Pilot plant synthesis of triaminotrinitrobenzene (TATB) from a novel process

Prepared For

2004 IM/EM Technical Symposium

November 15-17, 2004
San Francisco, CA

Sharon Dressen, Douglas Merrill,
Andrew Sanderson, Stephen Velarde*




Approved for public release by NSWC-Indian Head, 21September 2004

Outline

- Novel TATB process
  - Driving force behind development
  - Process overview
- Process results
  - Nitration
  - Alkylation
  - Ammonolysis
- Conclusions

Acknowledgements

- The funding for this work was provided to ATK Thiokol by the US Navy ManTech office
- Special thanks to Lori Nock, Al Stern, Chuck Painter, Jerry Salan, Tim Mahoney, John Brough (Navy), George Boyd (Andrulis), and Rob Schmidt, Alex Mitchell, Mike Coburn (DOE)

Why TATB Synthesis?

- Current production method produces undesirable waste (trichlorobenzene route)
- Future supplies of TCB uncertain – could become a major cost driver for future TATB production
- Development of an alternate route to ensure uninterrupted supply of TATB for needed applications
  - environmentally acceptable waste streams
  - cost-competitive product (vs. current TATB)
  - uncomplicated and robust process to be credibly transitioned to production

TATB Advantages

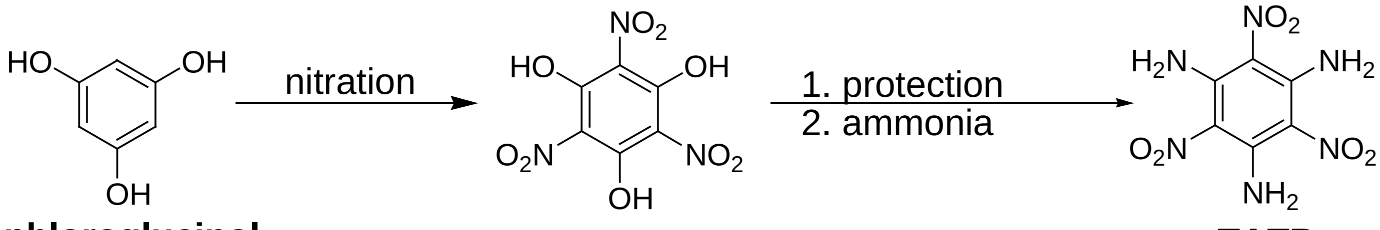
- TATB could be utilized as a valuable material for achieving IM compliance
  - High density - 1.93 g/cm^3
  - High thermal stability
  - Low shock sensitivity

Compound	Impact sensitivity (N m)	Confined detonation velocity (m/s)
Nitroglycerine	0.2	7600
RDX	7.5	8750
TNT	15	6900
TATB	50	7350

Source: Rudolf Meyer, Explosives (3rd edition), 1987, VHC Publishers, New York.

- TATB is already qualified in a number of systems

Alternate TATB Process Overview



- Three-step process was proposed starting from phloroglucinol
  - phloroglucinol found in naturally-occuring glycoside derivatives
  - worldwide, approximately 140-200 metric tons of phloroglucinol are produced each year
  - numerous synthetic industrial routes (including demil of TNT)
- Proposed process first reported in UK by Bellamy, Golding and Ward
- Considerable route development performed at ATK Thiokol
  - necessary for scale-up consideration

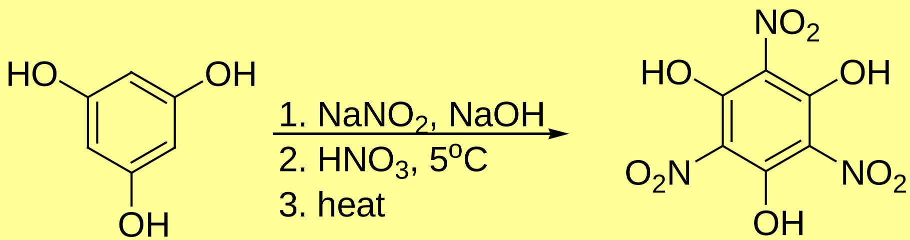
TATB Transition to Scale-Up

- **Lab scale**
  - **1 g to <1000 g**
  - **multiple laboratory fume hoods**
  - **5, 10, 22-L reactors (kilo bays)**

