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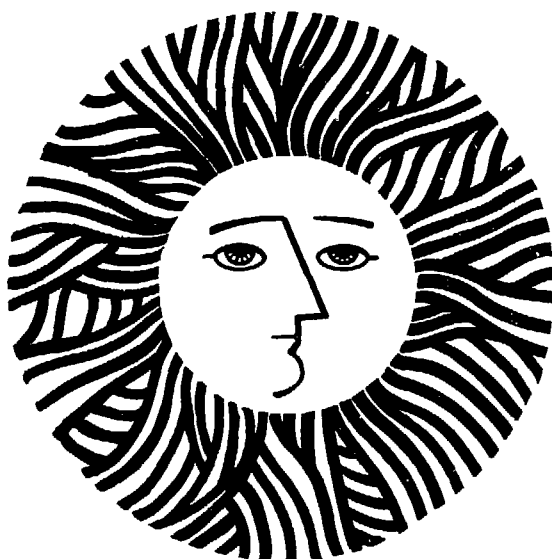
ENERGY & ENVIRONMENT DIVISION

OPERATING INSTRUCTIONS FOR LBL RADON MEASUREMENT FACILITIES

J. G. Ingersoll

June 1980

MASTER



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INTRODUCTION

This Manual is intended for users of the radon-measuring facilities of the Radon Project of the Building Ventilation and Indoor Air Quality Program at Lawrence Berkeley Laboratory. The Manual comprises three parts.

Part 1 sets out the steps involved in collecting, transferring, and counting radon. Radon is collected from a material that has been sealed in a chamber or from an air sample contained in any two-port canister. The chamber or canister is connected to the collection apparatus, and helium is blown through the system to carry the radon from the chamber or canister to a liquid-nitrogen-cooled glass-wool trap, where the radon is adsorbed onto the glass wool. The radon is then transferred from the trap to a scintillation flask (Lucas cell). Next, a photomultiplier tube is used to count the alpha particles emitted as radon and its daughters decay in the scintillation cell. A numerical calculation can then be performed to determine the radon activity that was initially present in the chamber or canister.

Part 2 describes the calibration of the transfer system and of the Lucas cells in the counting system. The systems should be calibrated periodically as a means of quality control.

Part 3 outlines the maintenance procedures for the facility. This part also deals with troubleshooting in the event of vacuum failures in the transfer or calibration system. Failure to achieve or maintain a vacuum, though infrequent, is the most common problem encountered in the facility.

Part 1

THE RADON-MEASURING FACILITY

I. Collection System

A. Sampling

1. Emanation chamber. To seal a sample of a radon-emanating material in a chamber, simply place the sample in the chamber, attach the lid, and tighten the bolts on the top of the chamber. NOTE: The two valves on the chamber must be closed. Record on the record sheet under "Start Collection" the time when you placed the lid on the chamber.
2. Two-port canister. To draw radon-containing air into a two-port canister, attach a pump to one of the ports. When you have pumped in sufficient air to produce at least five air changes in the container, stop the pump and close the ports. Record on the record sheet the time the pump was stopped.

