Synthesis of DNMT: A New Energetic Melt-Pour Ingredient

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Background

- TNT has been the backbone of melt-pour explosives for most of the 20th century.
  - Not IM enough for today's standards
- DNAN is:
  - quickly becoming the favored replacement for TNT due to its superior IM properties.
  - not very energetic and performance of DNAN-based explosives suffer as a result.
- Future melt-pour energetics need to have best of both worlds:
  - Superior IM properties
  - Good explosive performance

Program Objectives

- Identify and Prepare New Melt Pour Ingredients with Inherent Comp B Performance
- Evaluate Candidates Using Small Scale Safety and Performance Testing
- Evaluate Scalability of Synthesis
- Evaluate Formulation Characteristics

Selection Criteria:

- Melting Point in Desired Range (80-120C)
- Sufficiently High Predicted Density
- Perceived Ease of Preparation

Initial Candidate Compounds

DNP
m.p. ~ 85°C
calc. dens. ~ 1.87 g/cm³
energy out ~ 1961 cal/cm³

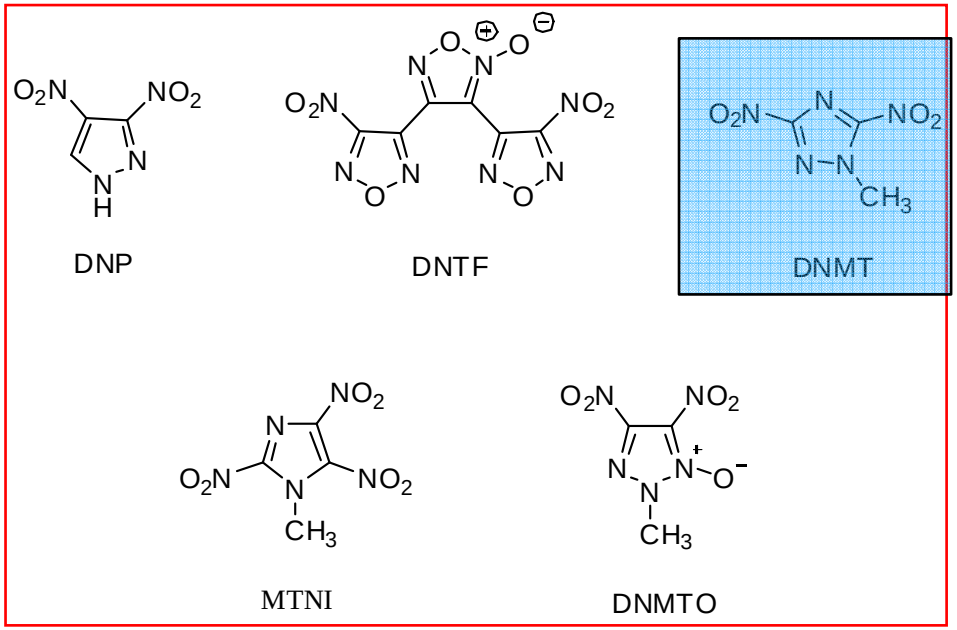
3 steps

MTNI
m.p. ~ 82°C
calc. dens. ~
energy out ~

6 steps

DNMTO
m.p. ~ 130°C
calc. dens. ~
energy out ~

2 steps



DNTF
m.p. ~ 108 °C
calc. dens. ~ 1.95 g/cm³
energy out ~ 2517 cal/cm³

4 steps

DNMT
m.p. ~ 95 °C
calc. dens. ~ 2.10 g/cm³
energy out ~ 1739 cal/cm³

2 steps

Comp B energy out ~ 1837 cal/cm³
LX-14 energy out ~ 2186 cal/cm³

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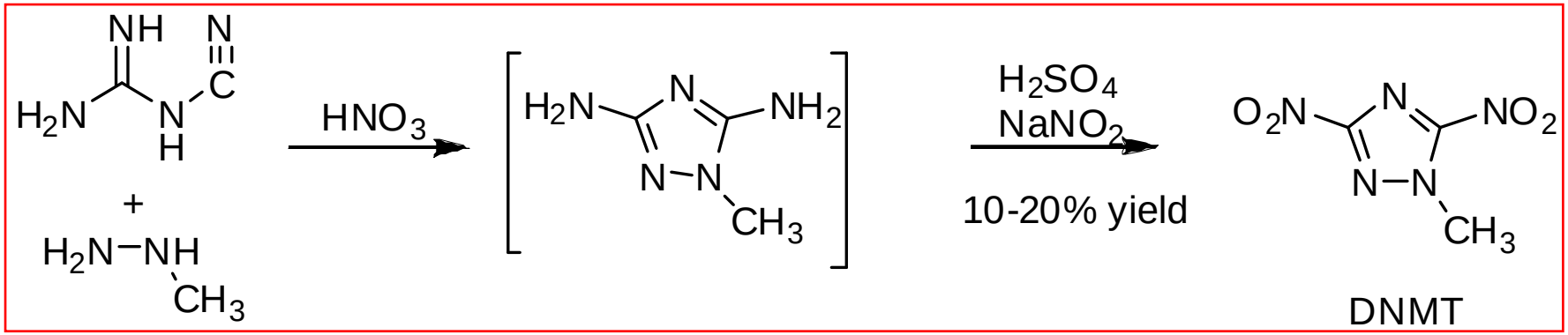
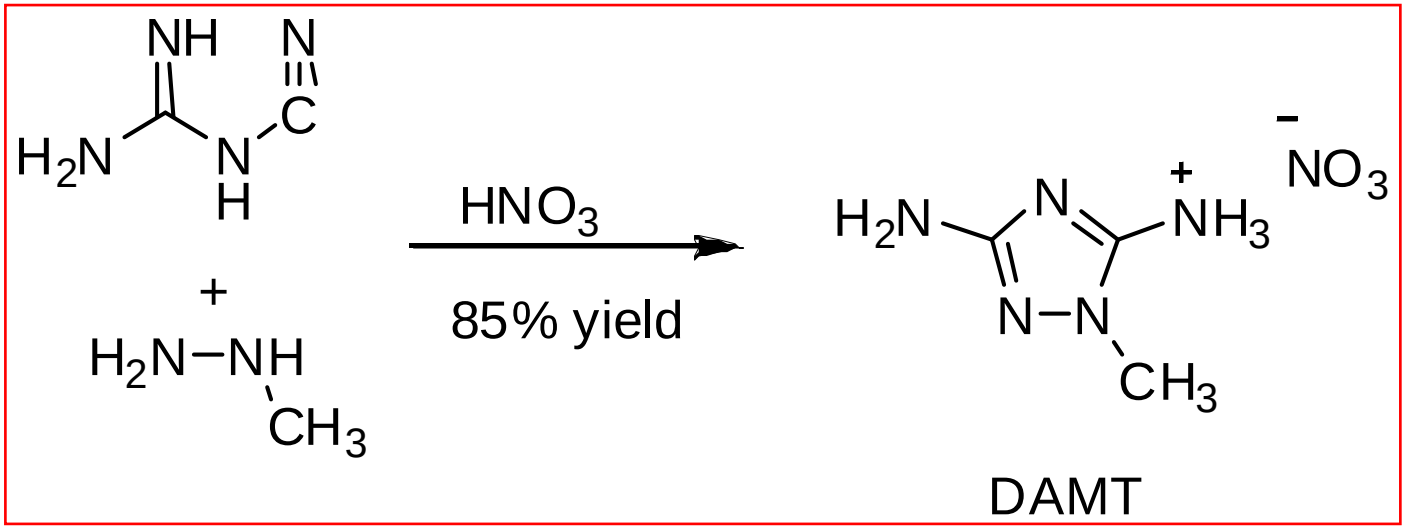
DNMT



- Original procedure:
  - Pseudo one pot reaction
  - Developed by Prof. Katritzky, et al.
  - DNMT soluble in acidic water
  - Extraction required
  - Synthesis/purification not optimized
  - 25 grams produced by this method

