Synthesis and Scale-Up of TATB

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TATB Program Goals

Low Cost/High Volume Supplier

- Inclusion in Many New IM Formulations
- Good Fit for Existing Holston Infrastructure
- Minimal Initial Capitalization
- Short Time to Production Quantities
- Equivalent Quality to Traditional TATB
  - Similar Shock Sensitivity in PBXN-7 needed…

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Technical Issues of Earlier TATB Efforts

- In PBXN-7, OSI 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 manufactured by NSWCIH Yorktown Det with ATK TATB	1.781	50% kbar increase	7464
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 of TATB (as compared to traditional TATB (50 micron)

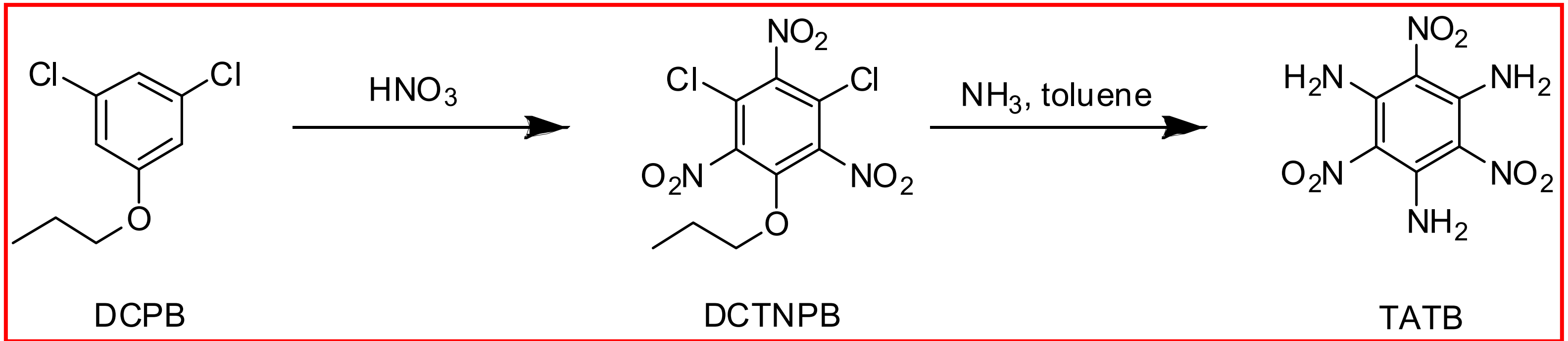
Available Technologies

- Traditional Trichlorobenzene (TCB) Route- “Benziger Route”
  - Harsh conditions; waste streams
  - TCB not domestically available

Holston TATB Synthesis Method

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

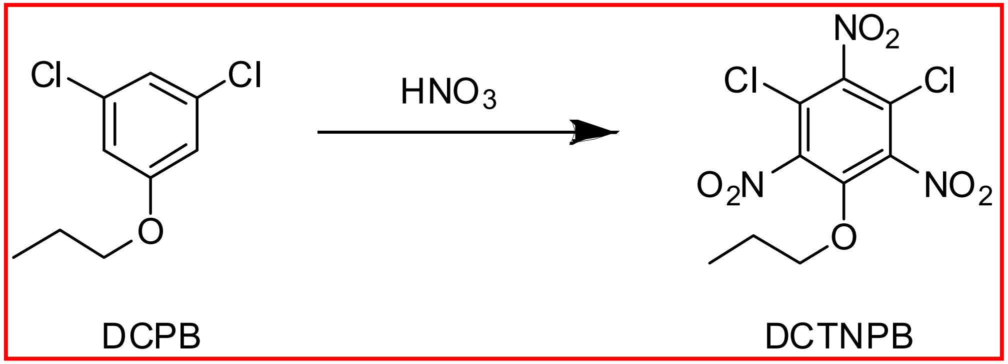
- Scalable on the Holston Infrastructure
- Good Fit for Agile Manufacturing Plant (G-10)
- Multiple Sources Identified for Raw Materials-Including CONUS



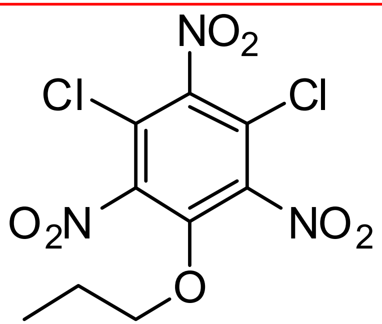
- Purity comparable to reference (Bridgwater)
- Particle size typically 40 microns
- Produced ~20 lbs TATB to date

