STANDARD OPERATING PROCEDURE

Use this form to document the Health and Safety information associated with the procedure.

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| **Procedure Title** | | Freeze dryer operator | | | | | | |
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| **Dept** | ABE | |  | **Bldg/Rm** | 4210 Sukup |  | **Supervisor** | Adina Howe |

**Procedure Overview** (brief description of the project)

To instruct in the operation of the FreeZone Benchtop Freeze Dryer system.

**Health and safety information for materials used (**briefly describe the hazards associated with the materials and/or equipment **OR** document your hazard assessment in Section I)

This process inherently contains two significant hazards, vessels under vacuum, which could implode, and extreme cold, which could cause frostbite. The contents of the samples to be freeze dried are a potential third hazard as they could become aerosolized as they sublime.

**Hazard Control Measures**

Please select which type of lab coat, eye protection, and hand protection will be used (Lab coat, eye and hand protection, and closed toe/heel shoes must be selected as required by Section D of the ISU Laboratory Safety Manual.)

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|  | Latex gloves |  | Insulated gloves |  | Face shield |  | Respirator |
|  | Nitrile gloves |  | Safety glasses |  | Lab coat |  | Fume hood |
|  | Neoprene gloves |  | Vented goggles |  | Apron |  | Biosafety cabinet |
|  | Vinyl gloves |  | Splash goggles |  | Dust mask |  | Glove box |
|  | Fully enclosed shoes | | |  | Flame resistant lab coat | | |

***Other Control Measures***

Be extra sure to wear safety glasses when working with objects under positive or negative pressure. The setup for freeze drying hazardous samples must include a cold trap on the vacuum line before the material reaches the vacuum pump.

**Methods** (Include step by step instructions detailing the process or attach this document to an existing method.)

Read freeze dryer’s manual before first use to become familiar with the machine.

Read EHS’s SOP on vacuum pump usage to become familiar with precautions and hazards with vacuum pumps.

**Prep work:**

1. Pre freeze vacuum flasks at -80°C

2. Check oil level of the vacuum pump. It should be between MIN and MAX. If the oil level is less than an inch (25.4 mm) above MIN, add oil to proper level.

3. If oil shows cloudiness, particles, or discoloration, drain the pump, and replace with fresh oil. Refer to chapter 7, section I of the manual.

4. Check that the vacuum pump power switch is turned on (on is default)

**To Begin a Run:**

1. The previous user should have left the machine in a clean, dry state. Please ensure that the following have been performed:

a. Remove the drying chamber from the collector chamber lid and using a soft, lint-free cloth or paper towel, wipe the port gasket(s) and sealing surfaces of the drying chamber and collector chamber lid to remove any dirt or contaminants that could cause a vacuum leak.

b. Remove baffle and wipe with a soft cloth or paper towel to remove any remaining moisture.

c. Ensure that the collector chamber and drain line are free of water. Place the drain hose in a suitable container to collect the condensate from the collector chamber. Insert the quick connect drain fitting into the quick connect drain coupling located on the left-hand side of the Freeze Dryer. Note: Freeze Dryer (Collector or Vacuum) will not start if moisture is detected in the drain line.

d. After completely draining the system, disconnect the quick connect drain fitting from the quick connect drain coupling.

e. Wipe the interior of the collector chamber with a soft cloth or paper towel to remove any remaining moisture.

2. The machine may have been left disassembled to facilitate drying. To reassemble:

a. Reinstall baffle in collector chamber. The arrow faces towards the front.

b. Reinstall the drying chamber on the collector chamber lid. Note: Vacuum grease is NOT required on the drying or collector chamber lid gasket to obtain a proper vacuum seal.

c. Replace the lid on top of the drying chamber.

d. Check that each sample valve is closed or in the “vent” position (bevel facing away from the sample port).

3. To start the machine, on the home screen, press AUTO, then press START

a. The COLLECTOR will start, then the VACUUM when the device reaches -40°C.

b. Labconco Freeze Dry Systems that are clean, dry and without samples attached should reach a vacuum of 0.133 mbar within 10 minutes and should achieve an ultimate vacuum of 0.040 mbar within 18 hours when the refrigeration is operating.

c. Having the vacuum set at 0.200 mbar is said to be more efficient.

