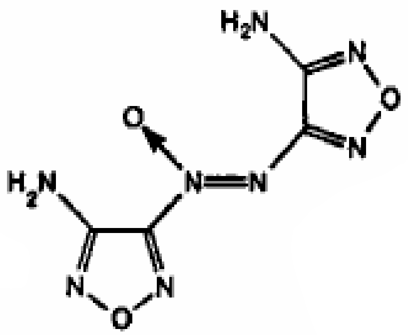
Synthesis Improvements of 3,3' Diaminoazoxy furazan (DAAF)

Elizabeth Francois, David Chavez,
Mary Sandstrom



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DAAF Outline

- The GOOD, BAD and UGLY of DAAF
- Original Synthesis Method
  - Features
  - Factors
  - Challenges
- DAAF recrystallization
  - DMSO
  - Acetonitrile
- Novel Synthesis Path
  - Features
  - Future work

DAAF: The GOOD

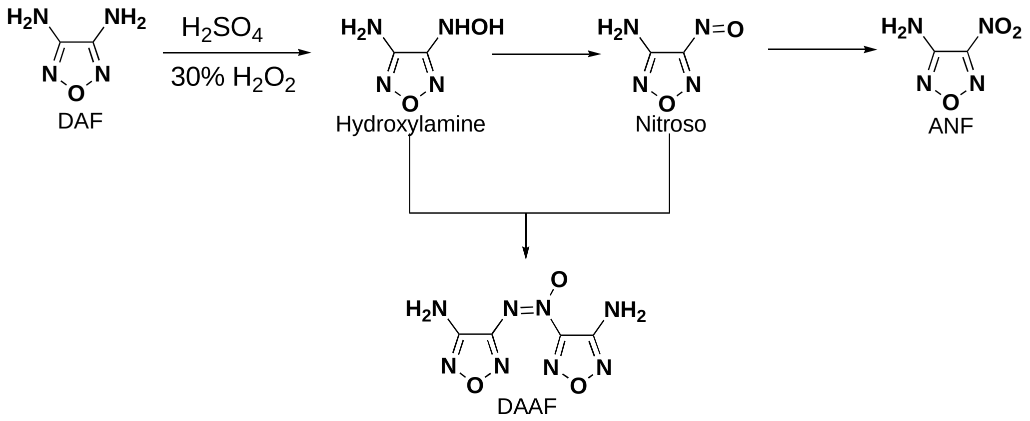
- Detonation Velocity 7.93 km/s

  (1/2" pellets pressed to ρ= 1.685 g/cm^3 neat)
- CJ pressure = 306 kbar
- Critical diameter < 3mm
- Drop height > 320cm, Friction >36 Kg
- Heat of Formation (ΔH_f) +106 kcal/mole

DAAF: The Bad and the Ugly

- Synthesis intermediates and side products are energetic and have low temperature onsets.
- 2-Step process involving synthesis and recrystallization with DMSO to achieve pure product.
- Significant amounts of highly acidic hazardous waste: 5Kg DAAF yields ~40+ gallons of waste.
- Recrystallized DAAF does not press into pellets easily. Pellets are fragile and have low density, affecting the performance.

