Novel Recrystallization for Small Particle Size HBDNQ

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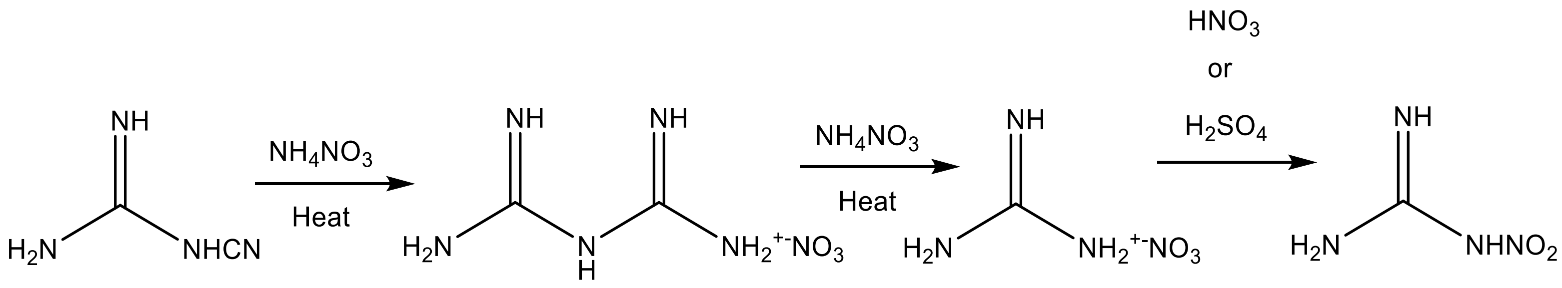
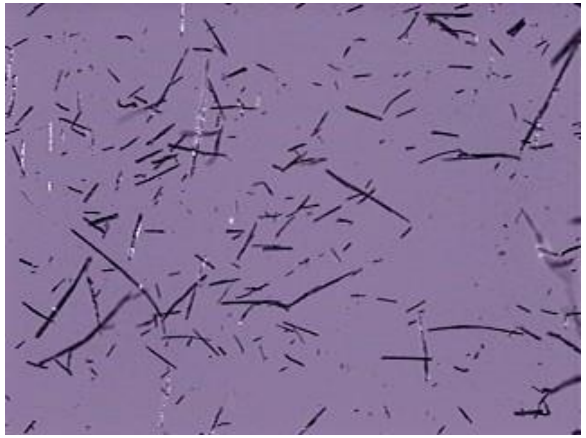
NQ (Nitroguanidine)

- NQ has long history of use in propellants (ca. 1930s)
  - Simple synthesis from 2-Cyanoguanidine
  - Highly insensitive to unplanned stimuli
- Limited use as explosive filler due to low bulk density
  - Bulk density (< 0.1 g/cc)
    - High aspect ratio needle shapes particles
- Development of High Bulk Density NQ (HBDNQ) allowed for incorporation into insensitive munition formulation IMX-101