- **Kilogram scale**
  - **multiple kilograms**
  - **dedicated facility including remote operation capability**
  - **50-L, 5 gallon reactors for batch processes**
  - **loop reactor and continuous reactor set-up for continuous operation**

- **Pilot facility**
  - **100's lbs.**
  - **20, 50, 200, 500-gallon reactors**
  - **remote operations**
  - **dedicated operators**

Nitration



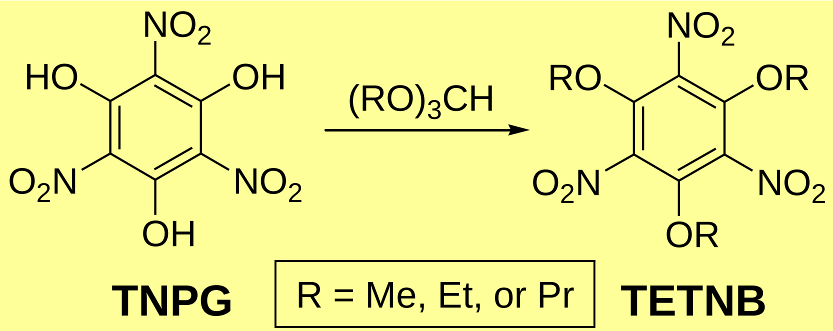
Phloroglucinol **TNPG**

- Reaction exotherm easy to control
- Over 1,000 lbs. (450 kg) of TNPG produced
- Reaction yields have been greatly improved (from ~ 75% to >92%)
  - excellent scalability and reproducibility

Small scale safety data

Safety Test	TNPG
ABL impact (cm)	1.8
ABL friction (lbs)	800 @ 8 ft/s
Electrostatic discharge	1.86 J (mass ignition @ 8 J)

Alkylation

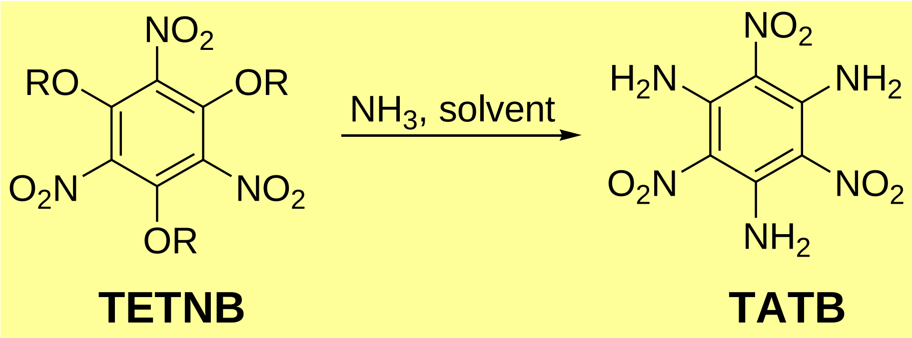


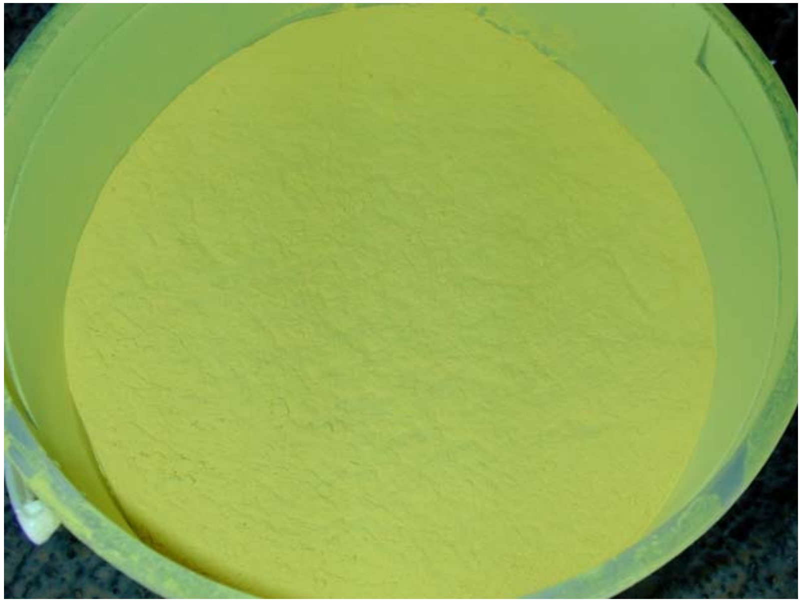
- Efficient, straightforward process
- High reactor loading levels
- Isolated yields >90%
- Triethylorthoformate used for cost considerations
- TETNB relatively insensitive and easy to handle