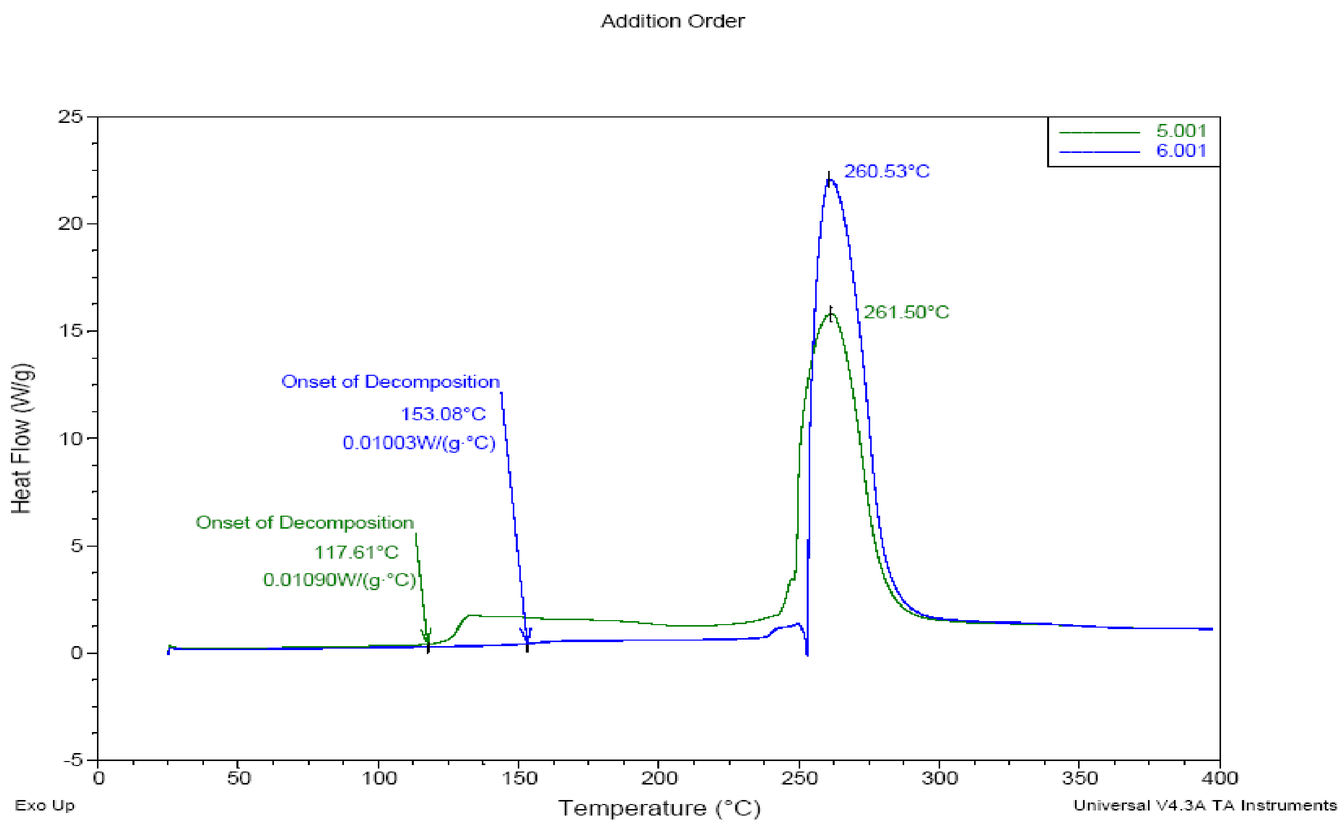
Synthesis of DAAF



Synthesis scheme

