Hello Everyone,

I would like to share with you some of the safety incidents and near misses reported to me from March, 2018 to date. Some other near misses were also reported, such as broken glass vessels, damaged laboratory equipment, uncapped needles and unchained gas cylinders found in the lab, etc. The general recommendations at the end of the document has relevant information to prevent these near misses from happening again.

1. **March 27, 2018: Closed reaction vessel overheated**
   Description: An undergraduate researcher connected a 3 neck round bottom flask containing a mixture of DDT, oleic acid, copper (I) chloride, and arsenic (III) sulfide, to the Schlenk line. In order to create an oxygen free environment, the researcher purged the line up to the teflon valve 4 times, and had left the valve open to argon, making sure that the vacuum valves in the Schlenk line was closed. Afterwards, the flask was heated to at 225 °C. Once the temperature reached 225 °C (approximately 7 minutes) the mixture was left unattended to react. After about half an hour, another researcher entered the lab and noticed that the Teflon valve attached to the vessel was closed. He tried to contact the owner of the experiment, but he was unsuccessful as the undergraduate student was in an area without signal, and decided on opening the valve carefully.
   Action: The researcher opened the valve slowly, while wearing appropriate PPE, as well as keeping the sash of the fume hood as low as possible. No damage was done in the experimental fume hood.
   Recommendations: The group will develop a general Schlenk line safety, to include the following recommendations: (i) **Never heat a closed vessel**; (ii) undergraduate students should always be supervised in the lab, and their experimental setup should always be checked by the graduate student mentor before being left unattended; (iii) when running an experiment do not go to a location where the phone has no service; (iv) have regular refresher training sessions on the use of Schlenk lines.

2. **April 17, 2018: Use of defective Liquid Nitrogen Dewar**
   Description: A researcher took a 35L liquid nitrogen Dewar to the Physics loading dock, for refill. The technician tried to fill it up, but liquid nitrogen sprayed out of the fitting on the Dewar, so he immediately stopped filling the tank and sent an email with pictures to the graduate students, postdocs, and professors involved, citing an improper fitting on the fill line.
   As a note: a similar leak had occurred on March 13th – Jason Davenport was notified verbally and the message was relayed to the user of the Dewar; however, no action was taken at that time. The tank was not used again until this incident.
   Action: The researcher contacted the manufacturer (Cryofab, Inc.) to request assistance with addressing the problem. The company stated there was an extra fitting on the fill line that could be removed and was unsure why that fitting was there. After removing the fitting and consulting with the technician, the tank could be filled with LN2 successfully.
   Recommendations: As soon as you are notified about faulty equipment, this must be taken out of service IMMEDIATELY due to safety concerns. Do not add any fittings to any Dewars without consulting with the manufacturer and if you add/remove fittings, make sure you document them (at this time it is still unknown when or why the fitting was added to this unit).

3. **April 18, 2018: Sample overheated in oven**
   Description: Samples of urea+glyoxal, phenol+glyoxal, melamine+glyoxal, urea+glutaraldehyde, phenol+glutaraldehyde were mixed together and placed into the oven. The temperature was set at 180^{o}C, which was 30^{o}C higher than previous experiments of this type. After monitoring the experiment for 15 minutes, the researcher left the lab. A smell was noticed in the lab by others in the building, about 30 minutes after the researcher left.

Action: After a member of staff tried unsuccessfully to reach the safety officer of the lab, they called the Fire Department to come investigate and remedy the situation. The oven was unplugged, and the windows were left open. The vacuum pump was run to remove odor.
Recommendations: Never leave an experiment unattended when performing it for the first couple of times after changing reaction conditions.

4. **May 9, 2018: Pressurized dissolution assembly**
Description: An undergraduate researcher started a dissolution of copper and Indium in dodecylamine and ethanedithiol procedure at 65^{o}C, procedure performed multiple times successfully in the past months. After checking the assembly and letting the graduate student mentor know he had finished, the undergraduate student left for the day, as he was tired. Later, another graduate researcher entered the lab and noticed that the Teflon valve on the assembly was not open. As the system was at 65^{o}C and also producing hydrogen as a dissolution byproduct, the unit was expected to be at higher pressure. The researcher opened the valve and depressurized the system. Fortunately, the solvents used in this dissolution were high boiling solvents and the reaction is extremely slow, thus the pressure build up within one hour was not high.
Action: The graduate researcher depressurized the vessel by opening the Teflon valve immediately after noticing the closed valve on the assembly.
Recommendations: **Never work in the lab when you are tired!** Undergraduate students should always ask their graduate student mentor to check their experimental setup before starting any experiment, especially if it involves working at high heat and/or pressure.

