An Improved Process for the Synthesis of TATB from TETNB

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Triaminotrinitrobenzene (TATB)

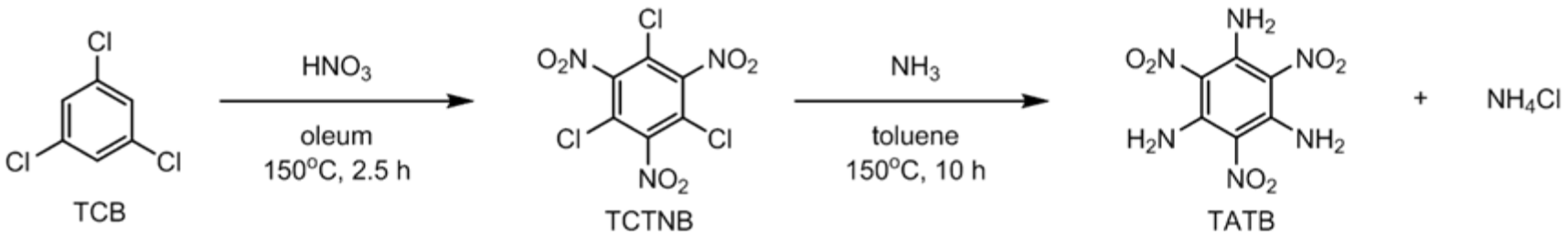
TATB is an important IM explosive material

- TATB is a key component in several important explosive formulations
  - PBXN-7, PBXW-14, PBX-9502, PBX-9503, LX-17
- TATB exhibits excellent balance of performance, thermal resistance and insensitivity to stimuli:

Compound	Sensitivity (Impact)	Deflagration Point	Density	Detonation Velocity
NTO	1200 N m	270 °C	1.91 g/cm^3	7860 m/s
TATB	**50 N m**	**384 °C**	**1.93 g/cm^3**	**7350 m/s**
TNT	15 N m	300 °C	1.65 g/cm^3	6900 m/s
RDX	7-8 N m	204 °C (mp)	1.82 g/cm^3	8750 m/s
CL-20	4 N m	195 °C (mp)	2.04 g/cm^3	> 9000 m/s

* Rudolf Meyer *et al*, Explosives (5th edition), 2002, Wiley-VCH Publishers, Weinheim

Legacy "Benziger" Process for TATB Production



- Starting material = Trichlorobenzene (TCB)
  - TATB from TCB produces environmentally undesirable waste stream
  - TCB is toxic; TCB synthesis from chlorination of benzene
  - PRC (China) is sole source of this material; future supply uncertain and the cost of TATB production may ultimately be affected
  - Currently no qualified supplier of legacy TATB available
    - Bridgewater, Royal Ordinance, McGregor have ceased production

ATK Navy ManTech TATB Process

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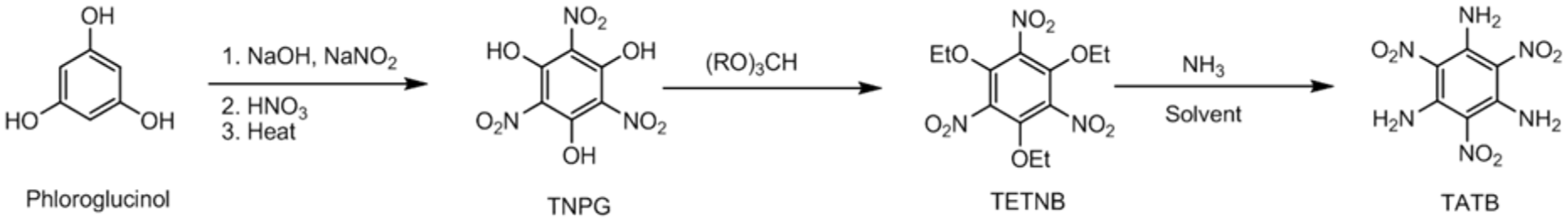
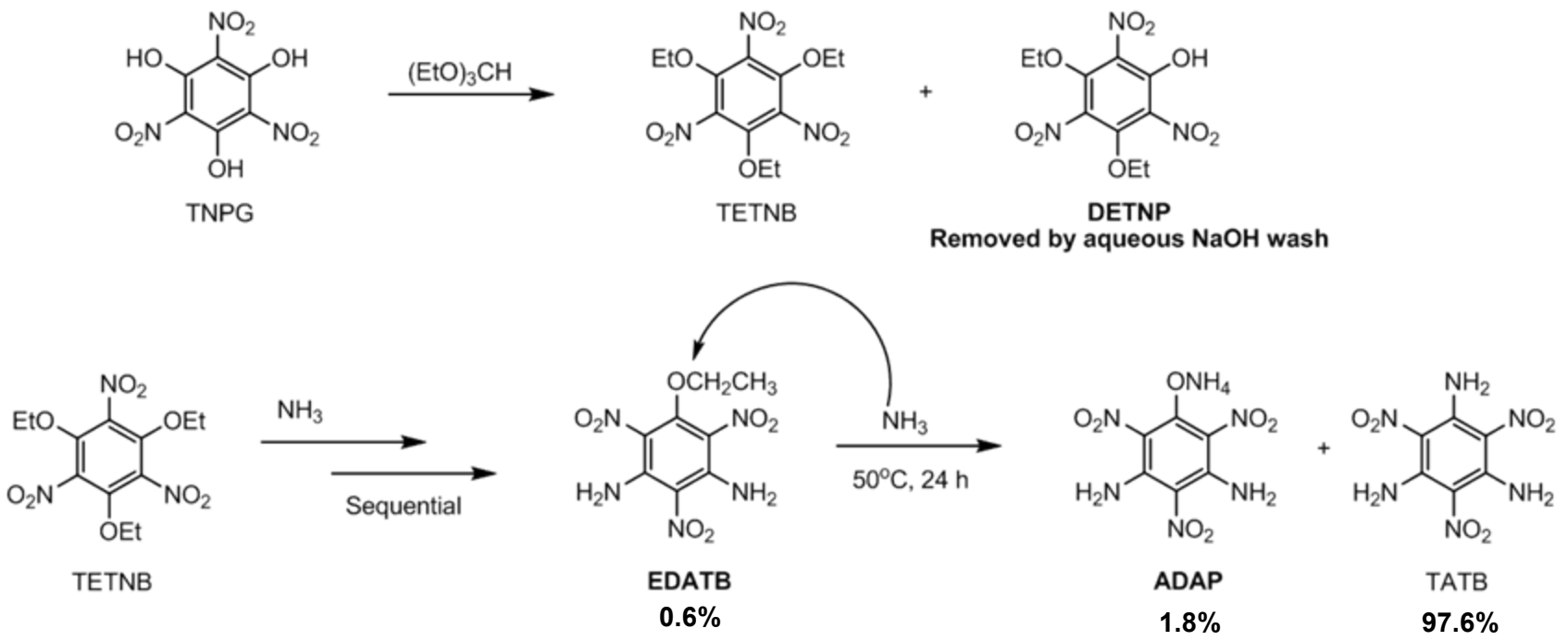