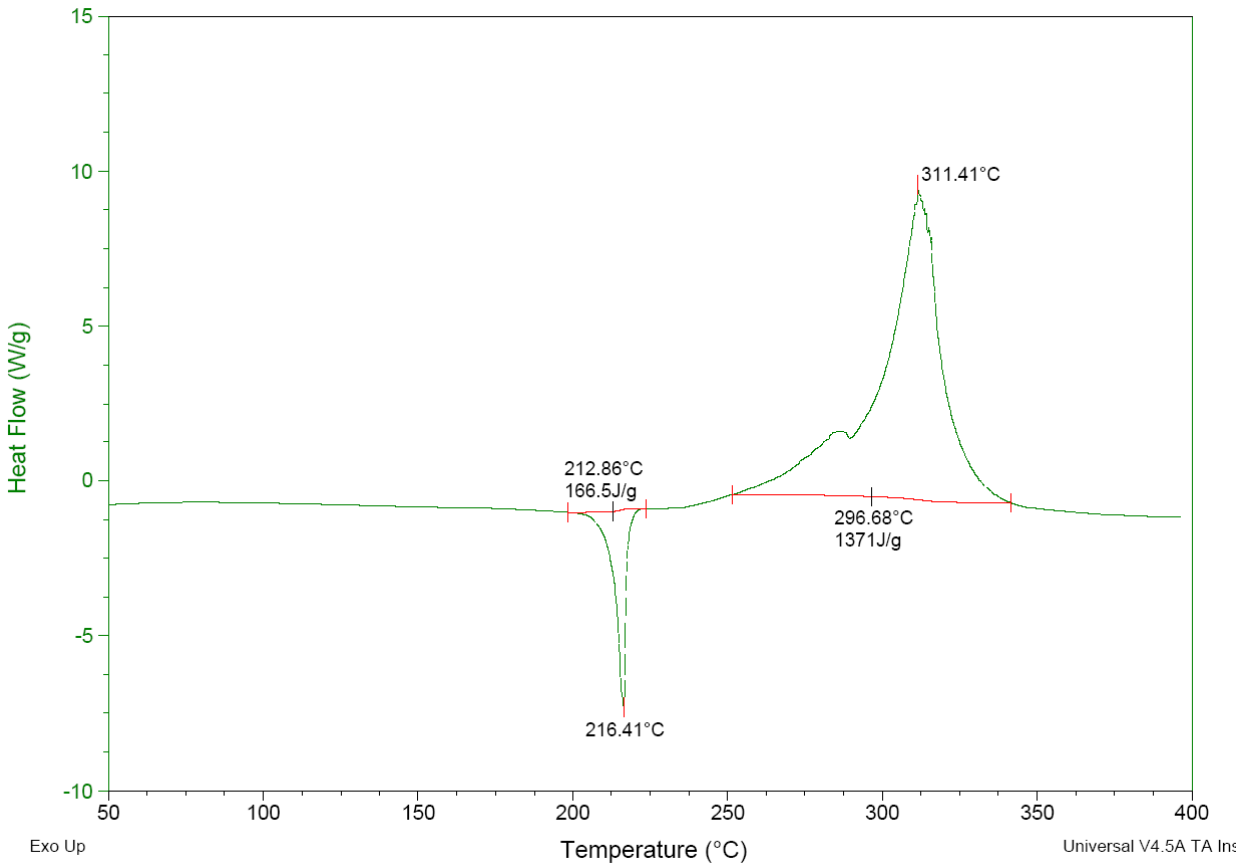
DNMT Improvements: DAMT synthesis



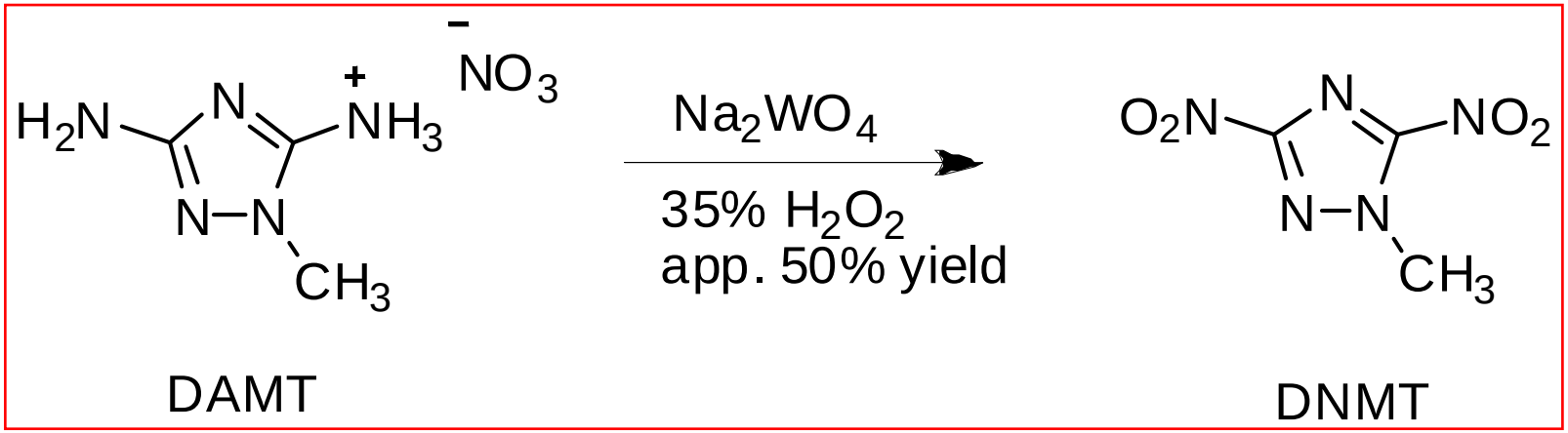
- Moved from one-pot to two-pot sequence
- 1st step:
  - Isolation of DAMT enhanced by minimizing water present in reaction
  - Product precipitates from reaction medium
  - Isolated by filtration

DAMT Properties

- DAMT shows good thermal stability.
- Calculated detonation velocity (~7800 m/s).
- Could find use as part of eutectic in melt-pour applications.