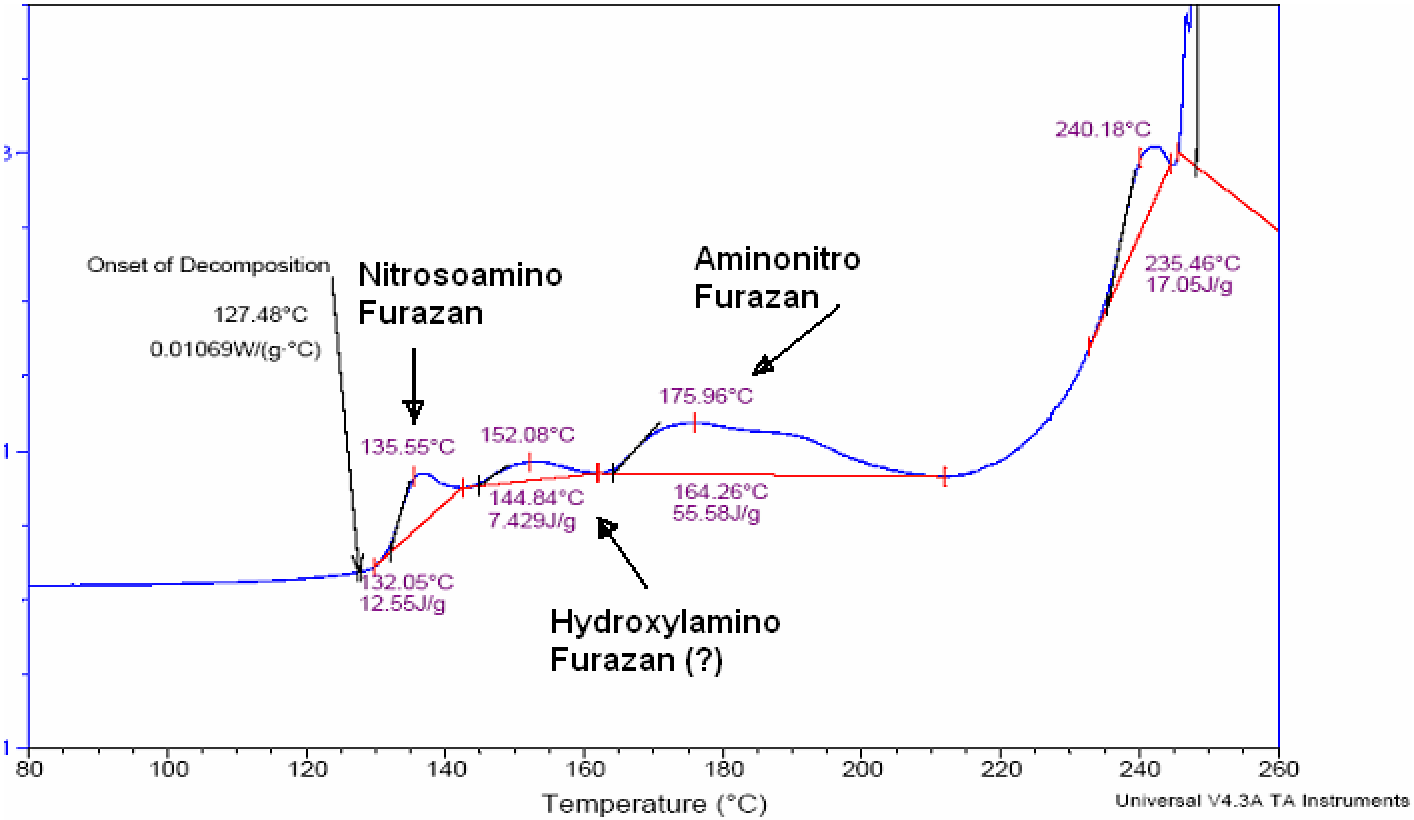
- Important Parameters
  - Order
  - Temperature

