Hello Everyone,

I would like to share with you a few safety incidents reported to me between July 2022 and October 2022. Near misses reported, such as broken glassware and unlabeled chemicals are not mentioned in this document. Ensure that all waste containers remain closed when not in use and gas cylinders are capped when not in use. It is important to promptly report safety incidents and near misses after they occur to ensure the safety of everyone in the department. No one will ever get in trouble for reporting a safety incident as it is a way to help keep our research community safer.

To submit a safety incident report/near miss, visit https://engineering.purdue.edu/ChE/aboutus/safety, fill out Safety Incident/Near Miss Report, and send it to me (chesafety@purdue.edu). It is also important to visit the ChE safety page often and review all the important information in the safety documents posted there.

1. **July 24, 2022: Flame Burst from Non-Oxidative Coupling of Methane Reactor due to Fitting Failure**

Description: A non-oxidative coupling of methane (NOCM) reaction with 80% methane (balance nitrogen) in the feed at 975°C and 7.75 PSIG was carried out. The flowrates for methane and nitrogen were 30 and 6.6 mL, respectively. The reaction takes place in a quartz U-tube reactor connected to the inlet and outlet pipelines using Ultra-Torr fittings and sealed using Chemical-Resistant Viton Fluoroelastomer O-Rings. A quartz thermocouple well is also connected to the reactor using Ultra-Torr fittings and sealed using Chemical-Resistant Viton Fluoroelastomer O-Rings. Around 20 minutes into the reaction (at 4:15 pm), a flame burst was seen from the top of the vertical furnace. No immediate damage was observed.

Immediate Action: The methane feed to the reactor was stopped by changing the position of a three-way valve positioned between the methane feed line and reactor inlet pipeline manifold and directing the flow to the vent. The nitrogen mass flow controller, positioned at the start of said manifold, was set to 100% open; the furnace heating was shut down and the methane cylinder and regulator were closed.

After the reactor cooled down, the system was inspected. The Ultra-Torr fitting connecting the quartz thermocouple well and the reactor got disconnected from the reactor, and the O-ring used for sealing the connection between the Ultra-Torr fitting and the reactor was melted to the fitting.

Recommendations and long-term actions:
The inlet manifold has a pressure relief valve set to 10 PSIG. Thus, it was decided that pressure tests should be performed just below this pressure (~9.0 PSIG), instead of 5 PSIG, value to which the system was pressurized during the tests previously. Additionally, it was set that the Ultra-Torr fittings should be inspected before each reaction and changed when there is visible damage (scratch, melted plastic, discoloration, etc.) and that new O-rings should be used for each reaction. Lastly, the integrity of the quartz reactor should be inspected before each reaction to ensure that it doesn't fail during the pressure test or reaction.

Other safety recommendations: the reactor should be made longer, and more insulation of the furnace should be added during operation to reduce the heat reaching the Ultra-Torr fittings and minimize their failure. Additionally, the current methane detector, which is connected to the methane shutoff valve, should be inspected for further use or replacement. A CO_2 detector should also be implemented and connected to the methane shutoff valve in case a flame occurs. An emergency button, closing the methane shutoff valve was also recommended.

2. July 25, 2022: Liquid Nitrogen Valve Froze Open, Liquid Nitrogen (LN2) Tank Allowed to Drain in Lab

Description: While a graduate student researcher was filling a small dewar with LN2 from a larger LN2 tank, the liquid flow valve became frozen open. There was a significant amount of ice deposited on the valve and the transfer line. The researcher tried to melt away the ice with a heat gun and to close the valve but due to the continuing flow of LN2, the ice on the valve could not be melted. Over time, the flow rate of LN2 from the transfer line increased.

Immediate Action: As the researcher and a lab mate were unable to close the valve, they opened the sash of the fume hoods in the lab to avoid the buildup of N2 in the lab. The transfer line hose was placed in a plastic bucket and an elephant trunk was placed on the bucket to minimize the N2 buildup in the lab. The two graduate researchers evacuated the lab leaving the LN2 tank to drain and keeping the lab doors open to prevent nitrogen build up. The tank drained after approximately 30 minutes. The graduate students waited for an additional 2 hours for the atmosphere in the lab to normalize before re-entering.

Recommendations: This incident occurred during a time when the lab was especially humid, due in part to issues with the building HVAC system. Indiana Oxygen was contacted and they suggested that while filling dewars with liquid nitrogen, the valve on LN2 tanks should constantly be in motion (slightly increasing, and decreasing flow) to ensure that ice build up does not freeze the valve open. If there is any ice build up near the liquid valve, it should be melted using a heat gun before the valve is opened. An O2 sensor was also installed in the lab to alert lab members if an asphyxiation hazard is present, should a similar situation arise again.

3. July 26, 2022: Melted Plastic on Hotplate and Burn Injury

Description: Although a "Hot" sign was in place for a hotplate, a graduate student did not realize that the hotplate located inside a glovebox was still "on" when they picked up a graphite plate sitting on the hotplate. This resulted in a blister on their finger. The researcher immediately dropped the hot graphite plate, and the plastic melted onto the hot graphite plate.

Immediate Action: The researcher rinsed their had with cold water. While a blister did form on the researcher's finger, the researcher did not seek medical attention.

Recommendations: While the research group did have a policy in place where a sign is posted on the glovebox when the hotplate is hot, the researcher had assumed that the hotplate was off, that the sign was placed a while ago, and the hotplate had cooled over time. The researcher neglected to check if the hotplate was still on. It is difficult to tell if something is still hot after a hot plate has been turned off especially inside of the glovebox. Students should allow ample time for something to cool down, especially if it is cooling down on the hot plate. The group has implemented a second sign to differentiate situations where the hotplate is on and situations when the hotplate is off and cooling down.

