

Case Study

A scientist in Huntsman Corporation was screening the acid for the acid-neutralized amines as polyurethane catalysts:

“Approximately 3–4 mL of 90% nitric acid was added to around 30 mL of a solution containing approximately 10% water and 90% of the amine. Upon addition of the nitric acid, the scientist noticed fuming along with the expected exotherm. The scientist halted the addition to allow the uncapped mixture time to cool... Approximately 3–4 min after setting the solution down, the chemical mixture spontaneously decomposed, exploding the glass bottle and emitting a burst of flames.”

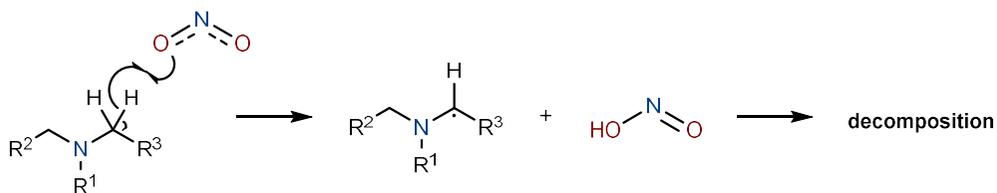
Meredith, M. T., *ACS Chem. Health & Saf.* **2022**, 29 (1), 72
<https://doi.org/10.1021/acs.chas.1c00042>.

Incident Analysis

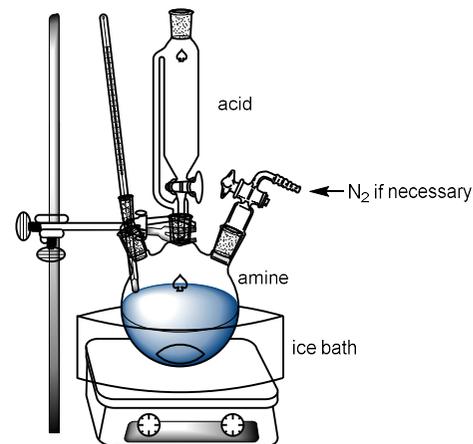
The hypergolic propellant experiments: excess of acid, minimal water
Their experiment: excess of amine, some water present

Some possible reasons:

1. The amine solution was warmed by the mixing of the amine and water.
2. The mixing was not adequate, the acid added might be at the bottom as a heterogenous region.
3. The excess of amine could only decay the decomposition.
4. The generation of amine hydrate could contribute to the dewatering of nitric acid.
5. The amine contains 5-7% impurities that have terminal –OH and –NH₂ group to go nitration or other accelerating reactions with nitric acid or NO₂.



Suggestion for Reaction Setup



1. Don't combine concentrated oxidizing acids with neat organic material without a full safety review
2. Use an appropriate reaction set up with thermally robust glass in a ventilated fume hood
3. Accurately consider hazards and emergency procedures

Safety with Oxidizing Acids

	Sulfuric Acid	Perchloric Acid
Oxidizing?	>90%	>73%, hot
Safety Risks in Reaction	large exothermic surges	oxidative decomposition, impact- and heat-sensitive salts
	Nitric Acid	
Oxidizing?	always	
Safety Risks in Reaction	NO ₂ & high-energy radicals, runaway exothermic decomposition	

Müller, H., *Ullmann's Encyclopedia of Industrial Chemistry*. **2000**, 35, 141
https://doi.org/10.1002/14356007.a25_635

Long, J. R., *Chem. Health & Saf.* **2002**, 9 (3), 12
[https://doi.org/10.1016/S1074-9098\(02\)00294-0](https://doi.org/10.1016/S1074-9098(02)00294-0).

Liu, W.-G.; Dasgupta, S.; Zybin, S. V.; Goddard, W. A.,
J. Phys. Chem. A **2011**, 115 (20), 5221 <https://doi.org/10.1021/jp202021s>.