DNMT Improvements: DNMT synthesis



DAMT

DNMT

2nd step:

- Switching from diazotization to oxidation:
  - Uses less reagents (cheaper)
  - Generates mostly aqueous waste (more environmentally friendly)
  - DNMT precipitates from reaction medium
  - Product isolated by filtration

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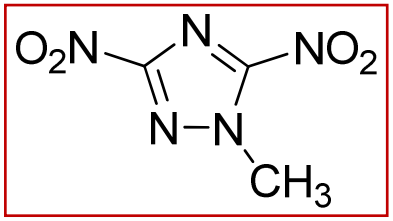
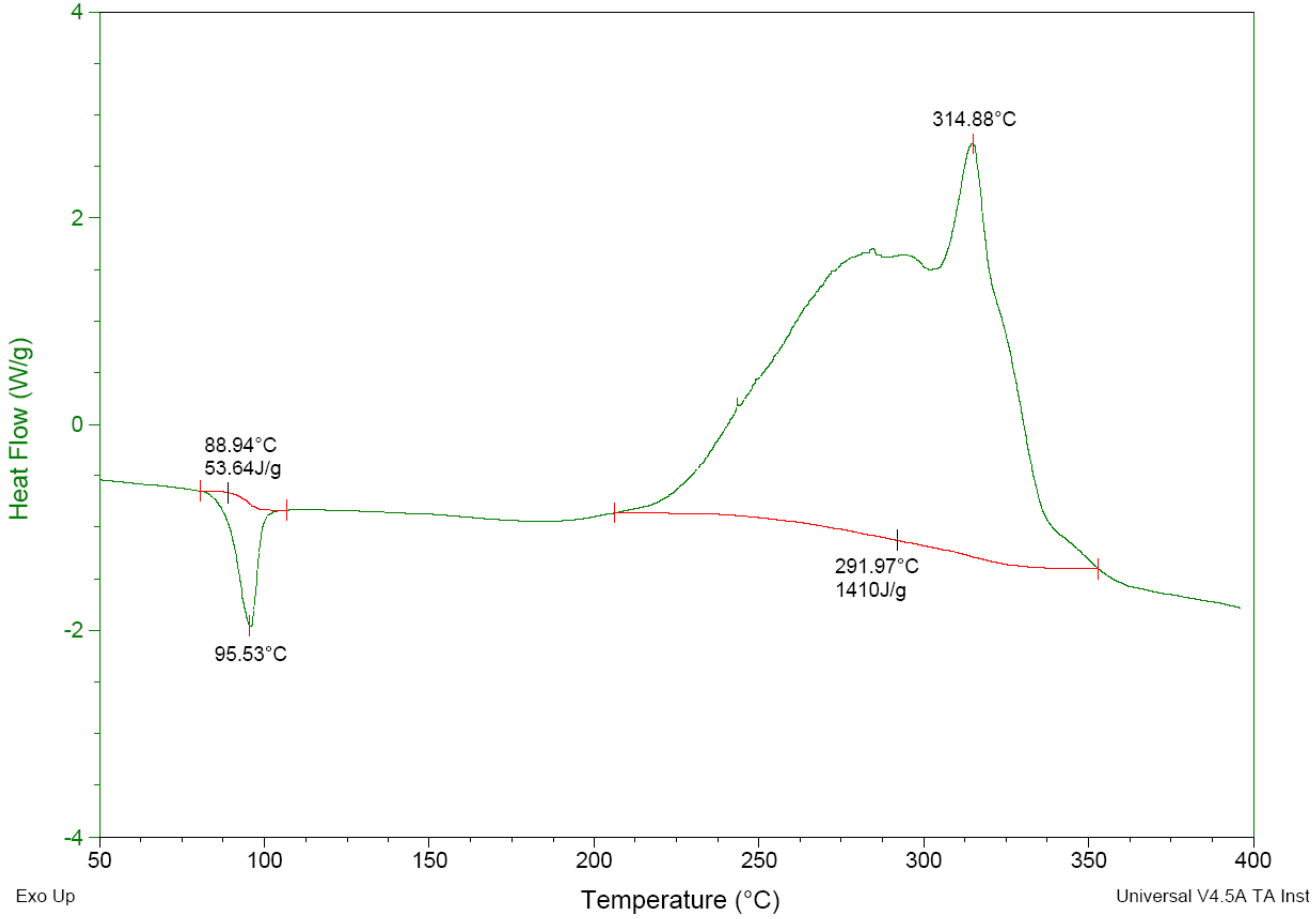
DNMT Properties