Laboratory 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)
- Yields ≥ 95%
- Purity typically > 99%



DCTNPB
(3,5-Dichloro-2,4,6-trinitropropoxybenzene)

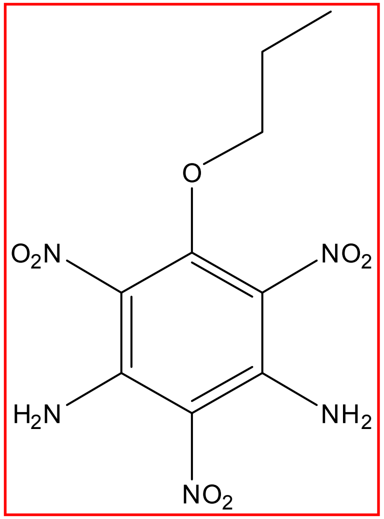


Chemical Formula: $C_9H_7Cl_2N_3O_7$
Molecular Weight: 340.07

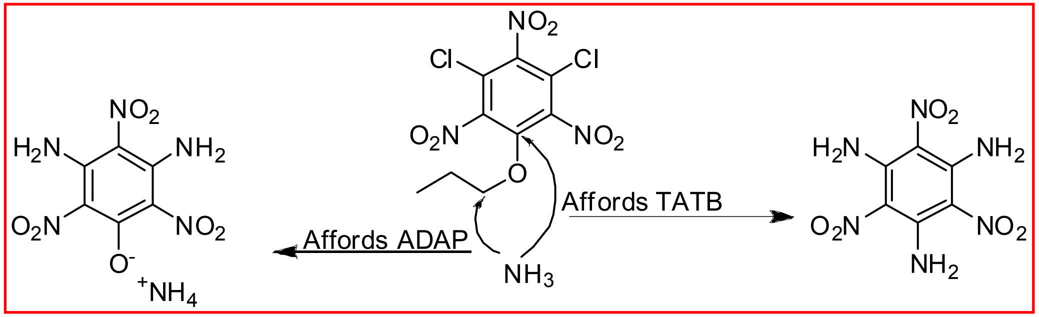
- Insensitive Intermediate
- Melting Point = 121°C
- Exotherm Onset = 220°C (as determined by DSC)
- Impact Sensitivity > 80 cm (Holston Method)

Laboratory 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)
  -3,5-Diamino-2,4,6-trinitro-propoxybenzene
  (PDAP-seen in early development, not detected in current process)
  -Mp = 214 C



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

TATB Analytical Summary

Batch 1

DSC	354°C
TGA	250°C (0.78%), 325°C (18.4%), 400°C (78.7%)
Mean particle size	40.7μm
% ADAP	0.03%
VTS (100C, 48 hrs)(mL/g)	0.1167
% Chloride	0.12%

Batch 2

DSC	354°C
TGA	250°C (0.42%), 325°C (16.6%), 400°C (77.1%)
Mean particle size	34.4μm
% ADAP	0.02%
VTS	N/A
% Chloride	0.10%