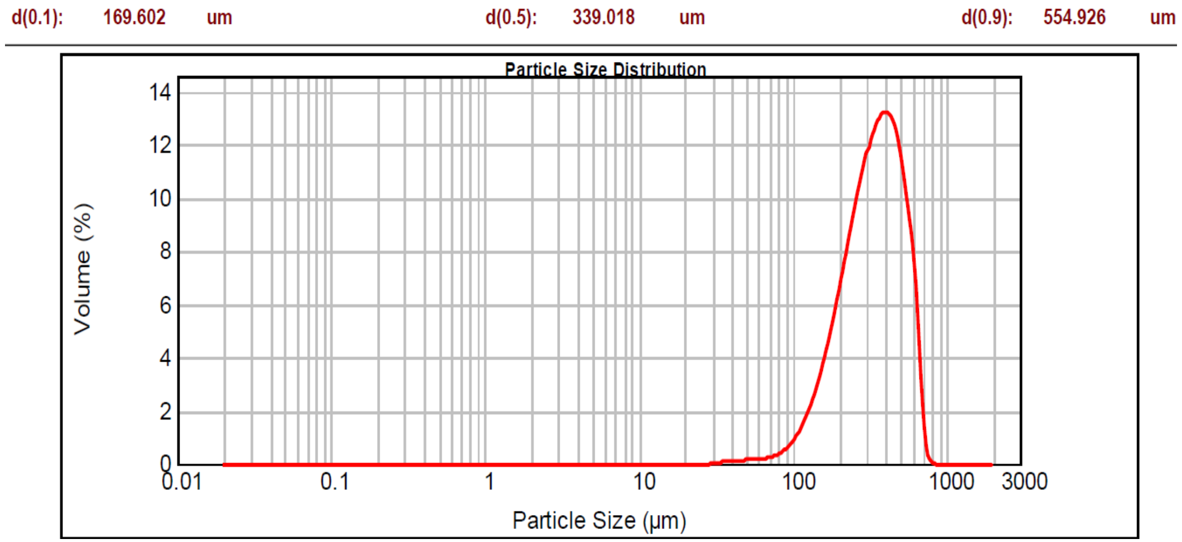


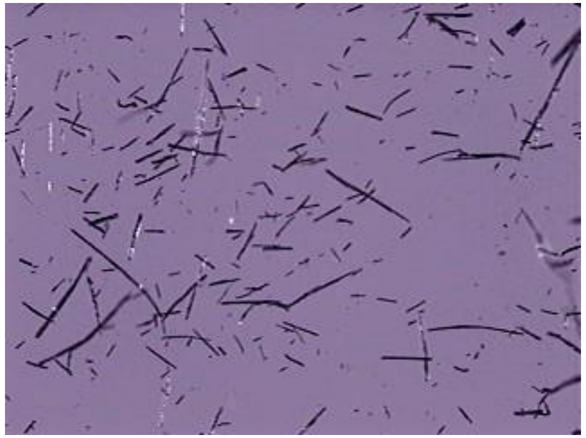


Low Bulk Density NQ

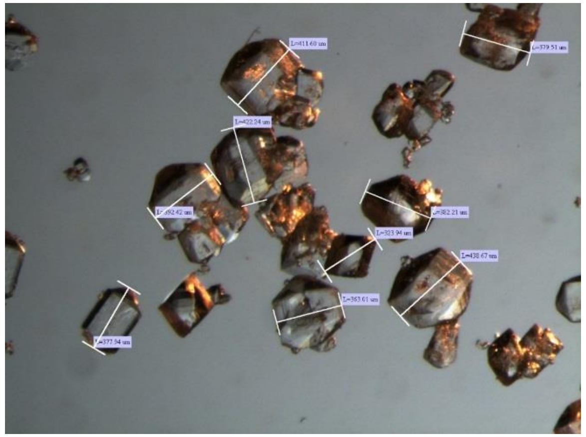
High Bulk Density Nitroguanidine (HBDNQ)

- High Bulk Density NQ (HBDNQ) developed at HSAAP in 2008
  - Dilute acid mediated recrystallization
- Typical particle size
  - d_{10} ~ 100 μm
  - $d_{50} \leq$ 400 μm
  - $d_{90} \leq$ 750 μm
- Acidity: ≤ 0.06%
- Purity: ≥ 98%
- Density: ≥ 0.8 g/cc
- Available at full production scale from HSAAP





Low Bulk Density NQ



High Bulk Density NQ

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New NQ Recrystallization Development

- Significant interest exists within DoD for additional classes of NQ for both propellants and explosive formulations
- A novel crystallization process would complement the current recrystallized HBDNQ at HSAAP
  - Simple and scalable procedure
- Decreased particle size could yield a less sensitive product
- BAE has developed a process for a new class of NQ meeting that requirement

	HBDNQ	1182-94	1182-96	1182-115	1182-126	1182-128
d(0.5) (µm)	300 - 400	50.51	31.71	32.22	89.64	82.10