- Navy-funded ManTech process developed at ATK LS between 2003-2007
- Starting material = phloroglucinol (PG)
- Phloroglucinol is a biosynthetically accessible material
- Triethylorthoformate is typically used for alkylation step (form TETNB)
- Main product impurities: ammonium diaminopicrate salt (ADAP) and ethoxydiaminotrinitrobenzene (EDATB; product of incomplete ammonolysis)
- Problem: PBXN-7 formulation using ATK ManTech TATB did not perform in all necessary configurations

Reaction Impurity Formation Mechanism



- Data has shown ADAP to have a minimal effect on TATB sensitivity (safety) and small effect on thermal stability
- Elevated temperature and NH_3 pressure were reported to convert ADAP to TATB (Bellamy)

ADAP Safety Data

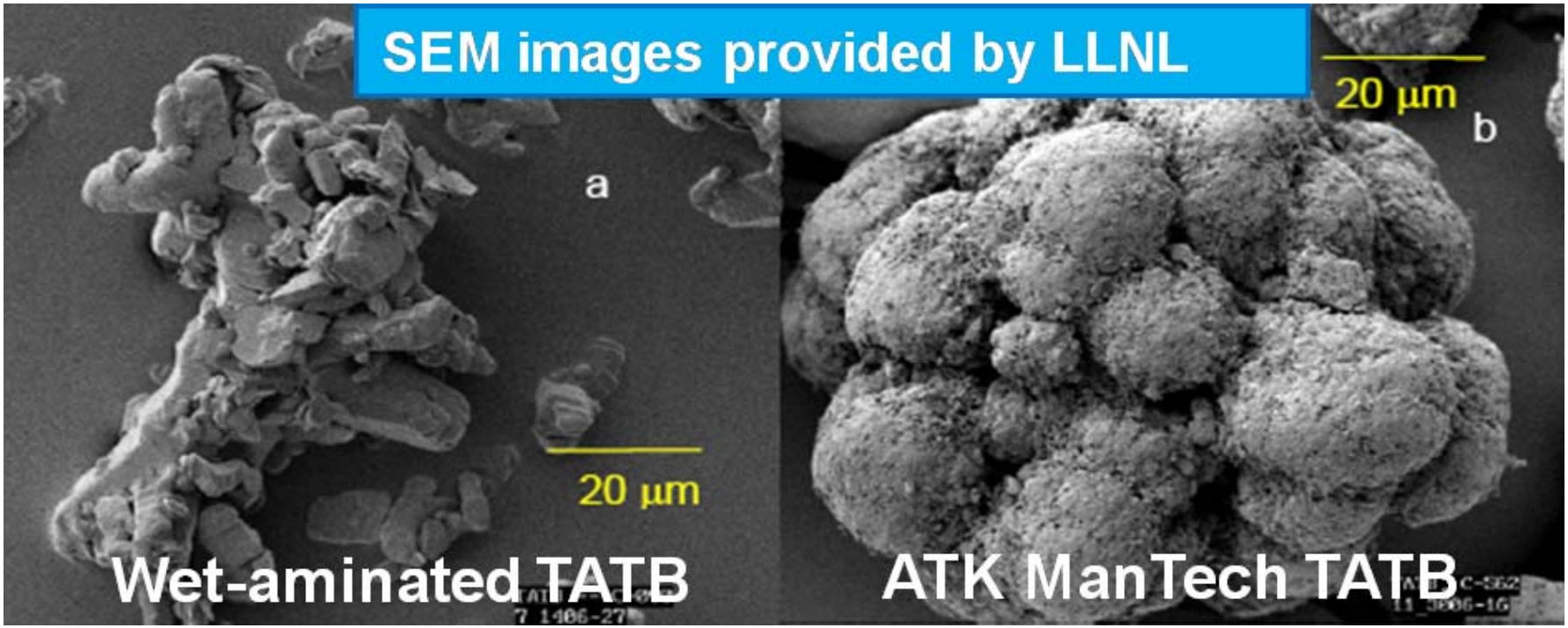
DSC onset/peak (°C)	303.5/306.1
HPLC purity	99.4 %
ABL impact	51 centimeters
ABL friction	800 lbs @ 8 feet per second

