TEX: A rational, next-generation IM explosive component

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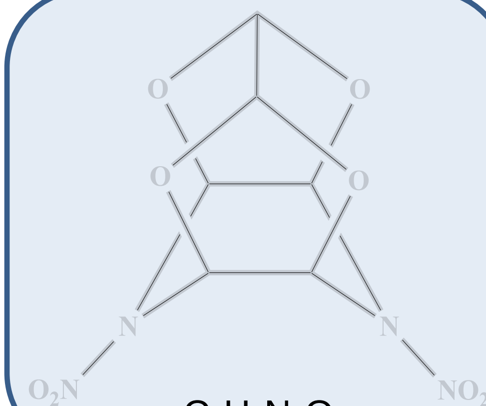
Overview

- TEX: What is it?
- Synthesis & scale-up
  - Production
  - Properties
- TEX: IM explosive ingredient
  - Explosive properties
- Summary

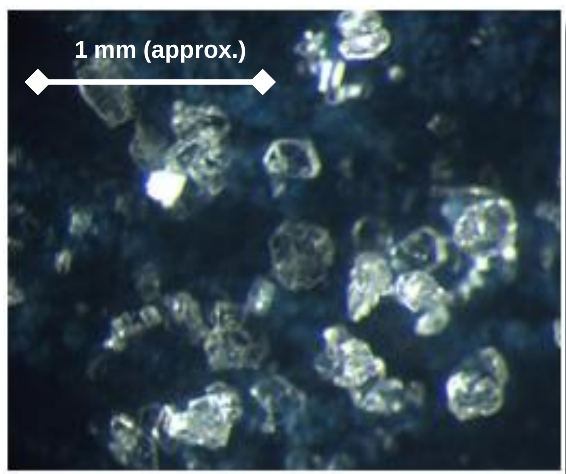
TEX: What is it?

- TEX = 4,10-dinitro-2,6,8,12-tetraoxa-4,10-diazatetracyclo-[5.5.0.$0^{5,9}0^{3,11}$]dodecane
- First reported by Boyer's group[1] in 1990
- High density, caged nitramine
  - **1.99 g/cm^3** (X-ray)
  - Structurally similar to CL-20



$C_6H_6N_4O_8$

TEX (Naked eye view)



TEX (30X)

TEX (1500X)

1. Ramakrishnan, V.T.; Vedechalam, M.; Boyer, J. H. *Heterocycles* (1990) *31*, 479.

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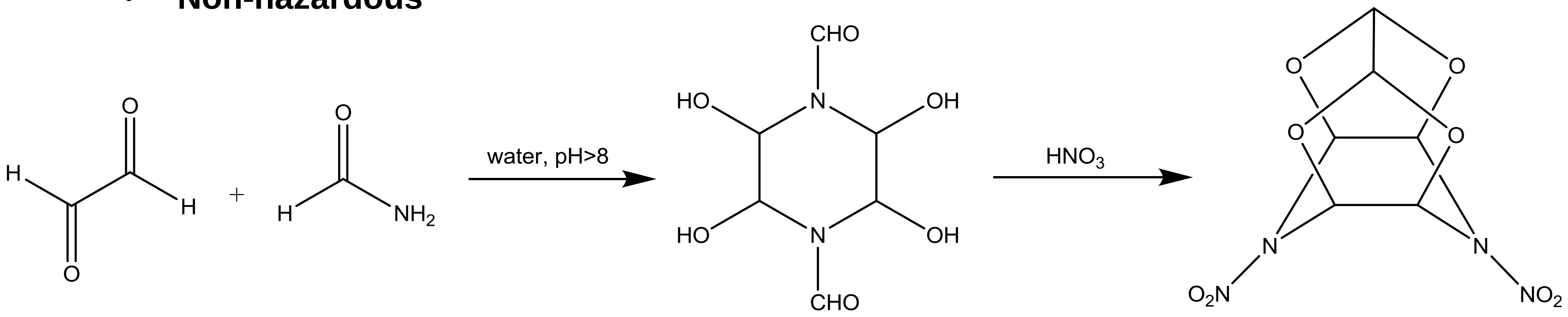
Synthesis & scale-up

- Simple, scalable two-step process
  - **Utilizes commodity starting materi**als
  - Precursor (THDFP) is non-energetic
    - THDFP produced in high yield
  - Nitration uses only nitric acid
    - **No specialized equipment required/no overt hazards encountered**
  - Manageable waste streams
    - **Non-hazardous**