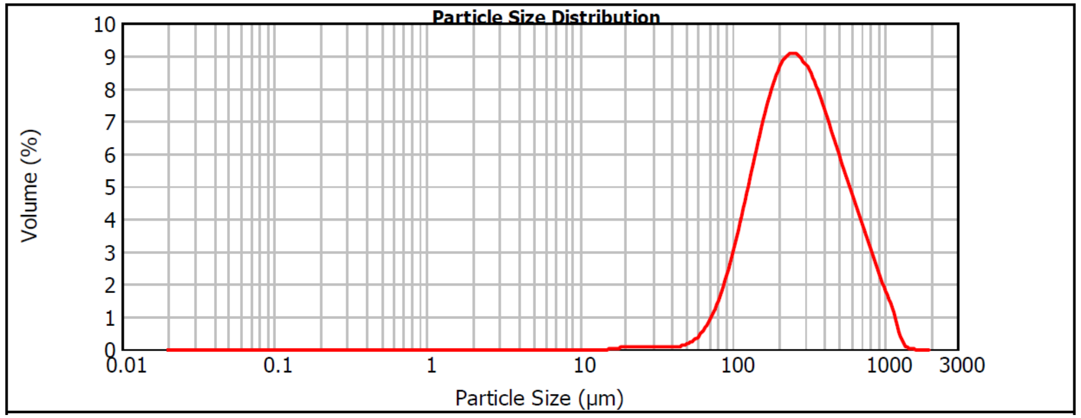
HSAAP HBDNQ (HBDNQ column) — Lab Batches of new NQ (1182-94 through 1182-128)

New NQ Recrystallization Development

- Both methods produce a monomodal distribution
- New procedure shows significant decrease in average particle size
  - Narrower distribution compared to standard high bulk density NQ