Small scale safety data

Safety Test	TETNB
ABL impact (cm)	64
ABL friction (lbs)	800 @ 8 ft/s
Electrostatic discharge	>8 J

Ammonolysis



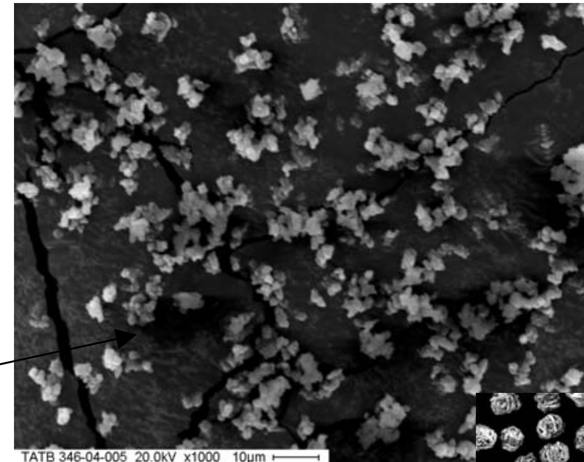


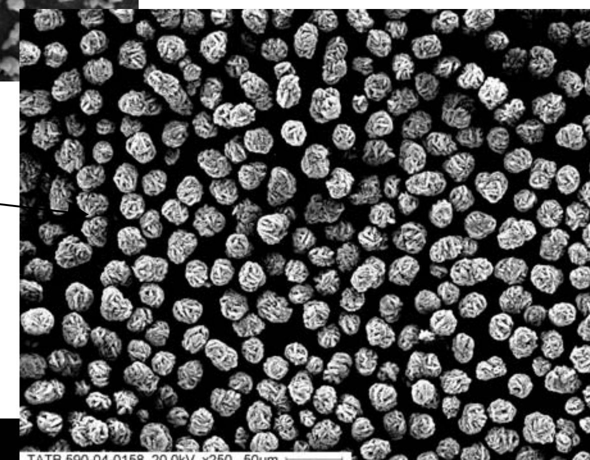
- Simple process that can yield complicated results
  - particle size & shape, purity
- Target particle size 40-60 micron (to match current TATB spec)
- Yield generally >98%

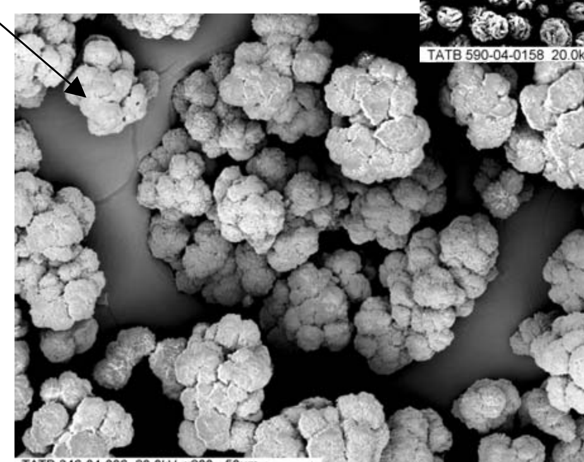
Small scale safety data

Safety Test	TATB
ABL impact (cm)	80
ABL friction (lbs)	800 @ 8 ft/s
Electrostatic discharge	2.7 J (no mass ignition)