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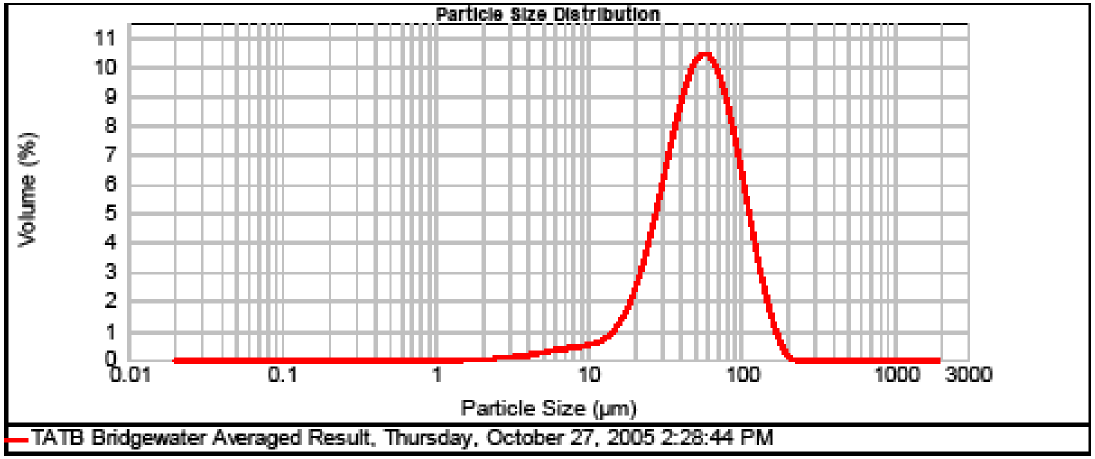
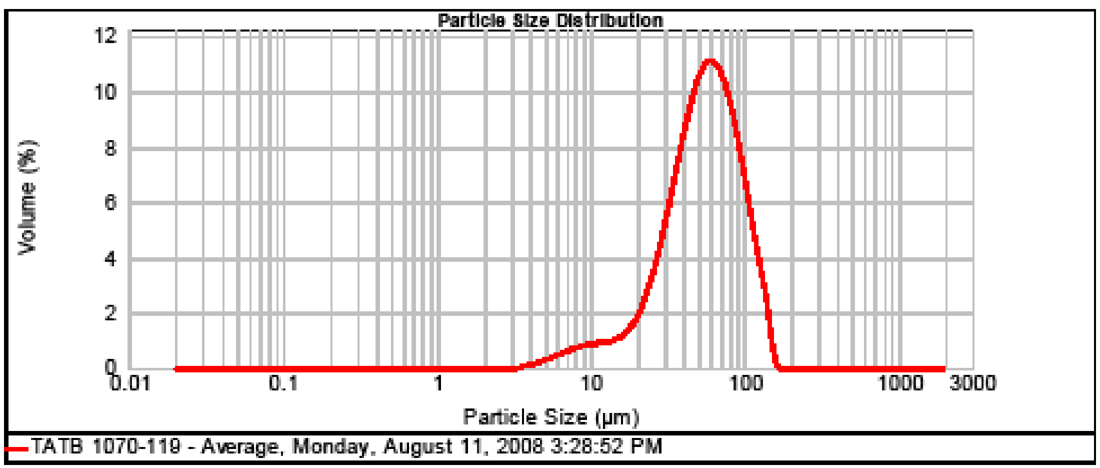
Particle Size Analysis

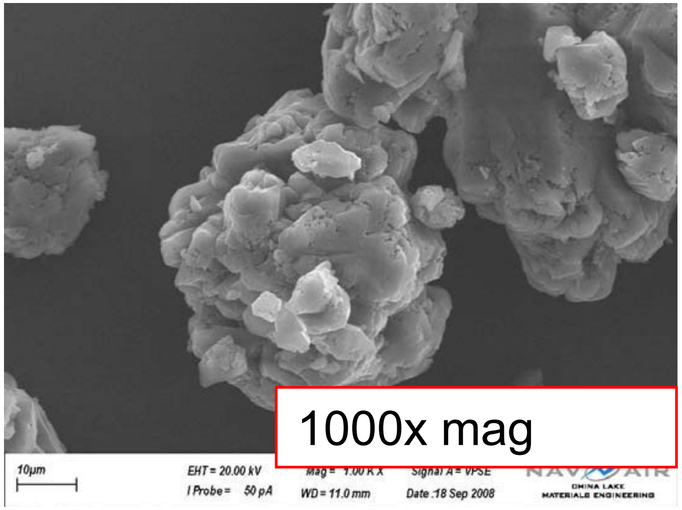
d(0.1): 22.889 um d(0.5): 52.910 um d(0.9): 104.938 um



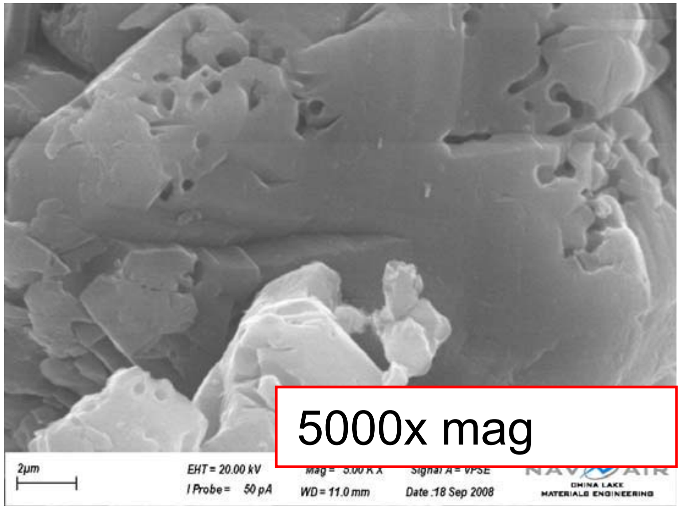
d(0.1): 22.216 um d(0.5): 53.906 um d(0.9): 100.625 um

