

Purpose

This SOP provides direction of the use and maintenance of the vacuum pump. Freeze drying (lyophilization) is a water removal process used to preserve perishable materials, to extend shelf life or make the material more convenient for transport. Freeze drying works by freezing the material, then reducing the pressure and adding heat to allow the frozen water in the material to sublime.

Materials

1. Vacuum grease
2. Soft cloth
3. Cleaning solvent (ex.: 70% ethanol)
4. Mild non-abrasive detergent

Operation

1. Complete the log sheet associated with this equipment.
2. Refer to SOP.005 for operation of the vacuum dryer before starting the freezer dryer.
3. Starting the freeze dryer
 - A. Do not leave the unit unattended when starting it. The unit will alarm when the temperature and/or vacuum are out of range. This condition must be addressed immediately.
 - B. The display panel provides step-by-step instructions for operating the unit.
 - C. All valves should be in the closed position.
 - D. The condenser should be dry and empty
 - E. Check the vacuum pump oil; it should be colorless and within the required range.
 - F. Press the RUN/STOP button 2 times. The system will power up in the correct sequential order.
 - I. The condenser should get below -40°C within 10 minutes.
 - II. The vacuum should go to 500mT within 1 minutes.
 - III. If these parameters are not met, an alarm will sound and "VACU >500mT, after 10min" will flash on the lower part of the screen.
 - IV. If the parameters are met, "READY FOR FREEZE-DRYING" will flash at the top of the screen.
4. Adding material for freeze drying
 - A. The material should be frozen in the flask or vial before attaching to the freeze dryer. The flask or vial should be tilted on its side.
 - B. Attach the flask or vial to the vacuum valve using a 3/4" to 1/2" adapter. The adapter should be greased.
 - C. Open the valve slowly to apply vacuum to the flask.
 - D. The vacuum may rise as flasks are added causing the "READY FOR FREEZE-DRYING" message to disappear. The message will reappear when the vacuum

reaches the proper level. Wait for this message to reappear before adding additional material.

5. Removal of material
 - A. To remove the flask or vial from the system, the vacuum in the container must first be removed.
 - B. Turn the valve stem past closed the release the vacuum. The vacuum is released through a small opening in the valve stem.
6. Defrosting the freeze dryer
 - A. Check the condenser after each cycle. If there is any frost or ice on the condenser, defrost the unit.
 - B. Remove any unfinished product.
 - C. Press the "DEFROST" BUTTON. The compressor will direct hot gas to the coils and will cycle on/off until all ice is melted.
 - D. Wipe the coils and chamber with a soft cloth.
7. Shutting down the freeze dryer
 - A. Drain any fluid using the drain on the back or side of the unit.
 - B. Flush the chamber and coil appropriately and wipe dry with a soft cloth. A cleaning solvent may be necessary to remove any residual materials.
 - C. Refer to SOP 001 for the procedure for shutting down the vacuum pump.
8. Maintenance
 - A. As needed (**TO BE PERFORMED BY RESEARCH LAB**):
 - I. Clean all spills, remove liquids from condenser chamber via drain or using a soft cloth
 - II. Clean lid and gasket using soft cloth and a mild non-abrasive detergent
 - III. Clean and apply vacuum grease to the vacuum valves when they become sticky or hard to turn.
 - B. Monthly
 - I. Check all rubber components for hardening or deterioration; replace as necessary.
 - II. Clean the condenser chamber lid using a soft cloth and a mild non-abrasive detergent.
 - III. Clean the exterior surfaces of the freeze dryer using a soft cloth and a mild non-abrasive detergent or a stainless-steel cleaner.
 - C. Annually
 - I. Clean the system condenser with a soft brush and a vacuum. This will require removing the cover of the equipment.
9. In the event of a problem, contact the Spokane lab services group (spok.labservices@wsu.edu, 509-358-7621)

Failure to use the equipment properly may result in a misuse fee to the last researcher or lab to use the equipment.