



U.S. Army Research, Development and Engineering Command

The Malcolm Baldrige National Quality Award logo, featuring a stylized gold star and ribbon design on a dark red background with binary code. The text "Malcolm Baldrige National Quality Award" is in a serif font, and "2007 Award Recipient" is in a bold sans-serif font below it.

Malcolm Baldrige  
National  
Quality  
Award  
2007 Award  
Recipient



***TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.***

## **Nitrotriazolone Process Optimization**

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**15 May 2012**



- US ARMY RDECOM
  - ManTech Funding
- US ARMY RDECOM-ARDEC
  - Munitions Engineering & Technology Center (METC)
    - Engineering & Technical Lead
    - Producibility Support
  - Project Management & Integration Directorate (PMID)
    - Program Support
  - Defense Ordnance Technology Consortium (DOTC)
    - Contracting Support
- US ARMY PD-Joint Services
  - Leveraged Funding
- US ARMY PM-CAS
  - Technology Transfer Customer
- BAE Systems
  - Research & Development Analytical Labs
  - Production / Design Teams



**BAE SYSTEMS**



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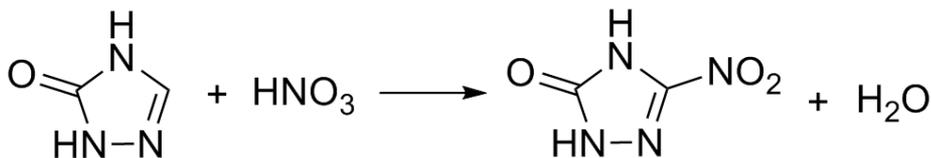


- Optimize NTO re-crystallization process to increase product yield without affecting quality
  - Identify key process metrics that can be optimized
  - Quantify and verify process parameters in laboratory environment
  - Implement and verify process improvements on manufacturing scale



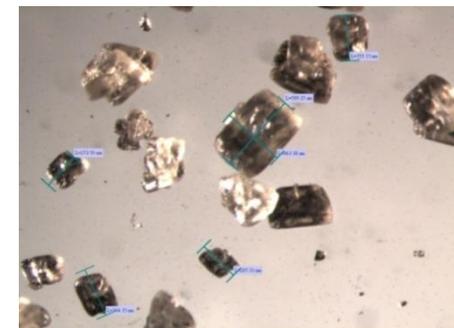
- Nitrotriazolone

- NTO is a key constituent being integrated into new energetic material formulations (IMX-101 & IMX-104)



Triazolone + 99% Nitric Acid  $\longrightarrow$  Nitrotriazolone (NTO) + Water

- NTO has similar performance to RDX with improved IM characteristics
- IMX explosive formulations are the high explosive loads in IM artillery projectiles and mortar cartridges





- Nitrotriazolone

- Demand for NTO will increase substantially to comply with insensitive munitions requirements
  - IMX-101 Qualified by the U.S. ARMY as main fill explosive in the 155mm M795 Artillery Projectile.
  - 2.1 M lbs annual NTO quantity required for IMX formulations
- It is anticipated that other munitions which use traditional energetics (Comp B or TNT) will utilize the same or variant compositions containing NTO
- NTO yield is not fully achieved. As a result, manufacturing costs are high (\$15/lb). Current manufacturing technique prevents full recovery of NTO



155mm M795  
Projectile



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- Develop / modify manufacturing techniques to permit full or nearly full recovery of NTO in bench scale environment
- Verify NTO crystal size, shape and morphology meets or exceeds military specifications
- From bench scale, develop design for facility modifications and/or equipment additions to optimize NTO manufacturing process
- Implement and verify process modifications at Holston AAP, G-10 Agile Manufacturing Facility



- Optimized NTO manufacturing process
  - Increased NTO yield and recovery
  - Increased yield per batch, lower NTO cost
  - Improved production control to attain consistent NTO yield and recovery

