March 01, 2022

Hello Everyone,

I would like to share with you a few safety incidents reported to me between December 2021 and February 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 or near miss, visit the ChE safety page (https://engineering.purdue.edu/ChE/aboutus/safety), fill out the 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. **July 10, 2021: Hastelloy Tubular Reactor Sudden Pressure Release/Explosion (reported February 14, 2022)**

Description: A 25 mL hastelloy tubular reactor was loaded with 10 grams of rare earth magnet, 0.015 g of NaCl and 15 mL of DI water, sealed following the Swagelok cap assembly procedure and placed in a muffle furnace at 250 °C for 18 hours. The reactor was then cooled to room temperature and fixed on a bench vise to be opened. When the top-side cap screw was loosened, the inside pressure caused by reaction products was suddenly and violently released (explosion like), and the cap hit the ceiling. The magnet pulverized in the reactor and spilled on the safety goggles, the mask, and the lab coat of the researcher opening the reactor. The researcher did not suffer any injuries.

The incident was not reported immediately because at that time the method was unlikely to be used in a pilot plant due to the high-pressure constrains. However, the method was revisited recently, which prompted the researcher to report the incident.

Immediate Action: The researcher involved thoroughly washed their face, hands, and any other part exposed to the magnetic powder.

Recommendations:

a. **Always report safety incidents and near misses promptly after the incident**. This will ensure that the particulars of the incident are fresh in the researcher's mind when writing the report.
b. When opening potentially pressurized reactor vessels, always point the cap away from any researchers to ensure that the cap does not injure anyone.
c. When performing pressurized reactions, it is important to account for gasses that may be produced during the reaction that may keep the reactor pressurized even after cooling.
d. If the group is continuing work using this method/reactor type, the safety committee is recommending the following:
    a. add a custom-made pressure relief valve to the reactor, use a larger reactor, or scale-down the reaction to 10% of the original quantities to prevent pressurization in the future.
    b. develop an SOP for operating the hastelloy reactor and consider all potential hazards before starting operation. Add engineering controls to the unit and have everyone in the lab trained on this SOP.

2. **December 09, 2021: Liquid Nitrogen Leak in Transfer Line**

Description: A researcher was performing temperature-dependent current-voltage measurements on a Lakeshore system. These measurements involve using an insulated transfer line to allow liquid nitrogen to flow from a tank into the Lakeshore system. After using the transfer line for ~8 hours, the researcher saw a large

volume of nitrogen gas leaking from the flow control valve of the transfer line. Using cryo-gloves, the researcher tried to close the flow control valve, but that seemed to make things worse. After ~2-3 minutes, liquid nitrogen started leaking (~2-3 ml per sec) from the flow control valve itself.

Immediate Action: The researcher opened the fume hood sash in the room and the door of the lab to maximize air flow into the room. The researcher called another lab member for assistance. With liquid nitrogen still leaking, they decided to remove the transfer line from the liquid nitrogen tank (nitrogen flow cannot be stopped if the transfer line is connected to the liquid nitrogen tank). They then stopped the nitrogen flow and let the transfer line warm up to room temperature.

Results of investigation: Knowing that nitrogen should not have leaked from the transfer line, the researchers disassembled the transfer line to look for possible reasons behind the malfunction. They determined that an O-ring in the transfer line was damaged.

Recommendations:
When working with cryogenic liquids or any chemicals or gasses, it is important to regularly inspect the equipment being used and ensure that any components that may wear down, such as O-rings, are replaced promptly when the first signs of damage are noticed.

3. December 12, 2021: Microwave Vial Explosion in Microwave Reactor

Description: A researcher was running a 24 hour microwave reaction at 250°C. They started the reaction on Saturday evening and the instrument would automatically stop the reaction on Sunday evening. This reaction was similar to many the researcher had done in the past, with the key difference being the use of a new sulfur source, carbon disulfide. He monitored the reaction for 1 hour, by which point the temperature and pressure seemed to have stabilized. Then the researcher left for the evening.

The microwave reactor was programmed to monitor and control pressure and temperature. If pressure exceeded 15 bar, it would automatically lower the temperature such that the pressure would have a maximum of 15 bar. The reactor would automatically shut off if the pressure reaches 20 bar. Despite these safety features being in place the microwave vial exploded and completely shattered 21 hours into the reaction. This explosion took place even though the pressure in the vial never exceeded 15 bar.