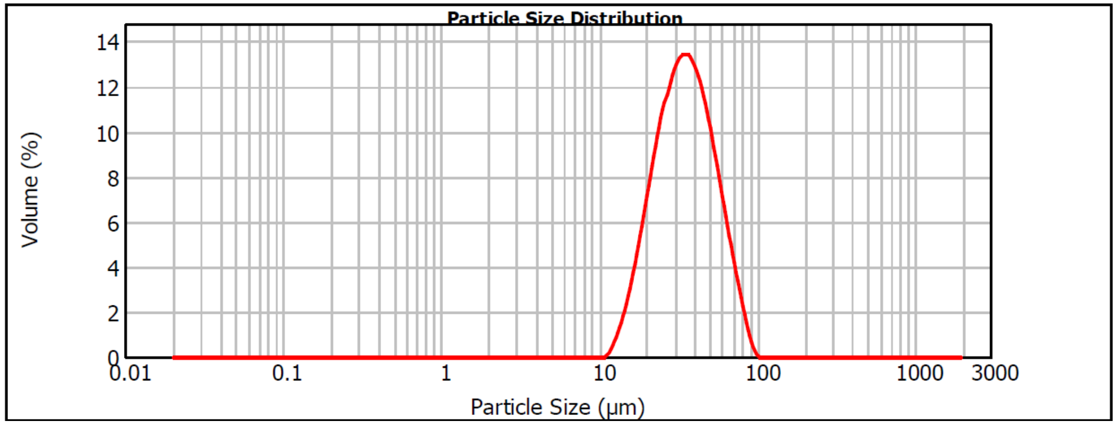
HSAAP HBDNQ

d(0.1): 121.175 um d(0.5): 272.519 um d(0.9): 664.332 um

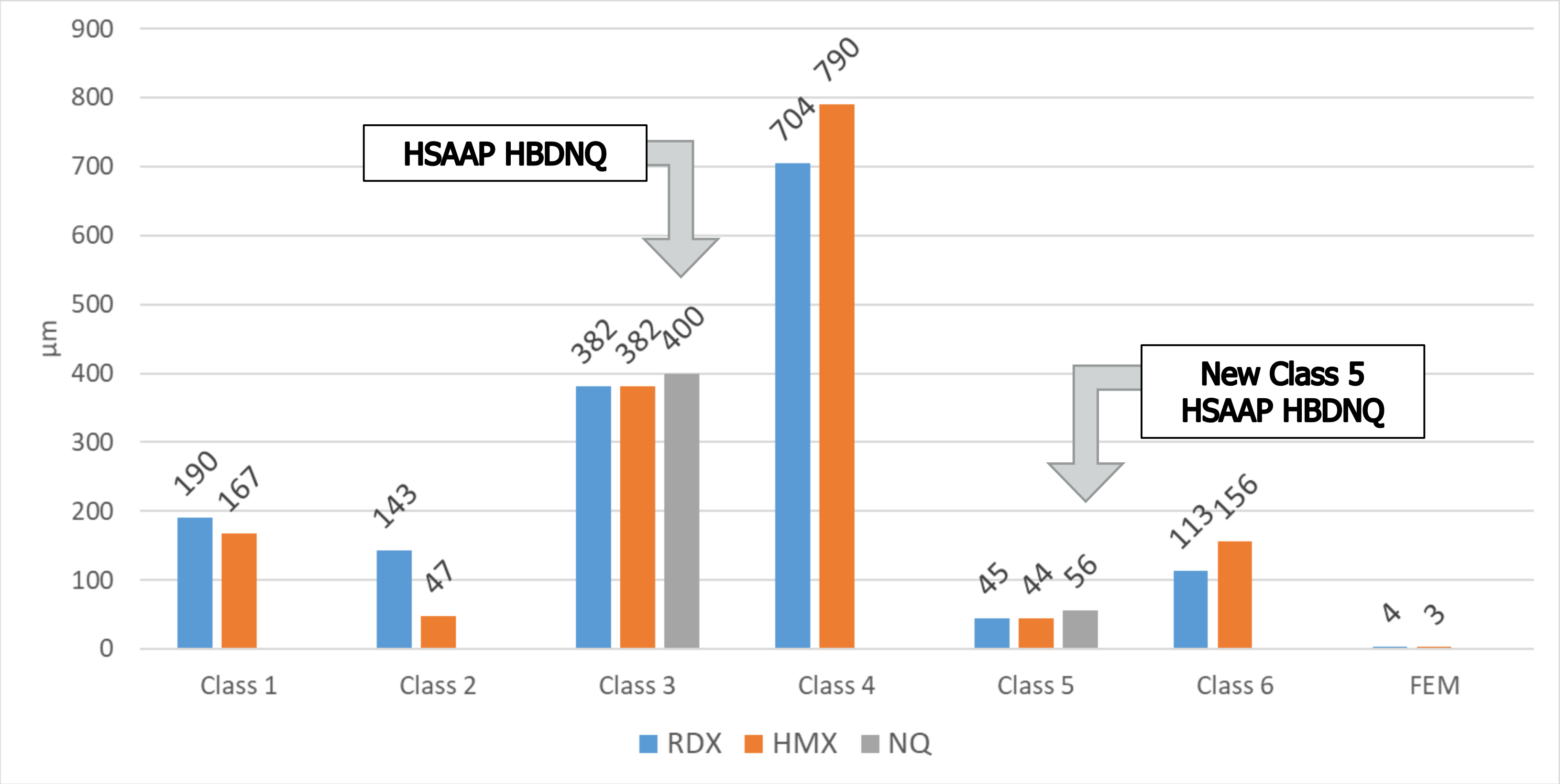


New ~ 50 μm High Bulk Density NQ

d(0.1): 19.888 um d(0.5): 34.728 um d(0.9): 60.350 um



New NQ Recrystallization Development



- Currently produced HBDNQ similar to Class 3 RDX / HMX
- New ~ 50 µm NQ most similar to Class 5 RDX / HMX

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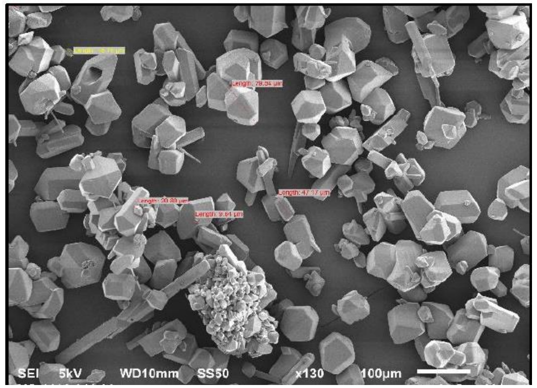
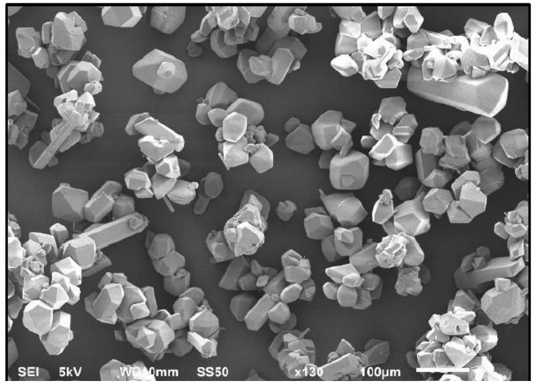
Temperature Control is Key

