

December 8, 2021

What Happened?

During a chemical procedure, a tube flask shattered when it was being inappropriately thawed for use in a Chemistry lab. The Graduate Student Researcher was performing a freeze pump thaw procedure to degas a solvent inside a California fume hood. The researcher was using a 350 mL Schlenk tube flask with 300 mL of toluene under inert atmosphere and slowly froze the solvent in the flask using a dewar of liquid nitrogen. When the researcher removed the flask from the dewar to hook it up to the high vacuum Schlenk line, and in doing so, the toluene in the tube flask did not thaw uniformly, causing it to expand and shatter the tube flask.

Incident causes

The direct cause of this incident is the removal of the flask from liquid nitrogen where the solvent warmed up too quickly and expanded in a closed system which caused a pressure build up causing the flask to shatter.

The root cause of the incident was the failure to follow the written/posted laboratory specific Standard Operating Procedure (SOP).

What can be done to prevent this from occurring again?

- Ensure that staff are trained and authorized to perform highly hazardous task and follow all applicable written Standard Operating Procedures (SOP) including procedures for Freeze Pump Thaw Degassing of Liquids. Also, staff should periodically review procedures and process SOPs before performing an experiment even if they have already performed this procedure before. Accidents can happen when staff become comfortable performing a procedure.
- Connect flask to the Schlenk line prior to freezing the solvent. It's difficult to connect the flask while holding up a dewar/keeping the flask submerged in liquid nitrogen to keep it frozen. It's much easier to connect a flask when it is not yet frozen/submerged.
- Slowly thaw the solvent by utilizing a room temperature water bath. Make sure the flask is submerged up to the amount of solvent in the water bath to ensure uniform thawing.
- Always use appropriate Personal Protective Equipment (PPE) like gloves, lab coat, safety glasses and closed toed shoes when working with hazardous chemicals; you may need to upgrade to goggles, face shield, etc. where there's a potential for explosion or fire. Refer to lab/equipment specific operating procedure which outlines appropriate PPE for the task being performed.

Additional Considerations for Freeze Pump Thaw

- Check all glassware for cracks or imperfections as they can affect the integrity of the glassware and cannot withstand the pressure of certain procedures.
- Only fill the flask with solvent to only 2/3rds of the volume of the flask. Overfilling the flask will cause the flask to shatter.
- Always ask for help if you need it and do NOT work alone.

Where to Get Help or More Information

For more information or questions, or to receive assistance, please contact EHS at (949) 824-6200 or at safety@uci.edu