Lot number	Particle size (µm) (10, 50, 90%)
346-04-001	18.9, **21.9**, 25.6
346-04-005	2.9, **4.3**, 6.8
590-04-0158	18.5, **24.6**, 35.4
346-04-006	32.7, **42.4**, 71.8
346-04-007	21.7, **33.2**, 65.0
346-04-008	18.7, **26.6**, 49.0
346-04-009	42.2, **65.1**, 131.3
346-04-010	37.1, **54.1**, 105.0
346-04-011	21.6, **31.2**, 59.6
346-04-012	60.5, **82.6**, 142.9

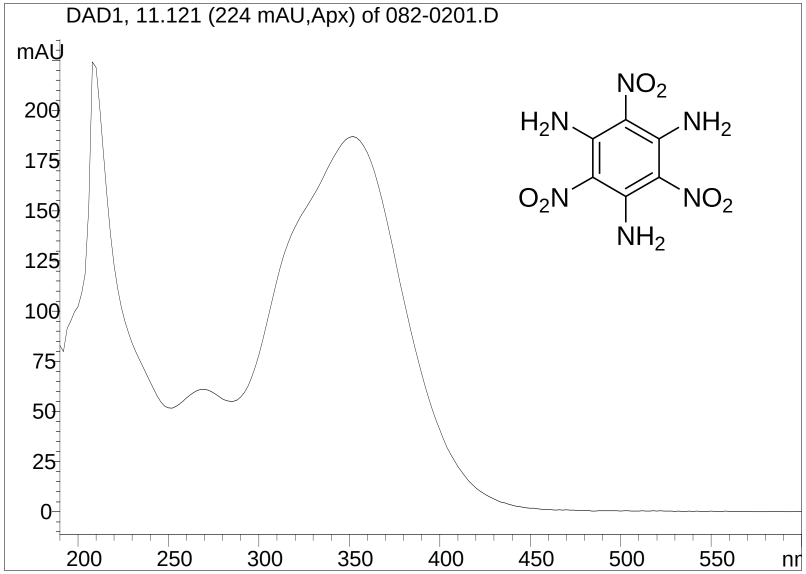




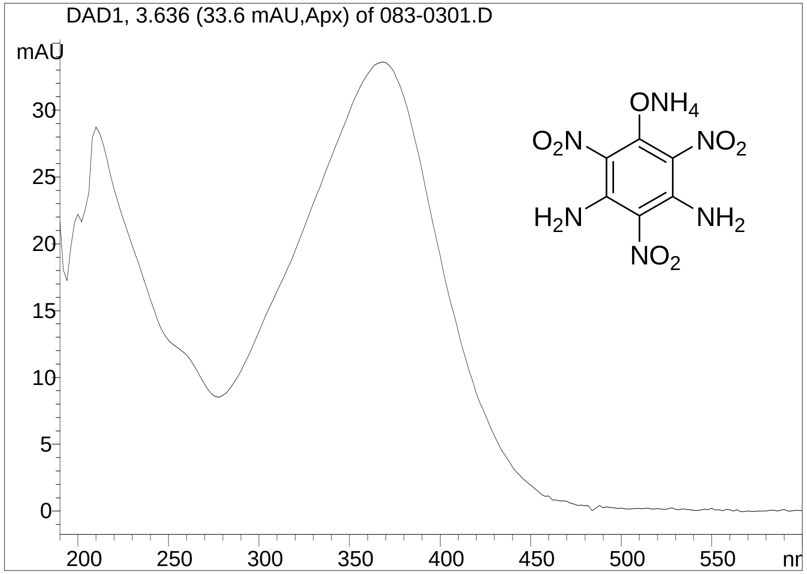


TATB

- Purity determination was a concern due to TATB insolubility
- HPLC/MS used to identify impurities
  - ammonium diaminopicrate and 1-ethoxy-3,5-diamino-2,4,6-trinitrobenzene are process impurities
  - quantification of impurities by HPLC an approximation due to similar absorbance spectra
  - TATB produced generally 95-99% pure

