Vacuum Ovens

Fisherbrand Vacuum Ovens and Cold Trap

Tool Type: Material Processing, Drying, Annealing
Manufacturer: Fisherbrand Oven and Welch Vacuum Pump
Location: Elings Hall 2440

Principal Scientist	Training and Operations Lead
Morgan Bates	Zachary Nett
morganbates@ucsb.edu	zjnett@ucsb.edu

About

Vacuum ovens are essential for various processes in synthetic polymer chemistry, particularly for drying non-volatile solids and polymer samples, as well as for material processing such as thermal annealing. They are especially useful for annealing materials that undergo self-assembly (e.g., block copolymers) and can be used for freeze-drying polymers from solvents like benzene.

To ensure proper functioning and longevity, vacuum ovens should be handled with care. Any spills or volatilized material inside the oven should be cleaned immediately to prevent contamination or damage. Additionally, liquid nitrogen should be used to condense volatiles, protecting the vacuum pump from potential damage.

User training and reservations through FBS are mandatory for use.

Vacuum Oven Standard Operating Procedure

1. Acquire Liquid Nitrogen:

- If there's liquid nitrogen (LN2) available in the dewar in E2 2120, you may use it, but please refill after use. If you’re coming from another lab, bring your own LN2.

2. Inspect Vacuum Manifold:

- Check the vacuum manifold for cracks. If any are found, report this finding to a BioPACIFIC MIP staff member, do not proceed to use the vacuum oven.

3. Prepare the Vacuum Trap:

- Ensure the vacuum trap is dry, then assemble it with an O-ring and clamp. Tighten the clamp finger-tight only to avoid cracking the glass. Never tighten while under vacuum.

4. Prepare the Vacuum Oven:

- Ensure both purge and vacuum knobs are closed. Place samples inside the vacuum oven chamber, latch the door shut, and write your name, date, and time on the whiteboard.

5. Start the Vacuum Pump:

- Close all Teflon knobs on the vacuum splitter, then turn on the vacuum pump. Ensure the vacuum gauge reads <100 mTorr.

6. Connect the Vacuum Pump to the Cold Trap:

- Open the Teflon knob on the vacuum splitter connecting the vacuum pump to the cold trap (0.5 to 1 rotation is sufficient). Ensure <100 mTorr is maintained on the gauge.

7. Set Up the Cold Trap:

- Fill a dewar with liquid nitrogen and lower the cold trap into it. Place color-coded foam insulation pads around the trap to ensure a tight fit. The trap should be lowered so the clamp touches the foam.

8. Connect the Cold Trap to the Vacuum Oven:

- Open the vacuum knob (4-5 complete rotations) connecting the cold trap to the vacuum oven chamber. Ensure the vacuum gauge reads <100 mTorr.

9. Heat Samples (Optional):

- To heat samples, set the desired temperature on the temperature knob and turn on the heater. Verify the correct temperature has been reached using a thermometer if needed.

10. Monitor the Dewar:

- Refill the dewar with liquid nitrogen in the morning and before leaving at the end of the day.

11. Check for Clogs:

- If vacuum pressure drops slowly (partially clogged) or rapidly (fully clogged), the trap may be clogged. Disassemble and allow the trap to dry before reassembling to ensure proper drying of your samples.

12. Access Samples During Operation:
    - To add or remove samples, close the vacuum knob and open the purge knob to equalize pressure before unlatching the vacuum chamber.
13. Re-evacuate After Sample Access:
    - After sample access, close the purge knob and reopen the vacuum knob (4-5 rotations). Minimize re-evacuations to prevent oxygen condensation in the trap, which is extremely dangerous.
14. Ending Operation:
    - Before finishing, check the vacuum gauge to ensure <100 mTorr. If the pressure is higher, liquid oxygen may be present. Place a blast shield around the cold trap, remove samples, close the Teflon knob connecting the vacuum pump to the cold trap, and open the knob connecting the vacuum trap to the vacuum oven. Allow the system to thaw for 24 hours and warn others to avoid the vacuum ovens.
15. Remove Samples:
    - Close the vacuum knob, open the purge knob, and remove your samples. Never open the purge knob while the system is under vacuum.
16. Shutting Down the Cold Trap:
    - Raise the cold trap out of the liquid nitrogen and close the Teflon knob connecting the vacuum pump to the cold trap.
17. Vent the System:
    - Open the knob connecting the vacuum trap to the vacuum oven.
18. Turn Off the Vacuum Pump:
    - If no other vacuum oven is in use, turn off the vacuum pump and open the middle Teflon valve on the splitter to vent the entire system.
19. Handle Remaining Liquid Nitrogen:
    - Pour any remaining LN2 from the dewar back into the 10 L dewar.
20. Clean the Cold Trap:
    - Allow the cold trap to thaw, then rinse with acetone and leave it to dry in a disassembled state. Clean up thoroughly for the next user.

Identifying and Handling Liquid Oxygen

Liquid oxygen has a distinctive light blue color, resembling blue Gatorade. If you observe a light blue liquid when raising the cold trap out of the liquid nitrogen (step 16), follow these steps immediately:

1. Place the cold trap back into the dewar and position a blast shield around the dewar.

2. Vent the system:

- Close the Teflon knob that connects the vacuum pump to the cold trap.
- Quickly open the vacuum valve that connects the cold trap to the pre-purged vacuum oven.

3. Allow the system to thaw for 24 hours and notify other lab users to avoid the vacuum ovens during this time.

If liquid oxygen is detected after venting, immediately place the cold trap back into the dewar, set up a blast shield, allow it to thaw for 24 hours, and warn other lab users to stay clear of the vacuum ovens.