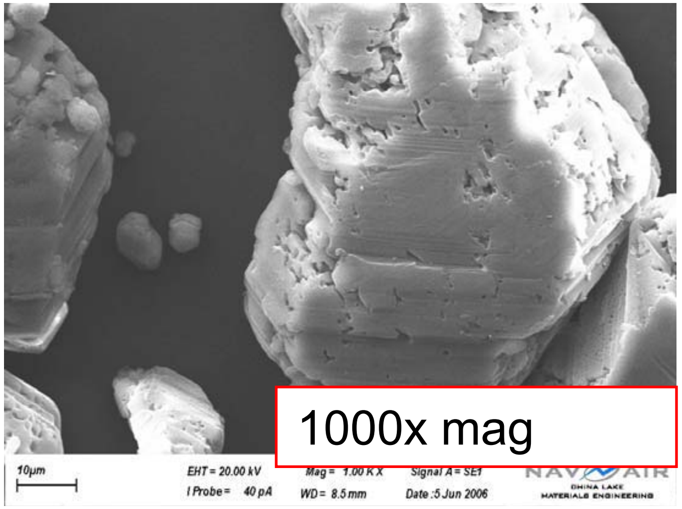


SEM Analysis

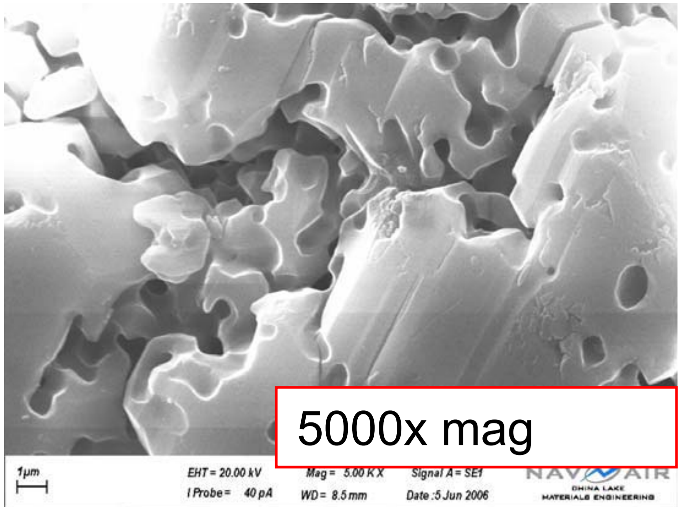


New OSI TATB



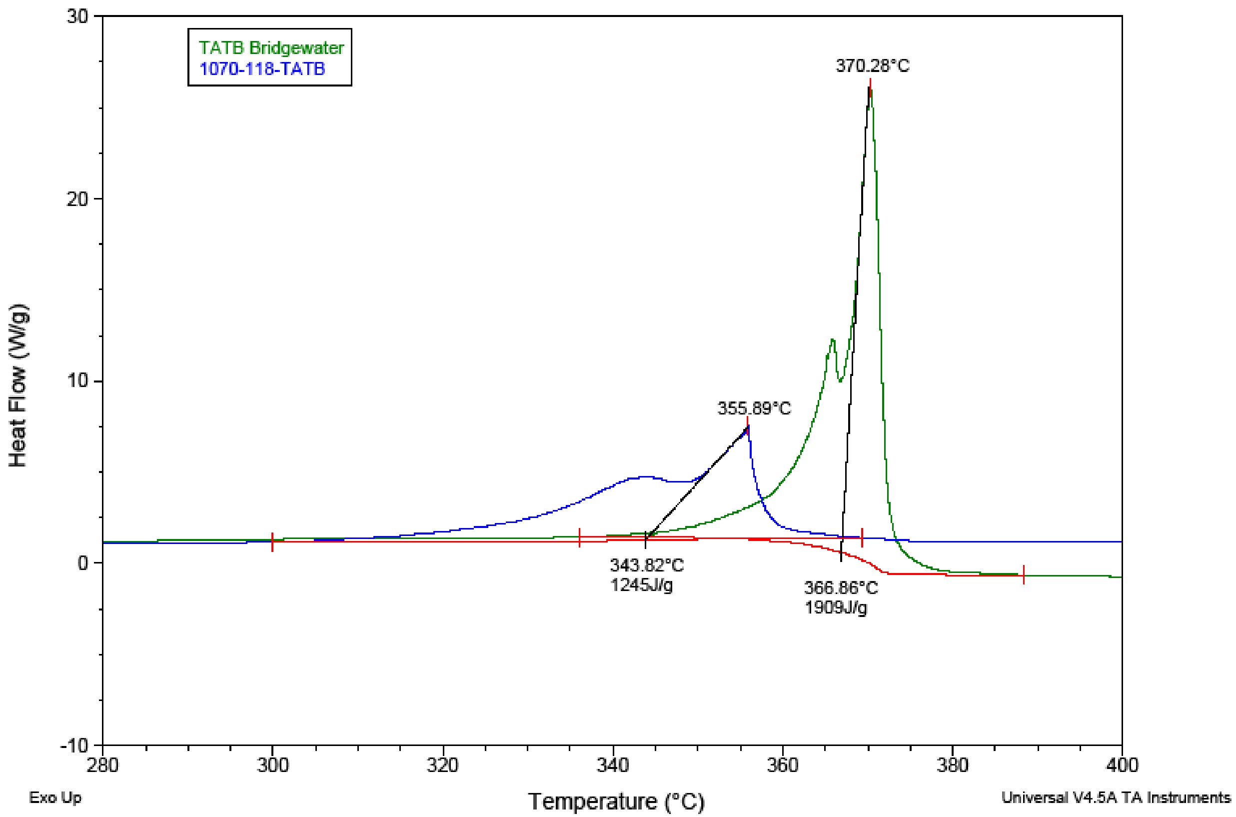


Traditional TATB



DSC phenomenon

- DSC of new TATB found to be significantly different than traditional TATB



•DSC* not affected by:

•Glass vs SS reactor

•Wet or dry amination

•Amination temp.