Addition Order- DAAF DSC



DSC courtesy of Mary Sandstrom

Synthesis of DAAF- Impurities

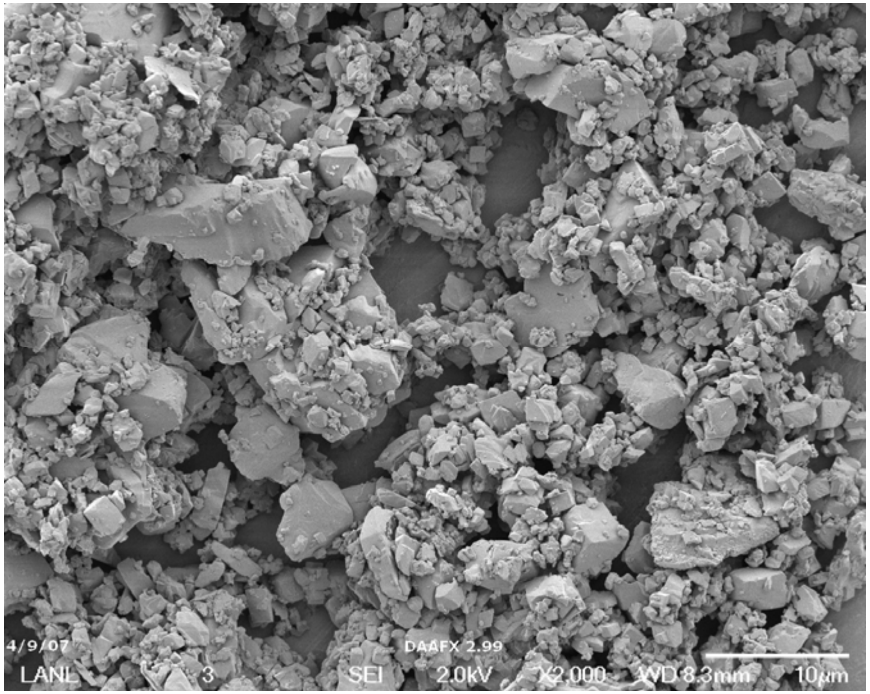


DSC courtesy of Mary Sandstrom

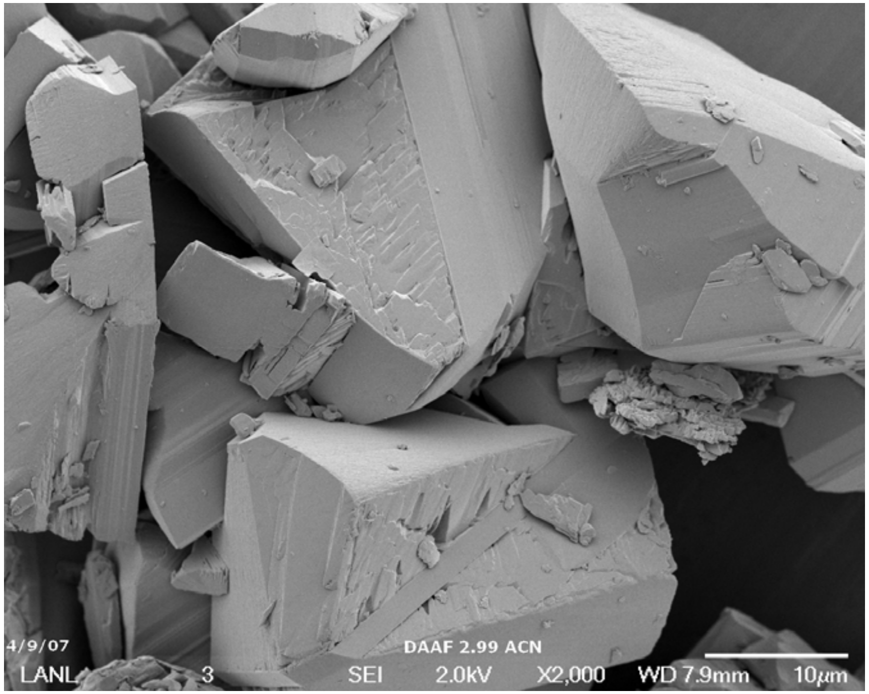
Recrystallization vs. Precipitation

- DAAF impurities can be easily removed with crash precipitation in DMSO and water. Overall yield is 80%
- Crash precipitation in DMSO creates small (<10μm) particles
  - Fragile neat pressed pellets
  - Poor pellet density
    - Neat: ρ=1.6 g/cm^3, TMD = 1.747
    - With Binder 3% KEL-F ρ=1.64 g/cm^3
- DMSO DAAF can be recrystallized in acetonitrile
  - Larger particle size, allowing:
    - Higher density of pressed pellets (1.69 g/cm^3)
    - Better performance due to higher density
    - Pressed pellets are less fragile
    - Becomes 3 step process with 50% yield