Navy: TATB & PBXN7 Performance Differences

	Legacy	ATK ManTech
1st (2nd) DSC Onsets	378	353 (368)
Purity (HPLC)	99+%	97-98%
Mean Size	48	56
Bulk Density (TMD = 1.937)	1.92	1.84
PBXN-7 Density Pressed (%TMD)	99.3-99.9	94.9
FMU-152 @ Low Temp (PBXN-7)	5/5 Go's	3/5 Go's
Large Scale Gap Test (PBXN-7)	207 cards	173.5 cards

- Formulations using ManTech ATK TATB did not demonstrate acceptable performance in FMU-152 fuses at low temperature
- Purity of ATK ManTech TATB significantly lower than legacy
- Densities, pressed densities and thermal onsets lower than legacy material

TATB Particle Morphology Variation



- TATB particle morphology was used as a key screening tool

Altering the ATK Navy ManTech TATB Process

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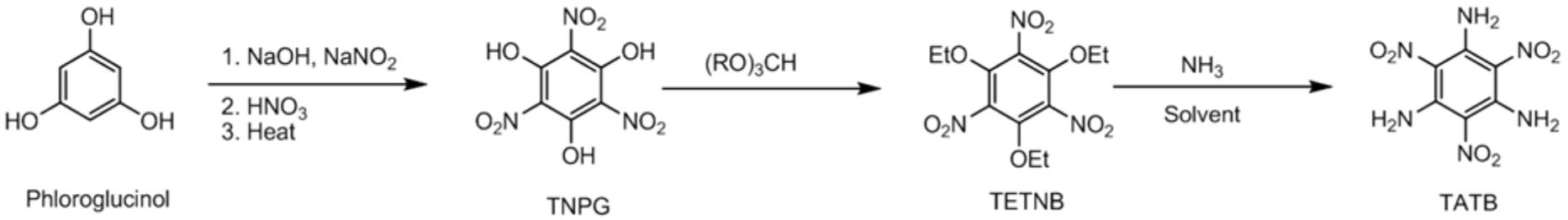
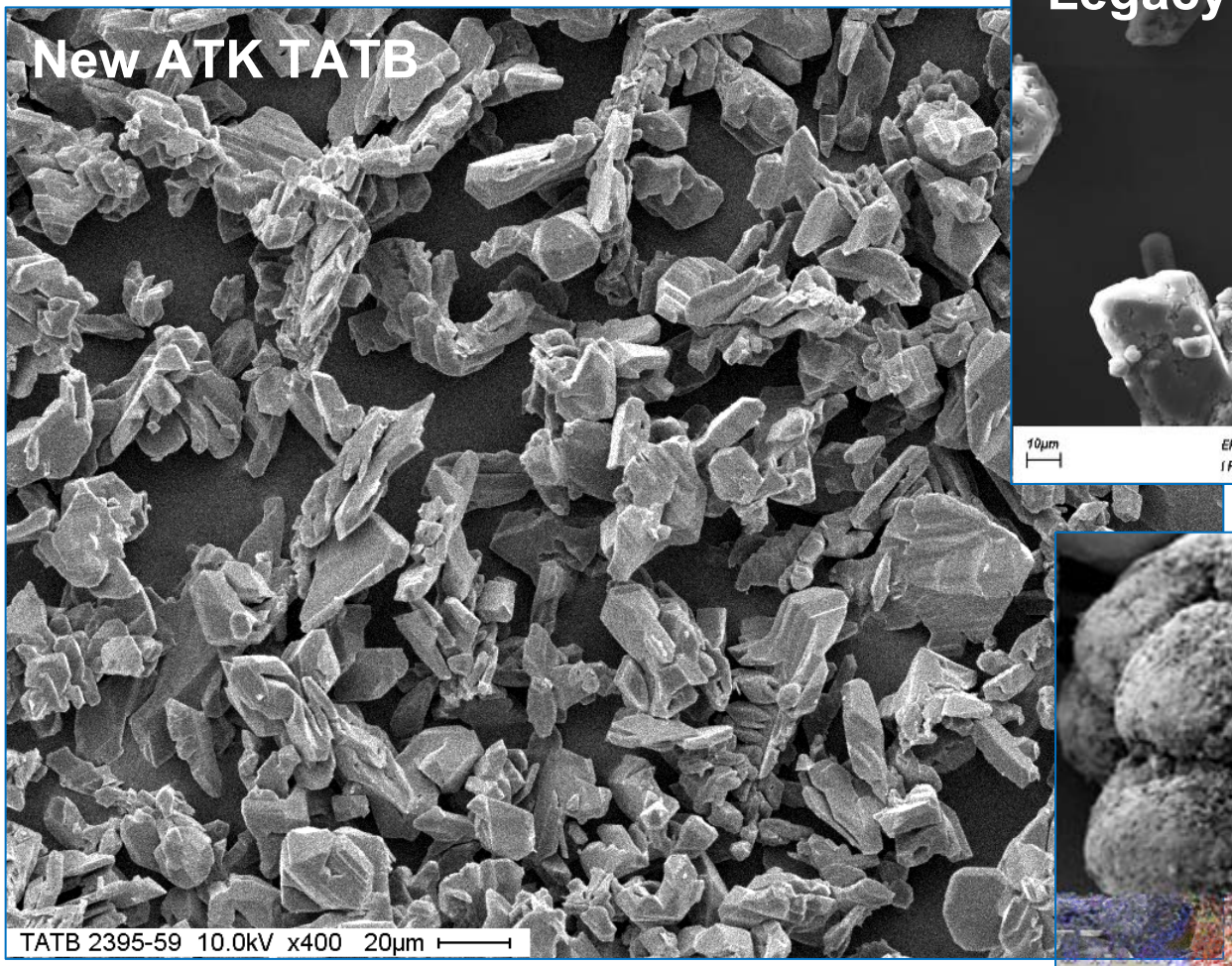
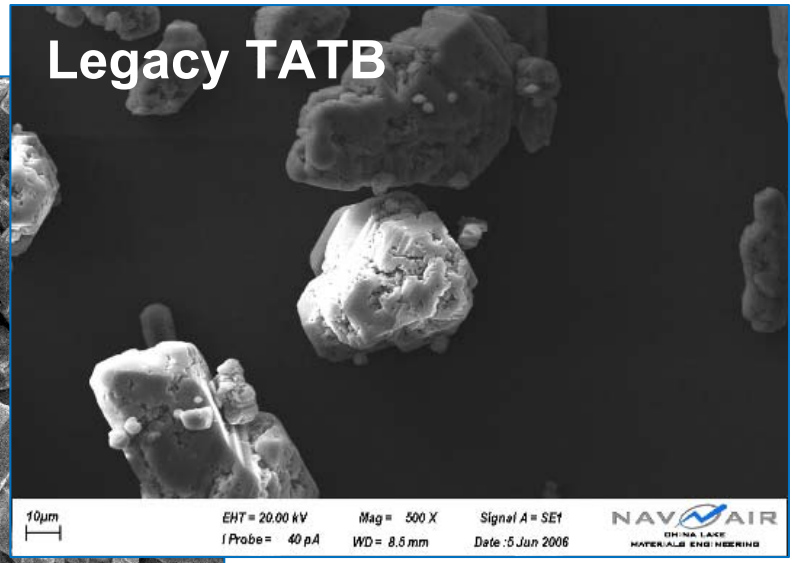