Immediate Action: Once the researcher found that the explosion had been contained by the microwave reactor and no hazardous chemicals had leaked, the researcher, with the help of the superuser of the equipment, dismantled and cleaned the shattered glassware using cut resistant gloves. The pressure and temperature sensors on the microwave reactor were checked to ensure that the instrument was detecting the correct pressure and temperature.

Recommendations:

a. While the cause of the explosion is unknown, it is always important to inspect all glassware being used in reactions for defects or cracks before use.
b. When running an experiment with a long reaction, it is important to ensure steady state is reached before leaving the reaction unattended and one should supervise the reaction for longer than usual whenever a new chemical or new procedure is being used.
c. Pressure and temperature sensors on reactors should be regularly inspected to ensure they are operating correctly.

4. December 20, 2021: Small Fire with Insulated Coiled Reactor

Description: A coiled tubular reactor was heated with heating tape (which is always connected to a temperature controller), and was contained by flame-resistant insulation wrapping. During the heating process, done under continuous observation, the researcher observed a glow from within the insulation followed by the presence of a small flame (equivalent to the size of a flame from a small pocket lighter).

Immediate Action: The researcher quickly grabbed the fire extinguisher and put out the flame. From ignition, this fire was present for 15 seconds before it was extinguished. The emergency venting was turned on and the heating was turned off. The system was then cooled and monitored to ensure that no more fires occurred. Additionally, the lab was closed for winter recess, with no experiments conducted until 2022.

Recommendations:

a. When using heating tape, one must ensure that the power supplied to the heating tape is low enough so that the heating tape does not overheat and cause the tape to melt.
b. Heating tape should be carefully installed on reactors such that wires of the heating tape do not cross one another to cause a short circuit (no overlapping).
c. Although the researchers were confident that they can manage the small flame using the fire extinguisher, in case of a fire**, the proper procedure is to call 911 to report the incident, and only after that use the fire extinguisher**. Alternatively, call 911 while someone else is using the extinguisher. In any case, the fire department needs to be informed that a fire extinguisher was used in the lab.

5. January 20, 2022: Cut from Clean Syringe Needle While Using Glovebox

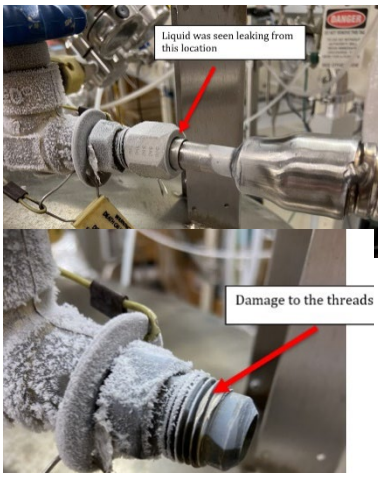
Description: While a graduate student researcher was training an undergraduate student on syringe and needle use in a glovebox, the undergraduate student accidentally stabbed their thumb with the needle.

Immediate Action: The graduate student researcher told the undergraduate student to remove their gloves and wash the wound with water for at least 5 minutes before a band-aid was applied. The undergraduate student was then asked to monitor themselves and immediately report to a hospital if any rash or irritation occurred around the puncture site.

Recommendations:

a. When training new researchers, especially undergraduate researchers, on new techniques, it is best to train them outside the glovebox first, then train them with the limited dexterity of the glovebox.
b. An SOP will be created to train students on how to safely uncap needles and use syringes.
c. Needles should never be re-capped unless absolutely necessary

6. February 7, 2022: Liquid Nitrogen Leak from LN_2 Tank Liquid Connection



Description: When a researcher was filling a Dewar with liquid nitrogen using a liquid transfer hose, they noticed that some liquid was spraying from the threaded connection between the hose and the tank.

Immediate Action: The researcher immediately turned off the valve to stop the flow and investigate. They unscrewed the hose and found that the threads on the liquid nitrogen connection on the tank were damaged. The threads on the hose itself seemed to be in good condition. The researcher posted a sign on the affected tank to prevent other users from using this connection and to notify Indiana Oxygen of the issue. The damage may have occurred while the tank was being filled by the vendor, or it may have occurred in the lab when someone was connecting a hose to the tank.

Recommendations:

a. For any gas cylinder or LN_2 tank, it is important to inspect threaded connections prior to connecting them to ensure that the threads are intact.
b. It is important to pay attention to the way a threaded connection moves while screwing the two halves together. Most regulators should be attached to a tank by screwing in until finger tight followed by $1/8^{th}$ of a turn using a wrench.
c. This near miss also highlights the importance of wearing a face shield when dispensing liquid nitrogen. Had the LN_2 port been pointed towards the researcher, the face shield would have been the last line of defense in preventing LN_2 from spraying on to their face.

