: OSI Scientists develop TATB manufacturing process starting from dibromoanisole.
  - Process affords 5 micron TATB
- 2007-2009: OSI Scientists develop TATB manufacturing process starting from dichloropropoxybenzene.
  - Process affords 30-40 micron TATB (very similar to Benziger TATB)

TATB Program Goals

We are proposing a new nomenclature system for TATB to avoid confusion and offer standardization when discussing and using TATB:

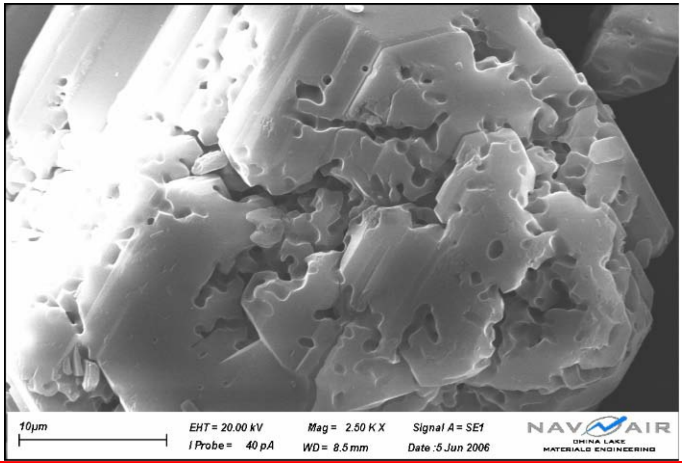
- **(Holston) Type 1**: Traditional Benziger TATB
- **(Holston) Type 2**: Small particle size (5 micron) TATB made from alkylated phenols
- **(Holston) Type 3**: Large particle size (30-50 micron) TATB made from alkylated phenols

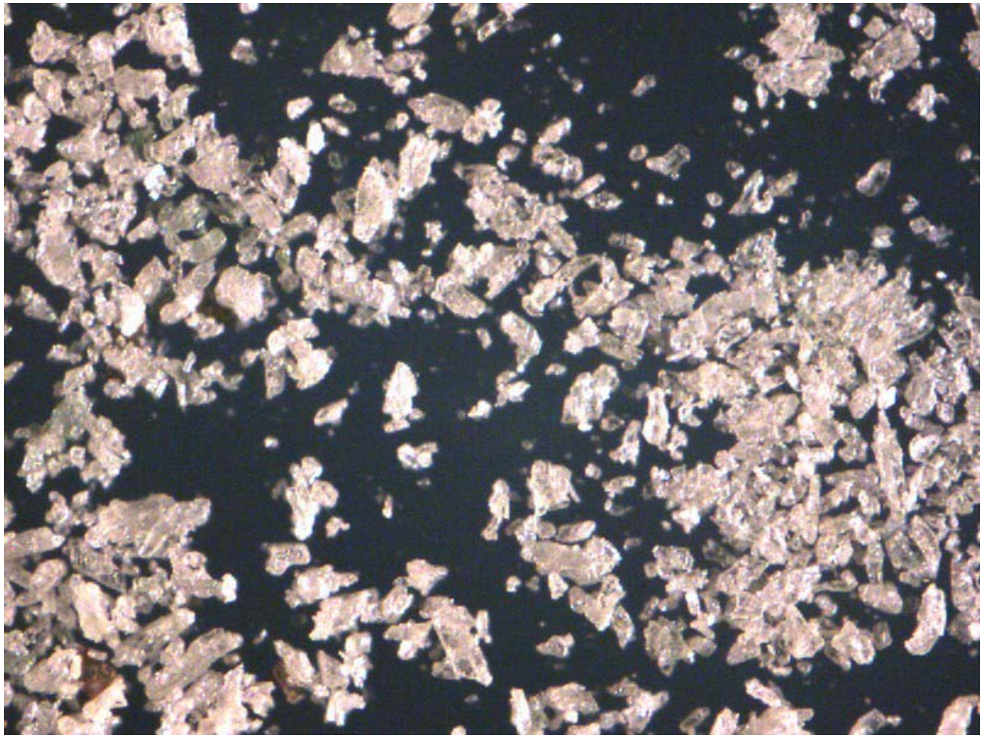
Benziger TATB: Type 1

- Benziger TATB starts with trichlorobenzene, an environmentally-unfriendly compound which is also not available from a US supplier.
- The nitration conditions are rather severe, requiring mixed acids and high temperatures.
- The nitration to obtain the desired TCTNB is also complicated by the generation of significant amounts of impurities (T3 and T4).
    - TCTNB purity is typically only 87-90%.
    - T3 and T4 are impurities that will be present in the final TATB and must be reduced to very low levels.