5. **May 31, 2018: Explosion of container with mixed acids solution**
Description: When the safety officer of a research group opened the acid cabinet in the lab, they noticed a strong smell in the cabinet, and that there was a container with ~50 mL of yellow colored solution in the back. Upon further inspection, they found a container labeled with 1:1 sulfuric acid: nitric acid that was uncapped. Debris from the cap were found on the nearby wall, and a large piece of the cap was blown off in the cabinet - the plastic lid seems to have melted or morphed. This container was located next to other glass bottles with acids in them, and luckily none of the other bottles were damaged.
Action: The safety officer moved the solution into the fume hood and isolated it as well as they could. Additionally, the lab PI was contacted, and they identified the person responsible for preparing, using and storing the solution, as neither of them was aware that this mixture was used in the laboratory. They verified the chemical contents of the solution and contacted REM to have the solution safely removed from the laboratory.
Recommendations: The visiting scholar responsible for the incident has had their lab access removed and can only work under supervised conditions. The PI and safety officer emailed the group, emphasizing safe practices such as always wearing PPE, avoiding dangerous chemical work, and discussing new experiments with the PI before beginning. The safety officer will hold a safety review session in the next group meeting, outlining the lessons of the safety incident. Namely, when mixed acid solutions are used (e.g. piranha etch, aqua regia) **only small quantities should be prepared**. Fresh solutions should be prepared every time, and they should be safety neutralized and disposed of immediately after being used instead of being stored.

6. **June 3, 2018: Hexane spillage**
Description: This incident happened as a researcher was trying to fill a glass beaker with hexane from a 4L solvent bottle. During the process, the bottle collided with the flame cabinet's door and fell on the floor, breaking, and spilling around 2L of hexane on the lab floor. No one was injured.
Action: The spill was contained using the spill kit and in accordance with the SDS of hexane. The shattered glass pieces were carefully gathered and placed into the contaminated glass waste container in the lab. The lab floor was cleaned to be free of any shattered glass pieces and liquid.

Recommendations: be alert at all times while working in the lab and use both hands, especially when lifting any heavy chemical containers (e.g.: 4L solvent bottle).

7. June 3, 2018: Dewar implosion

Description: While performing daily cleanup of benchtop area near vacuum Dewars, a researcher accidentally tipped one of the Dewars over the benchtop. The glass insulation inside the Dewar broke into small pieces, and although most of the broken glass pieces were contained by the outer wall of the Dewar, some of the glass pieces landed on the benchtop.

Action: The benchtop was cleaned and all glass pieces were placed in Dewar and labeled appropriately for disposal. There was no injury or other damage.

Recommendations: While working with, or near Dewars, pay extra attention as minor jarring on surfaces can cause enough disturbance to create implosion. As glass can be ejected several feet, keep face out of the way at all times when working with Dewars, and wear appropriate PPE.

8. June 16, 2018: Dewar implosion

Description: Around 9 am on June 16, 2018 a researcher observed high concentrations of NO and ammonia (>3000 ppm – about 10 times higher than normal), on one of the reaction units in the lab. The researcher noticed that the inlet balance nitrogen had run out, causing the other reactant gases to spike in concentration. The researcher, together with the owner of the experiment, observed the deposition of a salt (most likely ammonium nitrate) on the walls of a downstream bubble flow meter. The elevated concentrations of NO and ammonia caused this deposition, which could've plugged the exhaust line. The buildup of pressure could have caused the quartz reactor to explode. Additionally, there was no check valve on the nitrogen inlet line. This could have possibly caused backflow of the reaction mixture to the liquid nitrogen Dewar that supplies the balance nitrogen gas, if the reaction exhaust wouldn't have been under a slight vacuum.

This incident occurred when an individual incorrectly turned a valve on the liquid nitrogen Dewar, causing all of the liquid nitrogen to be released slowly into the room. Students have general access to this nitrogen source to dry glass or blow out powders from the UV-Vis cell in the lab.

Action: Upon noticing the high ammonia concentration in the reactor, the researcher found the owner of the experiment and together they shut down the system. All gas inlets were turned off, except CO_2, which was kept on to purge the system of NO and ammonia. It was decided that this liquid nitrogen Dewar will not be available to other students for general use. A separate purge will be provided for all drying and cleaning purposes.

Recommendations: Check valves will be installed on all gas inlet lines to prevent backflow. Also, a pressure relief valve will be added to the system to prevent the quartz tube from exploding

Some lessons learned and general recommendations:

1. Undergraduate students should be supervised at all times when working in the lab and only be allowed to perform a task without supervision when the mentor graduate student is sure they are trained and confident in performing the task safely.
2. Never work in the lab when you are tired, as this can lead to missing important steps in the performing the experiments safely.
3. Always wear the recommended PPE when working in the lab. Make sure the PPE used provides adequate protection from hazards and the necessary dexterity to avoid safety incidents.
4. Never work alone in the lab. It is recommended that at least two people be present in the lab when experiments are performed.
5. Always follow the standard operating procedures and pay close attention to details.
6. Never leave a full LN2 Dewar unattended on the corridors.

7. Never leave uncapped needles on the bench top or in the fume hood. Dispose of sharps, including razor blades and needles, following Purdue's waste disposal procedures.
8. Never leave a compressed gas cylinder on its cart for storage. Always secure a compressed gas cylinder to a stable designated gas cylinder storage area (wall or bench mounted) with the cap on or a regulator attached. Do not attach a regulator to a compressed gas cylinder while it is still on its cart because a cart is not a stable storage location.
9. When mixed acid solutions are used (e.g. piranha etch, aqua regia) **only small quantities should be prepared.** Fresh solutions should be prepared every time, and they should be safety neutralized and disposed of immediately after being used instead of being stored.
10. Ensure students, including undergraduate researchers, know and follow the incident report protocol.
11. 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