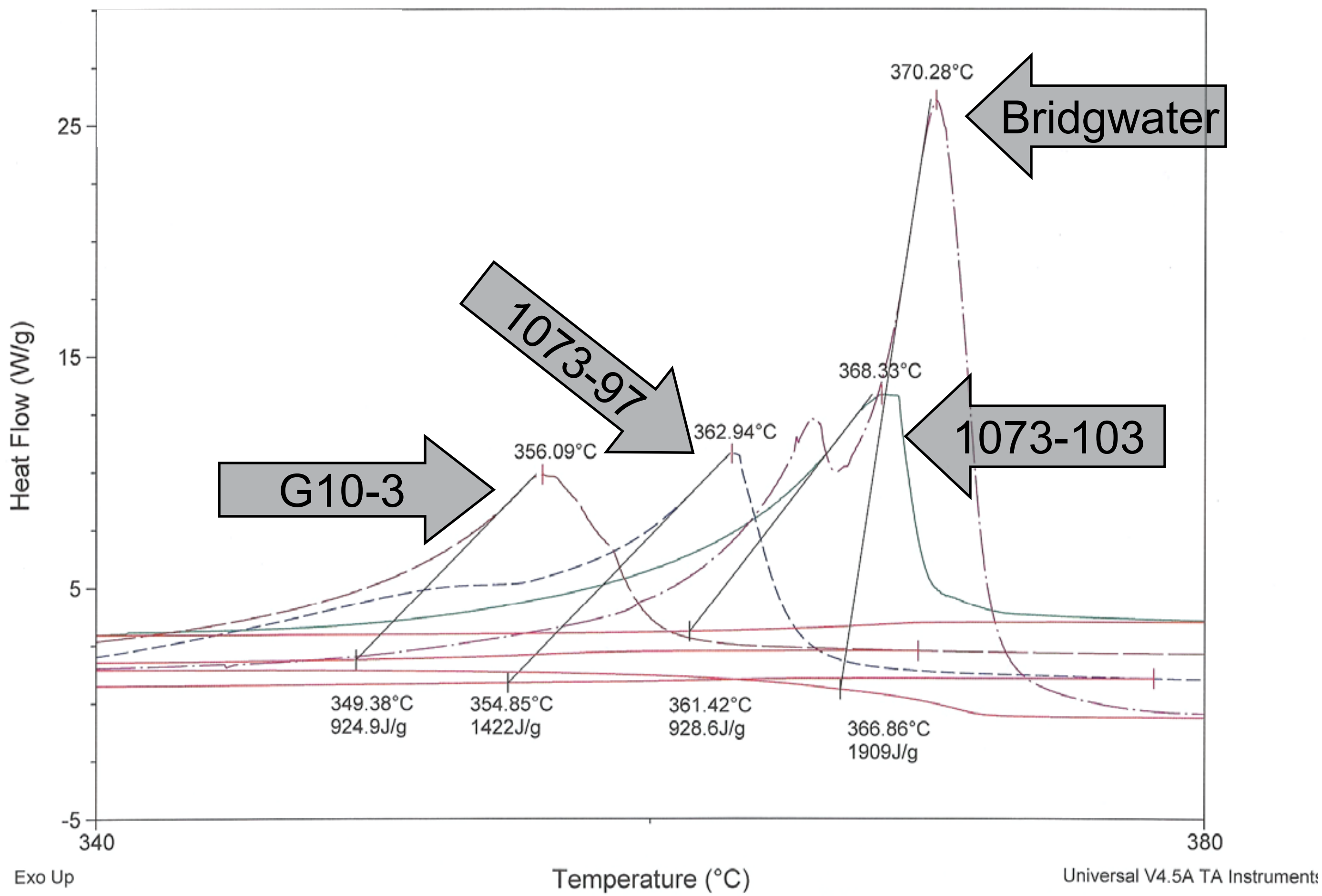
•Purity

•Digestion in DMSO

•Amination under N2

*All DSCs performed at 5C/min unless otherwise noted

TATB DSC (5°C/min)



- G10-3 is production run using previous dibromoanisole aqueous route.
- 1073-97 is from DCTNPB route, recrystallized from DMSO.
- 1073-103 is RT aqueous amination of DCTNPB (90% yield).

ADAP spiking of aminations- TATB DSC (5°C/min)