Benziger TATB: Type 1

- TCTNB is then aminated in toluene at high temperatures to form Benziger (Type 1) TATB.
- Conversion of TCTNB to TATB is nearly quantitative





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Holston Type 2 TATB Synthesis Method

- Based on Chemistry Developed by Benziger and Ott
- New Process/Synthesis Route Developed by OSI Scientists
- Readily Scalable (and scaled) on the Holston Infrastructure
- Good Fit for Agile Manufacturing Plant (G-10)
- Multiple Sources Identified for Raw Materials
  - Including CONUS
- Affords 5 micron (nominal) TATB

Type 2 TATB Production at Agile Manufacturing Plant

- Nitration
- 3,5-Dibromoanisole (2500 lbs) is Melted and Fed as a Liquid into 98% Nitric Acid at or Below 50 C in a 2000 gal. glass-lined reactor
- Initial Reaction is Mildly Exothermic
- Reaction is Complete in 4-5 hrs. at Reflux, or 24 hrs. at Ambient Temperature
- Yield is Essentially Quantitative (~3600 lbs. DBTNA after quench and wash)
- Product (DBTNA):
  - Insensitive Intermediate
  - Melting Point = 140 C
  - Exotherm Onset = 288 C
  - Impact Sensitivity > 80 cm (Holston Method)
  - DBTNA not isolated; Slurried and pumped directly to amination vessel

Type 2 TATB Production at Agile Manufacturing Plant

- Amination
  - DBTNA slurry is pumped to 6000 gal. still
  - Slurry is dewatered with wand filter
  - 29% aqueous ammonia is pumped in; agitation started
- Reaction Occurs Over 36 hours at 25 C
- Main By-Product is NH_4Br
- Known Impurities
  - Ammonium diaminopicrate (ADAP)
  - Starting material - DBTNA
- Yields are ~ 90%

Type 2 TATB Production at Agile Manufacturing Plant

- Collection in Filter Press
  - TATB slurry is pumped to filter press
  - Blown down and collected; nominal yields ca. 2150 lbs.
  - NH_3 is stripped from reaction filtrate using eductor
  - Used to neutralize spent acid from nitration step

Nutsches of Type 2 TATB

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Technical Issues of Early Type 2 TATB Efforts

- In PBXN-7, OSI Type 2 TATB (5 micron) performed well in all examined aspects except:
  - Shock sensitivity:

Material Tested	Average Pellet Density, g/cm^3	NOL LSGT, cards/kbars	Detonation Velocity, m/s
PBXN-7 with OSI TATB (supplied by OSI)	1.789	70% kbar increase	7572
Historical data [a]	1.78	[redacted]	7660

  - Reduction in sensitivity thought to be caused by small particle size and/or crystal morphology (lack of voids) of TATB (as compared to traditional TATB (50 micron)…

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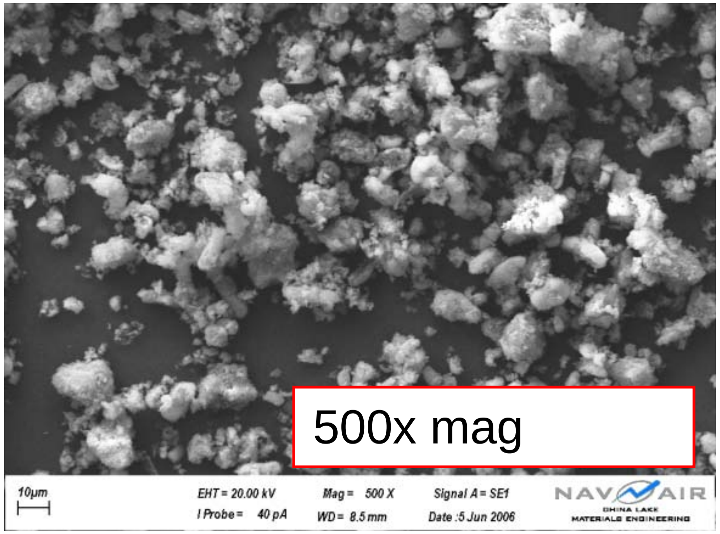
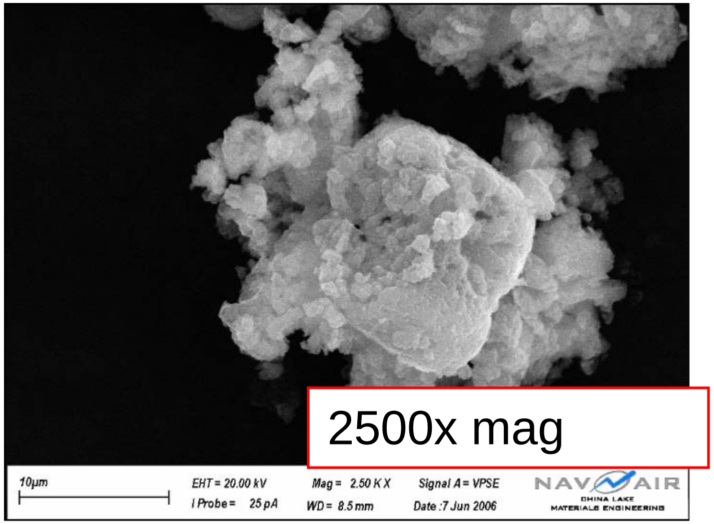
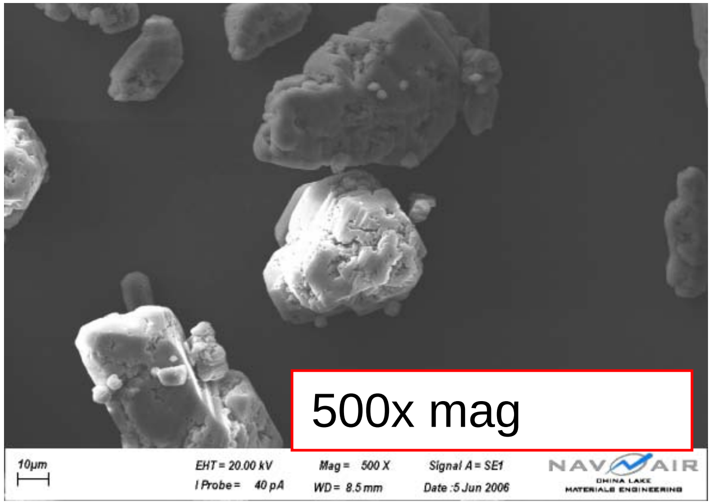
SEM Analysis

