OPERATING INSTRUCTIONS FOR THE LABCONCO FREEZE DRYER SYSTEM AND STOPPERING TRAY DRYER

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

CHEMISTRY PROGRAM

TALLAHASSEE, FLORIDA

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OPERATING INSTRUCTIONS FOR THE LABCONCO FREEZE DRYER SYSTEM AND STOPPERING TRAY DRYER

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) outlines the use, precautions, and maintenance of the laboratory's Labconco 12-L freeze dryer system and stoppering tray dryer. The procedure is applicable to all users of this instrument. Improper operation of the freeze dryer can easily result in severe damage to the unit and loss of samples. Refer to the User Manuals for detailed programming instructions.

2. SUMMARY OF THE METHOD

Freeze drying is a process of drying frozen materials without going through the liquid phase. This process, called sublimation, takes a solid material directly to a gas phase without first becoming a liquid. An example of sublimation is dry ice which converts from a solid to a gas (sublimes) at atmospheric pressure at temperatures above the freezing point of carbon dioxide. Frozen water sublimes when under vacuum below 6.11 mbar. Freeze drying is useful because it avoids the application of heat, commonly used in most other drying techniques.

- **Water-Based Samples Only** -The LABCONCO 12-L freeze dryer and stoppering tray dryer are only suitable for drying aqueous-based samples. Samples containing strong acids or organic solvents may damage the instrument.
- **Programing** -The LABCONCO freeze dryer and tray system is programmable for walk-away operation. The instrument may also be used in Manual Mode, which should be used primarily for trouble shooting or diagnostics.
- **Flexibility** - The tray dryer allows the user to simply place sample vials, bottles, or jars directly into a large 3-tier cabinet for freeze drying.
- **Sample Heating and Cooling** - The tray dryer is equipped with a heater and cooler from 40 to -40 °C. This allows samples to be frozen in the freeze dryer instead of requiring that the samples be pre-frozen. This feature can save time when the User needs to start the Freeze-dry process before the samples are pre-frozen. In this case, the instrument will automatically go through a sample freeze program and then start the freeze-dry program.
- **Isolation Valve** – The stoppering tray is equipped with an isolation valve so that Users can access their samples without completely shutting down the freeze dryer unit.
- **Stoppering Feature**– The tray is equipped with a stopping feature to seal samples with a fill-gas or under vacuum.

3. APPARATUS AND EQUIPMENT

3.1. Labconco FreeZone 12 Liter Freeze Dryer, 230V model

3.2. Labconco FreeZone Stoppering Tray Dryer, 230 V model

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Figure 1. Labconco 12-Liter Freezer Dryer and Tray

3.3. Labconco Isolation Valve

3.4. Edwards Pump 3-L

3.5. Large ChemWipe Towels.

4. REAGENTS AND CHEMICALS

4.1. DI Laboratory Reagent Water – for wiping down and cleaning unit.

4.2. Inland 45 Vacuum Pump Oil – for Freeze Dryer Unit

4.3. Lexol 542 Heat Transfer Fluid – for Tray Dryer Unit

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the specific analytical method SOP for sample collection, preservation, and handling procedures. Sample conditions will impact drying time; you can expect (1) samples with larger surface areas will dry faster; (2) thick samples will dry slower; (3) large sample size will dry slower and may overload the system.

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6. FREEZE-DRYING PROCEDURE

Three checklists are provided below including a **Pre-Operational, Operational, and Post-Operational** list. Please go thought the checklists carefully. More detailed, advanced procedural instructions for the Freeze Dryer and Tray Automated Program are provided in the Instrument User's Manuals. Please review the User's Manual for additional details.

Note: Failure to follow the procedure may result in serious damage to the vacuum pump and loss of samples. New users should receive hands-on training from an experienced staff member before attempting to use the instrument.

6.1. Pre-Operation Checklist for Freeze Dryer Base and Tray Units

- Samples must be pre-frozen or frozen in the temperature-controlled tray dryer prior to starting the freeze dry process.
- Wipe the interior of the Collector Chamber and Tray Drier with a ChemWipe paper towel to remove any residual moisture or particles. The collection chamber must be clean and dry.
- Wipe the interior of the Tray Drier
- Inspect the instrument - Report any instrument corrosion or damage to your Supervisor.
- Place the sealing lid over the collection chamber.
- Check the collector chamber drain hose to ensure it is free of moisture and the drain plug is securely installed.
- Check the Isolation Valve Position between the Freeze Dryer and Tray Unit. The valve is closed when the handle is horizontal and vertical when the valve is open. For most applications the valve should be open, unless the User needs to isolate the sample chamber from the freeze dry unit.
- Close and Lock the Tray Unit Door.
- Turn the Stopper Control Knob on the Tray Unit to "Lower" position.
- Turn the Vacuum Release Knob on the Tray Unit to "Closed" position.
- Check the Auto Program settings on both the Freeze Drier and Tray Units (See Table 1).

Table 1.

