

# CHEMISTRY OF DIFLUORAMINE DERIVATIVES



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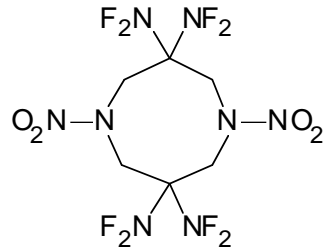

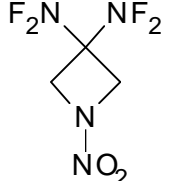
# Report Documentation Page

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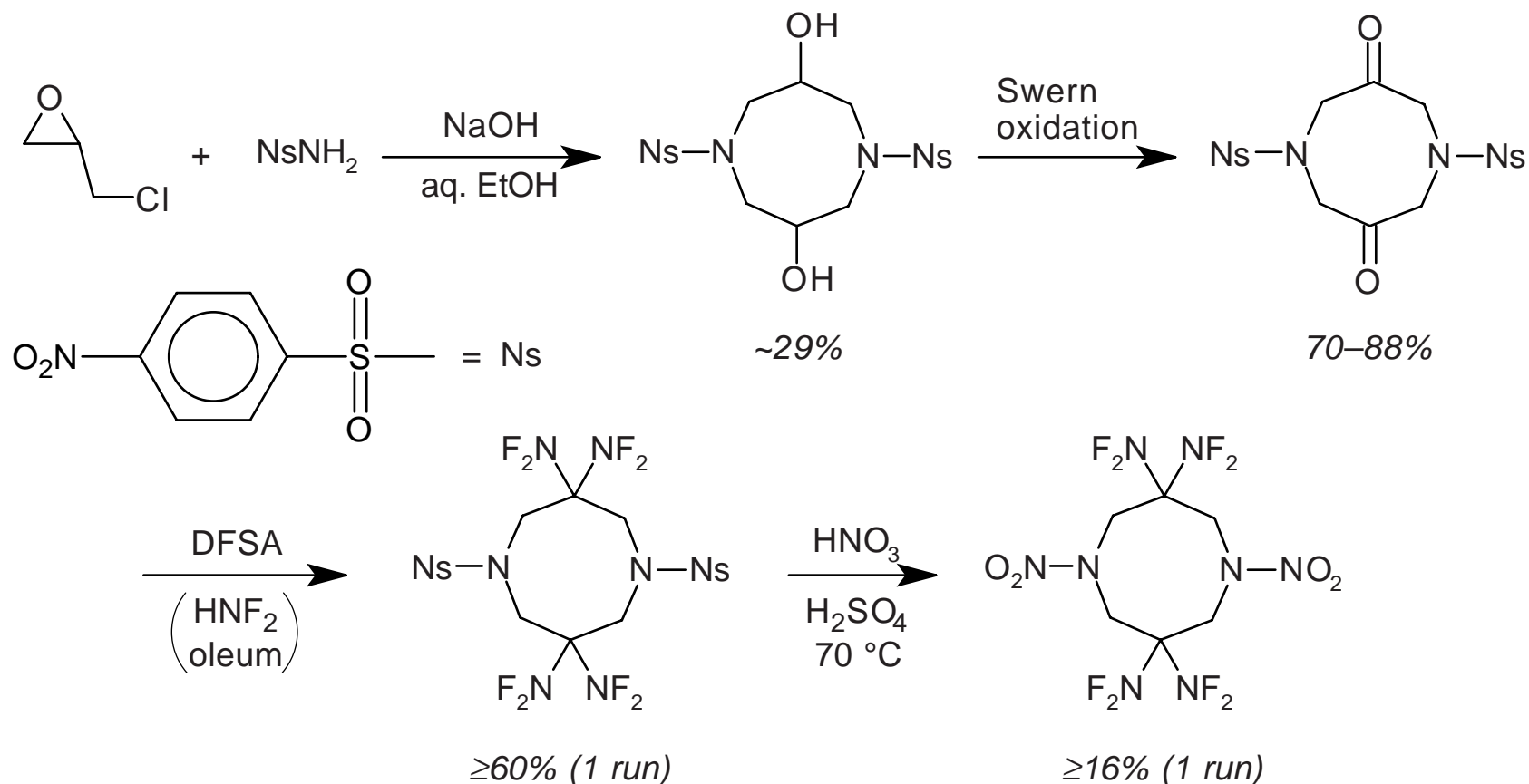
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# Calculated Performance Characteristics