- DNMT has melting point almost identical to DNAN.
- DNMT demonstrates good thermal stability.



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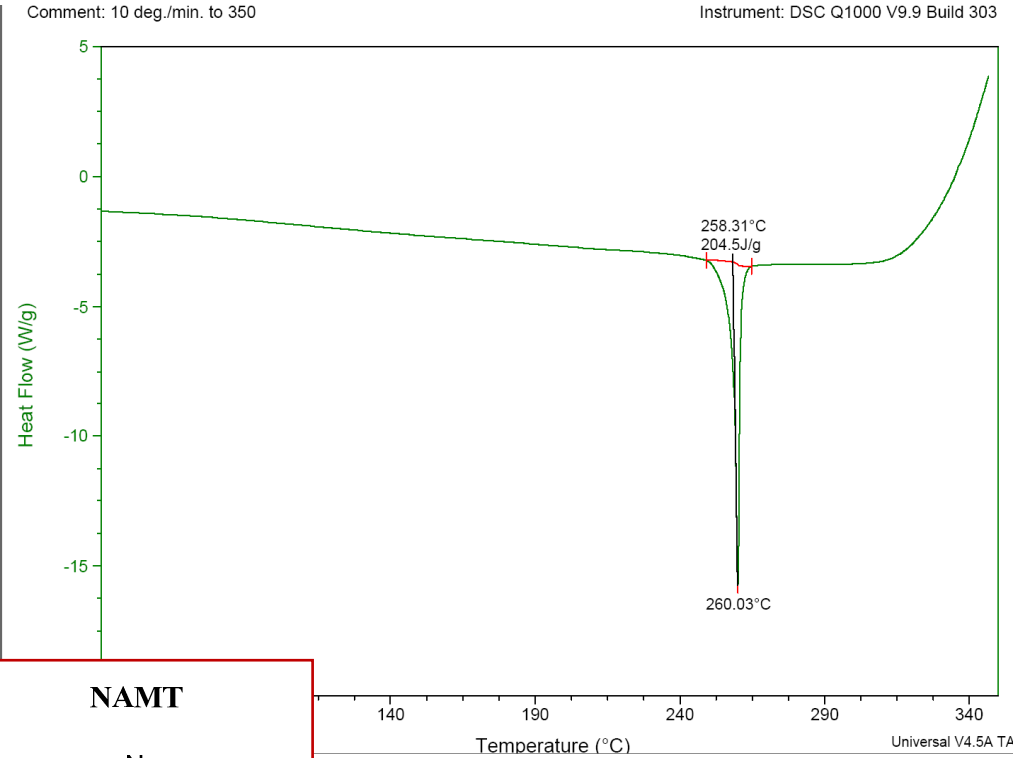
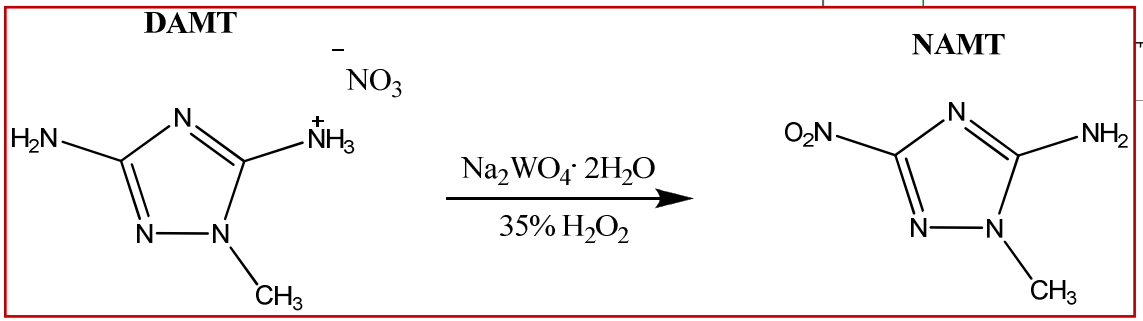
NAMT Properties