General Instrument Conditions:	
(Actual conditions required will vary depending on the sample matrix.)	
Condition	**Setting**
Sample Temperature	– 30 °C is adequately cold for most samples
Collector Temperature	-50 °C
Vacuum Set Point	~0.12 mBarr
Time Typically Required	< 24 hours

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6.2. Operation Checklist for Freeze Dryer Base and Tray Units

- Push the Freezer Dryer Base and Tray Unit main power switches "On". The main switches are located on the left side panels. The refrigeration unit should be set to "Manual" program and the tray Unit set to "Auto" program.
- Push the REFRIGERATION button to turn ON the refrigeration unit on the Freeze Dryer Base in "Manual" mode. The refrigeration compressor will come on and start cooling the collector chamber coil to less than –40 °C. The base unit needs the refrigeration started in "Manual" mode in order for it to reach temperature while the Tray Dryer program processes its first segment (Do not start the base unit's refrigeration button using automatic mode). When the Tray unit is ready to dry the samples, the vacuum pump will start automatically.

  [In Full Manual Mode make sure the Freeze Dryer coils are less than -40 °C before attempting to turn on the vacuum system. When the system vacuum is below 0.133 mBar, the green LED will no longer flash and the unit is ready for Freeze drying. Under most conditions full manual mode will not be used.]
- Record information in the Sample Log Notebook.
- Open the tray door and place samples onto trays.
- Place tray thermocouple sensors near samples to monitor temperature. Good thermal contact is needed to insure correct temperature readings.
- Close the Tray Door and Lock.
- Open the Isolation Valve between the Freeze Dryer and Tray Unit (handle will be vertical). You should see the vacuum pressure on the Freeze Dryer rise immediately, but within a few minutes it will recover and return to 133×10^{-3} mBar.
- Verify the Freeze Dryer and Tray Unit Programs (See General Instrument Conditions below).
- Start the Freeze Dryer Program by pushing the "Run" Button on the Tray Unit Controls.
- At the end of the programmed cycle, the Tray Unit will automatically stop, but the Freeze Dry unit will still be running.
- Stoppering (for special applications) – To use this function see User Manual for details.
- Gas Backfilling (for special applications) – To use this function see User Manual for details.
- Once the program is complete - Close the Isolation Valve (handle will be horizontal when closed).
- Break the vacuum of the Upper Tray unit by turning the Vacuum Release valve to "open". The base unit will still be cold and under vacuum.
- Open the tray door and remove the samples.

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- Complete the sample information in the Sample Log Notebook.
- After all samples are removed, the User has two options:
  - Option 1 - Add more samples and start another drying batch.
  - Option 2 - Shut down the Freeze Dryer Base Unit.

6.3. Post-Operational Freeze Dryer Base and Tray Shut Down

Once the samples are dry the freeze dryer must be shut down, defrosted, and cleaned.

- Turn OFF the main power to the Upper Tray unit (the bottom Freeze dry unit will still be running).
- Release the Freeze Dryer vacuum by opening the Isolation valve or pulling the collection chamber drain plug.
- Immediately press the VACUUM Switch on the control panel to turn the vacuum pump OFF.
- Next press the REFRIGERATION Switch to turn OFF the refrigeration system.
- The system must now be defrosted and cleaned.
- Pull the collector chamber drain hose out from the panel of the unit and remove the drain plug. Place the hose in a suitable container to collect the condensate off the collector coils.
- Press the DEFROST switch on the control panel and a green LED will come on indicating it is ON. Allow the unit to operate until the condensate is defrosted from the collector coil.
- Any ice plugs may be lifted out manually, without force.
- Press the DEFROST switch again on the control panel and the green LED will turn OFF.
- Rinse the collector chamber with DI water and wipe the chamber dry with a Chemwipe.
- Replace the collection chamber drain plug and push the hose back into the chamber.
- Turn OFF the main power to the Freeze Dryer base on the left-hand side of the cabinet.
- Please be considerate of other users and leave the freeze-dryer clean and in good working order.

-CAUTION -

- Do not attempt to chip ice from the condenser coils.
- Never attempt to start the vacuum pump with liquid in the collector chamber.

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7. ROUTINE MAINTENANCE SERVICE

- Check pump oil level (H=high; M=medium; L=low). Note level in logbook. If low, unscrew cap, add oil, and screw cap back on. If oil in pump looks cloudy, it will need to be changed. Change the pump oil every 6 months (or every 500 hours of operation). Consult Owner Manual for details.
- Check coolant level in the Stoppering Tray unit. Add Lexol 542 as needed.
- Record all service information in the maintenance logbook.

8. SAFETY/HAZARDOUS WASTE MANAGEMENT

Refer to the laboratory Health and Safety Plan and Waste Management procedures listed in the Reference section for details.

8.1. **CAUTION:** Always use appropriate protection in the laboratory!

8.2. Gloves and lab coats should be worn when handling all samples.

9. REFERENCES

9.1. Labconco Corporation, FreeZone 6, 12, and 18 Liter Freeze Dry Systems, User's Manual, Document #7403400, Rev E, ECO E091.

9.2. Labconco Corporation, FreeZone Stoppering Tray Dryer, User's Manual, Document #7353900, Rev B, ECO E139.

9.3. Florida Department of Environmental Protection, *Health and Safety Plan*. Jan. 2020.

9.4. Florida Department of Environmental Protection, Standard Operating Procedure **OG-001** *Safety and Waste Management Practices in the Organic Analysis Groups.*

APPENDIX OF CHANGES

07/09/2020 Updated change to Chemistry Program, corrected typos. and updated References.

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