Particle size examples DMSO & ACN



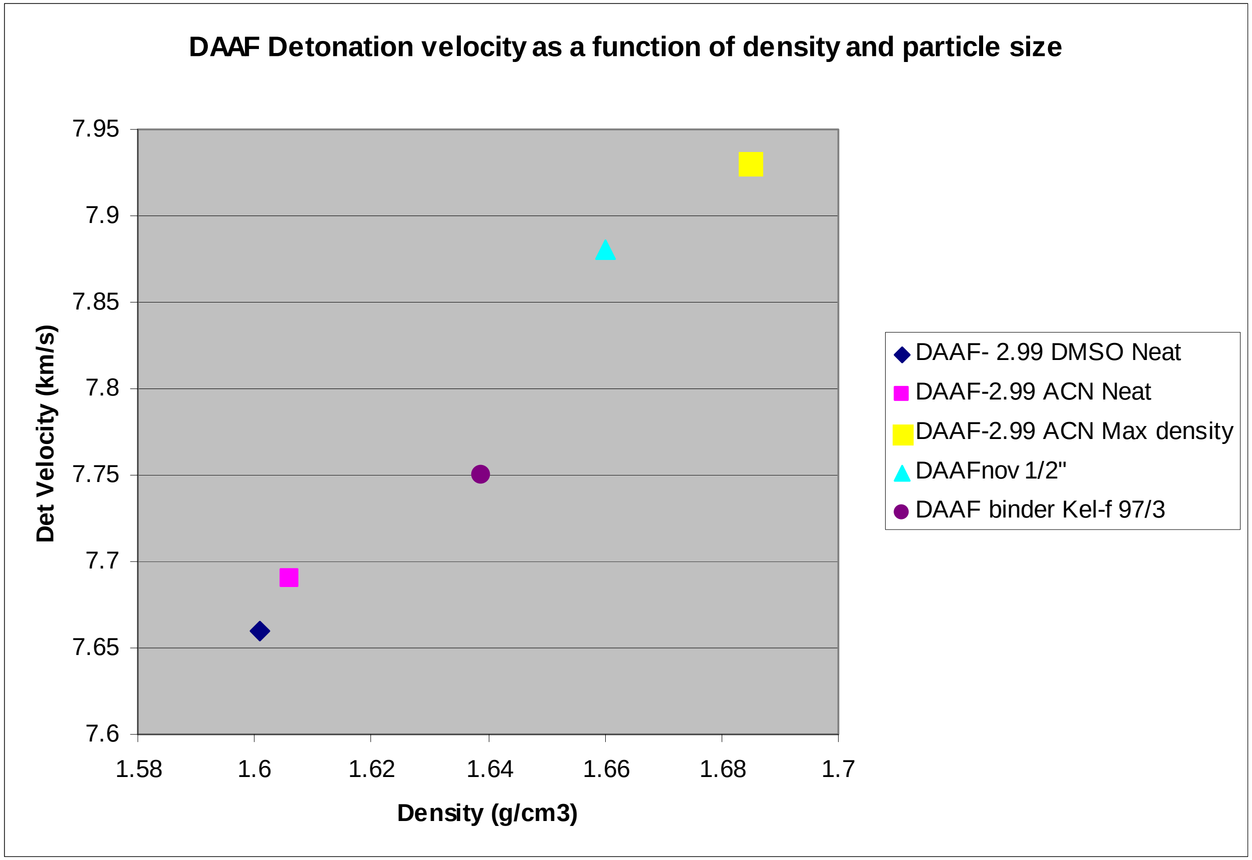
DMSO recrystallized DAAF; Particle size <10μm



Acetonitrile recrystallized DAAF; particle size ~20μm.