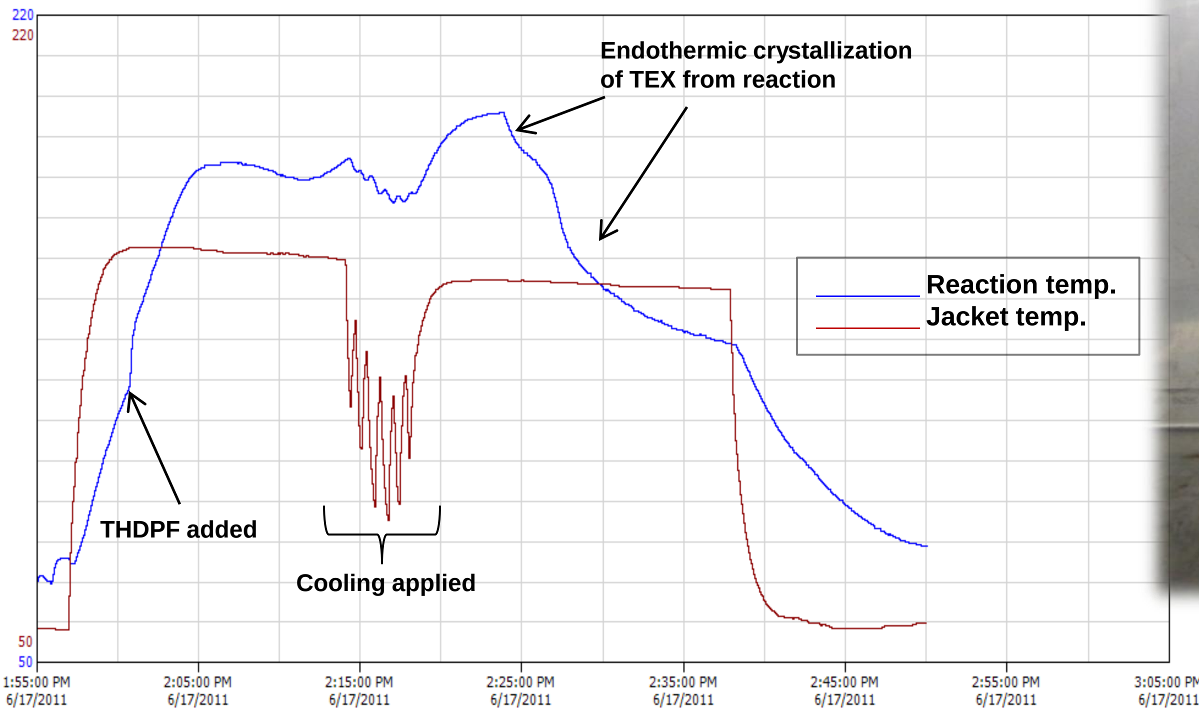
2,3,5,6-tetrahydroxy-1,4-diformylpiperazine (THDFP)

TEX

Use of low-cost, commodity chemicals is key to controlling production cost!

Production

- Pilot runs used 4 liter open-top, jacketed reactor
- Acid pre-heated, THDFP added in a single portion
- Virtually <u>zero</u> downtime between runs (i.e. min. clean-up)
- Reactor yield ~ 0.6 lb/gal