<u>Compound</u>	<u>Density</u> <sup>1</sup> $\rho$ (g·cm <sup>-3</sup> ) (HMX = 1.894)	<u>Detonation pressure</u> <sup>2</sup> $P_{CJ}$ (KBar) (HMX = 390)	<u>Specific impulse</u> <sup>3</sup> $I_{sp}$ (sec) (HMX = 265)
	1.999	474	285
	1.973	444	274
	1.949	443	291

1. Ammon, H.L. (University of Maryland, College Park), personal communication.
2. Adolph, H.G.; Koppes, W.M.; Lawrence, G.W. (Naval Surface Warfare Center, Silver Spring, MD), personal communications.
3. Baroody, E. (Naval Ordnance Station, Indian Head, MD), personal communication.

# The Best Demonstrated Route to HNFX

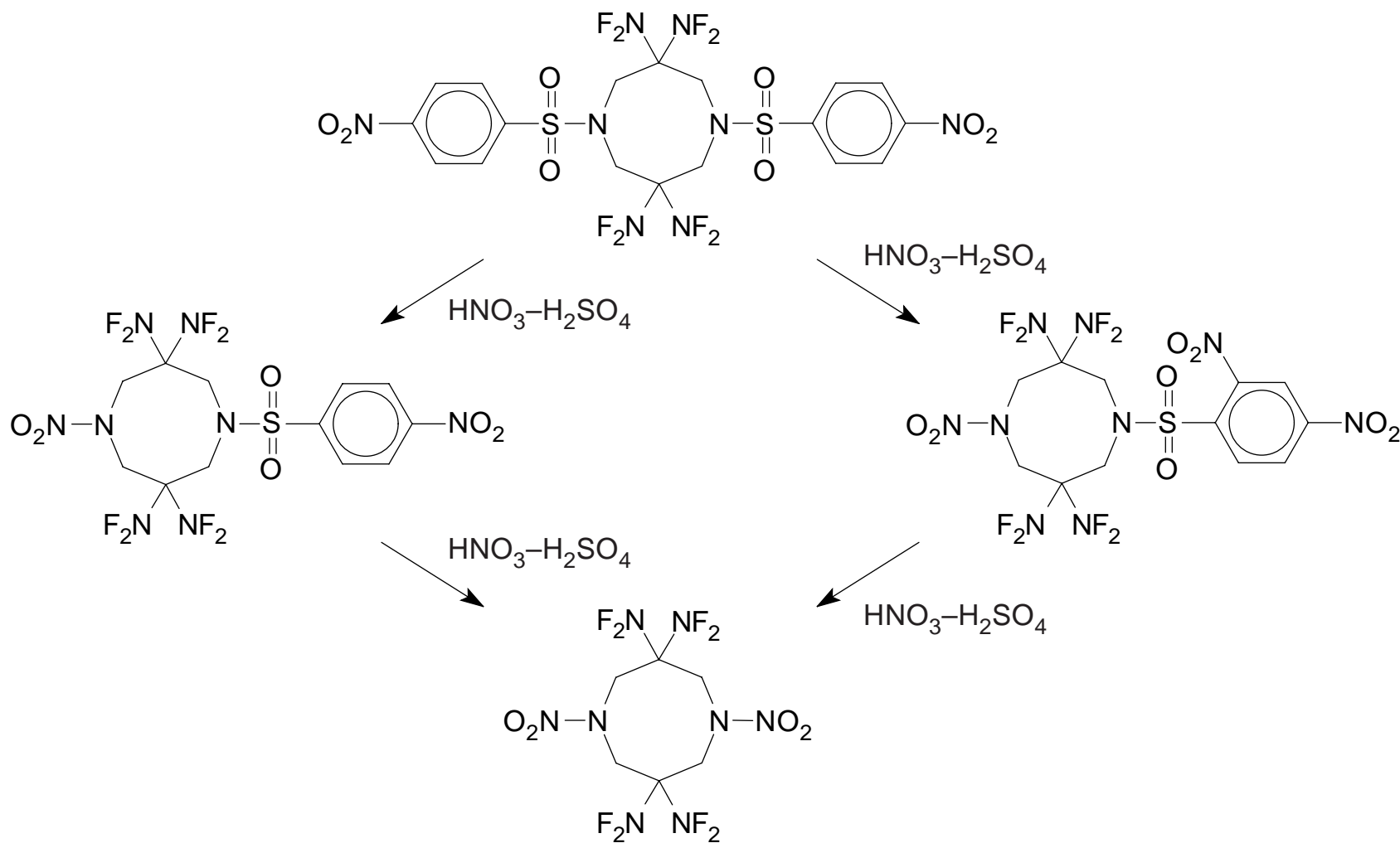


Chapman, Kreuzberger & Welker / TPL, Inc.

≡ **TPL, Inc.** ≡

# Complications in Nitrolysis

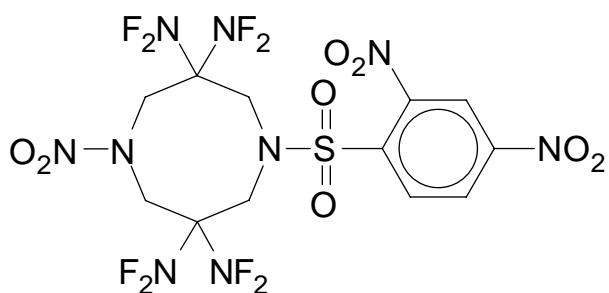
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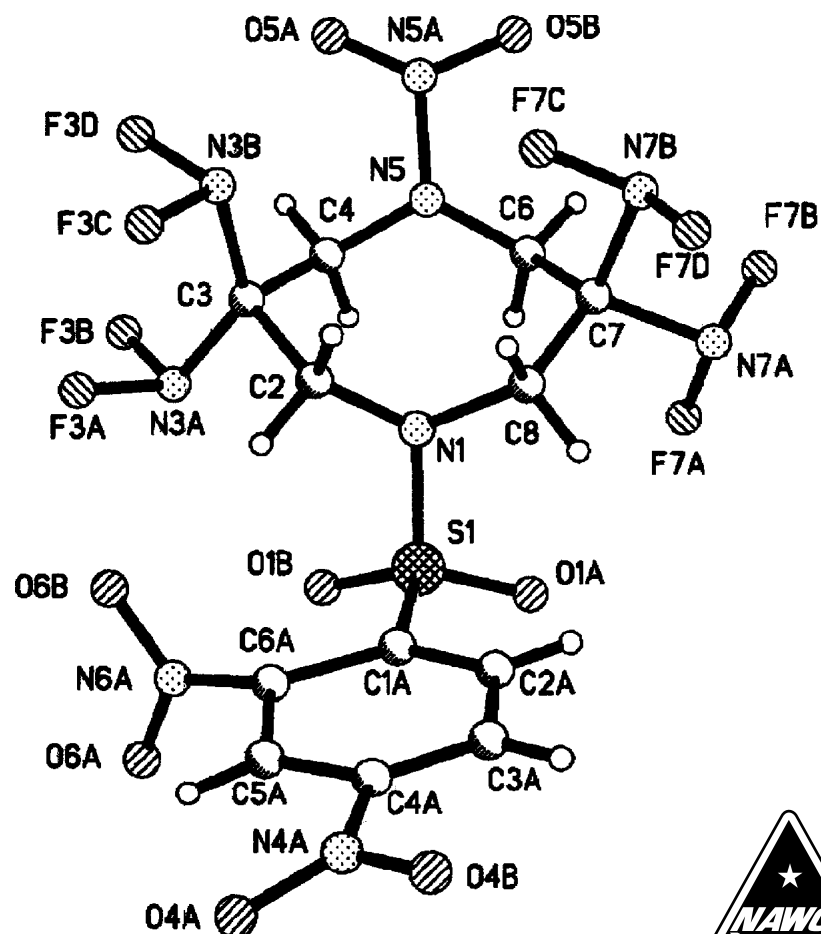
Welker / TPL, Inc.