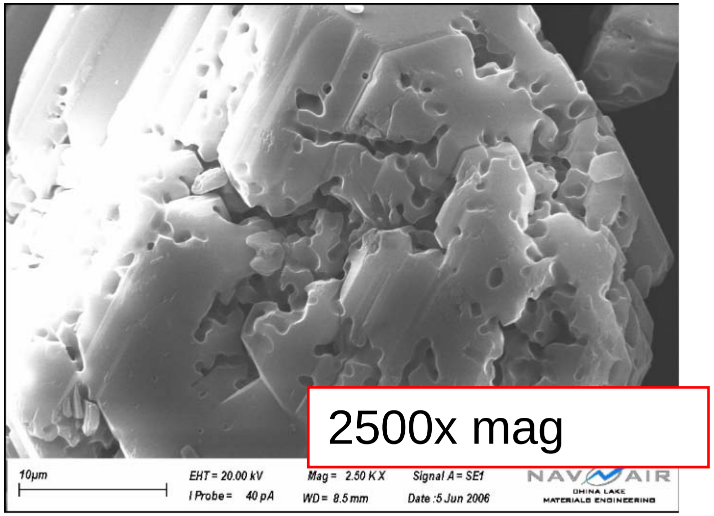


Holston Type 2 TATB





Traditional Type 1 TATB



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Holston Type 3 TATB Synthesis Method

New 2-Step Process/Synthesis Route Developed by OSI Scientists

- Scalable on the Holston Infrastructure
- Good Fit for Agile Manufacturing Plant (G-10)
- Multiple Sources Identified for Raw Materials-Including CONUS

- Purity comparable to reference Type 1 TATB
- Particle size typically 30-40 microns
- Produced ~20 lbs TATB to date

Nitration of DCPB

- DCPB is fed as a liquid into nitric acid
- Initial reaction is mildly exothermic
- Reaction performed several times in 5 gal reactor (10 lb batch size)
- DCTNPB (product):
  - Yields > 95%
  - Purity typically >99%
  - Insensitive Intermediate
  - Melting Point = 121 C
  - Exotherm Onset = 220 C
    (as determined by DSC)
  - Impact Sensitivity > 80 cm (Holston Method)

Amination of DCTNPB

- DCTNPB is aminated in toluene with gaseous ammonia at high temperature and under pressure (similar to Benziger route)
- Reaction Scaled to 1 mole (2 gal Parr)
- Yields are ~ 75%
- **Known Impurities:**
  -Ammonium diaminopicrate (ADAP)
    -Mp = 214 C
    -Accounts for most of missing mass

Formation and Elimination of Ammonium Diaminopicrate (ADAP)

