Recent Developments in CL-20 Synthesis and Processing

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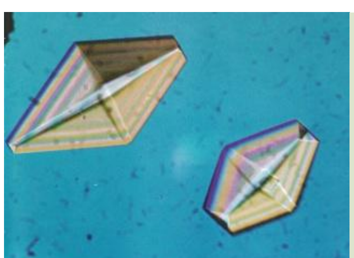
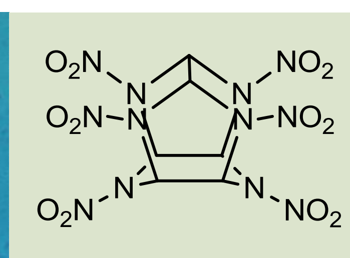
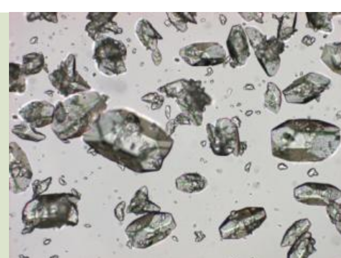






Outline

- Acknowledgements
- Overview
- CL-20 synthesis
  - Process improvements
- Use in formulations
- Summary

Acknowledgements

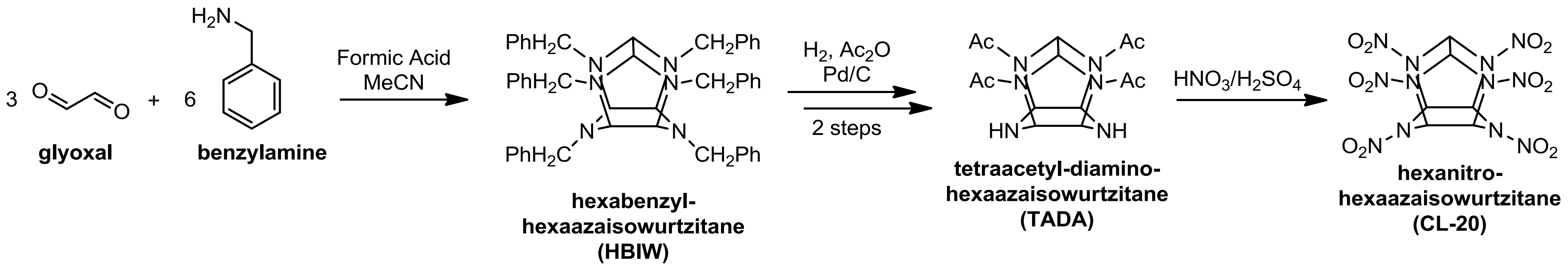
- Support of CL-20 from many DoD and DOE partners
- Dedicated team at ATK Aerospace
  - PM: Nathan Seidner
  - Scientists: Dr. Mike Killpack, Michael Adams
  - Analytical team: Max Patterson, Brian Rosa, Erin Anderson, Ken Spaulding, Joanne Bingham, Dr. Shawn Parry, Dr. Ping Li
  - Operations: David Schmidt
  - Quality: Kirk Bailey
  - Safety: Arlan Brandt

Overview

- CL-20 is one of the most interesting energetic molecules to be developed since WW II
- Original CL-20 synthesis route developed at NAWC by Dr. Arnold Nielson, 1987
- TADF process pioneered by Thiokol in early 1990s provided access to thousands of pounds of material for development programs
  - Synthesis was not optimal
- TADA, discovered by Dr. Robert Wardle in 1988, was not considered a viable precursor until independent development of a large-scale process for its synthesis was revealed by Asahi in 1997 time frame
  - TADA nitration produces higher yield and purity product and lower, more easily controlled exotherm than the TADF process
- During the past decade regular improvements in the synthesis and crystallization have been made

CL-20 Synthesis Summary

- Four step process from commercially available feed stock:



- Recrystallization is required to obtain the desired ε polymorph - crude CL-20 from nitration is a mixture of α and γ

- Recrystallization is essential to achieving desired particle size, morphology, and purity
  - Therefore any optimization of the recrystallization process is expected to have a positive impact on material cost and product quality