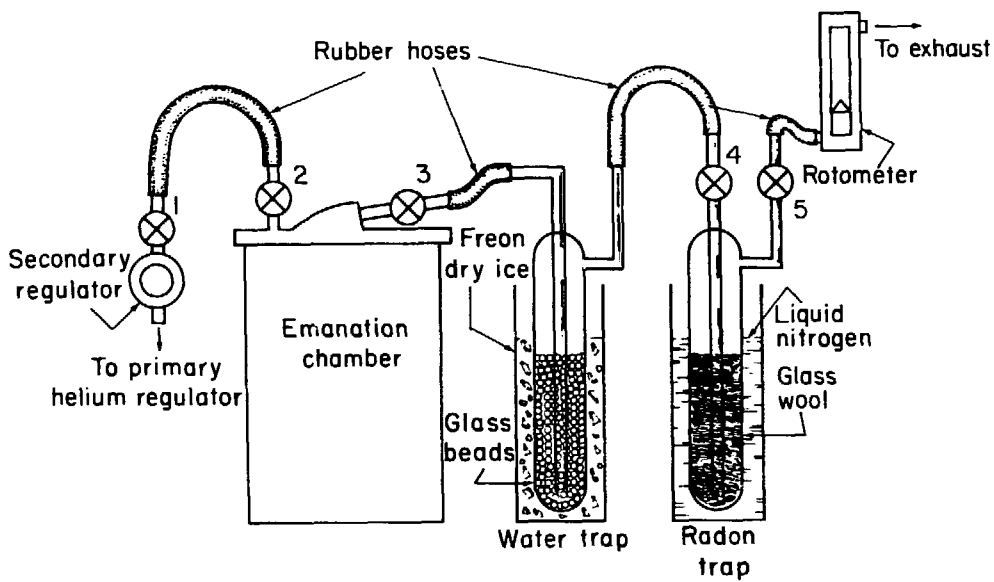
B. Operation

All of the instructions given below are applicable whether you are using emanation chambers or two-port canisters.

1. Prepare one Freon dry-ice Dewar and one liquid-nitrogen Dewar for each chamber to be processed. As many as six chambers may be done simultaneously. Since room-temperature Freon bubbles furiously when dry ice is added, and will

immediately overflow a small container, it is best to put the Freon into a large Dewar with pieces of dry ice of about 1/2 inch in diameter so that it becomes chilled, and then to pour the chilled Freon slowly into a smaller Dewar containing more dry-ice chips.

2. Place a water freeze-out trap in each Freon dry-ice Dewar and a glass-wool trap in each nitrogen Dewar. Make sure that the level of the glass beads in the water freeze-out trap and of the glass wool in the glass-wool trap is slightly below the level of the Freon and liquid nitrogen in the Dewars. Add more nitrogen if necessary.
3. While keeping the glass-wool traps immersed in liquid nitrogen, evacuate the vacuum bulbs by attaching the vacuum line to each bulb in turn, rotating the stopcock so that the small opening faces the outlet and allowing a vacuum of approximately 50 millitorr to be achieved.
4. Connect the emanation chamber, glass-bead trap, glass-wool trap, and rotameter to the system as shown in Figure 1. All stopcocks and valves should be closed. Record glass-wool trap number(s) on the record sheet.
5. After making sure that all secondary regulators are closed by turning the knobs fully counterclockwise, bring the outlet pressure of the primary regulator up to 50 - 60 psig.



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Fig. 1. Collection System.

6. Open each secondary regulator so that its outlet pressure is approximately 3 psig.
7. Open valve (1). Check regulator pressures and adjust if necessary; you should maintain the pressure levels given in steps 4 and 5.
8. Open stopcock (4) so as to equalize the pressures in the freeze-out and glass-wool traps. Then open valve (3).

Complete instructions 9, 10, and 11 for each chamber, finishing one chamber before going to the next.

9. Open stopcock (5).
10. Open valve (2) carefully, so as to begin the flow of helium, and watch the rotameter closely.
11. Wait several seconds for helium to enter the chamber. When the float in the rotameter starts to move, adjust the flow of helium to approximately 1 l/min. (The calibration should be marked on each rotameter.) Be careful not to open valve (2) too much or the rotameter float will immediately jump above 1 l/min. If the carrier gas moves too quickly through the glass-wool trap, radon may slip past without condensing on the glass wool. Under no circumstances should the flow exceed 2 l/min.
12. Record under "Flush Start" on the record sheet the time when helium starts to flow through the last chamber.