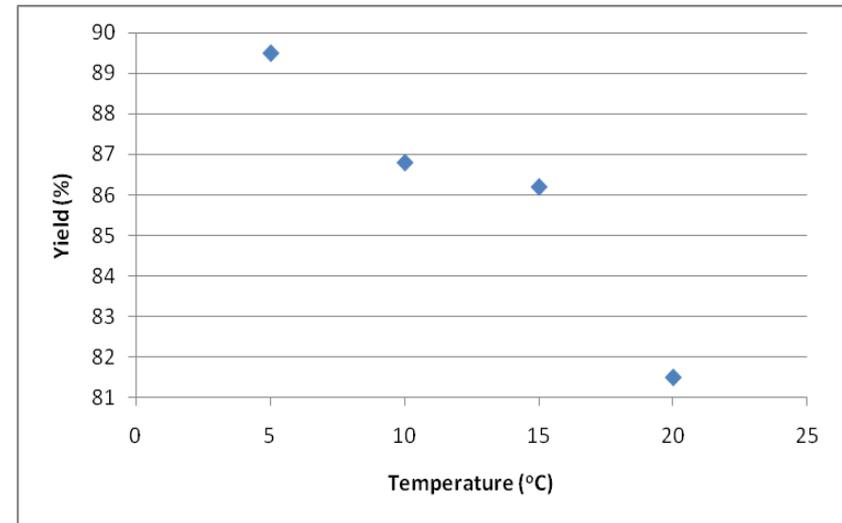
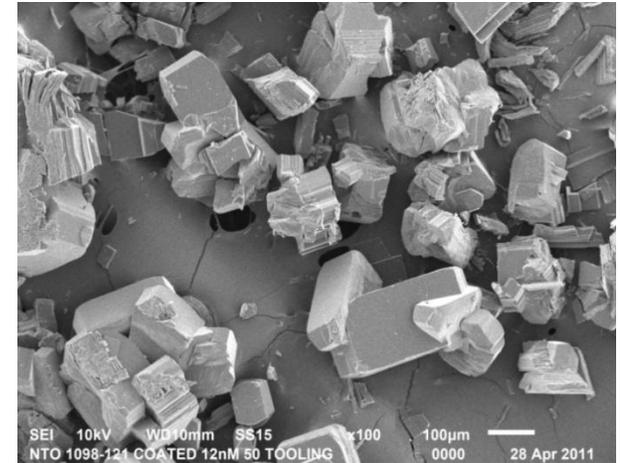


- Analytical Analysis
  - Equipment
    - 2-L Mettler-Toledo Reaction Calorimeter
    - Fully automated, integrated with PC for data collection
- Design of Experiments
  - DOE Variables: Final Temperature, Cooling Rate, NTO Concentration, Agitation, Hold Temperature
  - Experiments were set up so that each time a variable was changed (i.e. cooling rate, NTO concentration, etc), the yield and quality of NTO produced was looked at each of the four final temperatures (20, 15, 10, 5 °C)





- DOE Results/ Conclusions
  - Lowering the final temperature has the most noticeable impact on yield.
    - Changing the baseline parameters rarely changed the crystal size & morphology
    - Verified crystal structure and morphology using optical and scanning electron microscopes
    - NTO purity verified thru HPLC
  - Lowering NTO temperature down to 5deg C has no little affect in quality
  - An increase as high as 10% NTO yield
  - DOE results fed into the process design



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- Completed Process Design
  - Incorporating chiller / refrigeration system
    - Due to the energy consumption in the chiller, the design incorporates river water to initially cool during re-crystallization.
    - “Chilled” water will then cool to desired temperature
  - Integrating new chiller controls with existing control system
- Implementation
  - Procured long lead process equipment
  - Anticipated equipment arrival April - May 2012



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- NTO production facility scheduled for 6-month shut down
- Install, integrate, and prove-out facility improvements
- Verify NTO production yield and quality
- Transition to production by end of CY2012



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- Decreasing NTO re-crystallization temperature increases NTO product yield
  - At 5 deg C, NTO yield increases by 10%
  - Low re-crystallization temperature does not affect the overall quality of NTO
- FY12 milestones include procuring, installing, and integrating refrigeration / chiller system. As well as, prove-out process improvements and verify program metrics.





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