The University of Akron
College of Polymer Science and Polymer Engineering
Office of Operations - Safety Office

LESSONS LEARNED

November 2017 – Azide Synthesis Explosion

The round bottom flask and water condenser on the left was obliterated into the glass chards in the picture on the right.

What happened?

Researcher set up an azidonation reaction the night before a scheduled 2pm NMR time the next day. The reaction utilized sodium azide, 3-bromo-1propanol and water. At approximately 1:40 pm the next day the researcher needed to quickly stop the reaction to make the reserved NMR time, not waiting for the reaction to cool completely opened the top stopper and the azide group reacted rapidly producing the N2 gas causing an explosion. Researcher was not wearing a lab coat or safety glasses but was working with a lowered sash which protected from further injury to the face and torso. The researcher did know the health toxicity of the chemicals involved and quickly rinsed his arms in the sink with water and ethanol. The Safety Officer was called by a student who heard the explosion and then the researcher was driven to the hospital by a group member. The researcher received treatment for multiple minor lacerations.

What was the cause?

The reaction vessel was heated to 100° C and was a closed system without utilizing pressure rated glassware. Materials with azide functional groups are extremely reactive, especially those not bound to long alkyl chains. The high temperature and pressure of the vessel upon manipulation and the extreme reactivity of azide starting material and azide-based products caused the explosion.

What went wrong?

- **Student was not wearing lab coat or safety glasses.**
- Researcher did not have SOPs for this experiment.
- Researcher is not experienced in synthesis.
- Researcher created a closed system reaction vessel using glassware not rated for high pressure.
- Researcher's lab notes did not include specific information, such as amounts of starting materials involved or what glassware was used to set up the reaction, from 2 prior attempts at experiment.
- Researcher rushed the experiment and left personal protective gear in the office because of time constraints.
- There was no unattended lab experiment form submitted for the overnight reaction.
- **Researcher was driven to the hospital with a personal vehicle instead of ambulance.** (See previous Lessons Learned)

What went right?

- Researcher was working with hood sash lowered to a safe working position.
- Group members immediately helped and called Safety Officer - **Please call 330-972-2911 immediately**, then Safety Officer if there is an injury or high hazard associated with the accident.
- Researcher was aware of the health hazards associated with the chemicals that were used and knew right away what to do.

What corrective action was taken?

- Standard Operating Procedure (SOP) will be written for experiments.
- Safety in the lab will be discussed at group meetings.
- Unattended Experiment forms will be used for any overnight or unsupervised experiment.
- Researcher will be provided more oversight with synthesis experiments.
- Alternative, less hazardous, reaction routes will be utilized.
- Recommendation to use commercially available intermediates instead of synthesized when high hazardous chemicals are involved.

How can incidents like this be prevented?

- Mandatory lab coats and safety glasses in the lab.
- Do not rush experiments.
- Written SOP should be utilized for routine experiments.
- Contact the advisor, or EOHS for questions regarding experiments in areas of inexperience.
- Better time management when scheduled for NMR.

Resources:

Laboratory Safety Fact Sheet: Synthesizing, Purifying and Handling Organic Azides
http://www.ehs.ucsb.edu/files/docs/ls/factsheets/Azides_FS26.pdf

"Organic azides are potentially-explosive substances that can and will decompose with the slightest input of energy from external sources (heat, light, pressure). Additionally, small molecules containing the azido functionality tend to decompose violently which may result in injury if proper safety precautions are not utilized."