SEM images courtesy of Ed Roemer

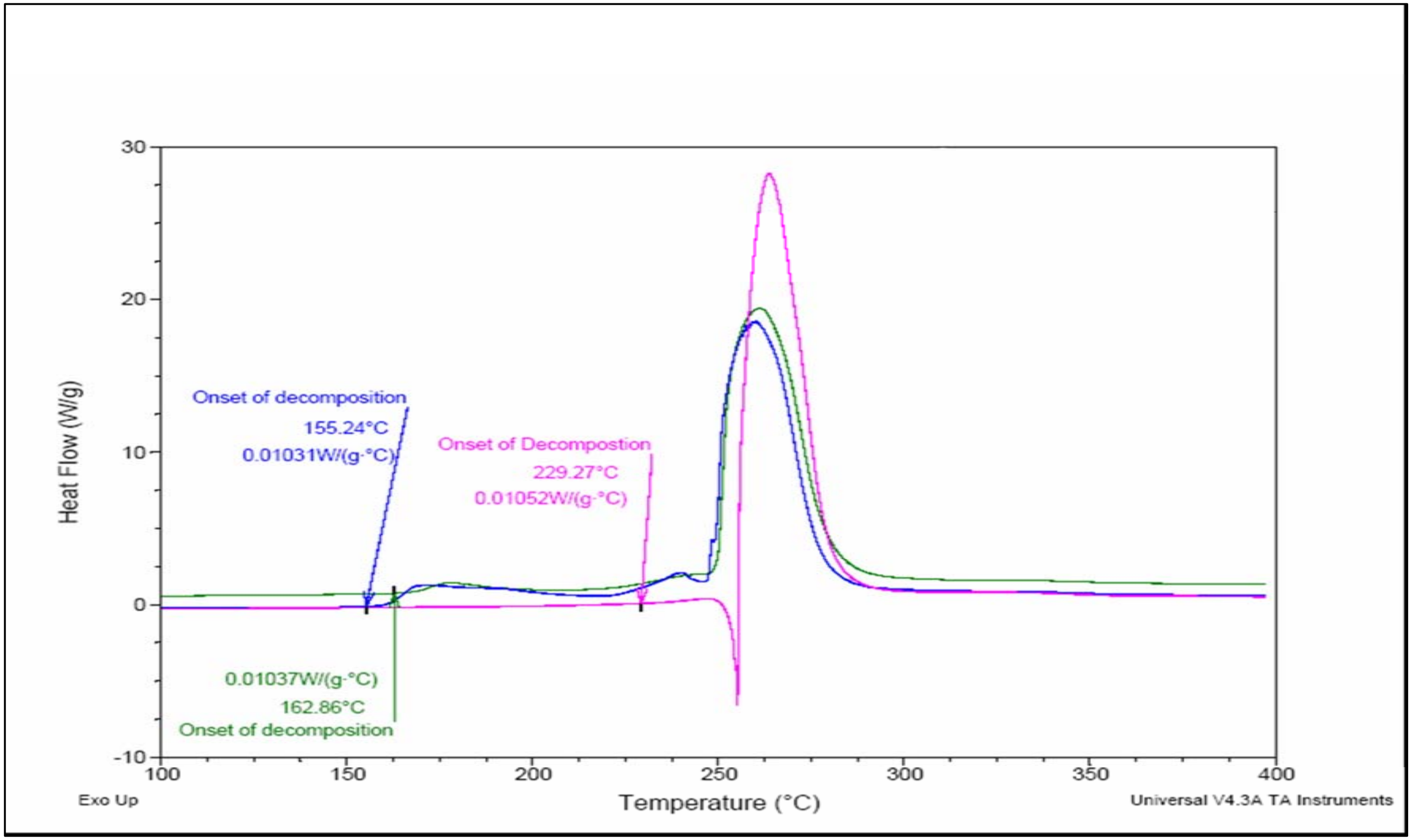
Performance comparison



Novel DAAF Synthesis

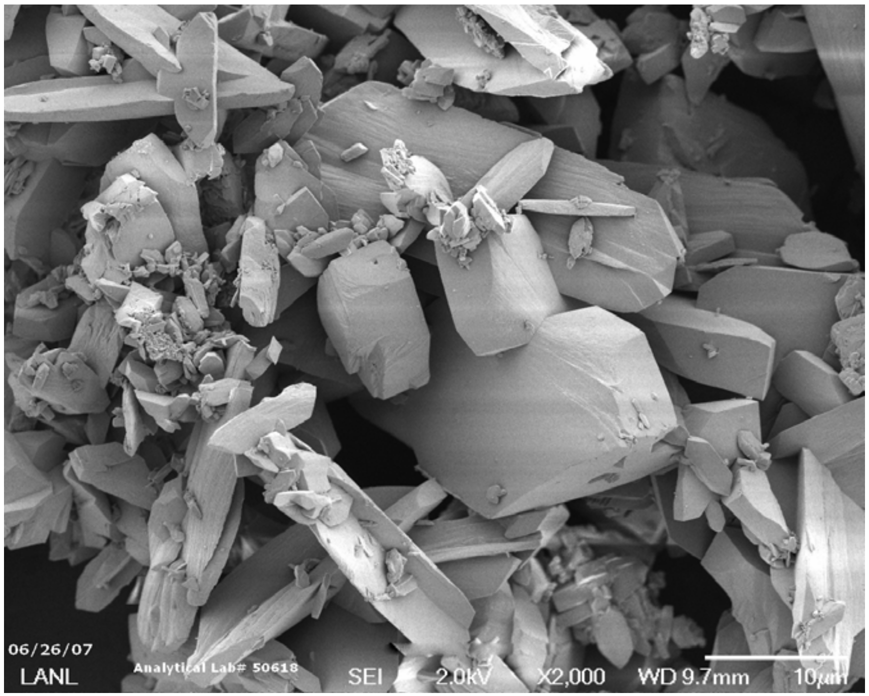
- Due to waste issues, new DAAF synthesis was tried.
- DAAF synthesized in water with commercially available oxidizer rather than creating one with H_2O_2 and H_2SO_4.
- Amount of oxidizer is controlled with this method.
- Did not initially make pure DAAF
  - The same impurities found in the original synthesis method were present.
- By varying the pH of the solution, an improvement in purity is observed.

