Dangers of: Azide Reactions, Large Scale Reactions

Incident
An explosion occurred during a 200g scale synthesis of trimethylsilyl azide.

During the distillation and purification phase of the synthesis, the distillation pot spontaneously exploded which created glass shrapnel, splashed hot solvent and demolished the fume hood.

This incident injured the researcher performing the synthesis, sending them to the hospital with glass shrapnel, burns and temporary hearing loss. The researcher has since recovered. The damage to the fume hood was irreparable and it has been decommissioned.

How did this happen?
Although we will never know the exact cause of the explosion, we do believe the following to be the most likely contributing factors:

- A reagent change may have introduced water to the reaction causing the formation of explosive azide byproducts.
- Difficulty stirring the thick reaction solution could have produced superheated spots which could have caused an explosive decomposition.
- The literature failed to sufficiently warn against the explosive nature of this synthesis.
- The reaction scale overwhelmed all appropriate laboratory controls.

How can you prevent a similar incident from occurring in your lab?
Researchers working with chemicals that pose physical hazards (reactive materials) should share this Safety Alert with their group and discuss how the following topics are applicable to your work.

PIs, Lab Managers, Directors and Safety Committees:

- Consider setting upper limits for classes of reactive materials used in your department or lab, above which procedures should be prohibited without additional review and approval.
- Train researchers using these materials to understand and recognize the factors affecting the probability and severity of an explosion occurring when working with these materials. It is not sufficient to simply recognize that something can be reactive without considering the potential energy released.
  - Characteristics which may affect the probability of an uncontrolled reaction include: temperature, pressure, atmospheric composition, light sensitivity, solubility, purity of solvents and reagents, mixing, incompatible materials and containers etc.
  - Characteristics affecting the potential severity of reaction include: scale and inherent properties of individual reactants, products and potential byproducts.
Experiment Planners (PIs, post-docs, senior graduate students):

- Design your experiment around the capabilities of the primary reaction vessel, apparatus or container.
  - Analyze your reaction to determine if the primary reaction vessel is sufficient to contain the reaction should something go wrong.
  - Check the maximum pressure, temperature and compatibility with your experimental conditions.
  - Discuss the risks and warning signs of adverse events with researchers who will conduct the experiments.

Individual Researchers:

- Follow group policies, including wearing appropriate PPE
- Communicate with others including signage to indicate hazards in process.
- Do not alter reactants when conducting high hazard reactions without thorough discussion and approval of your PI.
- Be mindful.
  - Being tired, distracted or complacent increases risk of incident (experienced researchers may become overly comfortable and miss important warning signs)
  - Recognize process problems, such as issues with stirring or unexpected sublimation as indications of a possible safety issue. If you detect something unusual or unexpected, pause and consult with colleagues and experienced investigators.

Additional Information
Details of the incident have been shared with the chemical and safety industries through news forms such as the C&EN Safety Zone blog and through presentations to the UMN community and the safety community at large.

If you have questions regarding this alert please contact Anna Sitek, DEHS, at engl0131@umn.edu, or Jodi Ogilvie, DEHS, at jogilvie@umn.edu

Resources for Predicting Hazards of Reactions

- CAMEO Chemicals – NOAA, EPA, USCG
- Chemical Reactivity Worksheet (CRW) – NOAA, EPA, AICHE, and Dow Chemical
- Identifying Chemical Reactivity Hazards: Preliminary Screening Method – EPA
- MANAGING CHEMICAL REACTIVITY HAZARDS – EPA
- Essential Practices for Managing Chemical Reactivity Hazards – CCPS, OSHA, EPA, & American Chemistry Council, the Synthetic Chemical Manufacturers Association, and Knovel Corporation
- A Checklist for Inherently Safer Chemical Reaction Process Design and Operation What You Need to Know – CCPS
- Reactive Material Hazards What You Need to Know – CCPS
- Chemical Reactivity Hazards – OSHA, Dow Chemical
- Dow Safety Academy – Dow Chemical, see “Resources” tab
- Academia and Industrial Pilot Plant Operations and Safety
- Bretherick’s Handbook of Reactive Chemical Hazards