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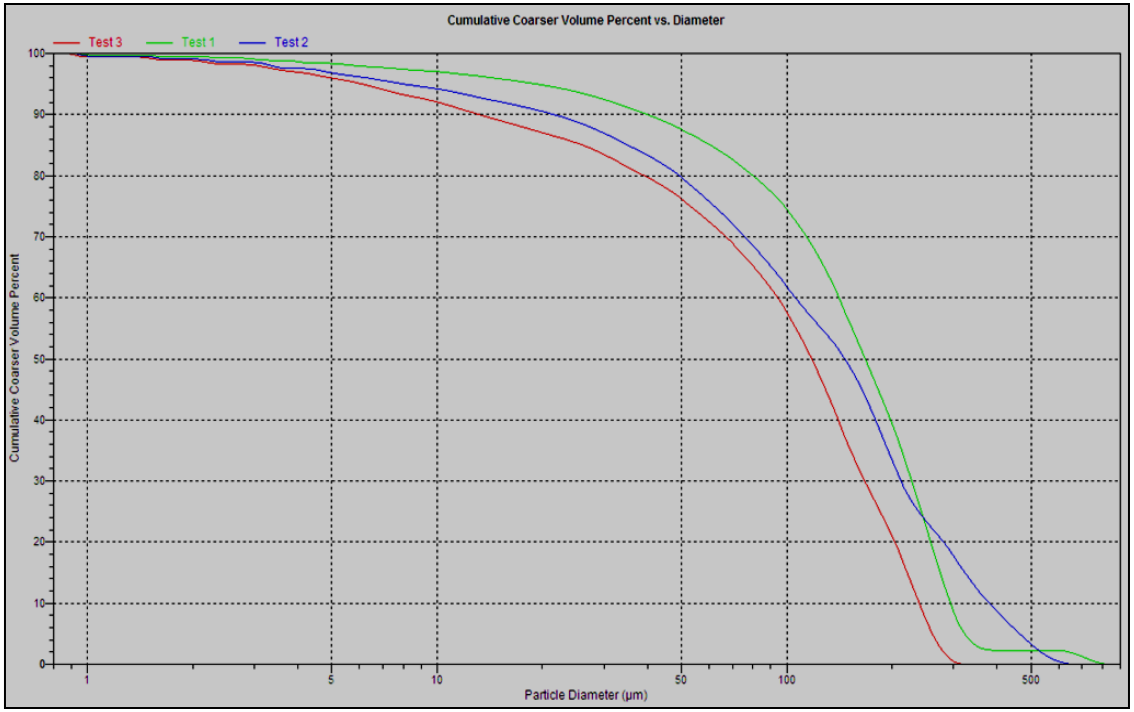
Properties

- Good small scale sensitivity
- Excellent purity (98-99%, HPLC)
  - No recrystallization
- No significant entrained acid
  - Total acidity ≤ 0.2%
  - No recrystallization
- Consistent particle size distribution (run-to-run)
  - 118-168 µm (50%)
  - Ability to modify PS in-situ

	Threshold initiation limit (TIL) values			
Material	ABL impact (cm)	ABL friction (lb @ 8 fps)	ESD (J)	Decomp. onset (°C)
TEX	80	490	0.0785	304
RDX	26	170	0.0251	219
CL-20	3.5	100	--	248
TATB	80	700	0.0251	375
TNT	51	720	1.3	291
NTO	80	1700	0.0783	274



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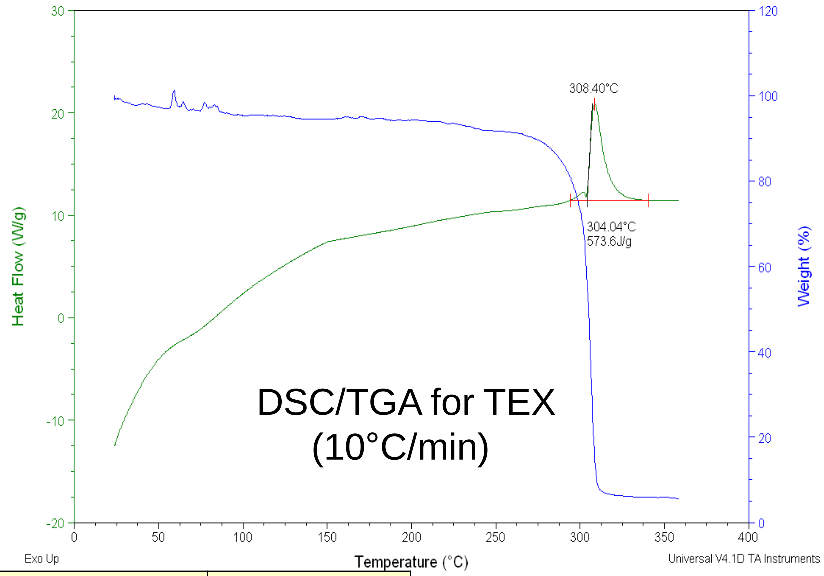
Thermal Properties

- TEX is extremely thermally stable
  - DSC exotherm onset/peak = 304/308°C
  - TGA shows very little residue upon decomp.
- TEX is compatible with most EM & binders
  - 1:1 DSC screening shows no significant ΔT



Neat Materials	Exo onset (°C)	Exo peak (°C)	ΔT onset (°C)	ΔT peak (°C)
TEX	304	308		
NTO	274	277		
NQ	250	255		
GuDN (FOX-12)	222	226		
RDX	219	248		
NC	203	214		
NG	187	204		
1:1 Material Mixtures				
TEX:NTO	260	267	-14	-10
TEX:NQ	245	251	-5	-4
TEX:GuDN	220	223	-2	-3
TEX:RDX	222	250	+3	+2
TEX:NC	203	214	0	0
TEX:NG	181	201	-6	-3