- NAMT demonstrates good thermal stability.
- Presence of nitro group and amino group suggests high degree of insensitivity, similar to TATB and DADNE





Safety Testing

ARDEC-Picatinny Arsenal

	ERL Impact (cm)	BAM Friction (N)	ESD (J)
DNMT	>100	>252	>0.25
RDX	25.4	>144	>0.25

OSI-Holston

Impact Sensitivity (cm), Navy method		
	Pre-melt	Post-melt
DNMT	92.7	171.0

- Melt-recrystallization might provide amorphous character
- Potentially remove crystalline defects/hot spots
- Appears valid for DNMT

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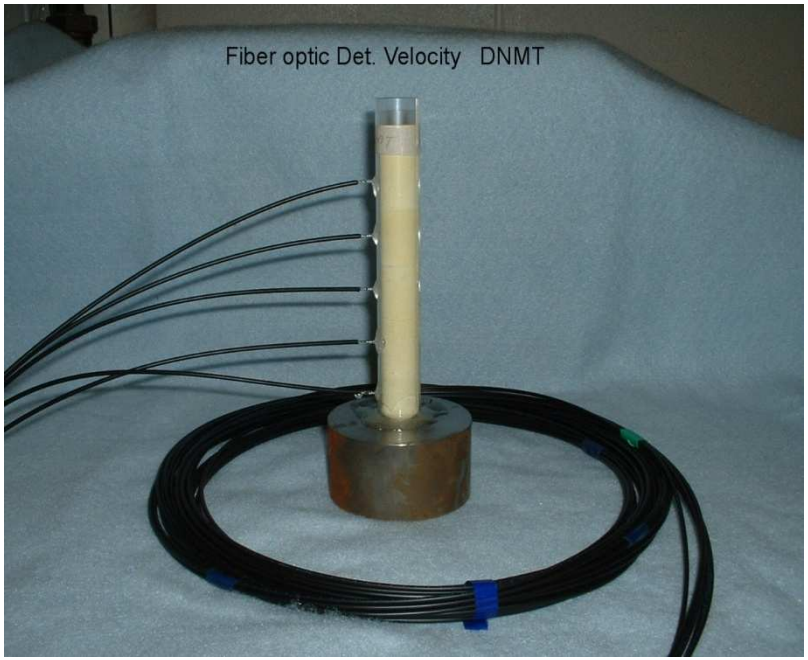
Performance-Rate Stick/Plate Dent