13. Allow helium to flow through the chambers for a time sufficient to produce approximately five air changes.
14. Close valves (1) and stopcocks (4) and (5). Record the time on the record sheet under "End Collection."
15. Remove the rubber hoses from the glass-wool traps, keeping the traps immersed in liquid-nitrogen Dewars.
16. Disconnect the emanation chambers and open them to the atmosphere so that the samples are not exposed to the helium any longer than necessary.
17. For each glass-wool trap:
 - (a) Connect the central leg to the vacuum system using the valve and stem near the pump, evacuate to 50 millitorr and turn the stopcock so that the small opening faces upward; a significant loss of vacuum indicates that a leak has occurred and that the results are not reliable.
 - (b) Connect the offset leg and proceed as in (a).
 - (c) Turn the stopcock of the offset leg so that the large opening faces upward and evacuate the trap to 50 millitorr.
18. Remove glass-wool traps from the liquid nitrogen and allow them to warm to room temperature. A heat gun may be used to speed up this process. Be careful not to heat the traps

too long, as too much heat can cause any water in the traps to vaporize, increasing the risk that moisture will contaminate the transfer system. Moisture contamination could lead to a vacuum failure in the transfer system (see "Troubleshooting").

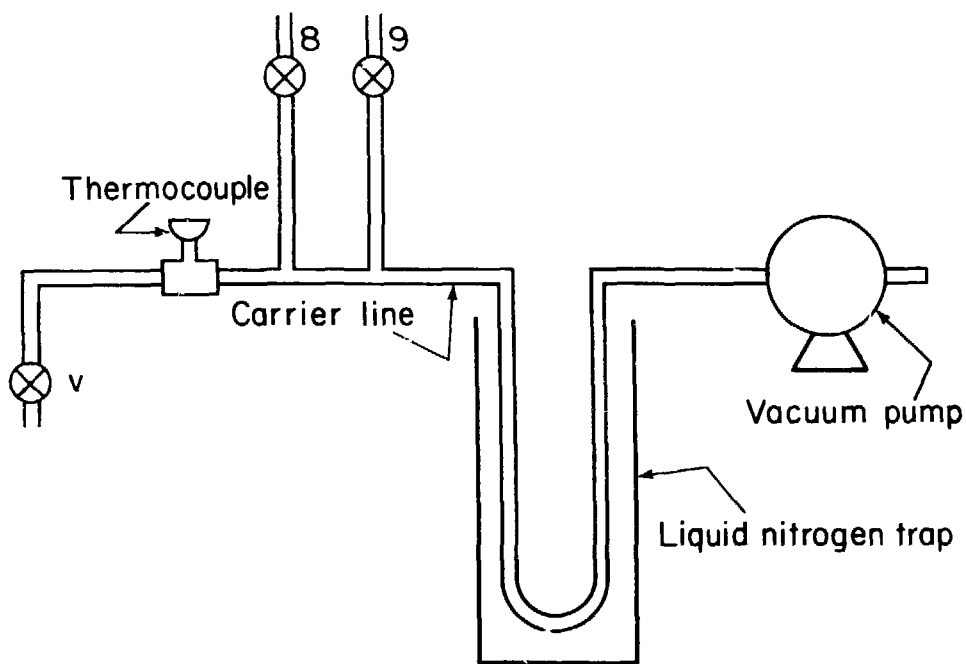
19. A second set of chambers may be prepared for collection while the glass-wool traps from the first set are warming up. This time may also be used to seal new samples in the emanation chambers just flushed.

20. After the glass-wool traps have reached room temperature, transfer the radon into Lucas cells and count it. (See "Transfer System" and "Counting System.")