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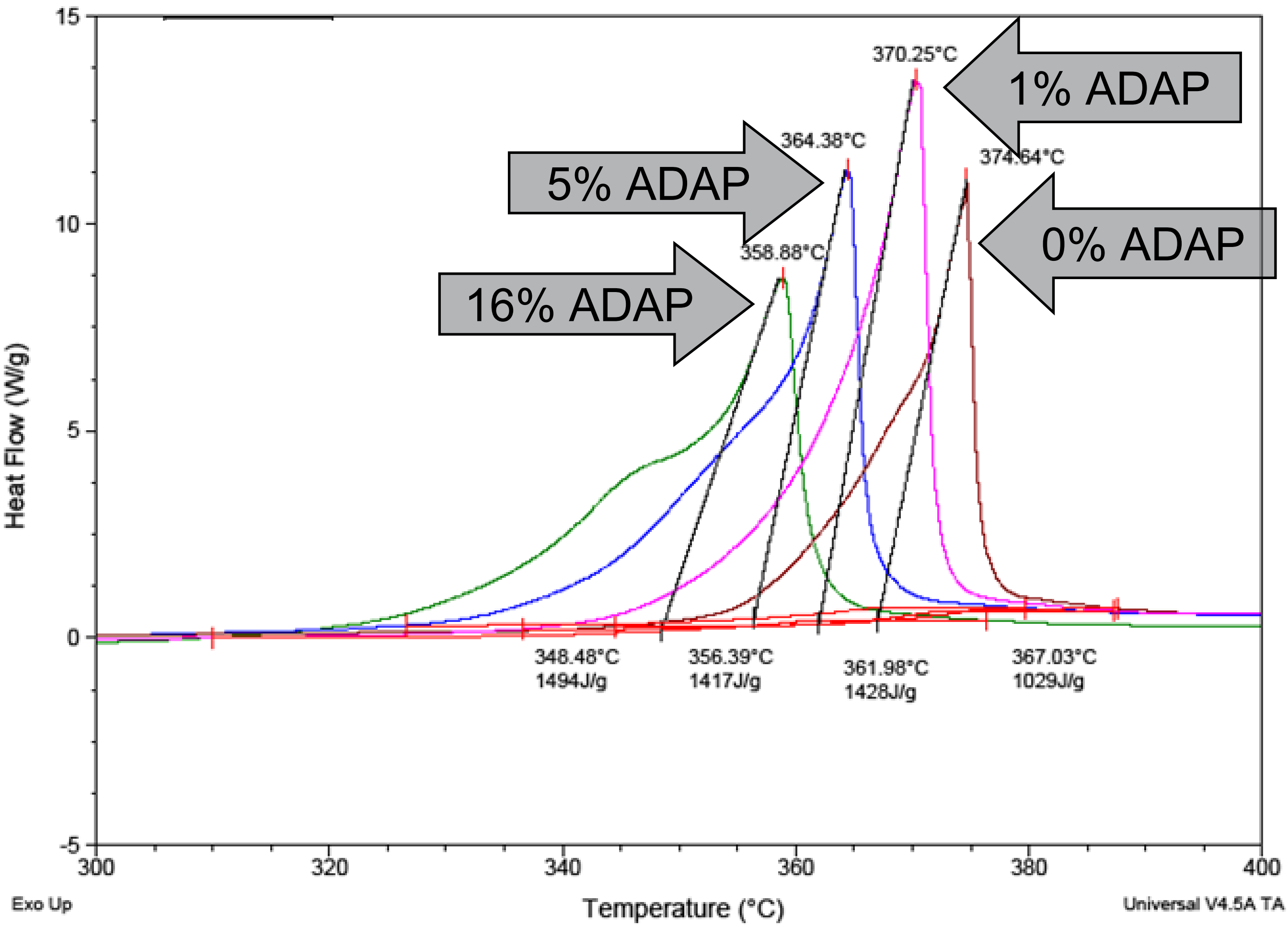