A "First Report of Injury" (FROI) form should be filled out for any injury (big or small) that may occur on campus, even if medical treatment is not required. It is recommended that this form be completed within 24 hours of the injury, but it can be completed at any time (the sooner the better). By submitting the FROI form Purdue workers compensation will cover eligible medical expenses related to the incident. Even if no treatment is required, this form should still be submitted. This form can be found at https://www.purdue.edu/ehps/rem/froi/ai.html. (**First Report of Injury (FROI) & Supervisor's Incident Investigation (SII) form)**

4. July 28, 2022: Needle Stab During Titanium Chloride Chemical Uptake

Description: When taking titanium chloride from a stock sure-seal bottle, a graduate student researcher used a long metal needle with some lack of proficiency. The researcher did not secure the front part of the needle and when the needle front came out from the sure seal it fluctuated and punctured the researcher's left-hand index finger. Titanium chloride is a highly toxic chemical.

Immediate Action: The wound was put under flowing water for 15 mins with continuous squeezing of blood from the punctured part. After 15 mins with nearly no blood coming out, the researcher was taken to ROCC for further inspection. The researcher was then monitored for several days, continuously screening the injured finger according to the information of titanium chloride from SDS sheet. There was a follow-up appointment with ROCC after several days of monitoring to confirm the recovery of the wound. A "First Report of Injury" (FROI) form was submitted by the researcher and their PI.

Recommendations:
Students will be extensively trained before they use needles for chemical uptake including instruction on how to prevent long needles from fluctuating. It is also recommended that another researcher is present for chemical uptake for students who lack proficiency.

5. September 2, 2022: Small Cut on Hand from Razor Blade During Sample Case Prep

Description: During the cleaning process of reusable sample cases, a researcher attempted to remove an old paper label using a razor blade. Upon insertion of the razor blade underneath the old label, the blade slipped, and made a small cut on their left hand near their thumb.

Immediate Action: The researcher rinsed their hand with water for a few minutes, then washed the wound and their hands with soap and warm water. A "First Report of Injury" (FROI) form was submitted by the researcher and their PI.

Recommendations: Whenever possible, removal of old labels should be done without the use of a razor blade. Common solvents such as isopropyl alcohol (IPA) can often dissolve adhesive, making label removal easier. When the use of a razorblade is necessary, the user should always use cut resistant gloves and position the blade to face away from their hand to prevent an injury.

6. September 20, 2022: Bubbling from Neck of Schlenk Line Reaction Flask Due to Pressurization During Heat Up

Description: A graduate student researcher was preparing a 25 mL, 3-neck, round-bottom flask for a nanoparticle reaction containing zirconium and sulfur precursors. Upon assembly and purging of the flask, the researcher checked that everything was ready, and they started the heating phase. When the reaction flask reached ~100 °C, some type of decomposition occurred in the reactor and a large amount of gas was released. A yellow fluid from the flask also began bubbling and leaking out of one of the necks of the flask. The researcher realized that they had not opened the PTFE valves in the rubber tubing (thus, leaving the system closed to the Schlenk line) and their reaction vessel has been pressurized.

Immediate Action: When the bubbling began, the researcher immediately turned off the heating mantle. They then did a quick inspection of the rest of their system and noticed that the PTFE valve was closed. They immediately opened the valve.

Recommendations: Students should always double and triple check all connections before beginning heating in any type of reaction system (e.g.: a tube furnace, a Schlenk line reactor, autoclave). A visual diagram has now been posted above the fume hoods for an easy checklist that students should check in addition to the checklist already posted. Furthermore, graduate students will always check their Schlenk line reaction setups with a lab mate before beginning heat up.

Some lessons learned and *general* recommendations:

1. Visit the ChE Safety website (https://engineering.purdue.edu/ChE/aboutus/safety) often and review the information in the safety documents posted. Discuss these in your safety group meetings regularly.
2. Keep PPE on at all times in the lab, and only remove it when you are sure you leave the lab.
3. When dispensing liquid nitrogen from a LN2 tank, it is possible for the valve to get frozen open. Take precautions and prevent the valve from freezing open by wiggling the valve increase/decrease flow during the dispensing process.
4. When working in the vicinity of hot plates, never assume that they are cool. Always check before taking something off a hotplate.
5. If you are not confident with a certain lab procedure, such as the use of a needle for extracting chemicals, be sure to seek assistance.
6. Always check your experimental set up for worn down parts, failing o-rings, and proper ventilation before beginning heat up phase.
7. While working with glass and sharp edges, use cut-resistant gloves whenever possible. For operations where cut-resistant gloves are not viable, take adequate precautions. Point razor blades away from you when using them.
8. Ensure that all chemical waste is always closed and that gas cylinders are capped when not in use.
9. Per Purdue guidelines, whenever an employee is injured (no matter how small the injury is), a "First Report of Injury" (FROI) should be completed and sent through the FROI portal First Report of Injury (FROI) within 24 hours. This will ensure that the University stays in compliance with OSHA and if needed, medical attention related to the incident (even if this is later than the incident) is covered by the Purdue worker's compensation plan. Even if it takes longer than 24 hours to submit the FROI, it should still be submitted.

Incidents and near misses are great tools to learn from previous situations/events. Please continue to report any safety incidents and near misses that occur in your work area (chesafety@purdue.edu); sharing them with everyone in our School will raise safety awareness and prevent similar situations from happening.

Sincerely,

Gabriela
On behalf of the ChE Safety Committee