Standard Operating Procedure

Task: Working with Lecture Gas Bottles on a Schlenk Line

Date: 5/13/2014

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Background:

Lecture bottles are small compressed gas cylinders, typically 12-18 inches long and 2-3
inches in diameter. These small cylinders are typically used in applications where large
quantities of gases are not used.

Training Requirements:

- Lab safety training
- · Changing gas cylinder training
- Working with CO training (if working with CO)

Potential Hazards:

- Inhalation of potentially toxic and corrosive gases
- Injuries from failure of pressurized gas lecture bottle

Special PPE Requirements:

Carbon monoxide (CO) detector must be worn if working with CO

Materials Needed:

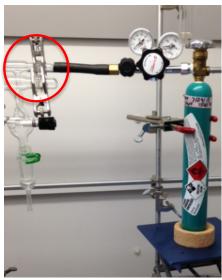
- Schlenk line
- Cylinder regulator CGA 170 or 180
- Crescent wrench
- Teflon Tape (optional)

Procedure:

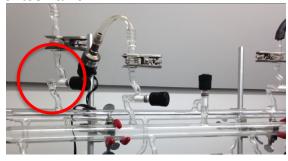
- 1. Set up vacuum manifold and traps and pump down the vacuum line.
- 2. Connect the lecture bottle regulator to the lecture bottle. Review the SOP "Changing Gas Cylinders" for more detailed information on regulator installation and use.
 - Regulator information. Note: there are two CGA numbers for lecture bottles! Ensure that you are using the correct regulator! In general, CGA-170 is designed for non-toxic, non-corrosive gases. CGA-180 is for corrosive or toxic gases. However, most lecture bottles can be purchased with either regulator type.
 - A plastic washer is required to achieve a proper seal between the regulator and the lecture bottle in some cases. Locate and inspect the washer for scratches or other defects before use. Spare Parts: https://store.mathesongas.com/cga-washers-teflon/
 - Align the regulator and cylinder threads and hand-tighten the nut while supporting the weight of the regulator.

- Use a crescent wrench to fully tighten the nut (usually another 1/4-1/2 of a turn).
- If the regulator has not been used recently or you are unsure of the pressure setting
 — turn the regulator dial all the way to the left so that it will be set to its lowest pressure
 delivery setting.
- A comment about Teflon tape
 - Different laboratories follow different guidelines for the use of "Teflon tape", which
 is a thin, pliable film of poly(tetrafluoroethylene). In some groups, Teflon tape is
 used frequently when installing regulators, while in others this practice is not
 allowed. Some regulator manufacturers warn against the use of Teflon tape.
 - Those in favor of Teflon tape point to improved seals and more reliable operation when applied properly.
 - Those against the use of Teflon tape argue that properly maintained cylinders and regulators should make a tight metal-metal connection without leaks and warn that improper application of Teflon tape can stress the regulator components.
 - When using Teflon tape: Make sure that any old bits of tape are completely removed from the threads of the regulator and the cylinder. Use a plastic object to gently scrap off tape if needed (do not use razor blades, syringe needles, or other metal tools to avoid damaging the delicate threads). Apply one (and only one) complete wrap of new Teflon tape to the exposed threads (usually the nut on the regulator), working opposite to the direction of tightening. Use the thinnest tapes possible and do not wrap multiple times: thick layers of Teflon tape will put strain on the regulator as it is tightened.
- 3. Connect the regulator outlet to the line with a short length of tubing. The lecture bottle can be connected directly to the gas manifold or it can be connected to the gas manifold through one of the 24/40 ports. To connect to the gas manifold directly, remove the o-ring joint end cap of the gas manifold. Using a hose barb to o-ring joint adapter, connect the short rubber tuning from the regulator to the line, clamping the connection. To connect to the gas manifold via a 24/40 port, use a 24/40 to hose barb adapter to attach the short length of tubing from the regulator. This method carries a risk of accidentally exposing the contents of the lecture bottle to vacuum. Be sure that the Kontes valve leading to the vacuum manifold is shut when manipulating the lecture bottle. The Kontes valve leading to the gas manifold will be used to admit gas from the lecture bottle to the manifold.

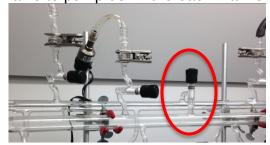




- 4. Check that the gas manifold is connected to a bubbler that is properly venting towards the back of the hood. Check that the vacuum is properly venting into the hood. This is absolutely critical for toxic or hazardous gases.
- 5. If working with a toxic or flammable gas, ensure gas cylinder is in a well ventilated area and a detector is on your person, if applicable.
- 6. Perform a leak test as follows. Shut the outlet needle valve on the regulator. Turn the regulator dial counter-clockwise to zero delivery pressure. Open the lecture bottle valve to pressurize the regulator. Close the lecture bottle valve. Note the starting pressure, then wait 3 minutes and see if any pressure drop occurs. If a pressure drop occurs, troubleshoot the regulator connection. If a new tank is being used, note the starting pressure, the date, and your initials on a piece of tape and place on the tank.
- 7. Shut the special gas inlet Kontes valve



8. Open the crossover Kontes valve to pump down the back manifold.



- 9. Shut the crossover valve. Open the lecture bottle valve and adjust the regulator to 1-5 atm. Shut the lecture bottle valve. Open the needle valve to fill the back manifold with gas, then shut the needle valve. Note: Subjecting the regulator to dynamic vacuum may damage it. An alternative is to purge the system of air and moisture using a "T"-purge valve (See Aldrich Technical Bulletin AL-151 in References and Related SOPs).
- 10. Repeat steps 8 and 9 until three cycles are done. On the third cycle leave the lecture bottle valve open.
- 11. Maintain only a very light bubble when working with lecture bottles. Once the back manifold is filled with gas use normal Schlenk techniques to perform your chemistry. *Note: For low pressure lecture bottles, a continuous bubble may not be possible. Know the expected pressure of your lecture bottle and use small volume manifolds if needed.*
- 12. When finished, shut the main valve and allow the pressure in the regulator to dissipate through the bubbler before shutting the needle valve.
- 13. Pump down the back manifold by opening the crossover valve to remove the gas from the back manifold.
- 14. Shut the crossover, and then open up the special gas inlet (usually nitrogen) to purge out the back manifold with nitrogen.
- 15. Allow the vacuum pump to run for at least 10 minutes before dropping traps as some gases might condense. Active vacuum should remove any condensed gases.
- 16. Shut off special gas (nitrogen) flow, and remove the regulator, replacing the end cap.
- 17. Remove the regulator from the lecture bottle, cap the bottle, and place back in storage.
- 18. Purge out the regulator by opening the needle valve and running nitrogen through the regulator for 30 seconds.

References and Related SOPs:

- Changing gas cylinders SOP
- Working with CO SOP
- Oregon State University Lecture Bottle Safety Fact Sheet: http://oregonstate.edu/ehs/sites/default/files/pdf/si/lecture_bottles_si.pdf
- Aldrich Technical Bulletin AL-151: http://www.sigmaaldrich.com/content/dam/sigmaaldrich/docs/Aldrich/Bulletin/al_techbull_al151.pdf
- http://www.ilpi.com/inorganic/glassware/regulators.html