4. Prepare samples for drying

a. See Freeze Dryer sample preparation SOP for initial sample preparation.

b. Number sample tubes and lids to ensure the same lid will go on each tube after the run.

c. Uncap samples and place them, along with their lids in a frozen vacuum flask.

d. Return prepared samples to freezer until freeze-dryer is ready.

i. It is important to keep everything as cold as possible for as long as possible; this can be difficult in the summer.

5. Once the vacuum has reached 0.133 mbar (or equivalent), attach one pre-frozen flask containing a pre-frozen sample to a sample valve on drying chamber using insulated gloves.

a. Note: Samples must be fully frozen – Improper freezing can result in damage to the machine.

b. Connect a pre-frozen sample to a sample valve on the drying chamber using an adapter. Turn the plastic valve knob to the “VACUUM” position to open the valve, which connects the attached sample to system vacuum. The bevel on the knob should be positioned toward the sample port to apply vacuum to the sample.

c. Before adding another sample, allow system vacuum to return to 0.133 mbar or lower. Any combination of valves and sample sizes may be utilized at one time provided that the system vacuum and collector temperature remain sufficiently low to prevent melting of the frozen sample.

i. Do not freeze dry more than four flasks per run in the summer. If more samples are added, the machine cannot keep them cold enough and your samples will boil – which is bad for your sample and the machine.

d. It is recommended that only one or two flasks are removed from the -80°C freezer at a time when adding them to the machine to ensure samples are the correct temperature. This is especially important in the summer.

e. Place paper towels under the flasks in the summer to collect condensation.

**When the run is finished:**

1. When all the frost has disappeared from the outer surface of the sample container and no cold spots can be detected by handling the container, the sample is nearly dry. To be certain of low final moisture content, dry the sample for several hours past this point.

2. Before stopping the machine, release the vacuum by turning the plastic knob on a valve that has no sample attached to the open position. If the samples are manure, it is recommended that the operator back away from the machine for a few seconds at this point. Wait for the sound of air flowing to stabilize.

3. Go to Home screen, press VACUUM, then STOP to turn the vacuum pump OFF. Press COLLECTOR, then STOP to turn the refrigeration system OFF.

4. To remove a container after drying is complete, turn the plastic knob on the valve to the “VENT” position, which closes the valve and vents the container. The sample container may now be removed. In the vent position the bevel on the valve knob should point away from the sample port.

**Defrosting:**

1. The hot gas defrost function can be used to speed up the defrosting process. Defrost can be activated as follows: Go to Home screen, press COLLECTOR button, press Defrost Options, then press START.

2. To turn off Defrost: Go to Home screen, press DEFROST button, press STOP. The defrost function will turn off automatically if the collector temperature reaches +60°C or if defrost has been running for 2 hours.

3. Remove the drying chamber and set it aside.

4. Remove the baffle and rinse it well in the sink. It can be washed with lab detergent if it has significant deposits on it or is particularly smelly.

5. If rapid defrost is desired, pour warm water over the collector coil. Do not allow the liquid to enter the vacuum port on the upper rear wall of the chamber.

6. Place the drain hose in a suitable container to collect the condensate from the collector chamber. Insert the quick connect drain fitting into the quick connect drain coupling located on the left-hand side of the unit.

7. Flush the collector chamber with water and wipe chamber dry.

8. Disconnect the quick connect drain fitting from the quick connect drain coupling.

9. Repeat steps 5-8 several times to remove all the ice and rinse the coils.

10. Wipe down the collector chamber and coil with a clean, dry cloth.

**Clean up:**

1. Wipe down all surfaces with a damp cloth including the drying chamber and sealing surfaces.

2. Place the drying chamber on top of the collector chamber

a. The baffle and the lid of the drying chamber may be left to the side to facilitate air drying of the equipment.