Average % ADAP Pre-Wash

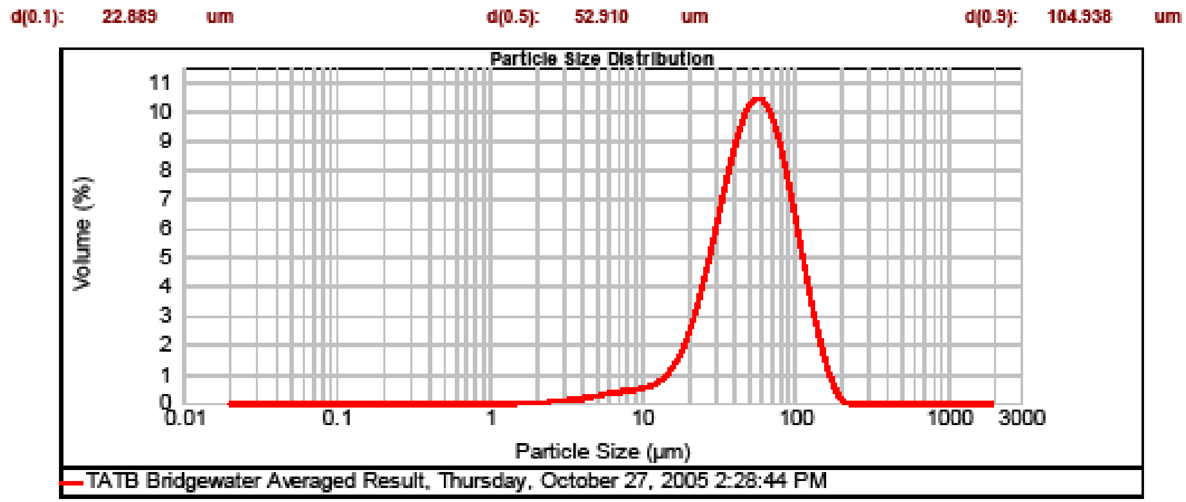
Lot 1	0.15%
Lot 2	0.15%
Lot 3	0.58%

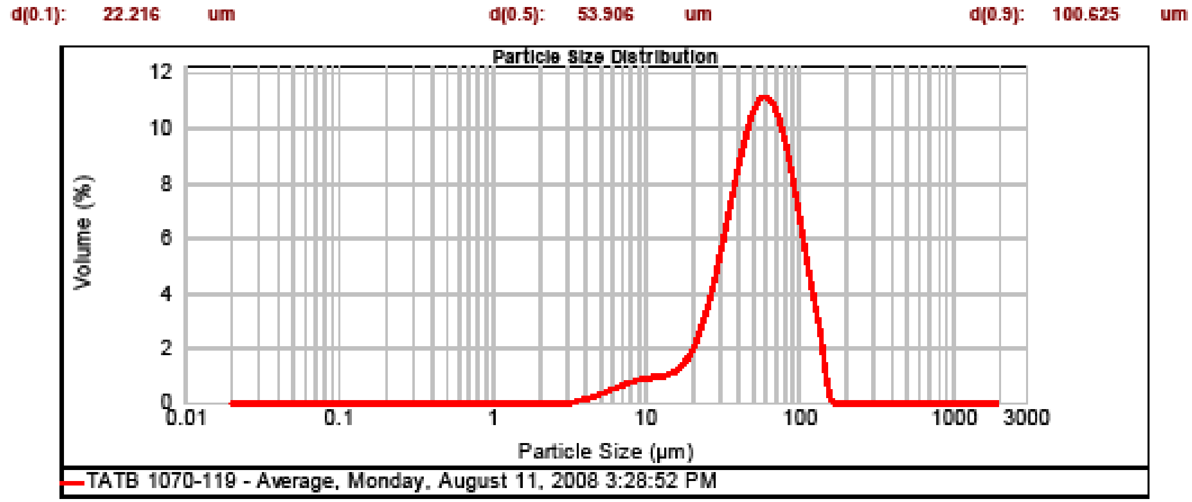
Average % ADAP Post-Wash

Lot 1	0.04%
Lot 2	0.02%
Lot 3	0.02%

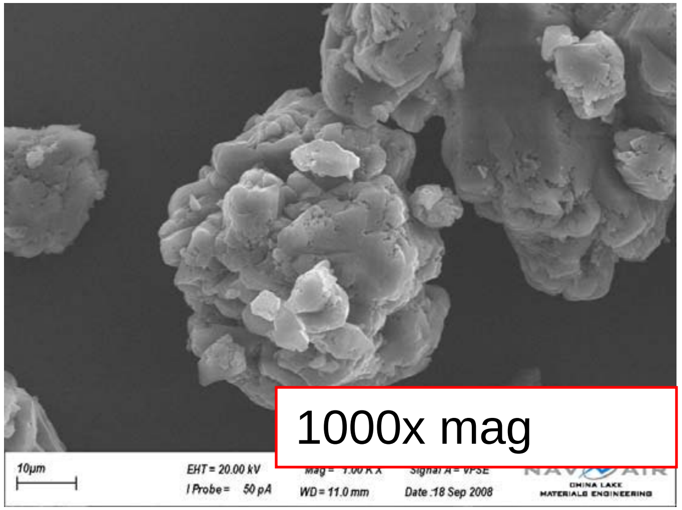
•Washing with hot water until wash water becomes light yellow lowers ADAP contamination considerably