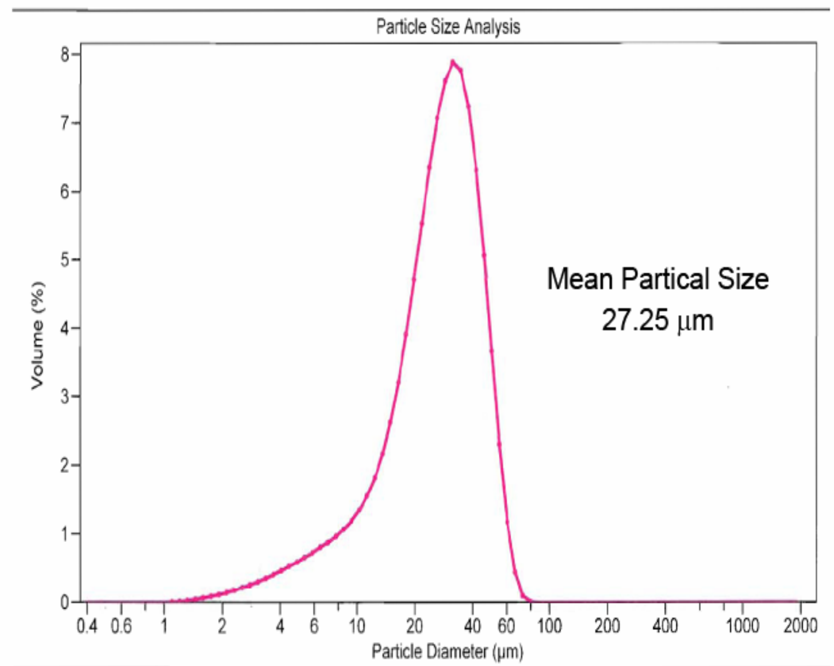
pH comparison



DSC courtesy of Mary Sandstrom

Novel Synthesis Particle Size Improvements





SEM courtesy of Ed Roemer

Analysis courtesy of Kien-Yin Lee

Novel DAAF Synthesis- Comparison

Original DAAF synthesis

- Impurities which lower the Onset of Decomposition
- 2-step process to remove impurities
- Small particle size of final product (<10μm)
- Low pressed density: 91% TMD, poor performance
- Lengthy synthesis process:

  100 hours
- Large quantity of hazardous waste

Novel DAAF synthesis

- No impurities, high Onset of Decomposition
- 1-Step process
- Larger particle size (~28μm)
- High pressed density 97% TMD, good performance
- Fast synthesis: 4 Hours
- Non-hazardous waste: “ salty water”

What's Next?

- Scale up process to multi kilogram level, maintaining desirable characteristics.
  - Currently at 150g level with favorable results
- Critical Diameter and Diameter Effect tests in progress.
- Mechanical Strength tests in progress
- Mini-Gap tests to determine particle size effects on shock sensitivity planned

Acknowledgements

- Blaine Asay and Bert Harry
- Dave Chavez- Chemistry
- Mary Sandstrom- DSC, Design of Experiments
- Ed Roemer- SEM imaging
- V. Eric Sanders and Joe Lloyd- Performance tests and shot data analysis
- Kien-Yin Lee- Particle size analysis
- Bryce Tappan and Dave Oschwald- Critical diameter and diameter effect tests
- Darla Thompson and Racci DeLucca- Mechanical testing
- Karen Mehlin- HPLC analysis