

Standard Operating Procedure

Task: Dispensing solvent from the solvent purification system

Date: 5/13/13

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Background:

- Purification of organic solvents using a low temperature drying column has supplanted distillation as the method of choice for removal of water. Most columns are packed with activated alumina, and the solvent is passed over the desiccant using a positive pressure of argon or nitrogen.

Training Requirements:

- Users must be trained by the Solvent System Czar before first use

Potential Hazards:

- Inhalation of toxic fumes
- Fire or explosion if sparked (do not use cell phones or other electronic devices while dispensing solvent).

Materials Needed:

- Appropriate solvent receiving flask (usually a "Straus flask")

Procedure:

- Do not use the solvent system until trained by Alex or the Czar.
- Full guidelines are located on a clipboard attached to the solvent system
- General dispensing guidelines:
 1. Attach an **oven-heated** flask to the desired solvent port using **Teflon (Krytox) grease** and a green Keck clip. Grease must be used when attaching ground glass joints, but no other grease is suitable for solvent dispensing. **Use only Krytox!**
 2. The top valve should be pointing to vacuum. The bottom right valve is then turned downward to evacuate the flask. Allowing the flask to cool under vacuum. Evacuate for at least 5 minutes. Once cooled, turn the top valve to argon, refilling the flask with argon, and then turn the valve back to vacuum to evacuate for 1 minute. Complete 5 evacuate/refill cycles. When you are done, the top valve should be pointing to argon.
 3. Turn the bottom right valve to "closed" position (**static vacuum**: neither Ar or vacuum). To dispense solvent, **slowly** open solvent valve. When the desired amount of solvent has been dispensed, close solvent dispensing valve. Slowly turn the other valve to Argon to blow the last of the solvent into the flask, and close the Teflon stopcock on your flask.
 4. Remove flask from port, flushing with Ar for a few seconds to remove residual solvent. Replace stopper on port, turn valve to vacuum. Evacuate the manifold for at least 5 minutes. Open ballast for the last minute before shutting off vacuum.
- GlassContour (part of Pure Process Technologies) contact info:

- Phone: (603) 598-0691
- Directions for refilling solvent kegs are located in the “Glass Contour Solvent System Manual” that should remain connected to the clipboard
 1. Only users trained to refill kegs should do so
 2. Users should assist in at least one refill before doing so themselves

Dispensing Solvent

Do not use the solvent system until trained by Alex or the Czar.

Instructions for dispensing into flasks under air.

1. Check that the solvent purification system is not in use by another researcher:
 - a. No flasks attached to the ports
 - b. All argon/vacuum valves (with metal piece) pointed to vacuum
 - c. Argon tank pressure gauge reads 8-12 psi
2. Turn on the vacuum pump (bottom right). Turn valve on main steel vacuum manifold to evacuate manifold.
3. Attach an **oven-heated** flask to the desired solvent port, using **Krytox grease** on the ground glass joints and a **green Keck clip** to support the flask.
4. Turn the bottom right valve downward to evacuate the flask. Make sure the Teflon stopper is open. Allow the flask to cool under vacuum, at least 3 minutes.
5. Check that the valves on the purifying columns are all open. **The bottom valves on the dichloromethane columns should be closed when not in use.**
6. While gently supporting the flask by (gloved) hand, slowly turn the top valve from vacuum to argon, refilling the flask with argon. Then turn the valve back to vacuum and evacuate for 1 minute. **Complete 5 refill/evacuate cycles.**
7. After the last evacuation cycle, close the bottom right valve to place your flask under **static vacuum**.
8. Turn the top valve from vacuum to argon. To dispense solvent, **slowly** open the solvent valve. The static vacuum will “pull” the solvent into the flask. When the desired amount of solvent has been dispensed (no more than 3/4 capacity of flask), close solvent dispensing valve. While supporting the flask by (gloved) hand, slowly turn the bottom right valve to Argon, blowing the last of the solvent into the flask and providing an inert atmosphere.
9. Close the Teflon stopcock on your flask.
10. Remove flask from port, flushing with Ar for a few seconds to remove residual solvent. Replace stopper on port, turn valve to vacuum.
11. Evacuate the manifold for 5 minutes. For the last minute, open the ballast on the pump to clean out any residual solvent. Then close the vacuum manifold valve and turn the pump off. If using dichloromethane, close the bottom valves on the purification columns.

Instructions for dispensing into flasks under vacuum.

1. Check that the solvent purification system is not in use by another researcher:
 - a. No flasks attached to the ports
 - b. All argon/vacuum valves (with metal piece) pointed to vacuum
 - c. Argon tank pressure gauge reads 8-12 psi
2. Turn on the vacuum pump (bottom right). Turn valve on main steel vacuum manifold to evacuate manifold.
3. Attach a Teflon-sealed, **previously evacuated** flask to the desired solvent port, using **Krytox grease** on the ground glass joints and a **green Keck clip** to support the flask.

4. Turn the bottom right valve downward to evacuate the volume between the Teflon stopper and the solvent outlet. Evacuate for 3 minutes.
5. Check that the valves on the purifying columns are all open. ***The bottom valves on the dichloromethane columns should be closed when not in use.***
6. While gently supporting the flask by (gloved) hand, slowly turn the top valve from vacuum to argon, refilling the flask with argon. Then turn the valve back to vacuum and evacuate for 1 minute. ***Complete 3 refill/evacuate cycles.***
7. After the last evacuation cycle, close the bottom right valve to place the volume above the Teflon stopper under ***static vacuum.***
8. Open the Teflon stopper on the flask, such that the whole system is under static vacuum.
9. Turn the top valve from vacuum to argon. To dispense solvent, ***slowly*** open the solvent valve. The static vacuum will “pull” the solvent into the flask. When the desired amount of solvent has been dispensed (no more than 3/4 capacity of flask), close solvent dispensing valve. While supporting the flask by (gloved) hand, slowly turn the bottom right valve to Argon, blowing the last of the solvent into the flask and providing an inert atmosphere.
10. Close the Teflon stopcock on your flask.
11. Remove flask from port, flushing with Ar for a few seconds to remove residual solvent. Replace stopper on port, turn valve to vacuum.
12. Evacuate the manifold for 5 minutes. For the last minute, open the ballast on the pump to clean out any residual solvent. Then close the vacuum manifold valve and turn the pump off. If using dichloromethane, close the bottom valves on the purification columns.