# The Proof

- X-ray analysis (Richard Gilardi, NRL) of a minor impurity crystal in HNFX



Interestingly,  
 $\rho = 1.863$



# Polymorphism in HNFx

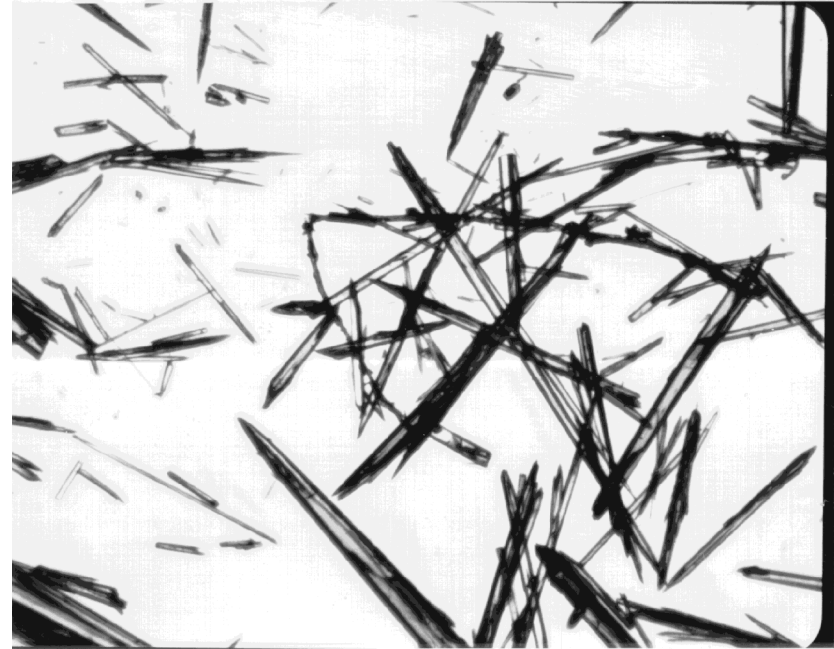
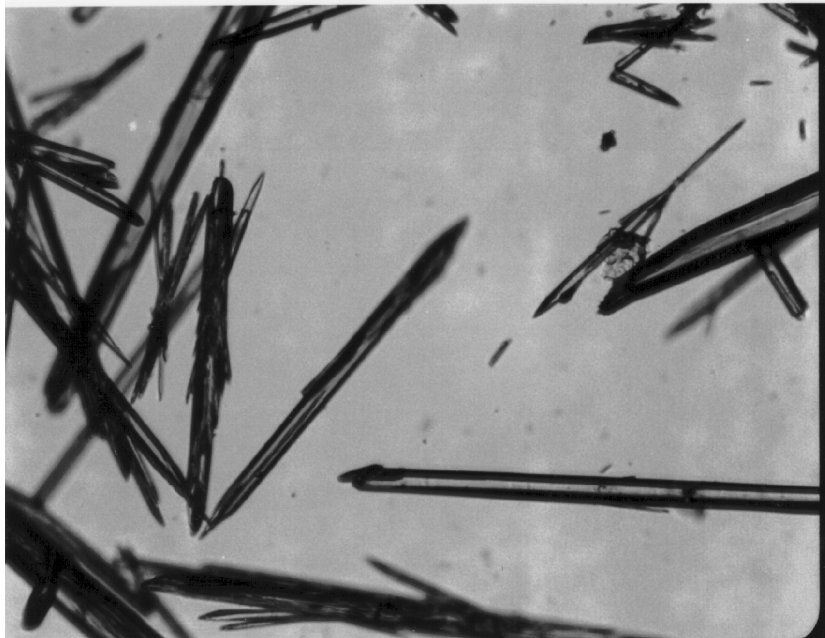
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- Original crystal preparation (needles) had solvent channels  $\rightarrow \rho = 1.784$  (theor.  $\rho = 1.999$ )
- New recrystallization from  $\text{CHCl}_3$  gives m.p. 216–218 °C without solvent (old m.p. 202~203 °C), but same polymorph!
- New recrystallizations from acetone give *new morphologies*: prisms and plates rather than needles