- Considered options to realize utilizable TATB by minimally altering ATK ManTech process
- TATB is difficult to modify post-process due to insolubility in solvents
- As a result, ATK LS used IR&D funding to explore numerous significant process changes to final ammonolysis step
- > 50 lab-scale synthetic experiments were run to date and > 200 supporting analyses have been made on the resulting materials
- ATK LS has seen many promising results as a result of this IR&D effort

SEM of TATB from Newly Developed Method

- **New ATK TATB "looks" more like legacy material by SEM**





SEM of New ATK TATB & Legacy TATB

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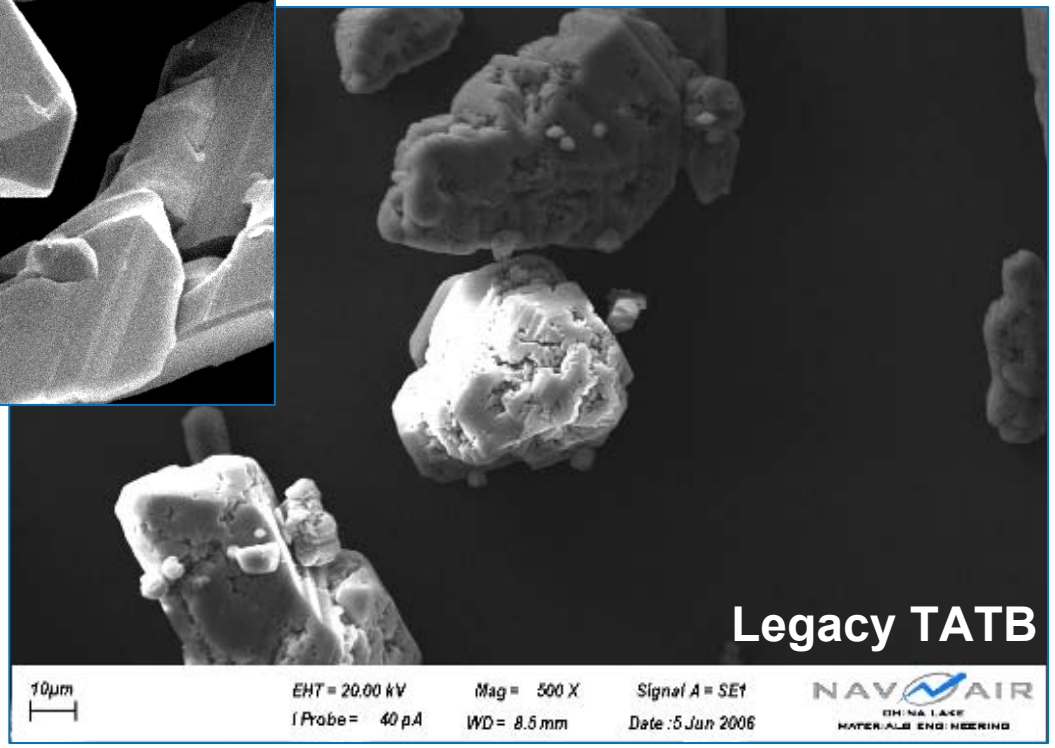