Type 3 TATB: Particle Size Analysis

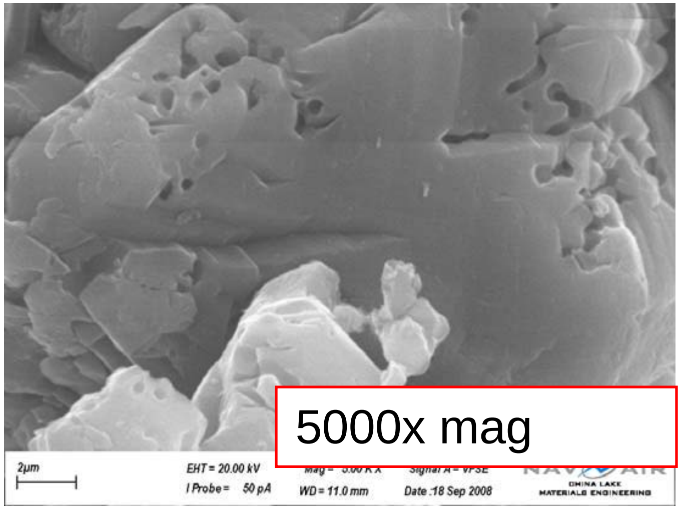


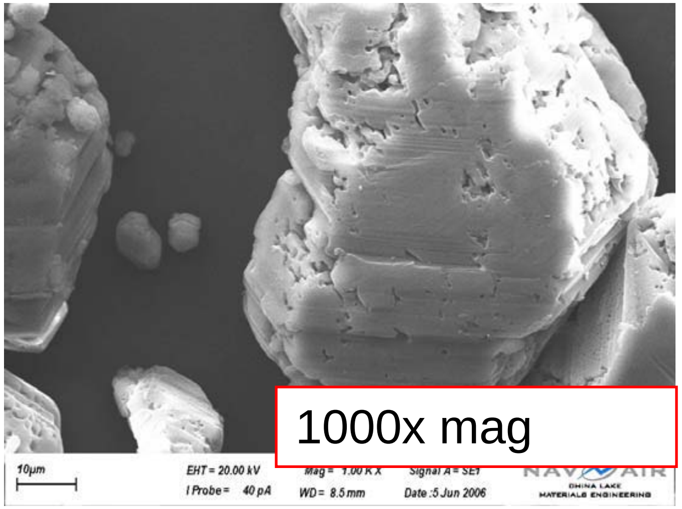


SEM Analysis

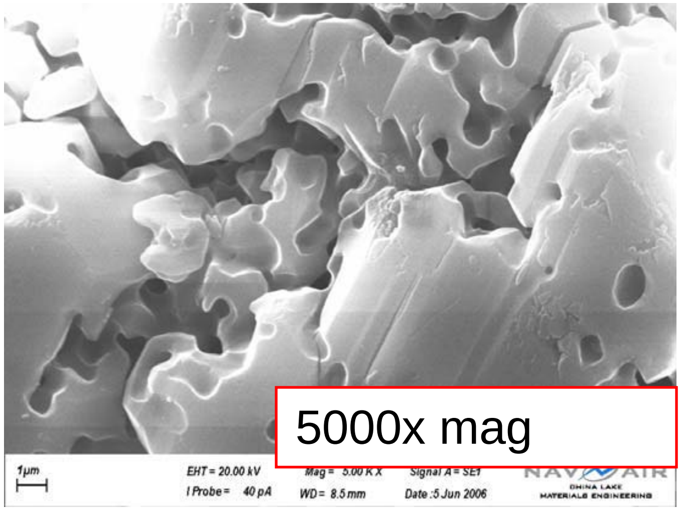


Holston Type 3 TATB





Traditional Type 1 TATB



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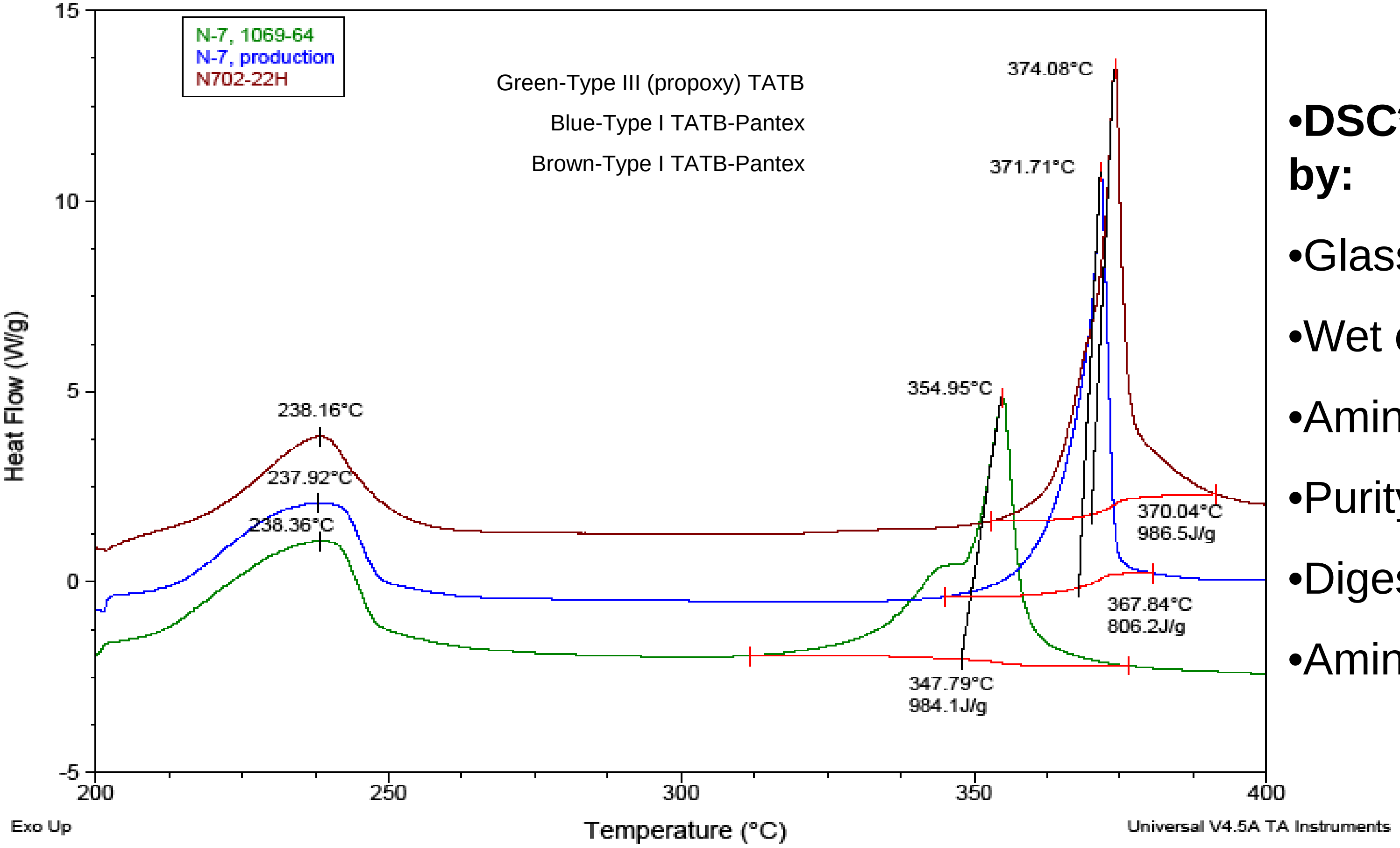
Formulations: PBXN-7

- Several lab batches made with Type 3 TATB
- Consistent process and product

Screens (%Pass)	batch 1	batch 2	batch 3	composite
#6	met spec	met spec	met spec	met spec
#14	met spec	met spec	met spec	met spec
#18	met spec	slightly out	met spec	met spec