# Typical HNFx Needles

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~80x



# New HNFX Crystal Habits

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Prism  $0.1 \times 0.02 \times 0.02$  mm



Plate  $0.80 \times 0.22 \times 0.02$  mm

- Unit cells measured by Prof. Alan Pinkerton by CCD detector
- But...all crystals are the same polymorph!
- Can you believe it?



# HNFX Properties

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- M.P. 216–218 °C(dec)
- Readily forms solvent adducts as HMX does
- “Solvent-channel” form (“ $\alpha$ -HNFX”)  $\rho$  1.784
- Comparison of solvent adduct densities:

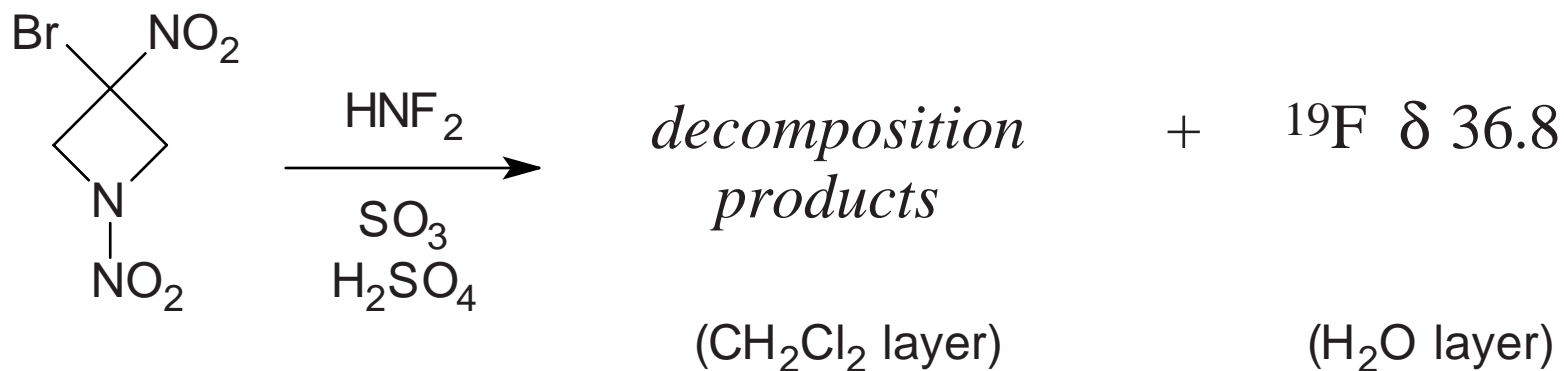
$\alpha$ -HMX	$\rho$ 1.838	← ← $\Delta\rho \approx 0.25$	HNFX·solvent $\rho = 1.784$
HMX·DMF	1.607		⇓ $\Delta\rho \approx 0.25$
HMX·NMP	1.570		HNFX $\rho \approx 2.03$

- Impact sensitivity of “ $\alpha$ -HNFX” comparable to PETN

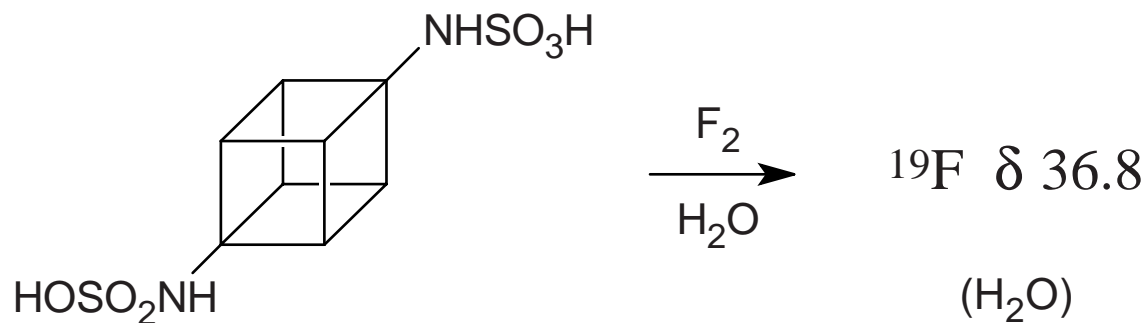


# Difluorosulfamic Acid (DFSA): A Recent History

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- Chapman, Fluorochem, Jan. 1990

