June 16, 2022

Hello Everyone,

I would like to share with you a few safety incidents reported to me between April 2022 and June 2022. Near misses reported, such as laboratory broken vessel or small, incidental spills are not mentioned in this document. However, please use caution and don't rush through tasks while working in the lab, as this can easily lead to more serious safety incidents. 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 (nagyg@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. April 7, 2022: Minor Cut on Hand

Description: An undergraduate researcher completed lab work for the day and removed their PPE. Before leaving the lab, the researcher saw a tray with clean glassware and decided to put them away in the designated location. While doing this, one of the flasks fell and broke; this resulted in a scratch/minor cut on hand.

Immediate Action: The researcher washed their hands with soap and water and used the first aid kit to apply antiseptic and a bandage. Another researcher in the lab assisted with this task and with cleaning up and disposal of the broken glass.

Recommendations: Keep PPE on at all times until you leave the lab, even for minor tasks and don't rush through tasks. Review broken glass clean-up protocols regularly.
Note: As the researcher was an undergraduate student, the completion and submission of an FROI was not needed in this case

2. May 02, 2022: Microwave Vial Leak from Borehole in Microwave Reactor

Description: A researcher was performing a nanoparticle reaction with the intent of forming indium selenide nanoparticles. The initial conditions were to run the reaction for 5 hours at 250 °C. At ~2.5 hrs, the microwave cut the power due to an error indicating pressure had increased to unsafe levels (the pressure curve on the screen had indicated otherwise). The researcher decided to collect some of the reaction product for characterization and proceed to "finish" the reaction with the remaining product. After ~1 hour, the microwave cut the power again due to a sudden decrease in pressure indicating a vial rupture/leak. Upon opening the microwave lid and pulling out the vial, the researcher found a borehole caused by solid material superheating on the wall of the vial.

Immediate Action: Initial inspection showed that the vial holder was stuck in the vial as the borehole was just beneath where the holder body ends. Upon trying to file down the borehole to remove the vial holder, the vial broke into two pieces. No shards or jagged edges formed as a result. The researcher removed the vial holder and discarded the glass waste. Due to the incident occurring later in the day, the researcher notified the group that the microwave was not to be used until it had been investigated and properly cleaned. The superuser of the instrument investigated it the following morning and found no damage to the microwave. After the spilled/leaked material was cleaned, the group was notified that the microwave reactor was safe to use again.

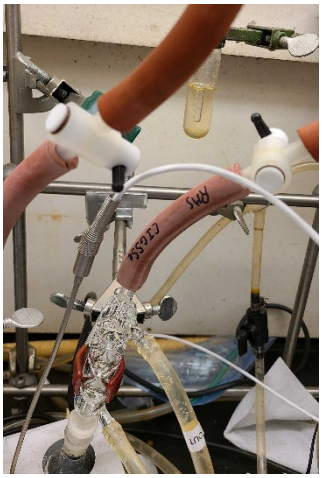
Recommendations:

- When performing microwave reactions, ensure that mixtures are well dissolved/dispersed and that no solid material is trapped on vial walls.
- Ensure solvent levels are above manufacturer recommended minimum

3. May 24, 2022: Heating of Closed Container on Schlenk Line

Description: On a Friday, a researcher set up a multi-day reaction of copper and selenium containing material on the Schlenk line at 65°C, under a flow of argon. The dissolution was in a 15mL round bottom flask with a condenser, rubber tubing, valve, and more rubber tubing forming the connection between the flask and Schlenk line. The researcher let the setup reach 65°C for at least 5 minutes as recommended in their group's checklist before leaving for the weekend. The researcher checked the setup on the following Monday and didn't notice anything unusual. On Tuesday, another researcher in the group noticed that the connecting valves had been left closed, meaning that a volatile solvent had been heating in a sealed container.

Immediate Action: Once the group member alerted the researcher running the experiment of the situation, the researcher asked them to open the valves. The researcher later came in to inspect the setup and found no evidence of a leak or a rupture from over-pressurization.



Recommendations:

- While no over-pressurization occurred on this occasion, this is not the case in all situations. This near miss emphasizes that researchers should not get comfortable with lab procedures. Be sure to follow every step of the SOP and get clarification if in doubt of any steps.
- Whenever running heated reactions or reactions that produce gaseous byproducts outside of a pressure vessel (autoclave), it is vital to ensure that you have proper venting to avoid over-pressurization. Setups should be double checked, especially before leaving them unattended.