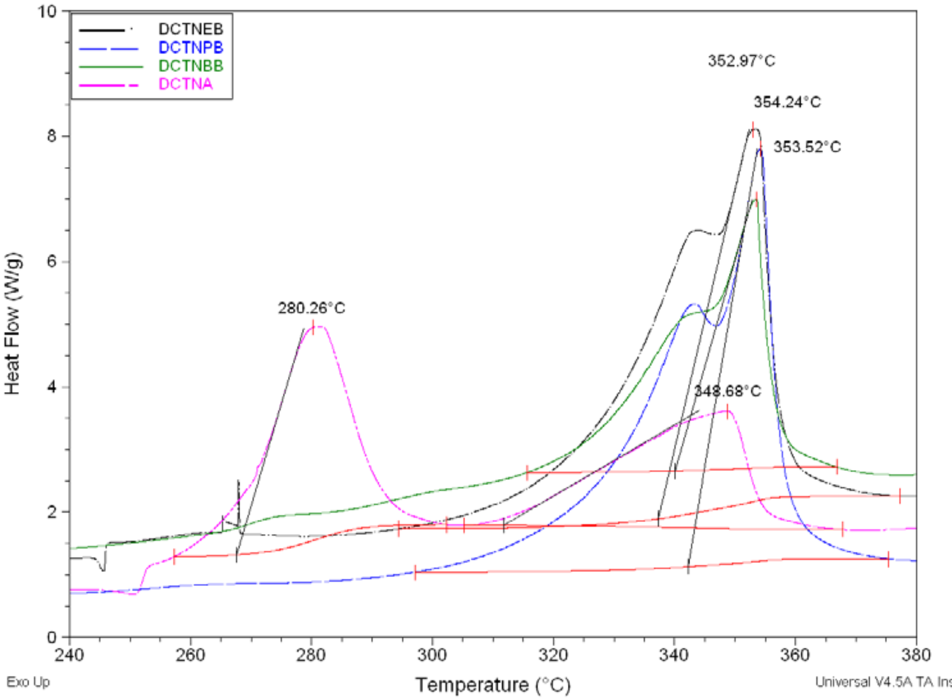
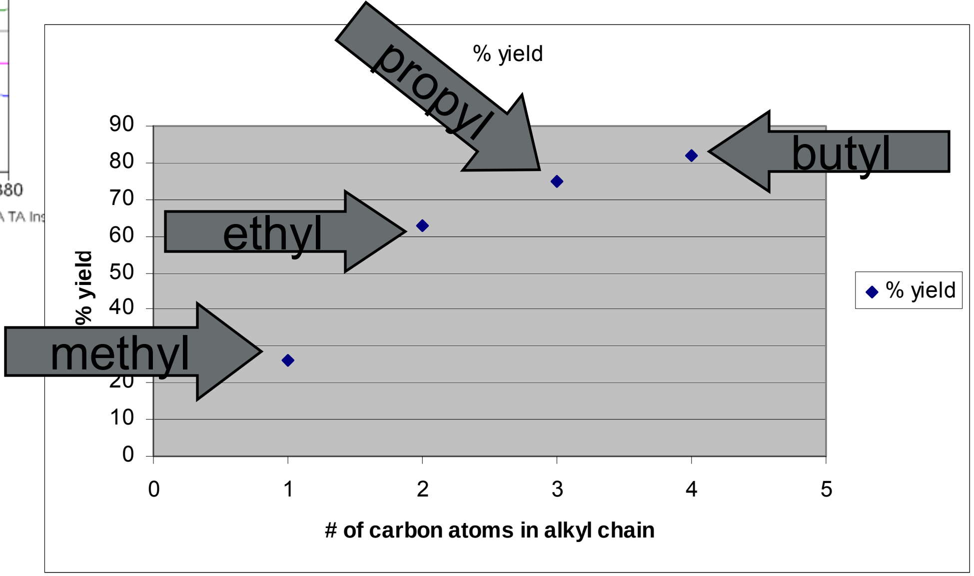