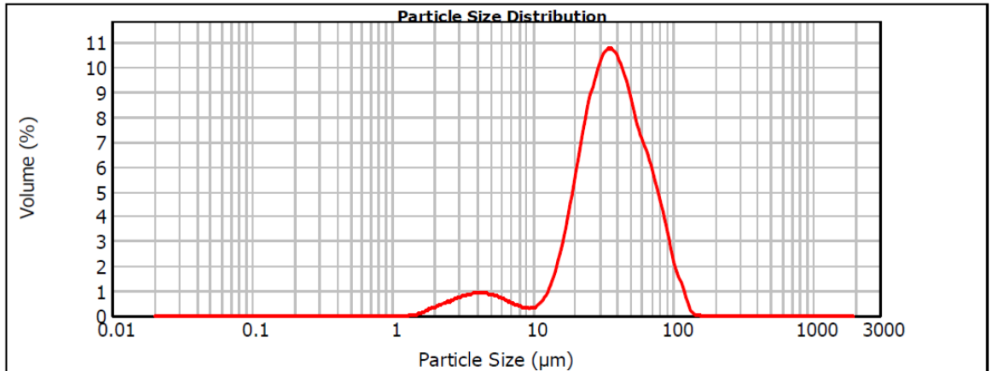
- Initial trials used standard ice bath to chill reaction
  - Increased particle size
  - Bi-modal distribution

- Insufficient surface area / interface between reaction vessel walls and cooling bath
  - Require extreme cooling control to afford sufficient cooling rate to achieve particle size control