#100	met spec	met spec	met spec	met spec
Bulk Density (g/cm^3) (Naval)	met spec	met spec	met spec	met spec
Composition	met spec	met spec	met spec	met spec
Moisture	N/A	N/A	N/A	met spec
Impact Sensitivity (ERL, cm)	N/A	N/A	N/A	met spec
VTS by PT Method (100°C, 48h)(mL/g)	N/A	N/A	N/A	met spec
Press Density (g/cm^3)	N/A	N/A	N/A	slightly out (low)
Comments				Blend of 1,2,and 3

PBXN-7 comparisons

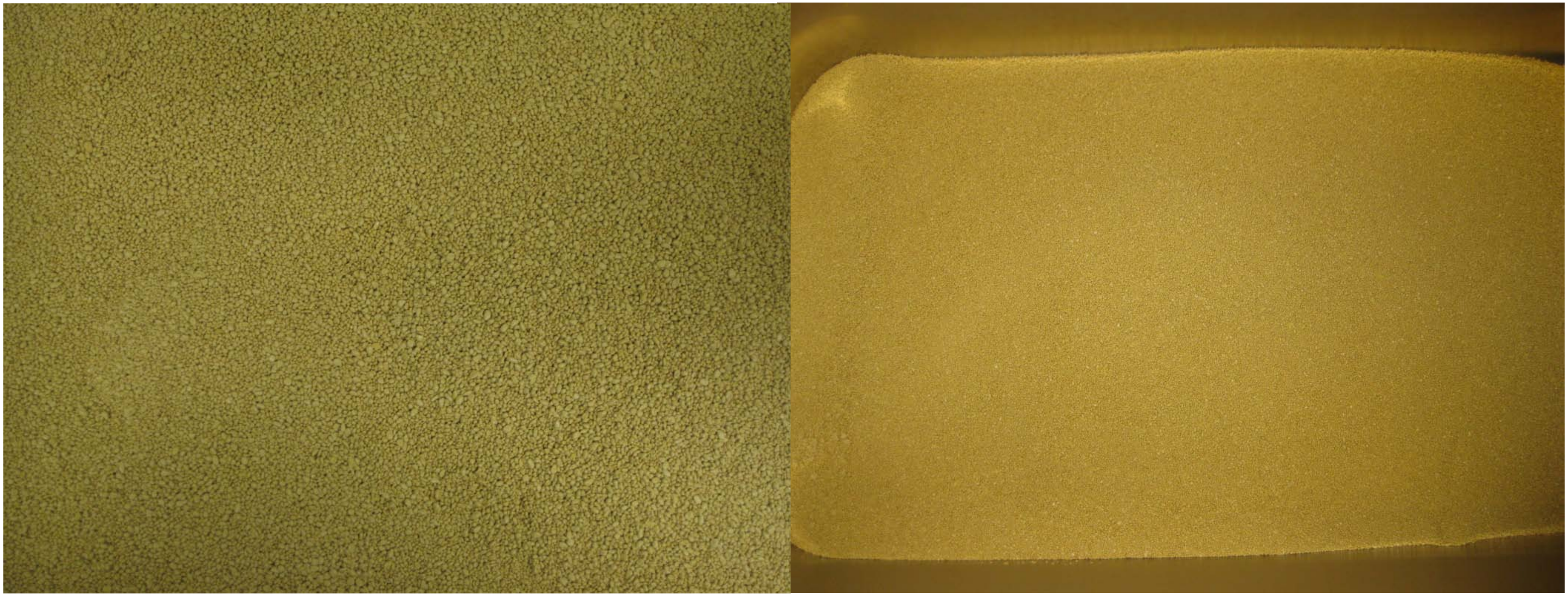
- DSC of new TATB (Type 2 and 3) found to be different than Type 1 TATB
- Phenomenon appears to be caused by presence of ADAP in amination