Legacy TATB DSC Data

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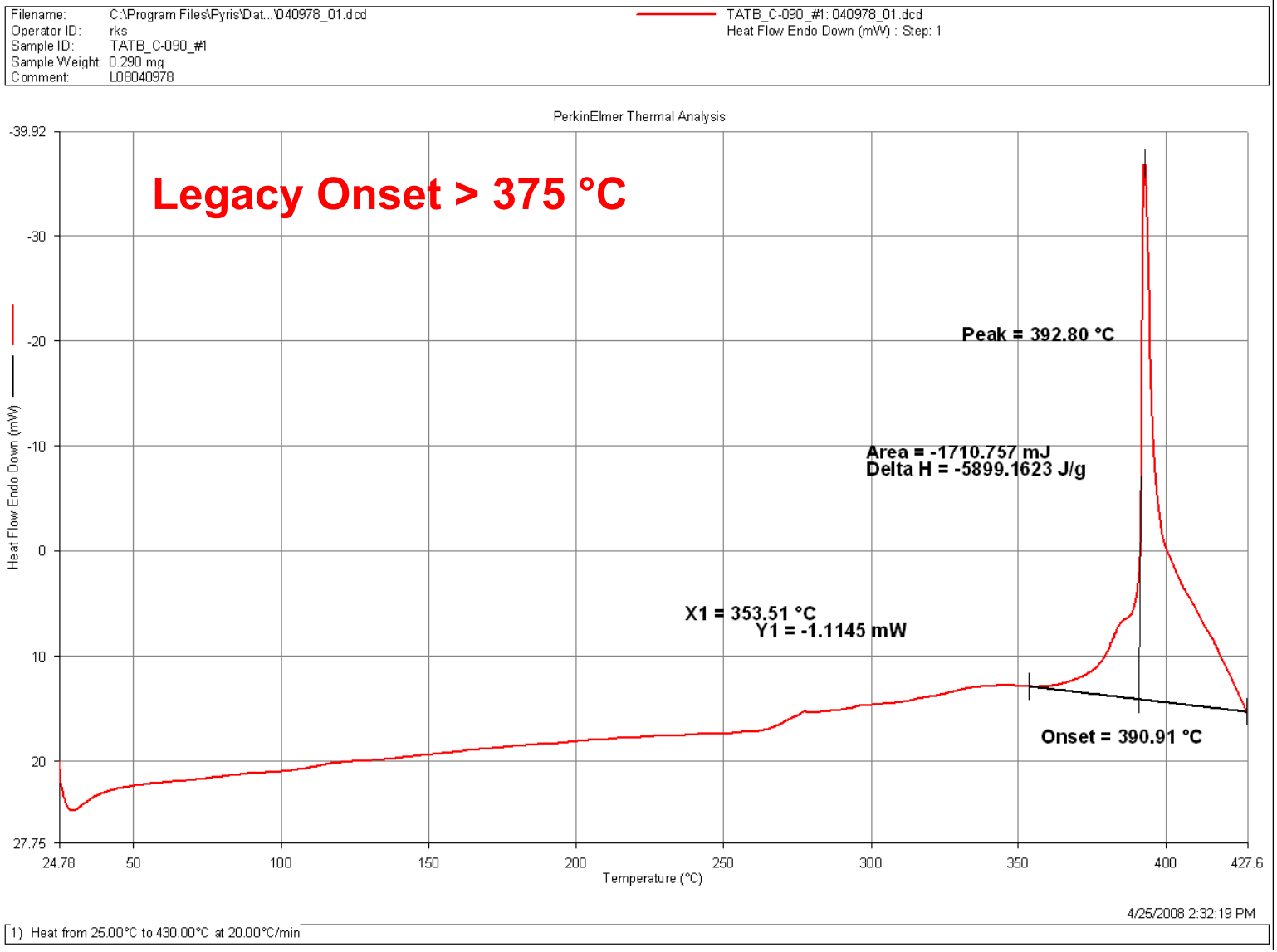