4. May 25, 2022: Reactor glass-lined tube rupture due to overheating

Description: The Effi reactor unit in Flex Lab is used for ethylene oligomerization reaction with hydrogen co-feed. This unit is composed of an enclosed hot box containing two furnaces and numerous fittings and stainless-steel tubing. One reactor tube could be loaded onto each furnace. This unit is able to automatically monitor and adjust the temperatures of the reactor tubes inside furnaces and hot box separately, as well as the pressures of the reactor tubes. An improper operational procedure occurred during setup on 5/25 at around 4:00pm. In the SOP, whenever the reactor tube is removed from the furnace and the heat box for sample loading/unloading, the thermocouple for the reactor tube/furnace has to be unplugged due to it being connected with the reactor tube. As this triggers an alarm due to the absence of the thermocouple, to temporarily mute the alarm, a dummy thermocouple is plugged in (as stated in the SOP). Once the sample is loaded and the tube is placed back into the hot box, the dummy thermocouple should be unplugged and replaced with the reactor tube thermocouple.

However, during the operation on 5/25, the researcher did not remember to replace the dummy thermocouple with the reactor tube thermocouple. As a result, the thermocouple that was supposed to monitor the temperature of the furnace was actually measuring the temperature of the hot box. Consequently, the reactor tube was overheated to an extremely high temperature (glowing hot) in order to increase the "furnace temperature" to 350 °C. The hot box temperature never reached 350 °C but stayed at 270 °C, so the furnace was

on full power overnight. The hot box temperature limit was set at 280 °C and did not trigger an overheating emergency shutdown. The situation remained unnoticed until the safety officer discovered it around 10 am on 5/26 and performed emergency shutdown for the system.
As a result of the above error, **the tube reactor ruptured due to extreme temperature** (unknown, but likely in excess of 1000 C) **and pressure** (20 barg). Combined with the pressure/temperature/flow rate reading history, the researcher and the safety officer believe the reactor tube burst at the 1-hour mark (around 4:40pm), which corresponds to a dramatic drop in pressure readings and a short fluctuation in flow rate readings at that time. Fortunately, the rupture happened during the warmup step of the experiment where only N2 was fed into the reactor tube, and the Effi reactor system did not proceed into the next automation step to start feeding H2, ethylene and N2 mixture. This could have potentially caused a major incident resulting in fire and a possible explosion, had either flammable gases been fed into the reactor tube after the rupture occurred, or if the rupture happened after high pressure flammable gas was already present.

Immediate Action: The safety officer performed emergency shutdown procedure at 10 am on 5/26, and informed the researcher responsible for the experiment. Together they carefully inspected the unit and disassembled the reactor from the heat box after everything was cooled down to room temperature.

Recommendations and long-term actions:

- Following the incident, the hot box temperature limit was reduced to 200 °C from 280 °C to ensure the alarm would trigger. (The normal operation temperature of the hot box is 80-180 °C). Alarm limits should not be too much higher than the standard range of operation.
- The SOP will be revisited and revised. The procedure will be modified so that the thermocouple would not need to be unplugged to load the reactor.
- In addition, if due to some special circumstances the thermocouple must be unplugged, a dummy thermocouple will never be used to bypass the alarm system**. It is never acceptable to bypass a safety system in place for the sake of convenience.**
- Reactors or any chemical reaction should not be left unattended during their heat-up phase. Reactors should be monitored at least until they reach steady state.

5. June 4, 2021: Scraped thumb while sealing ampules

Description: A researcher was sealing glass ampules using a propane torch in the fume hood when the propane torch ran low on fuel. Instead of stopping midway to refill the torch, the researcher tried to finish off the sealing. Because of the low flame, the glass cracked with sharp edges instead of softening. One of the edges scraped the researcher's thumb while they were inspecting it for any leaks.

Immediate Action: The researcher quickly removed their gloves, washed their thumb with soap under running water for several minutes, then put on a band-aid. The glass was not chemically contaminated. A "First Report of Injury" (FROI) form was submitted by the researcher and their PI.

Recommendations:

- When using a propane torch for sealing ampules, it is recommended to refill the torch after every sealed ampule.
- If fuel in a torch runs out mid-way through sealing, it is better to allow the glass to cool and then refill the torch than to try to melt and seal glass at lower temperatures.
- Ensure to melt away sharp edges on an ampule to protect the ampule's integrity and to protect yourself from getting cut.
- 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.

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. No one should work alone in the lab; it is good practice to always have at least one other person in the lab to assist each other in case of an emergency/incident.
4. When using an instrument that employs radiative heating, be mindful of heterogeneous solutions which may not heat evenly resulting in superheating of particles. These particles, if in contact with the edge of the reactor vessel, may cause ruptures and leaks.
5. Never heat a reaction containing volatile solvents or one that produces gaseous byproducts without proper ventilation unless it is being carried out in an appropriate pressure vessel with adequate controls.
6. The use of dummy thermocouples should be avoided to prevent unwanted accidental overheating of systems/reactions.
7. It is never acceptable to bypass a safety system in place for the sake of convenience.
8. Reactors or any chemical reaction should not be left unattended during their heat-up phase. Reactors should be monitored at least until they reach steady state.
9. 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.
10. 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.

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; 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