- **DSC* not affected by:**
- Glass vs SS reactor
- Wet or dry amination
- Amination temp.
- Purity
- Digestion in DMSO
- Amination under N2

Formulations: PBXW-14

- One batch made in lab with Type 3 TATB.
- Successful integration of TATB made from the new OSI method into the existing W-14 formulation procedure.
- No performance data at this time.



TATB Safety Data

Material	Source	ERL Impact Sensitivity	ABL Friction Sensitivity at 1000 lb	ESD Sensitivity at 0.25 J
Type 2	OSI	1/18 fires at 200 cm 2/2 no-fires at 158 cm	10/10 no-fires	10/10 no-fires
Type 2	OSI	4/15 fires at 200 cm 5/5 no-fires at 158 cm	10/10 no-fires	10/10 no-fires
Type 2	OSI	3/16 fires at 200 cm 4/4 no-fires at 158 cm	10/10 no-fires	10/10 no-fires
Type 3	OSI	10/10 no-fires at 200 cm	10/10 no-fires	10/10 no-fires
Standard-Type 1	DOE	10/10 no-fires at 200 cm	10/10 no-fires	10/10 no-fires
RDX standard	N/A	17 cm	550 lb_f	10/10 no-fires

PBXN-7 Qualification Small Scale Safety Data

PBXN-7, BAE06L382-015, OSI Type 2 TATB					
Parameter	Aged “0” months	Aged “2” months	Aged “4” months	Aged “6” months	Units
Impact Sensitivity	103.1	110.3	107.2	108.2	cm
Impact Reference (RDX)	16.6	16.6	16.6	16.6	cm
Friction	>360	>360	>360	>360	Newt.
PETN Reference	48	48	48	48	Newt.
VTS	0.09			0.02	ml/g
DSC	242.3	242.6	242.3	242.3	º C

PBXN-7 Qualification LSGT Data

		Baseline "0" Months		Aged "6" Months	
Material Tested	Ave. Pressed Density gm/cc	Shock Sensitivity Cards	Shock Sensitivity kbar Pressure	Shock Sensitivity Cards	Shock Sensitivity kbar Pressure
PBXN-7 Manufactured with **Benziger TATB Type 1** Lot # BAE07B382-014	1.79	205 - 210	18.621 – 19.627	206.1	19.398
PBXN-7 Manufactured with **OSI TATB Type 3**	1.78	203.8	19.883	---	---
PBXN-7 Manufactured with **OSI TATB Type 2** Lot # BAE06L382-013	1.79	155.8	35.939	165.8	31.517