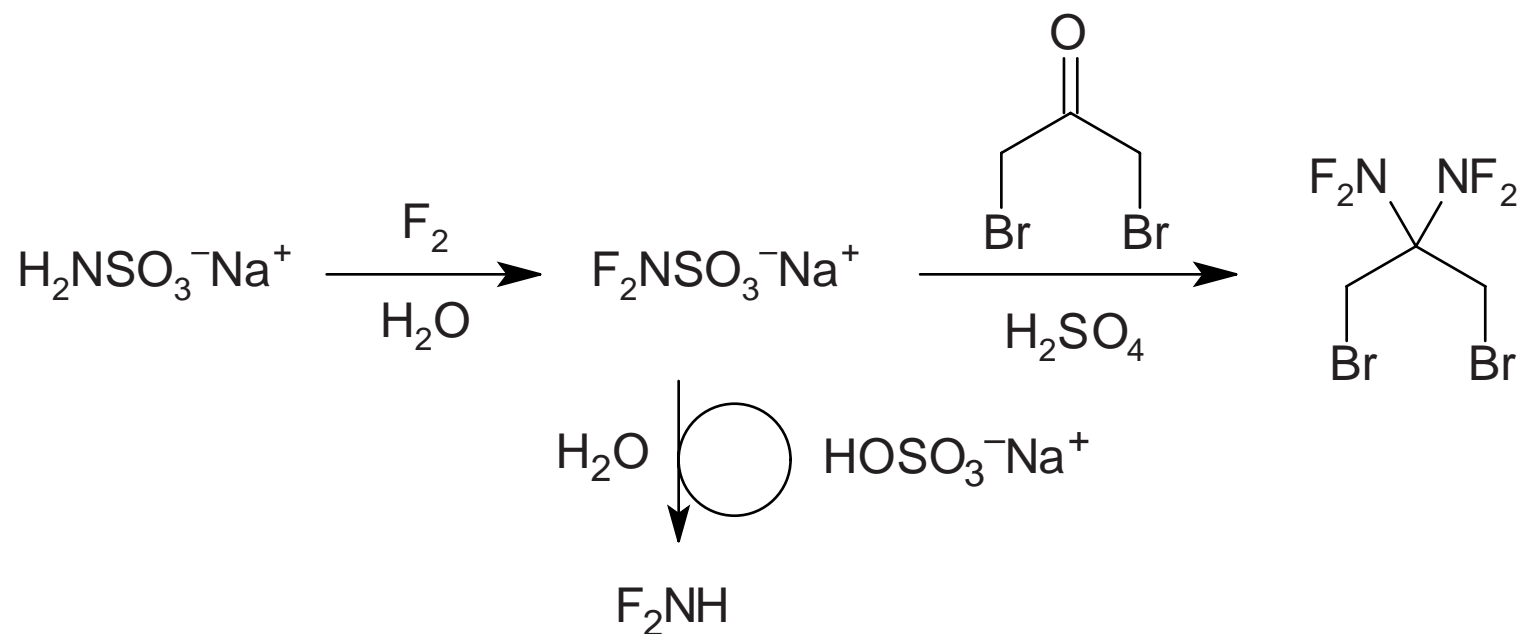


- Archibald, Fluorochem, Jan. 1990

⇒ “Could this be aqueous F<sub>2</sub>NSO<sub>3</sub>H?”

# Sodium Difluorosulfamate

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- But dilute aqueous  $\text{F}_2\text{NSO}_3^-\text{Na}^+$  is stable for weeks at pH 7–8



# Acknowledgments

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