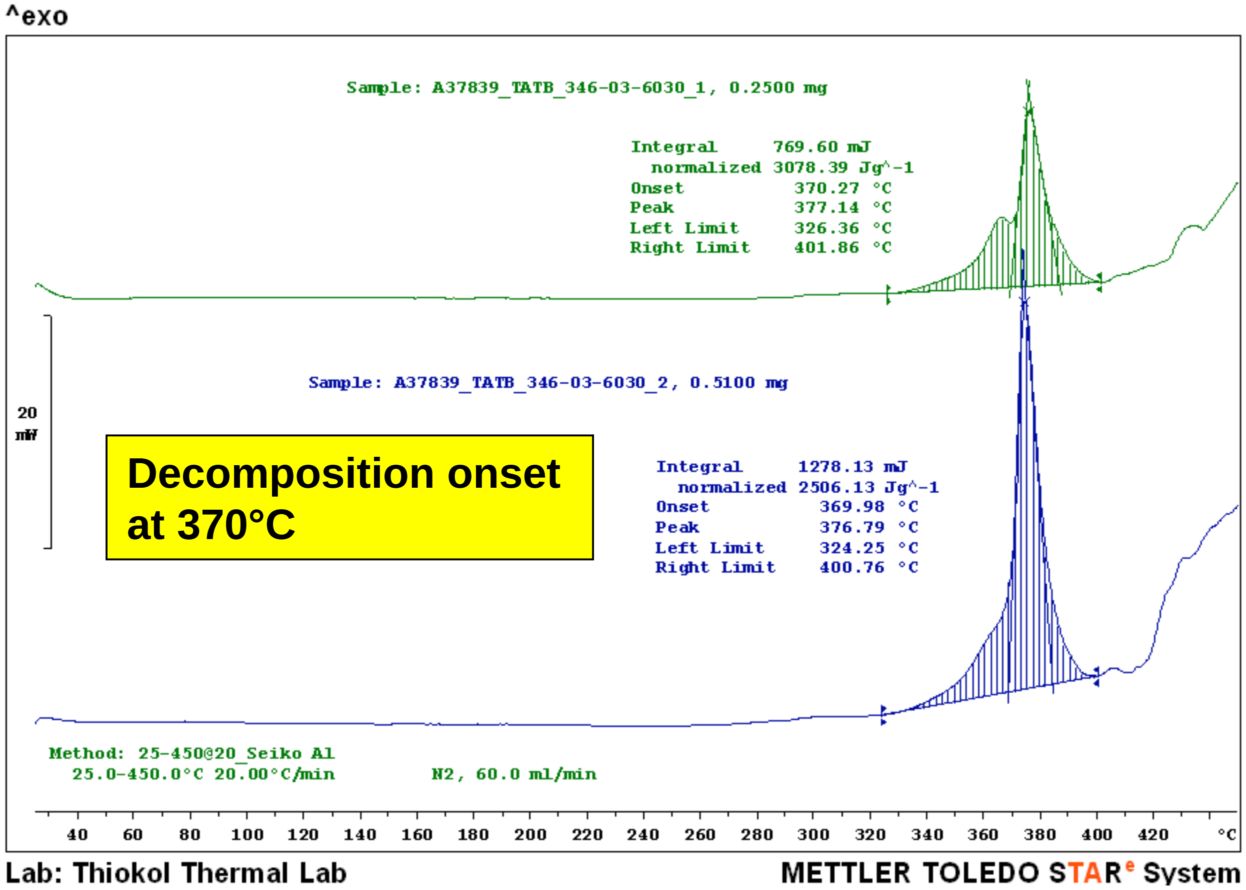


UV spectrum of TATB



UV spectrum of ADAP

TATB Thermal Behavior



- TATB possesses excellent thermal stability

Conclusions

- A novel process for producing TATB from phloroglucinol has been successfully transitioned from the lab to the pilot plant level
- The three step process reduces the environmental impact of TATB production when compared to the traditional trichlorobenzene route
- TATB particle size control possible via the current route
- The TATB produced under this effort will undergo extensive testing and evaluation at the completion of the program