3. Wipe up any water that may have spilled on the counter or floor near the machine.

4. Wash the vacuum flasks and lids with laboratory soap (not dish soap). Let them air dry before replacing them in the -80°C freezer or cabinet.

Notes:

1. Do not change the vacuum ballast setting from closed (the “o” symbol) during operation

a. If you know what you’re doing, do it between runs.

2. Disconnect the quick connect drain fitting before starting the vacuum pump. Failure to remove the fitting will result in a large vacuum leak.

3. Utilization of acid requires immediate cleaning and neutralization after defrosting or physical damage to the collector chamber and collector coil will result.

4. Do not attempt to chip ice from the collector coil as damage may occur to the coil.

5. Never attempt to start the vacuum pump when there is liquid in the collector chamber. This could result in damage to the vacuum pump.

6. As needed, clean lid and gasket using soft cloth, sponge or chamois and a mild, non-abrasive soap or detergent.

7. Check the collector chamber for condensed or frozen solvents and dispose of appropriately. Completely empty the collector chamber before the next run or before attempting to start the vacuum pump.

8. If sample valves leak or stems become difficult to turn, pull the stem out of the rubber valve body and apply a thin coat of vacuum grease to the stem. Reinsert the stem into the valve body.

**Waste Disposal Procedures**

As per the SDS for the sample’s contents.

**First Aid Procedures**

**For frostbite**: Gently rewarm frostbitten areas: Soak frostbitten fingers, toes, or other extremities in warm water — 105 to 110 F (about 40 to 43 C). If a thermometer isn't available, test the water by placing an uninjured hand or elbow in it — it should feel very warm, not hot. Soak for 20 to 30 minutes or until the skin becomes its normal color or loses its numbness. For the face or ears, apply a warm, wet washcloth. Don't rewarm frostbitten skin with direct heat, such as a stove, heat lamp, fireplace, or heating pad. This can cause burns. (Source: Mayo Clinic)

**For cuts**: If you sustain a minor cut or puncture to your fingers/hands, wash your fingers/hands with soap and cold water in the lab sink for 15 minutes. Once the bleeding stops, apply antibiotic ointment and a band-aid. If injuries are more significant, apply pressure and seek immediate medical attention and/or call 911.

**Spill/Release Containment, Decontamination, and Clean Up Procedures**

As per the SDS for the sample’s contents in the case of spill. See protocol for system cleaning instructions.

**Using Substances Requiring Special Procedures?** No  Yes

(If Yes; identify authorized personnel, designate a use area and specify specialized safety precautions here. Refer to Section B in the ISU Laboratory Safety Manual for details.)

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| **Written By** | Lorien Radmer |  | **Date** | 4/17/24 |
|  |  |  |  |  |
| **Approved By** |  |  | **Date** |  |

(PI or Lab Supervisor)

1. **HAZARD ASSESSMENT**

Use the hierarchy of controls to document the hazards and the corresponding control measure(s) involved in each step of the procedure.

Consider *elimination or substitution* of hazards, if possible.

***Engineering Control(s):*** items used to isolate the hazard from the user (i.e. fume hood, biosafety cabinet).

***Administrative Control(s****):* policies/programs to limit the exposure to the hazard (i.e. authorizations, designated areas, time restrictions, training).

***Required PPE***: indicate PPE including specific material requirements if applicable (i.e. flame resistant lab coat, type of respirator or cartridge).

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| **Task** | **Hazard** | **Engineering Control(s)** | **Administrative Control(s)** | **Required PPE** |
| Applying vacuum to a flask | Flask implosion – broken glass projectiles, samples contaminating lab |  | Training, visual inspection of glassware | Safety glasses, lab coat, gloves |
| Working with frozen samples and flasks | Frostbite from contact with frozen samples or flask | Thermal gloves | Training | Insulated gloves |
| Freeze drying sample with hazardous material | Sublimation of hazardous material – varies based on material | Cold trap | Training | Safety glasses, lab coat, gloves |
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1. **TRAINING RECORD**

Use the following table to record the training associated with this Standard Operating Procedure.

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| **Print Name** | **Signature** | **Trained By** | **Date** |
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