CL-20 Manufacturing Overview – Nitration

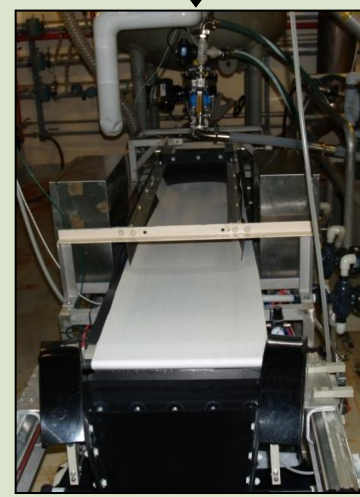
Nitric Acid Tank and Meter

TADA Addition

Dump Tank

Sulfuric Acid Line & Mass Meter

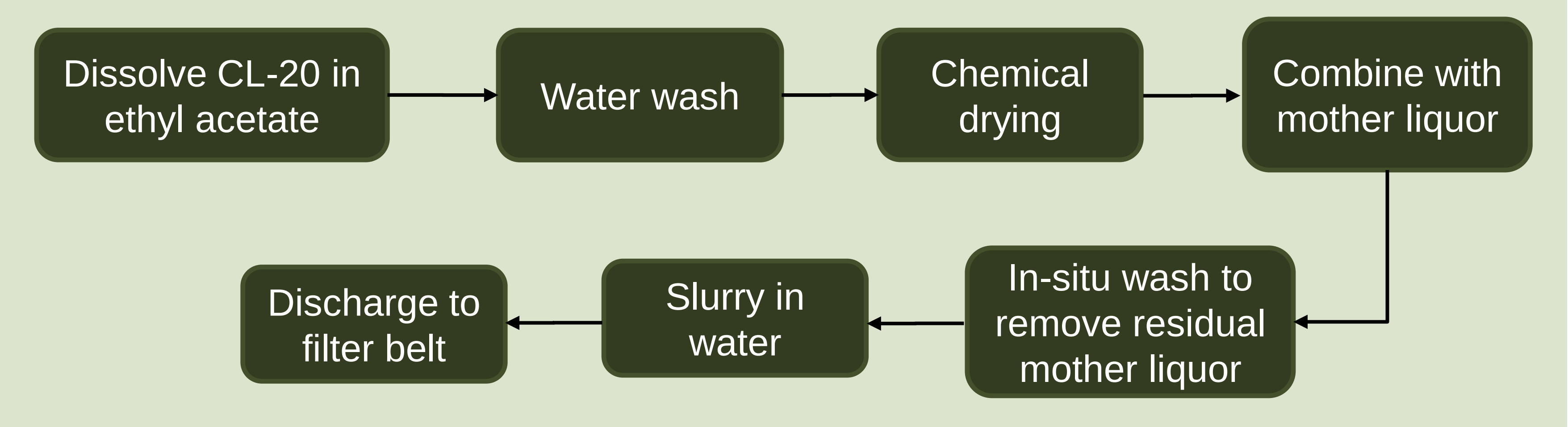
Jacketed, Glass-Lined Reactor System



Vacuum Belt Filter

Nitration process is being continually refined and improved

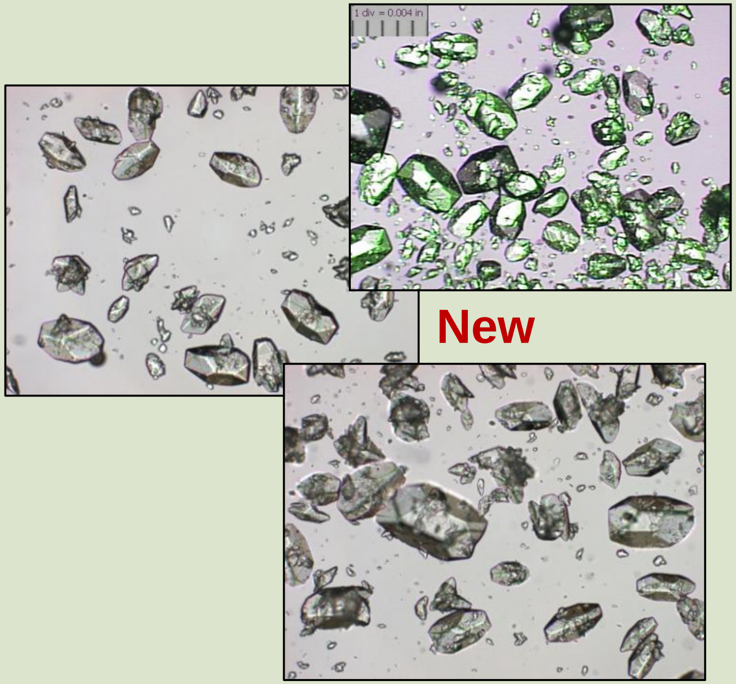
CL-20 Recrystallization Overview



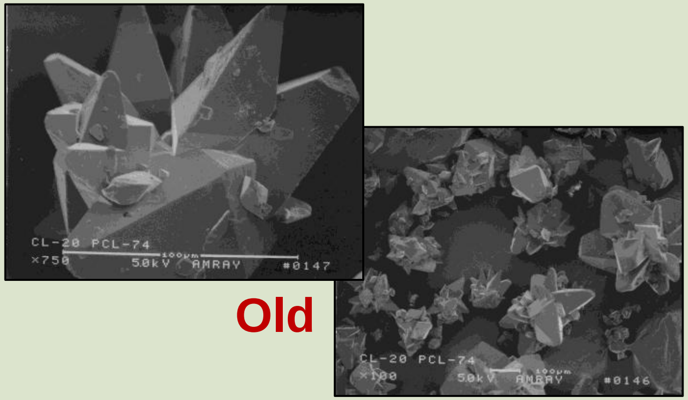
- **Process is the result of a 2003 Navy Mantech effort**
  - Determined that "evaporative recrystallization" was optimum for reproducibility
    - Scaled to multiple 500-gallon runs
    - Gives reproducible "unground" particle size
    - Consistently ε polymorph
    - Good particle morphology (distinct crystals)

Recrystallized (Unground) CL-20

- CL-20 is manufactured to an internal ATK specification
  - Specification is based on the existing STANAG for CL-20
- Improvements to crystallization step produces crystals that are more rounded and easier to process
  - Internal defects have been minimized