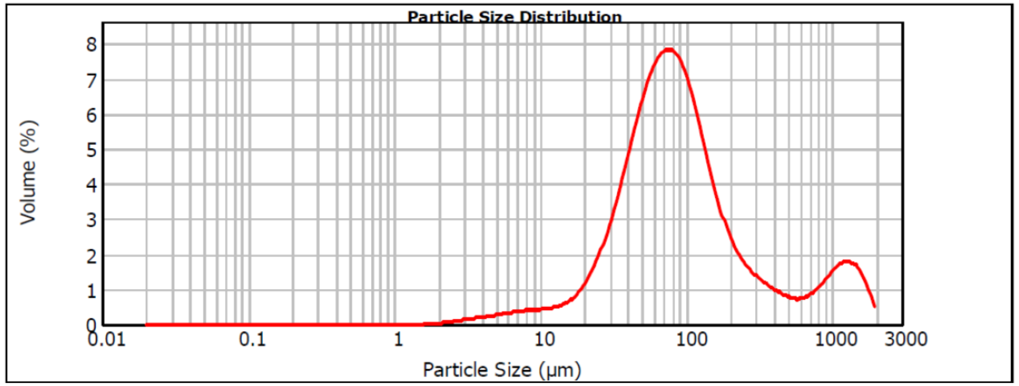




d(0.1): 15.672 um d(0.5): 35.989 um d(0.9): 74.699 um



d(0.1): 29.439 um d(0.5): 82.414 um d(0.9): 599.857 um

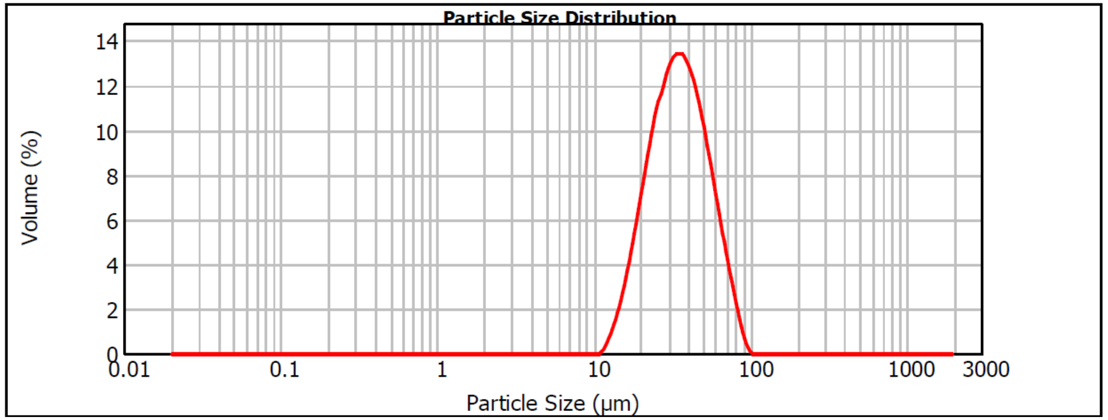


Temperature Control is Key

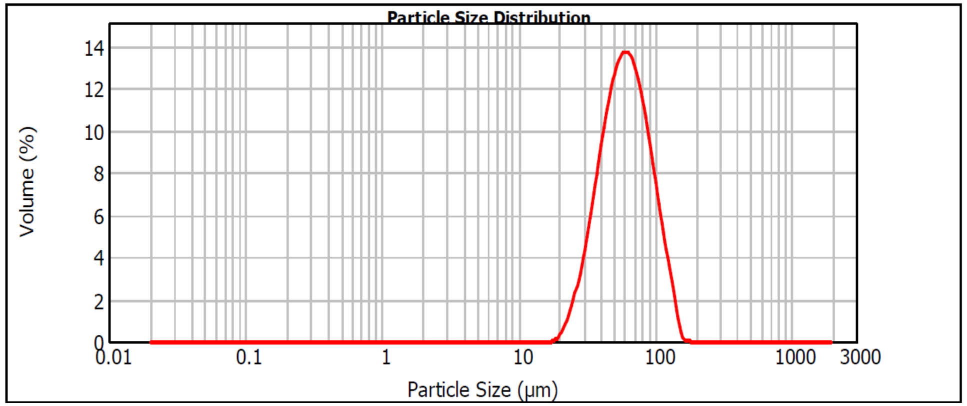
- New procedure requires high degree of temperature control
- At small laboratory scale (up to 1 L scale) this is easily achieved
- When scaling up, ability to remove heat from solution in key
  - Insufficient cooling results in increased particle size and broader distribution

