Standard Operating Procedure

Task: Dealing with Liquid Oxygen
Date: 5/13/2013
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Background

- Oxygen has a melting point of 54 K (–219 ˚C) and a boiling point of 90 K (–183 ˚C). It is a cryogenic liquid between these temperatures with an expansion ratio of 1:861.
- Liquid oxygen is a serious hazard in any laboratory that utilizes liquid nitrogen. The primary hazard presented by liquid oxygen is the explosion or detonation of a glass apparatus that contains liquid oxygen. This can be due to either rapid expansion tied to the large expansion ratio as well as violent reaction between liquid oxygen and organic solvents to produce highly sensitive peroxides.
- Any vessel cooled by liquid nitrogen poses a potential risk for liquid oxygen condensation, because liquid nitrogen has a lower boiling point (77 K, –196 ˚C) than liquid oxygen and therefore vessels cooled by liquid nitrogen can potentially condense oxygen from air. Safety controls must be in place to prevent air from entering a system cooled by liquid nitrogen. These include ensuring the vacuum system is leak-free and in working order, never cooling a closed system with liquid nitrogen (including systems under static vacuum), and utilizing cryogens that are incapable of condensing liquid oxygen (e.g. dry ice / isopropanol).
- This SOP is written assuming liquid oxygen is suspected in a vacuum trap assembly. Identification of liquid oxygen and appropriate actions are introduced first, followed by tips for avoiding condensation of liquid oxygen.

Training Requirements:

- Laboratory safety training

Potential Hazards:

- Explosion
- Lacerations from exploding glassware

Special PPE Requirements:

- Blast shield
- Face shield

Materials Needed:

- Vessel suspected of containing liquid O_2
- Blast shield
- Face shield

Procedures:

- Identifying liquid oxygen:

  - ***The presence of any liquid in a trap just after removal from a liquid nitrogen bath is cause for alarm,*** because most chemicals are solids at liquid nitrogen temperatures.
  - A pale blue color is ***sometimes, but not always,*** observed for liquid oxygen. However, do not rely on this hard-to-see color.
  - If the volume of material in the trap exceeds the volume of solvents evaporated, this may indicate condensation of a gas such as liquid oxygen.
  - Note that argon is also condensable at liquid nitrogen temperatures.
  - If liquids are present in the glass trap when the cooling dewar is lowered, return the trap to its original state if possible (i.e. raise the cooling dewar)

- If liquid oxygen is suspected in a system:
  - **Do not panic.**
  - Try to return the system to its original cryogenic state (vacuum pump on, cooling dewar raised).
  - Inform researchers in the lab of the possible danger, and have non-essential personnel leave the lab immediately. Utilize a blast shield and clearly label the area as dangerous.
  - ***Contact your PI*** *and the group safety officer* to plan next steps. The general approach typically involves (a) securing the area (see above), (b) identifying the source of the air/oxygen, and (c) resolving the problem while maintaining active vacuum. Once the leak has been stopped, the liquid oxygen will pump off slowly, even at liquid nitrogen temperature. The trap can be carefully lowered to check the liquid level after ca. 1 hour; the liquid oxygen should have evaporated substantially if the leak was resolved. Some possible points to discuss with PI and safety officer(s) include:
    - Are there any known problems with the pump or trap assembly? Is there an initial suspected leak point?
    - Have there been any recent changes to the pump or trap assembly, such as repairs or changed connections?
    - Does the trap appear to be operating? How does it sound?
    - Is there a local electrical problem or a building-wide power outage?
    - Is there organic solvent in the trap?
    - If the liquid nitrogen level in the cooling bath was low, it is possible the trap temperature is higher than expected, such that the trapped organic solvents are thawing. Try adding more liquid nitrogen to the bath to see if the trapped material freezes.
  - *Do not remove the trap from the line or glove box; letting the trap warm is extremely dangerous and could result in an explosion due to rapid expansion or violent reaction with organics!* Only in the event that the leak cannot be identified or fixed, or if there is no spare pump to replace a broken pump, should the trap be allowed to thaw. In this case, the trap is left in the cooling dewar behind a blast shield and with the hood sash down (if possible). The trap is allowed to warm very slowly as the liquid nitrogen evaporates; hopefully the oxygen will evaporate slowly, rather than rapidly expanding. Do NOT handle the trap until it is certain that all liquid in the trap has evaporated.

- After resolving the problem, consider the following:
  - What went wrong? Was a leak identified and corrected?
  - How can liquid oxygen be prevented from condensing next time?

- Tips for preventing liquid oxygen condensation:
  - ***Never*** **leave a system under static vacuum in a liquid nitrogen bath.** A small leak could lead to condensation of liquid oxygen. *An active vacuum is required when cooling with liquid nitrogen* so that any oxygen that leaks into the system is efficiently removed.
  - If adjustments are made to a pump apparatus which utilizes a liquid nitrogen-cooled trap, communicate those changes to labmates. This can help to ensure that the adjustment being made is safe while alerting users of the pump that the procedure for setting up the trap has changed.
  - Use dry ice / isopropanol baths for long-term trap utilization (e.g. overnight), or when working with solvents with high melting points (e.g. water, benzene).
  - Regularly check your line for leaks. Use the vacuum gauge to determine that the Schlenk line has pumped down to its normal state. Do not use cryogens on a Schlenk line that is not properly pumping down.
  - Have a contingency plan in place for power outages. Loss of power to the vacuum pump while the traps are being cooled with liquid nitrogen is a common source of liquid oxygen. Don't leave traps unattended unnecessarily and drop all traps promptly if a power outage occurs.
  - For shared traps, such as those on gloveboxes; communicate with labmates about dropping the trap (i.e. if you set it up but someone is using it; explicitly transfer the responsibility to drop the trap).

References and Related SOPs:

- Vacuum Traps SOP
- Degassing SOP
- Vac Transfer SOP