TEX: IM explosive ingredient

- TEX exhibits NTO-like performance with superior IM character
  - ATK Thiokol patent for castable explosives (Lund, et al.)[2]
  - Polish work on HTPB-based explosives (Vágenknecht, et al.)[3]

Table 1. Det. velocity & card gap results of TEX, NTO, and RDX based cast-cure explosives[2]

Energetic Solid	Cards	Reaction Type	Measured Detonation Velocity (m/s)
TEX	0	Sustained detonation	6811
TEX	70	No detonation, No deflagration	
NTO	0	Sustained detonation	6263
NTO	70	Sustained detonation	5571
RDX	0	Sustained detonation	7844
RDX	70	Sustained detonation	7790

Formulations: 70% solids (TEX, NTO, or RDX), isocyanate-cured DEGDN/PGN binder system (Pl:Po = 2:1)

2. Lund, G.K.; Highsmith, T.K.; Braithwaite, P.C.; Wardle, R.B. "Insensitive High Performance Explosive Compositions, US Patent 5529649, June 25, 1996.
3. Vágenknect, J.; Mareček, P.; Trzciński, W.A. "Sensitivity and Performance of TEX Explosives," *J. Ener. Mat.* (2002), *20*, 245.

Explosive Properties

- Evaluation of TEX in melt pour explosive formulation
  - 1:1 DSC of TEX:IM melt phase
    - Compatible
  - Small scale sensitivity
    - Reasonable sensitivity even at high solids loading

	Formulation (IM melt phase + crystalline nitramine)							
Test	**20% TEX**	**20% RDX**	**30% TEX**	**30% RDX**	**40% TEX**	**40% RDX**	**50% TEX**	**50% RDX**
ABL impact (cm)	**64**	**80**	**64**	**64**	**51**	**64**	**51**	**51**
ABL friction (lb @ 8 fps)	**495**	**395**	**490**	**310**	**540**	**305**	**500**	**295**

Explosive Properties

- Critical diameter & card gap testing
  - Melt pour formulation
    - 20 wt% TEX, 80 wt% IM melt phase
    - Formulation processed and poured well
    - 96% TMD
  - Critical diameter between 1" – 1.5"
    - Formulation not optimized for particle size
    - No other additives used (i.e. wetting agents or desensitizers)
  - Card gap results
    - NEGATIVE at 70 cards (3 shot trial)
    - POSITIVE (hole in witness plate) at 0 cards
      - Det. velocity = **7200 m/s**

Witness plates (70 cards)

Recovered 1" pipes (NO GO)

Pieces recovered from 1.5" pipe

Environmental

- Process waste streams are manageable
  - THDFP: aqueous (**NON-HAZARDOUS**)
  - TEX: spent acid $\xrightarrow{\text{neutralize}}$ no residual energetics (**NON-HAZARDOUS**)
- Aquatic toxicity
  - Initial toxicity screening shows TEX ≈ RDX to fathead minnow (*Pimephales promelas)*
  - Solubility of TEX in water (27 mg/L) ≈ RDX in water (33 mg/L)

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TEX in Summary

- Well-balanced energetic ingredient
  - Energy-density
  - Sensitivity
  - Affordability/producibility
- Potential applications for IM explosives
  - Cast cure
  - Melt pour
  - Pressed
- Plenty of room for development!

Material	Density[1]	Performance[2]	Reactivity[3]	Manufacturability[4]	Sensitivity[5]
TEX	1.99 g/cc	High	Low	Simple, low cost	Low
RDX	1.82 g/cc	High	Low	Simple, low cost	High
CL-20	2.04 g/cc	Very high	Low	Moderate, high cost	High
NTO	1.93 g/cc	High	Moderate	Moderate, med. cost	Low
TATB	1.93 g/cc	Medium	Low	Moderate, med. cost	Low
TNT	1.65 g/cc	Medium	Low	Simple, low cost	Low

1) TMD. 2) Based on calculated VOD and C-J pressure. 3) Potential for reaction based on solubility and presence of reactive groups/centers within the molecule. 4) Based on current processes including number of steps, costs of raw ingredients, and assumptions of full scale (> 10Klb/year) production. 5) Small scale sensitivity of neat materials.

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Questions?

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