- Aminated TCTNB w/ varying levels of ADAP present in reaction medium
- TCNTB does not afford ADAP.
- ADAP levels in TATB from spiked reactions are consistently low.

Effect of varying length of alkyl chain on yield and DSC (5°C/min)



- Amination of DCTNA (methyl group) gives low yields.
- Ethyl, propyl, and butyl groups show no effect on DSC.
- Yields modestly increase as length of alkyl chain increases.



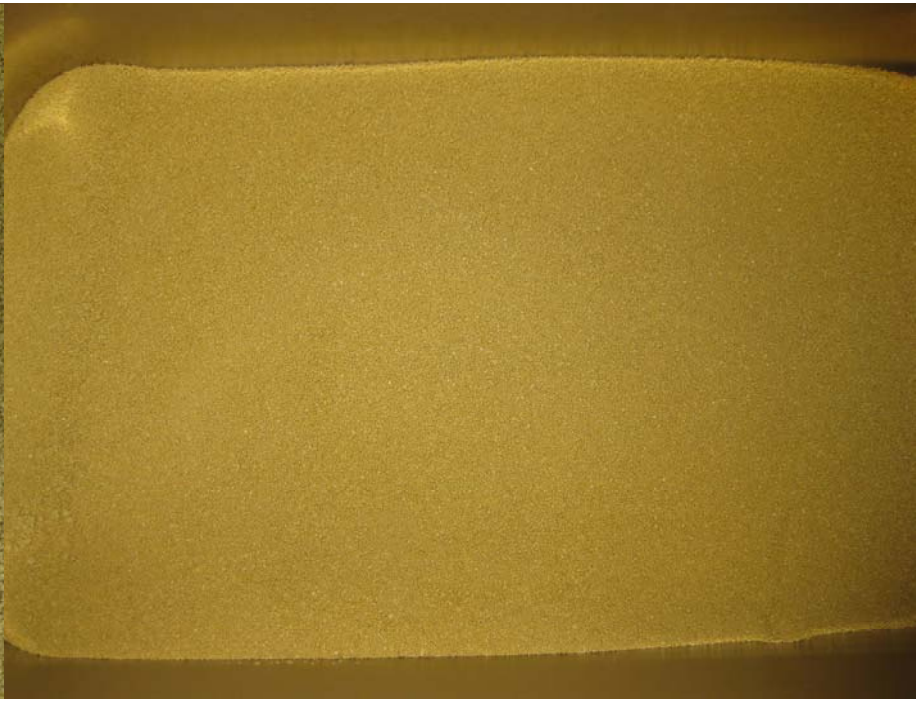
Formulations: PBXN-7

- Several lab batches
- 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

Formulations: PBXW-14

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

Conclusions

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

Acknowledgments

- BAE Systems:
  - Neil Tucker and Jim Haynes-Nitrations and Aminations (lots of them!)
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