II. Transfer System

A. Start-up

1. Place a large Dewar over the water trap on the carrier line, as shown in Figure 2, and fill it with liquid nitrogen.
2. Check all valves in carrier lines to see that they are closed.
3. Switch on pump, making sure that the fan on the noise suppression box is plugged in. Plug thermocouple junction into socket closest to pump. If all parts are functioning properly, you should be able to achieve a pressure of 10-15 millitorr in the carrier lines. The pressure in the



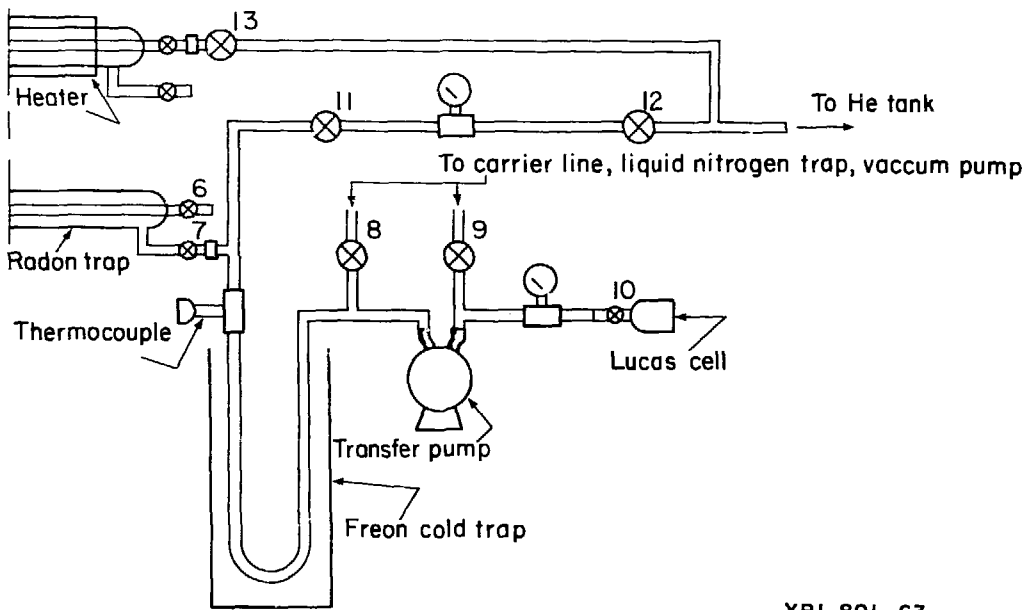
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Fig. 2. Carrier Line.

transfer and calibration systems should be no more than 50 millitorr.

B. Operation

1. Connect room-temperature glass-wool trap to the transfer apparatus with a 1/2"-long piece of 3/16" x 3/32" amber tubing, as shown in Figure 3.
2. Connect a Lucas cell to the apparatus as shown in the figure.
3. Put a new rubber hose in the transfer pump. The hose should be replaced daily. Moreover, if, in the same day, a low-activity sample is to be transferred after a high-activity sample, the hose should be changed between the runs. It is much easier to squeeze the hose into the pump head if both ends are connected to the copper tubing and the line is evacuated first.
4. Prepare a tall Freon dry-ice Dewar and place it under the U-trap so that the trap is immersed in Freon.
5. Connect thermocouple junction to socket in transfer system.
6. Open valves (8), (9), and (10). Evacuate system to approximately 50 millitorr. If it won't pump down, try turning on the transfer pump for a couple of seconds. This allows gas trapped in the pump to escape.



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Fig. 3. Transfer System.

7. The pressure in the helium-inlet line should be kept at about 6 psig (read gauge with valve (11) closed and valve (12) open). Close valves (8) and (9) and stopcock (10). Open valves (11) and (12).
8. Turn on transfer pump until gauge on Lucas-cell side of apparatus reads about 0 (atmospheric pressure). Turn off transfer pump. Close valve (11). Take care not to over-pressurize the Lucas cells.
9. Repeat steps 6 through 8 twice to flush out system.
10. Close valves (11) and (12); then open valves (8) and (9). Turn on transfer pump for a few seconds to flush out any gas trapped in tubing in the pump. Wait for pressure to drop below 50 millitorr.
11. Turn on transfer pump. Close valves (8) and (9). Open stopcock (7). Note whether the pressure registered by the thermocouple changes. If it changes more than 50 millitorr, the vacuum has not been maintained in the trap and the results should be disregarded.
12. Open valve (11) and immediately close it. Open valve (12) until pressure in reservoir between valves (11) and (12) returns to 6 psig. Close valve (12).
13. Note how much the pressure rises on Lucas-cell side. It should rise about 5" - 6" Hg, then stop. You can control how far it rises by varying the pressure in the helium

reservoir. If the pressure increase is greater than this, the run should be discontinued, since it will not be possible to transfer all of the trapped radon to the Lucas cell.