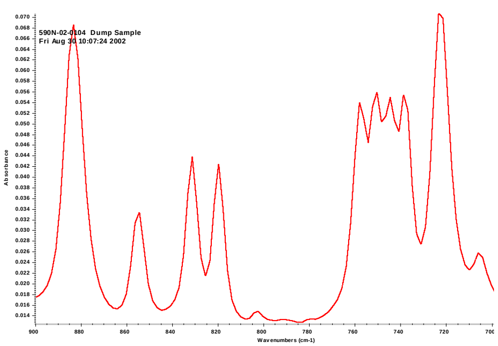


New

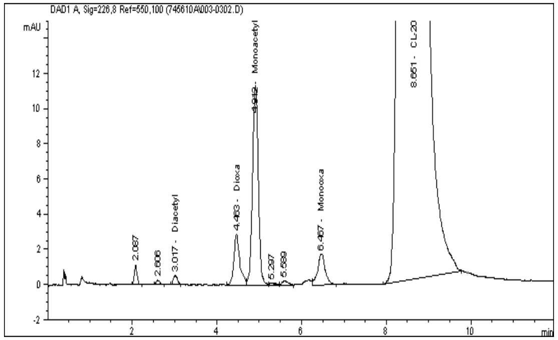


Old

Epsilon Polymorph



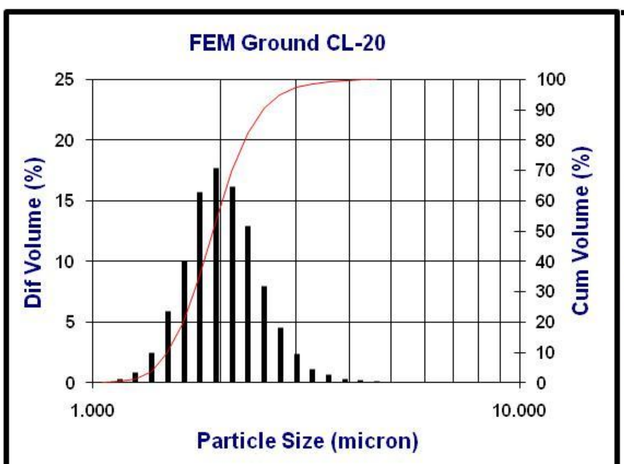
99.5% Pure by HPLC

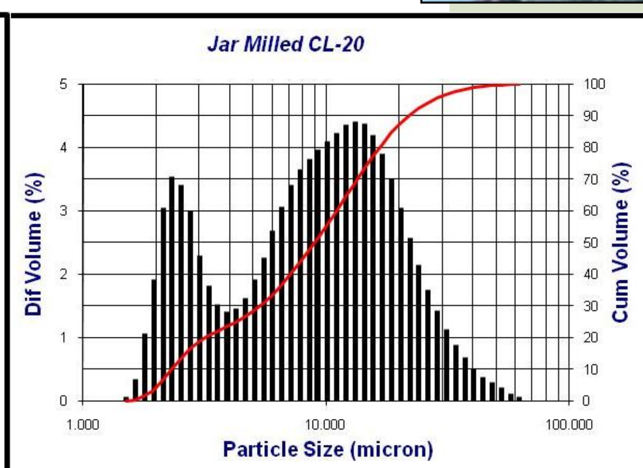


Ground CL-20

- **Two grades of ground CL-20 are produced on a routine basis**
  - **FEM ground**
    - Nominally 2 micron
  - **Jar milled**
    - Nominally 11 micron
- FEM facility has been recently refurbished and upgraded
  - Improvements to grounding, smooth walls, collector, etc.





Formulation and Application Advances

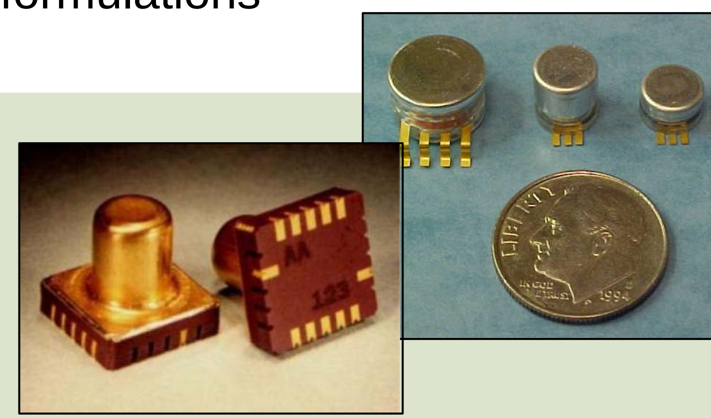
ATK

- **New formulations and applications involving CL-20 have emerged as a result of:**
  - Better CL-20 crystal quality
  - Availability of multiple and useful sizes of CL-20
  - Greater understanding of CL-20 binder filler interaction
- Notable examples include:
  - High solids cast cure explosives with good IM properties
  - Initiator systems that utilize CL-20 based formulations

Main body & closure

Copper liner

Mild Cook-off



Improved Initiators

Approach for CL-20 Cost Reduction

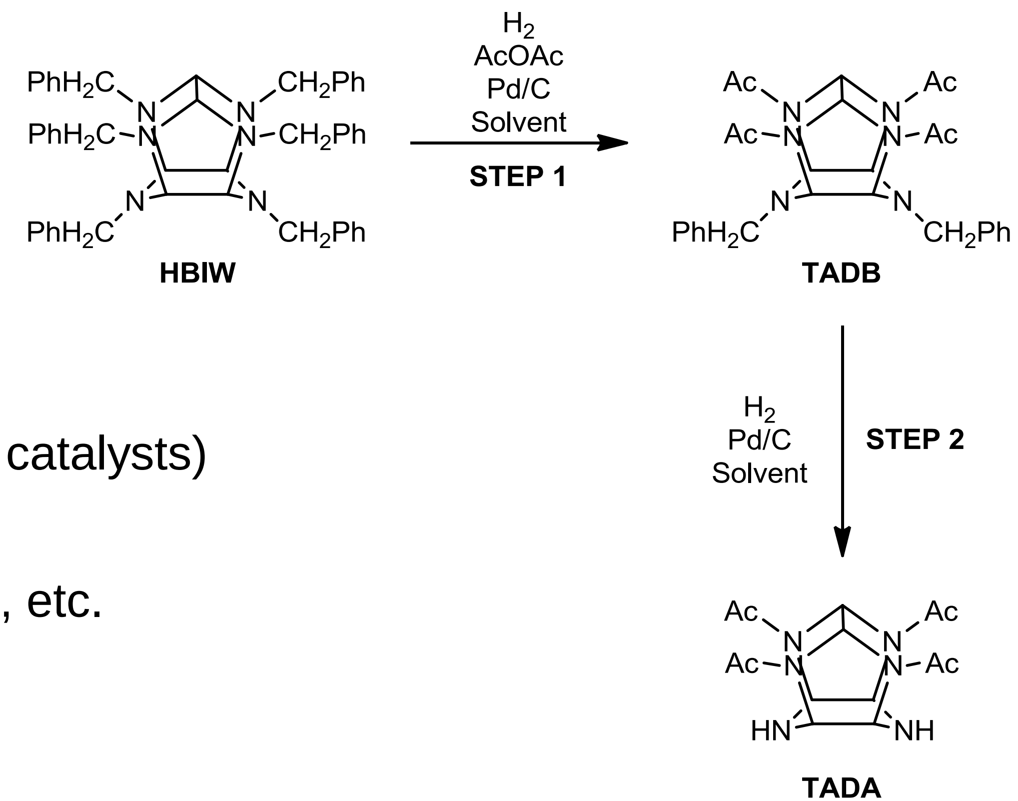
- ATK has invested substantial internal funds during the past three years to decrease CL-20 cost and improve the CL-20 manufacturing process
- The approach to cost reduction has focused in two key areas which have been attacked vigorously:
  - Process efficiency improvements
  - Precursor (TADA) cost reduction
    - TADA accounts for over 50% of the cost of CL-20
- Early studies were focused on process improvements
- More recent efforts have concentrated on developing a domestic TADA manufacturer and optimizing the TADA synthesis process
- Continued long-term objectives are being pursued