ATK ManTech Process TATB : DSC Data

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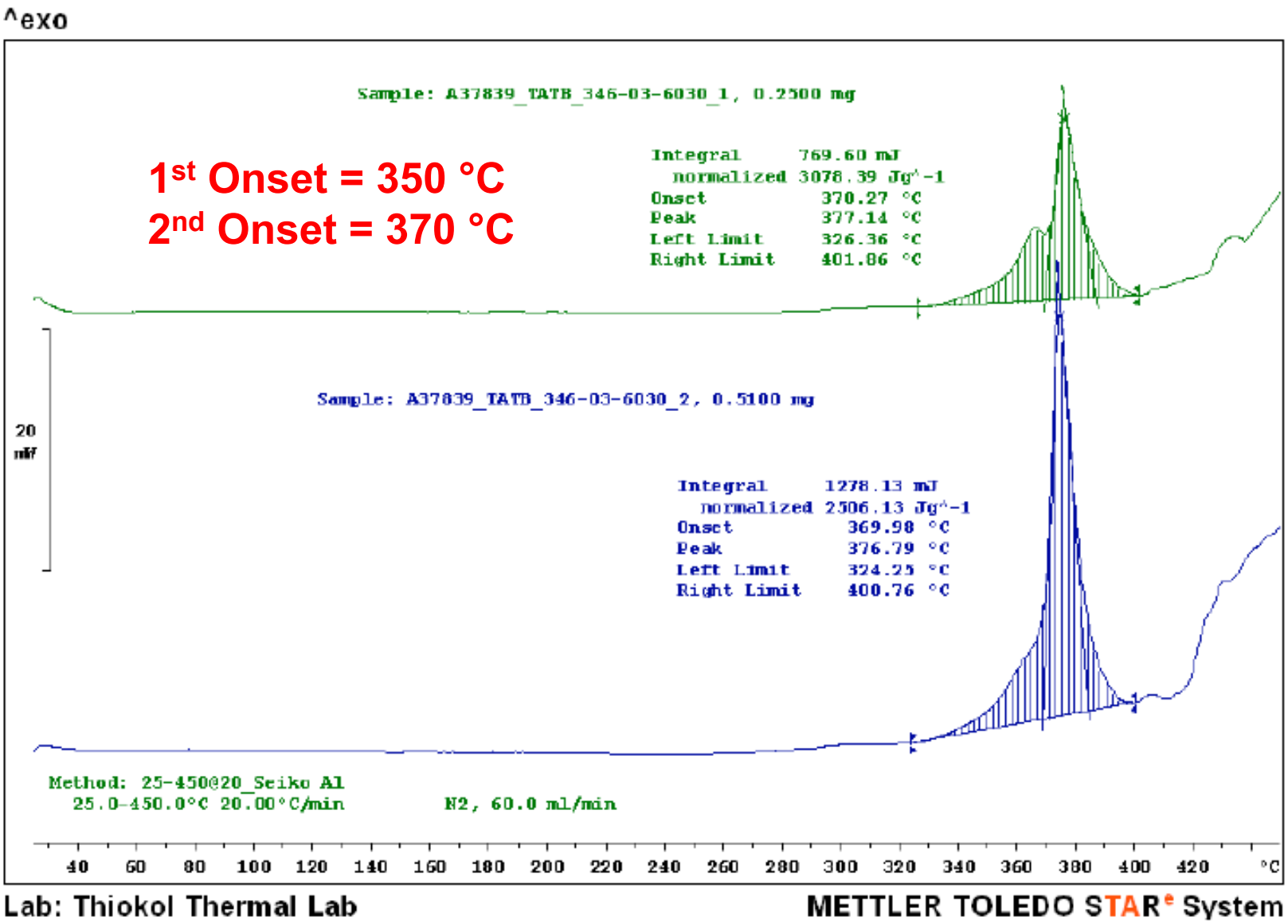