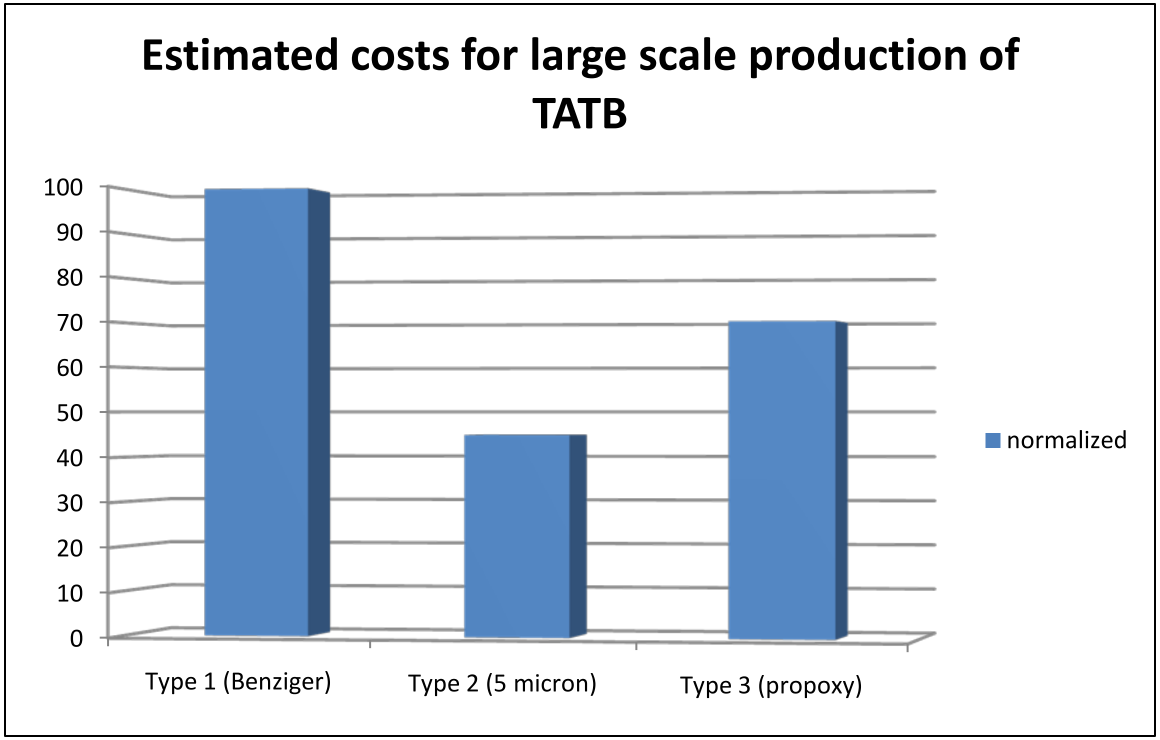
Initiation Validation Test at Cold Temperature

PBXN-7 LOT # BAE06L382-013 Manufactured with OSI Holston Type 2 TATB

Fuze Configuration	Pellet Density (gm/cc)	Dent Plate Hardness	Dent Depth (inches)	Dent Depth GO Criteria (inches)	GO/ NO GO
FMU-139	1.760	90	0.0565	0.0425	GO
FMU-139	1.760	90	0.0525		GO
FMU-139	1.800	82	0.0580		GO
FMU-139	1.800	88	0.0580		GO
FMU-139	1.800	85	0.0585		GO
FMU-152	1.760	90	0.0425	0.0346	GO
FMU-152	1.760	88	No Dent		NO GO
FMU-152	1.800	84	No Dent		NO GO
FMU-152	1.800	86	No Dent		NO GO
FMU-152	1.800	87	No Dent		NO GO

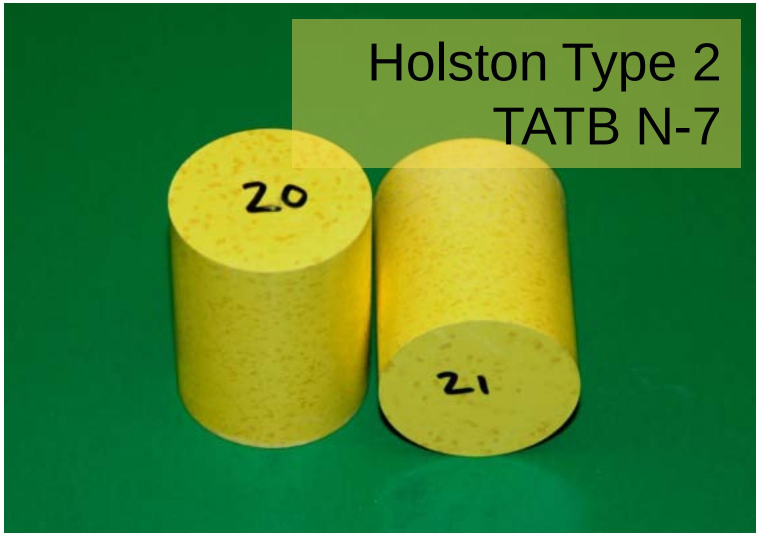
TATB Costs

- Estimates are based on R&D efforts, production efforts, and prior experience and knowledge.
- Costs are normalized to Type 1 cost estimates.
- Type 2 is less than half of the cost of Type 1, due to the simplicity of the process.



Conclusions

- Two TATB manufacturing processes developed at HSAAP (Type 2 and Type 3)
- Processes are robust and safe
- Competitive costs to Type 1 TATB
- Process and cost optimization ongoing
- Quality equivalent to traditional sources of “DOD grade” material
- Difference in thermal properties (DSC) appear to be caused by ADAP impurity in process
- Type 3 TATB currently appears to be a “drop-in” replacement in DOD formulations (waiting for further performance testing)



Acknowledgments

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