Precursor Improvement Initiative

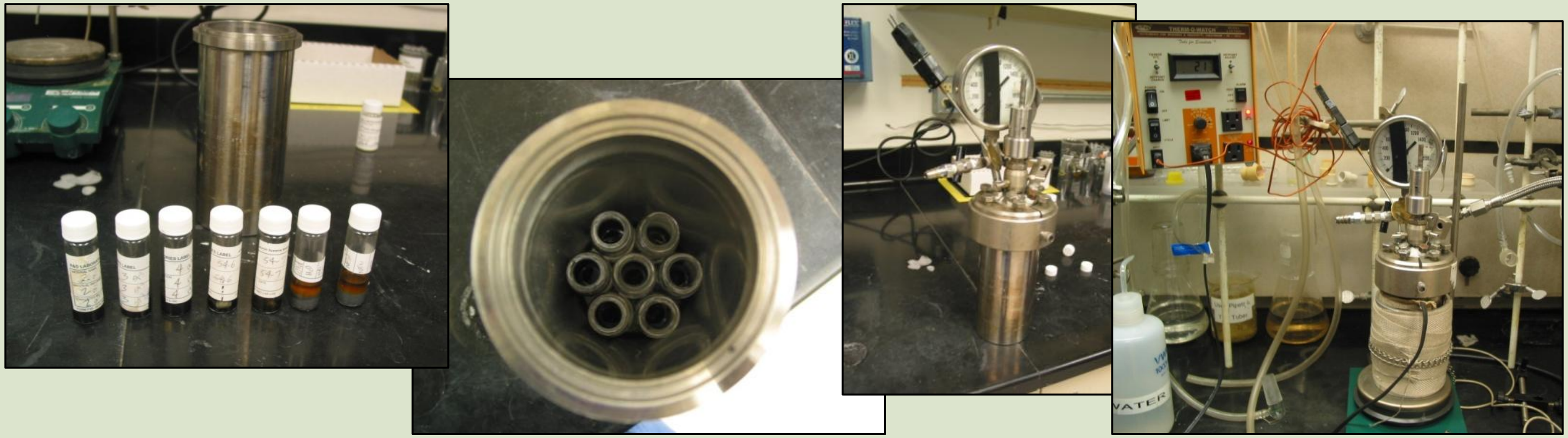
- Experiments were performed to understand reaction conditions and improve yield

  <u>Synthesis Step 1</u>
  - Reaction rates
  - Temperature
  - Pressure
  - Time
  - Wet catalyst vs. dry
  - Best catalyst (evaluated 18 catalysts)

  <u>Synthesis Step 2</u>
  - Filtration, H_2O, solvent rinse, etc.
  - Time
  - Temperature
  - Catalyst - one addition vs. two additions
  - Reuse of catalyst



Experimental Setup and Results



Seven experiments are run at once under identical pressure and temperature conditions

- **Work resulted in drastically increased yield and reduced catalyst costs**
  - Early improvements focused on Step 1
  - Later efforts refined Step 2
  - Substantial improvements were made in both steps
  - Efforts are continuing on work-up of the TADA

Summary

- **CL-20 provides enabling capability for several key areas**
- **Processing improvements, such as FEM grinding, make CL-20 a more viable ingredient for new state-of-the-art energetic formulations**
- **Synthesis of CL-20 continues to be refined and improved**
  - In recent years activities have focused in two areas:
    - Development of a domestic source for TADA
    - Cost reduction