

Lesson Learned

Unsealed cold trap using liquid nitrogen

July 2024

What Happened?

On June 6th, 2024, a graduate student in a campus chemistry laboratory was conducting an experiment in a glove box. After finishing his experiment, the student researcher attempted to remove the organic solvent vapors that were produced during his experiment from the glove box using a vacuum pump. To protect the vacuum pump from the organic solvents, this laboratory frequently uses liquid nitrogen in the dewar of the cold trap setup that is installed between the glove box and the vacuum pump (see Image 1 of a cold trap missing the bottom collection flask and the dewar below). When liquid nitrogen is used in a cold trap, liquid oxygen can potentially be collected in case the trap is not sealed properly. If liquid oxygen were collected in a cold trap setup, it would be visible by a pale bluish color of the liquid collected in the trap. The collection of liquid oxygen poses a hazard due to the possibility of rapid expansion of the liquid oxygen if the temperature increases or the violent reaction with organic compounds like organic solvents.

Image 1:



Image 2:



To set up the cold trap, the graduate student researcher put an O-ring in the groove of the lower part of the glass trap (see Image 2 above showing the collection flask of the cold trap plus the black rubber ring on top), put it under the glass connector leading to the glove box, clamped it tight using a glass joint clamp, turned the vacuum pump on and lowered the cold trap setup into the dewar filled with liquid nitrogen.

The student researcher involved was trained on the standard operating procedure that is used in the laboratory and he wore the appropriate PPE. Once the system was running, he stepped away from it. After about 5 minutes, a co-worker walked by the glove box and noticed a sound that led them to check the cold trap. It was determined that it was not sealed properly. The student researcher, as well as other members of the lab, were notified of this situation immediately. The graduate student researcher turned the vacuum pump off, removed the liquid nitrogen, and inspected the liquid collected in the trap. The liquid was clear and colorless, and the graduate student researcher deduced that no liquid oxygen was collected. He ensured the connection was properly sealed and started the vacuum pump again. This time, all connections were sealed properly, and the graduate student researcher was able to finish his experimental procedure.

Direct Cause:

The vacuum setup was improperly sealed, causing a potentially dangerous situation when liquid nitrogen was used in a cold trap setup.

Root Cause of the Incident:

The student researcher followed the lab-approved setup technique for the cold trap; however, he did not check if the vacuum lines were sealed properly once the vacuum pump was turned on, indicating that he was not following the SOP for this procedure.

Primary factors that contributed to the incident:

The student researcher had performed this procedure many times before, so he assumed the cold trap was sealed. He did not follow established procedures or check that the cold trap and tubing were appropriately sealed before beginning the procedure.

What steps can be taken to prevent this type of incident from occurring again:

- Update the SOP to set up a cold trap using liquid nitrogen as follows:
 - Assemble all parts of the cold trap, but do not close the two parts of the cold trap with a glass joint clamp yet. Turn the vacuum on, make sure the two parts of the glass trap stay together just through the vacuum application, and then close it tightly with the glass joint clamp. Then, lower the trap into the liquid nitrogen.
 - Review and train all graduate student researchers and other researchers on the updated SOP.
 - Wear appropriate PPE for this procedure.
 - Inform others in the lab that you are using a cold trap utilizing liquid nitrogen.
 - Have emergency equipment (shield) ready when using liquid nitrogen in a cold trap.

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