	Pcj, calc. (GPa)	Pcj, exp (GPa)	Energy out, calc. (cal/cc)	VOD, exp (Km/s)
DNMT	25.4	23.3	1739	7.850
Comp B	27.7	~27.6	1837	~7.960



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Compatibility Evaluations

- 1:1-Mass:Mass Physical Mixtures of HSAAP Formulation Ingredients
- DSC @ 5 °C/min. from 50 to 450 ℃
- Observe Changes in Exotherm Onset and Peak for Lowest Value Component
- Negative Deviations ≥10 ℃ Indicate “Fail”; Invoke VTS

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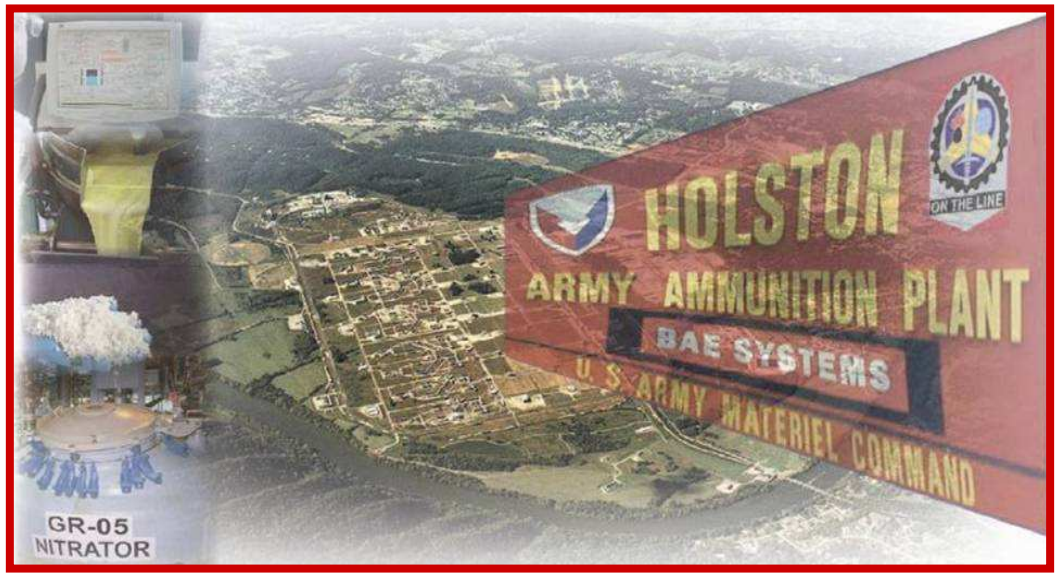
Compatibility Matrix

			DNMT		
			MP	Exo Onset	Exo Max
NEAT DNMT			95.7	260.6	280
RDX			88.2	204.3	230.9
MP	Exo Onset	Exo Max			
203.6	205.8	227.9			
HMX			93.7	223.4	252.8
MP	Exo Onset	Exo Max			
187.2	276.3	284.2			
NTO			97.7	176.9	231.4
MP	Exo Onset	Exo Max			
N/A	262.3	273.4			
TATB			97.6	227.1	243.5
MP	Exo Onset	Exo Max			
N/A	366.56	373.8			
DNAN			54.8	301.6	322.3
MP	Exo Onset	Exo Max			
94.2	326.9	342.9			
NQ			95.3	182.7	222.8
MP	Exo Onset	Exo Max			
N/A	195.2	202.8			
DNP			46.25	285.4	345.6
MP	Exo Onset	Exo Max			
86.5	275.8	296.9			
DNTF			81.1/92.5	233.8	268.7
MP	Exo Onset	Exo Max			
107.5	230.2	270.7			

- DNMT appears to be compatible with RDX, DNAN, DNP, and DNTF
- DNMT could have compatibility issues with HMX, NTO, TATB, and NQ.
- Further compatibility tests were conducted (VTS):
  - DNMT was found to be compatible with RDX, HMX, NTO.
  - No other materials were tested.

Conclusions

- DNMT shows great promise of performance, scalability and affordability.
- Program deemed successful by OSI in terms of identifying and executing scalable, safe processes.
- Future work to include lab scale-up to produce several pounds of DNMT for LSGT, VOD, and formulation activities.



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