7. February 9, 2022: Mass Spectrometer Transfer Line Heater Electrical Overload

Description: While a new mass spectrometer was being set up as a part of a CSTR system, the plug for the transfer line heater of the mass spectrometer was plugged in. Several seconds after the heater was plugged in, sparks and smoke were emitted from the location where the cord was connected to the heater. Heat generated caused damage to the wires and to the clamshell heater. There were no injuries.

Immediate Action: The heater cable was immediately unplugged followed by the main power cable of the mass spectrometer. The instrument was allowed to cool overnight then inspected for damage the following day.

Results of investigation: It was found that the heater was only intended to run at 40 volts and not the 120 volts that the wall outlet supplied. The normal cable for the heater had been modified to be fitted with a standard 120V plug and the heater was likely previously connected to a Variac controller, but there was no instruction or warning label on the instrument describing this limitation.

Recommendations:

a. Properly document and label any changes made to the electrical wiring of equipment so that it is not accidentally used inappropriately.
b. Be aware that most line heaters and heating tape need to be connected to a controller or Variac that allows for the heater current and voltage to be regulated.

8. February 14, 2022: Glass Laceration While Packing Column

Description: While a researcher was assembling a glass chromatography column in the lab, by holding a glass column in their left hand and pushing the column end piece into the empty glass column with their right hand, the glass column broke in the middle and cut the researcher's left hand. The researcher was working alone on the lab in the evening.

Immediate Action: The researcher flushed the wound under tap water immediately to remove any possible glass residue and to get a clearer look at the depth of the cut. The wound was pat dried using paper towel and wrapped tightly using pads and bandages in the first aid kit. The researcher then received medical attention at Franciscan Health Lafayette East Emergency Department, where the wound was eventually treated.

Recommendations:

a. When assembling glass columns, it is recommended to use a clamp to secure them.
b. Check any glass columns and glassware for damage (such as cracks or chips) before use.
c. Do not force in column parts when it is difficult.
d. As was done by this researcher, **if an injury occurs during a safety incident, a First Report of Injury (FROI) must be completed and submitted by the supervisor within 24 hours of the incident (https://www.purdue.edu/ehps/rem/froi/ai.html)** .
e. Please see the information at the following link regarding immediate care treatment options for work related injuries, including lab work injuries like this the laceration resulting from this incident: https://www.purdue.edu/hr/Benefits/WorkersComp/wcimmediatecare.php

Some lessons learned and *general* recommendations:

1. Visit the ChE Safety website (https://engineering.purdue.edu/ChE/aboutus/safety) often and review regularly the information in the safety documents posted. Discuss these in your safety group meetings on a regular basis.
2. Never work alone in the lab. Always have someone with you, who can assist you in case of need.
3. When handling cryogenic liquids, such as liquid nitrogen, always use the proper PPE, including a face shield for tasks as dispensing liquid nitrogen
4. The treaded connections on gas cylinders and LN_2 tanks should be inspected for damage prior to installing a regulator to help prevent leaks.
5. When performing pressurized reactions, it is important to account for gasses that may be produced during the reaction that may keep the reactor pressurized even after cooling.
6. Develop Standard Operating Procedures (SOPs) for hazardous operations/processes in your lab and train everyone affected (operating equipment, or even just being around equipment) before starting operations.
7. Before using any glassware, inspect it for any defects, cracks, or chips. Damaged glassware should never be used.
8. When heating tape or a line heater is being used, it should always be connected to a Variac or voltage controller to prevent it from overheating. Make sure that heating tape is installed properly and has no risk of creating a short (no overlapping of heating elements).
9. If a fire develops in your lab, call 911 and ask for help. Fire extinguishers are to be used by emergency responders. Only attempt to use a fire extinguisher if you are trained on how to use it, and you already called for help.
10. It is important to properly document and label any changes made to the electrical wiring of equipment so that it is not used inappropriately.
11. Ensure everyone in your group knows and follows the incident report protocol. The first bullet point under the "Workplace Injury Information" on ChE safety website (https://engineering.purdue.edu/ChE/aboutus/safety) has useful information on the proper safety incident/near miss reporting procedure. Direct link to the form: Safety Incident/Near Miss Report
12. Report any safety incidents or near misses to the group safety officer and to the safety committee chair, and discuss them in your group meetings. Sharing this type of information is key in increasing safety awareness.

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 the safety awareness and prevent similar situations from happening.

Sincerely,

Gabriela
On behalf of the ChE Safety Committee