Small scale / good cooling

d(0.1): 19.888 um d(0.5): 34.728 um d(0.9): 60.350 um

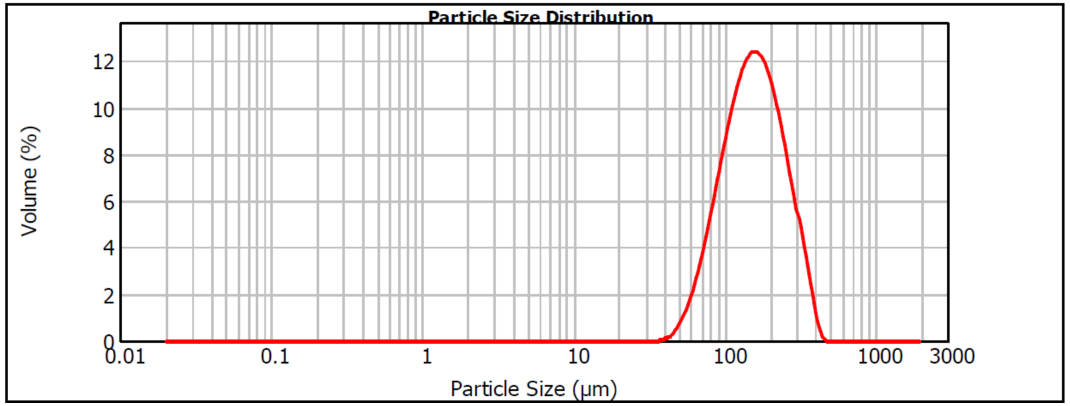


d(0.1): 34.782 um d(0.5): 60.374 um d(0.9): 103.222 um



Large scale / good cooling

d(0.1): 83.387 um d(0.5): 155.367 um d(0.9): 277.484 um



Large scale / insufficient cooling

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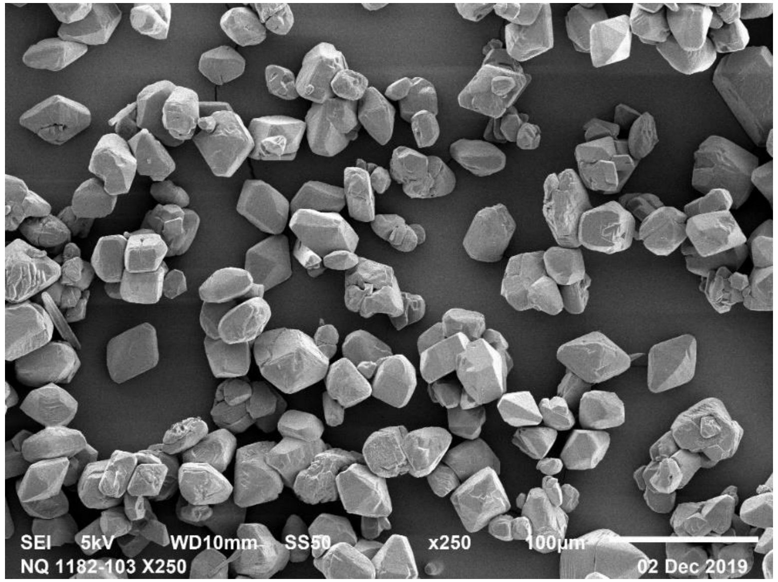
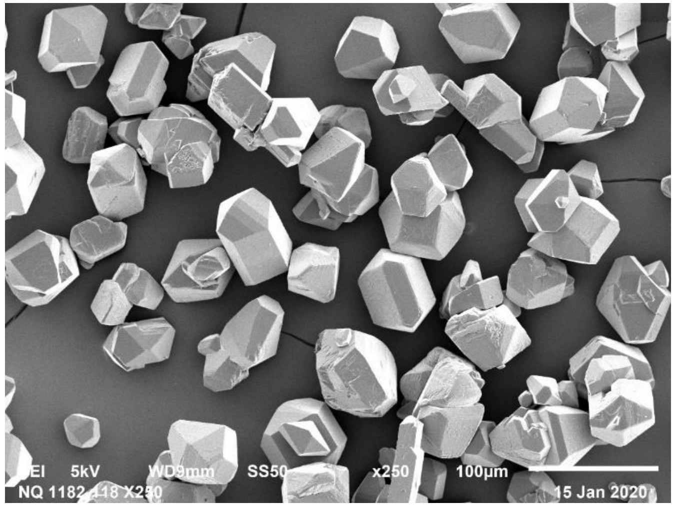
Small scale / good cooling

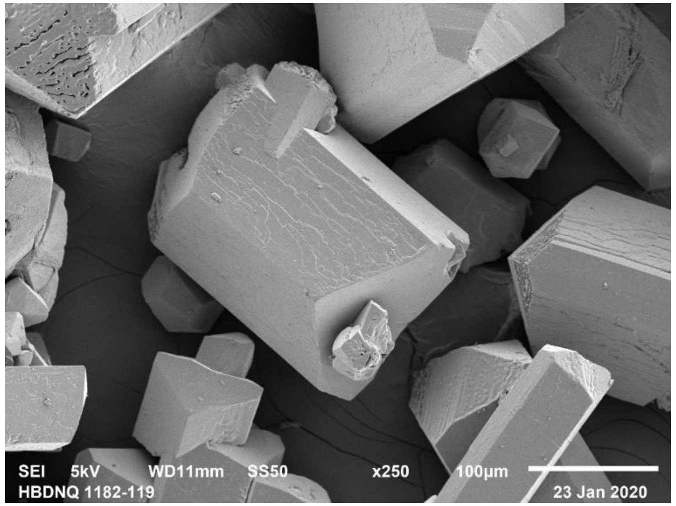


Particle Shape Analysis

- The crystal shape of the new class of HBDNQ is cubic / bipyramidal
- The smaller scale material is comprised of more uniform shaped particles
- Scaled up material is slightly elongated as compared to smaller scale samples
    - If adequate temperature control is not maintained during entire procedure, resulting particles begin to revert to column shaped elongated particles