New ATK Process TATB: DSC Data

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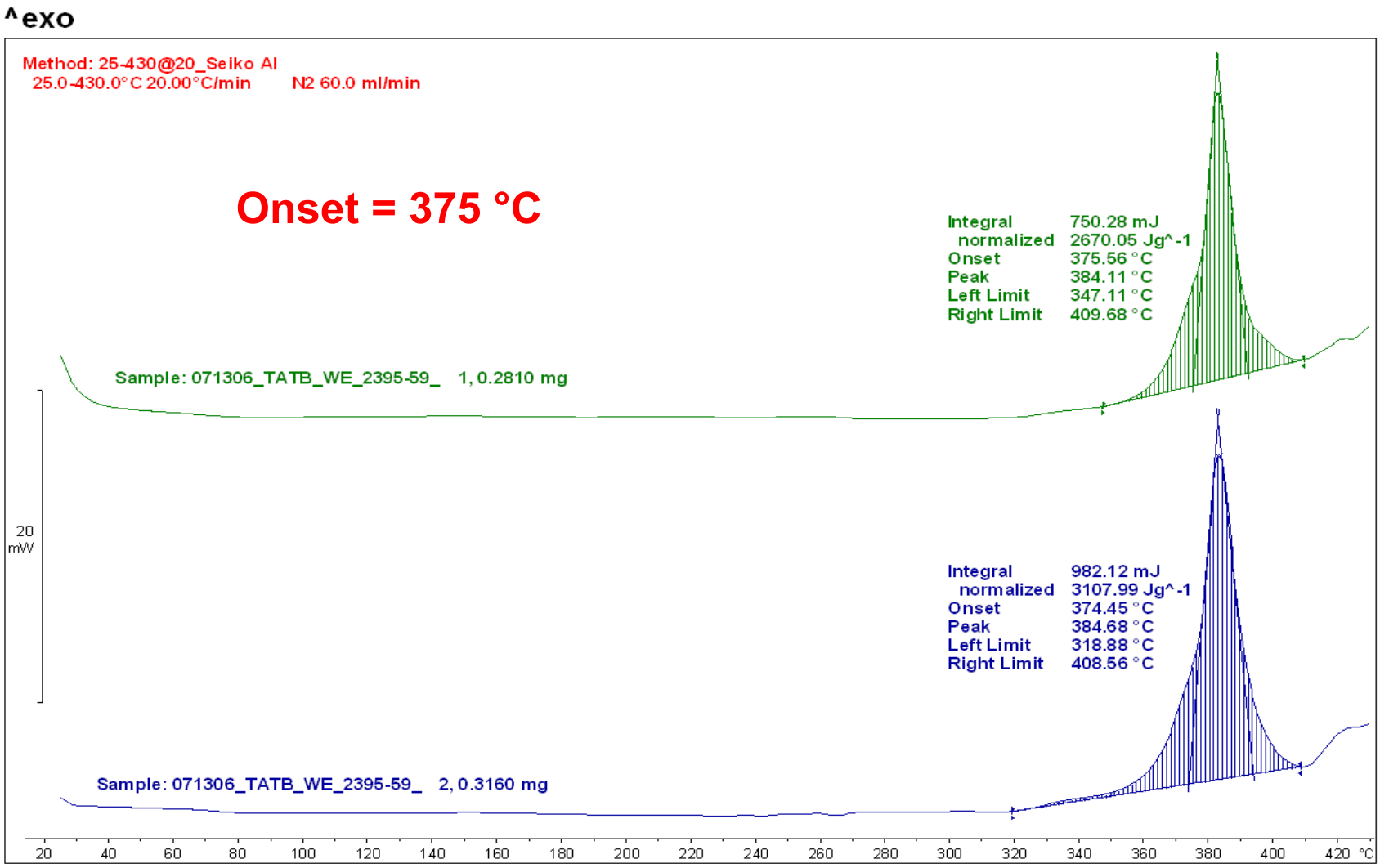

Lab: ATK Thermal Lab

STARe SW 8.10

Properties of the Newly Developed ATK TATB

	% TATB (HPLC)	ADAP	EDATB	50%Size (μm)	1st DSC Onset (°C)	Density (gas pyc)	C,H,N (27.92, 2.34, 32.55)
New ATK 1	99.5%	0.2%	0.3%	22.53	369.8		**28.20, 1.89, 32.07**
New ATK 2	99.3%	0.3%	0.4%	25.75	374.5	1.90-1.91	**28.21, 1.92, 32.00**
New ATK 3	99.5%	0.5%	0.0%	n/a	378.0		**28.14, 2.02, 32.04**
New ATK 4	99.7%	0.3%	0.0%	n/a	380		**28.11, 1.95, 32.03**
Mantech ATK	97.6%	1.8%	0.6%	54.3	368.9	1.83-1.84	**29.02, 2.04, 31.44**
Legacy C-090	100%	0%	0%	70.4	> 378	1.91-1.92	**27.92, 2.34, 32.55**

- DSC onset temperature may improve/become more consistent with refinements to process conditions
- Bulk density is much improved; comparable to legacy material

Conclusions

- New processing conditions for the conversion of TETNB to TATB have been developed
- New conditions yield product that is much more comparable to legacy materials than previous ATK process
- Purity/decomposition temperature improved over ManTech ATK process; not quite as good as legacy but process improvements continue
- Density of new product is much improved; comparable to legacy material
- New ATK TATB appears much more crystalline, “looks like” legacy material by SEM
- 25 gram sample has been sent to China Lake for further evaluation
- 25 gram samples have been prepared for shipment to LANL and LLNL
- ATK’s goal is to establish a viable process for making TATB that can be used by all potential users/consumers (DOD and DOE)

Acknowledgements

- Navy ManTech office
- Chuck Painter
- Tim Mahoney
- Lori Nock
- Becky Olinger
- Others