Large scale / good cooling



Large scale / insufficient cooling

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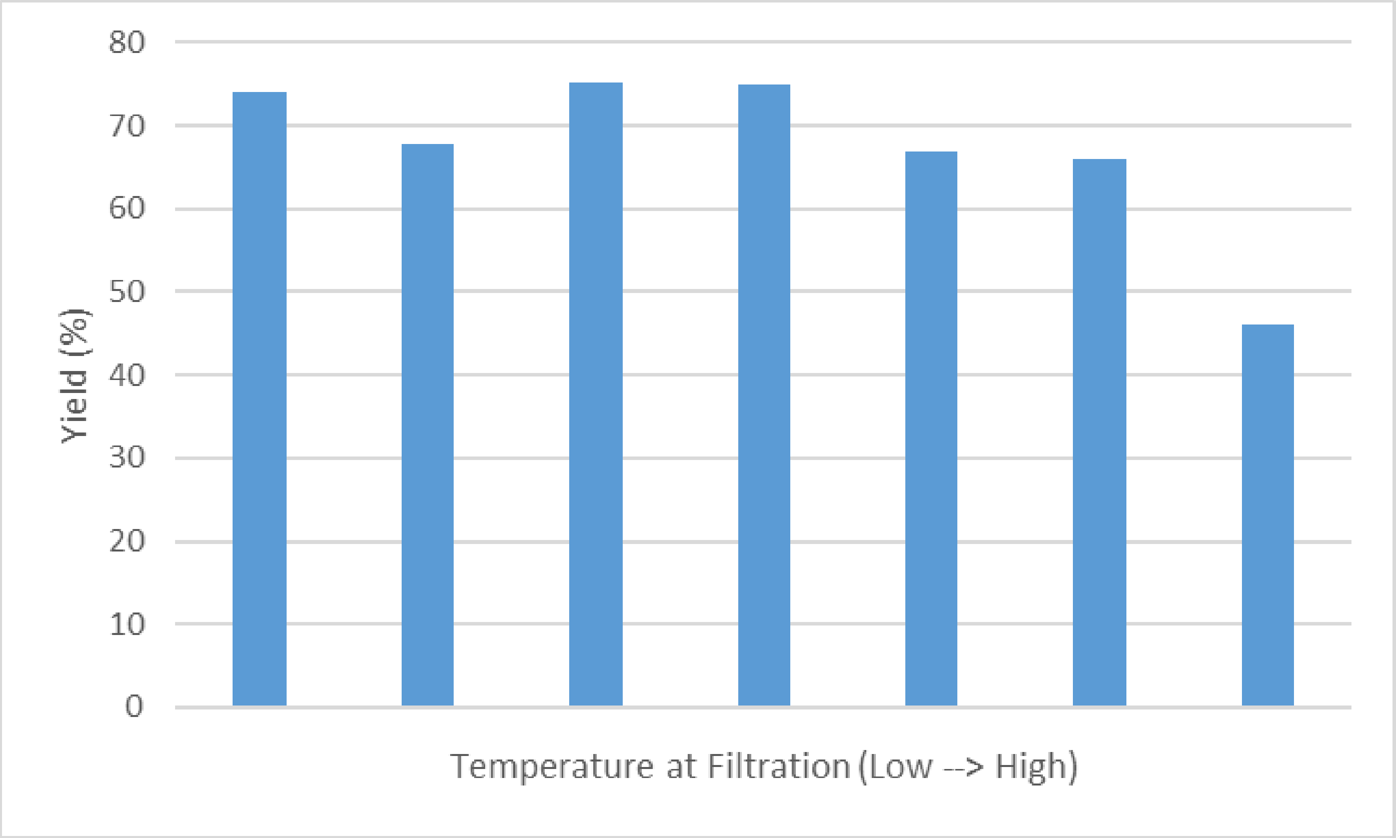
Recovery of Final Product

- Total recovery depended on the final temperature of recrystallization solution
- 60 – 75% recovery of NQ when sample is cooled extensively
- Drops to ~ 45% when sample is cooled insufficiently
  - > 30 % drop in recovery



Path Forward

- Transition to pilot plant scale procedure
  - HSAAP D-10 Pilot plant
  - > 100 kg scale
- Determine optimal processing parameters for production scale equipment
  - Additional baffling inside reactor should allow for further reduction in particle size, and lower requirement for highly controlled cooling
- Test new Class 5 NQ in IMX-101 and similar formulation to see effect on viscosity and pourability
- Evaluate Class 5 NQ in:
  - Propellants
  - Pressable and cast-cure formulations

Conclusions

- A new class of NQ has been developed by BAE Systems at HSAAP
  - Nominal particle size : ~ 50 µm
  - New designation: Class 5
- New material should allow for higher loading of NQ in propellants, and as an explosive filler with a diminished effect on viscosity of formulations

Acknowledgments

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