SEM from Other Explored TATB Conditions

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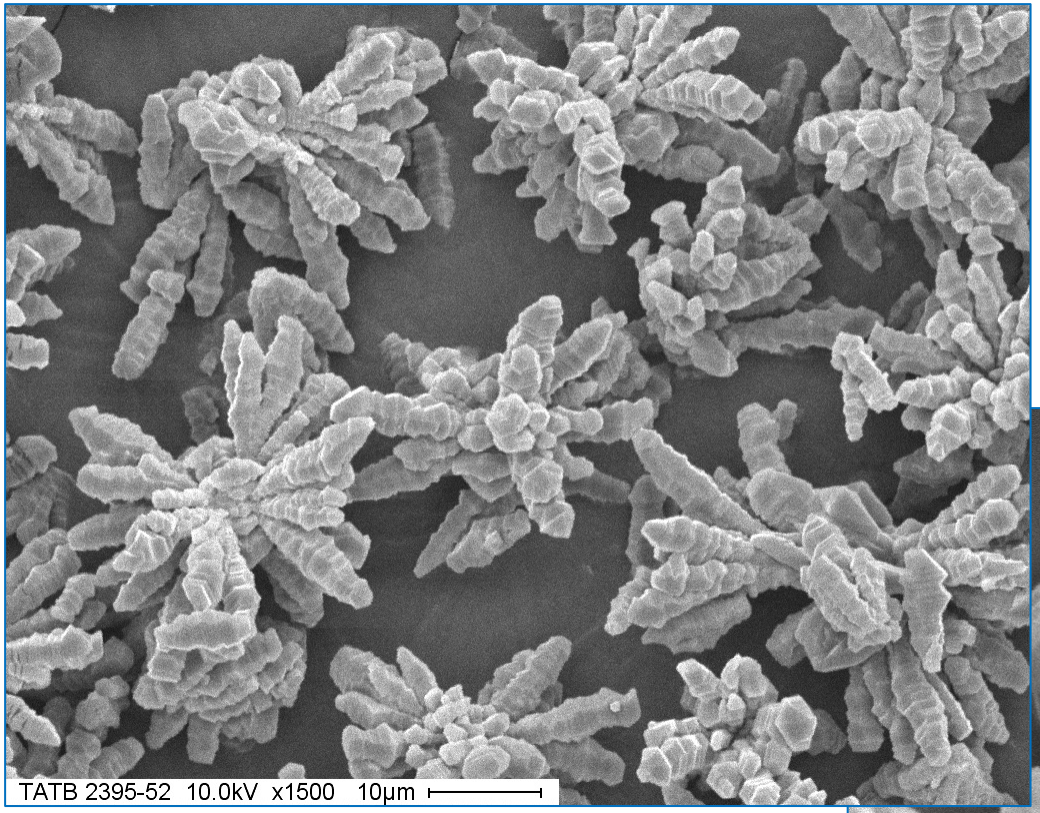

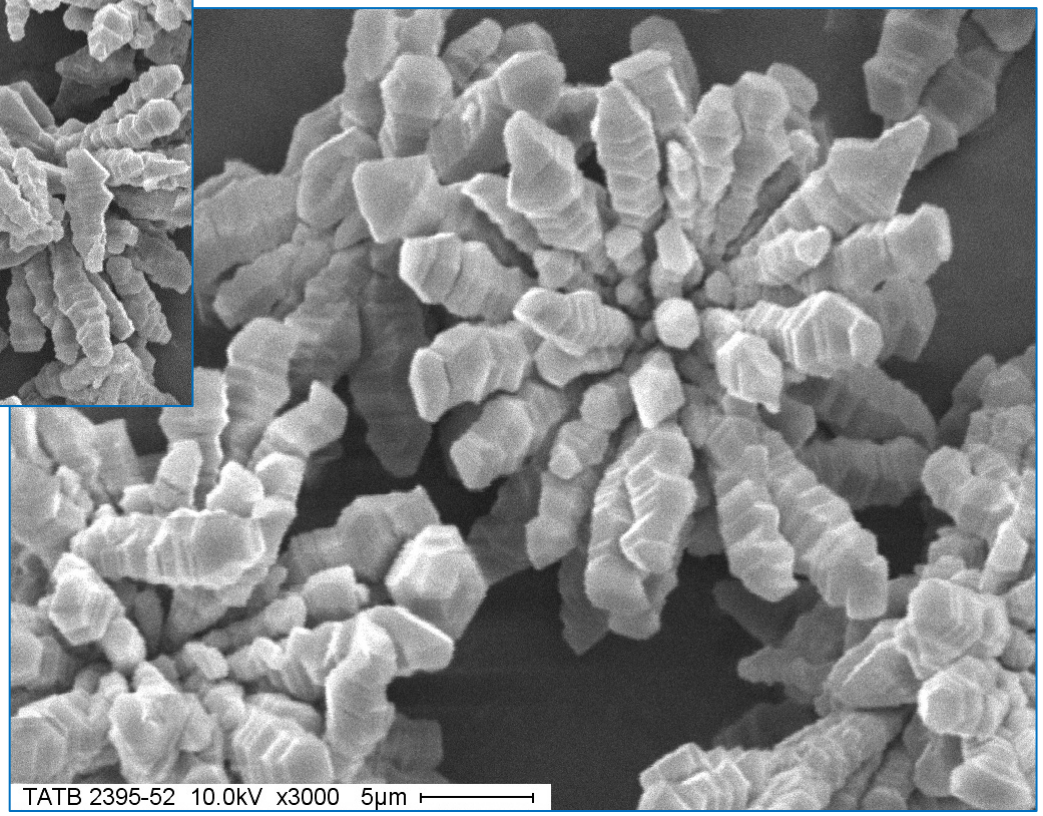

SEM from Other Explored TATB Conditions

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