14. Wait until vacuum gauge by Lucas cell stops moving.
15. Repeat steps 12 through 14 until pressure on Lucas-cell side is almost 0. Just before the needle on the vacuum gauge hits the peg, close stopcock (10).
16. Turn off the transfer pump. Remove the Lucas cell. Record the time on the record sheet under "End Transfer."
17. Slowly open stopcock (6). Remove the glass-wool trap and place it in the heater above the transfer apparatus. Turn on the rheostat connected to the heater, and set it for 90 volts. As the temperature of the heater will rise to 100°C, be careful not to touch it or to handle any parts that have been in contact with it until they have cooled.
18. Open valve (13) until a small stream of helium flows through the trap (place a finger over the open tube of the trap to feel the gas flow). Let the helium flow for 8-10 minutes. This step will purge the trap of water that may have accumulated during the collection period. It is important to remember that, in letting gas into the trap, the valves should be opened slowly so as to prevent a blast of air from shooting through the trap and possibly dislodging pieces of glass wool.

19. You may have to readjust the pressure in the helium reservoir to 6 psig after valve (13) has been opened.

C. Shutdown

1. Remove liquid-nitrogen Dewar from U-trap. Shut off vacuum pump.
2. Immediately open the valve (V) in the line carrier line and allow air to fill the (Figure 2). Failure to do this will cause oil to be sucked out of the pump and into the carrier line.
3. Close valve.
4. When you have finished for the day, shut off the helium at the tank to prevent leaking, and turn off the heater.

III. Counting System

Two stations are available for counting Lucas cells.

A. Preparation

After Lucas cells have been filled, they must sit at least three hours before they can be counted. During this time the short-lived daughters come into secular equilibrium with the radon.

B. Operation

1. Shut off high voltage and turn out room lights before removing cover of the photomultiplier tube (PMT).
2. Place a Lucas cell on the PMT and put the light-night cover over it. Turn on room lights.
3. Turn the high voltage back on and wait one minute before starting the counting in order to ensure that the voltage has stabilized.
4. On the record sheet, record the time that counting was started under "Count Start." Record the counting interval (in minutes that you read off the scales) under "Counting Time." Record the number of counts under "Counts." The counting time should be long enough to allow at least 200 counts to be registered.

5. After a cell has been counted, fill it with helium and evacuate it at least four times in order to flush out the radon. If building materials rather than air samples have been measured, evacuate Lucas cells for several hours.
6. You should routinely count the helium-filled Lucas cells for background before using the measuring facility.

Part 2

CALIBRATION

Figure 4 illustrates the measuring facility set up for calibration. The emanating flask contains a solution of radium-226 in a known amount.

1. Connect apparatus as shown in the Figure. Close all valves and stopcocks. Clean and regrease all ground-glass fittings.
2. Prepare two Freon dry-ice Dewars.
3. Place Dewars on empty water trap (B) and glass-bead water trap (C).
4. Fill two Dewars with liquid nitrogen and place them on glass-wool traps (A) and (D).
5. Test for leaks by opening valves (19) and (17) and letting the system evacuate. Connect a thermocouple junction to one of the sockets in the calibration system. You should be able to pump the pressure down to about 50 millitorr if there are no problems in the system.
6. When a 50-millitorr vacuum has been achieved, close valve (19) and let helium in by slowly opening stopcocks (14) and (15). The helium pressure upstream from stopcock (14) should never be allowed to rise above 2 or 3 psig. Vent the helium by opening valve (20).

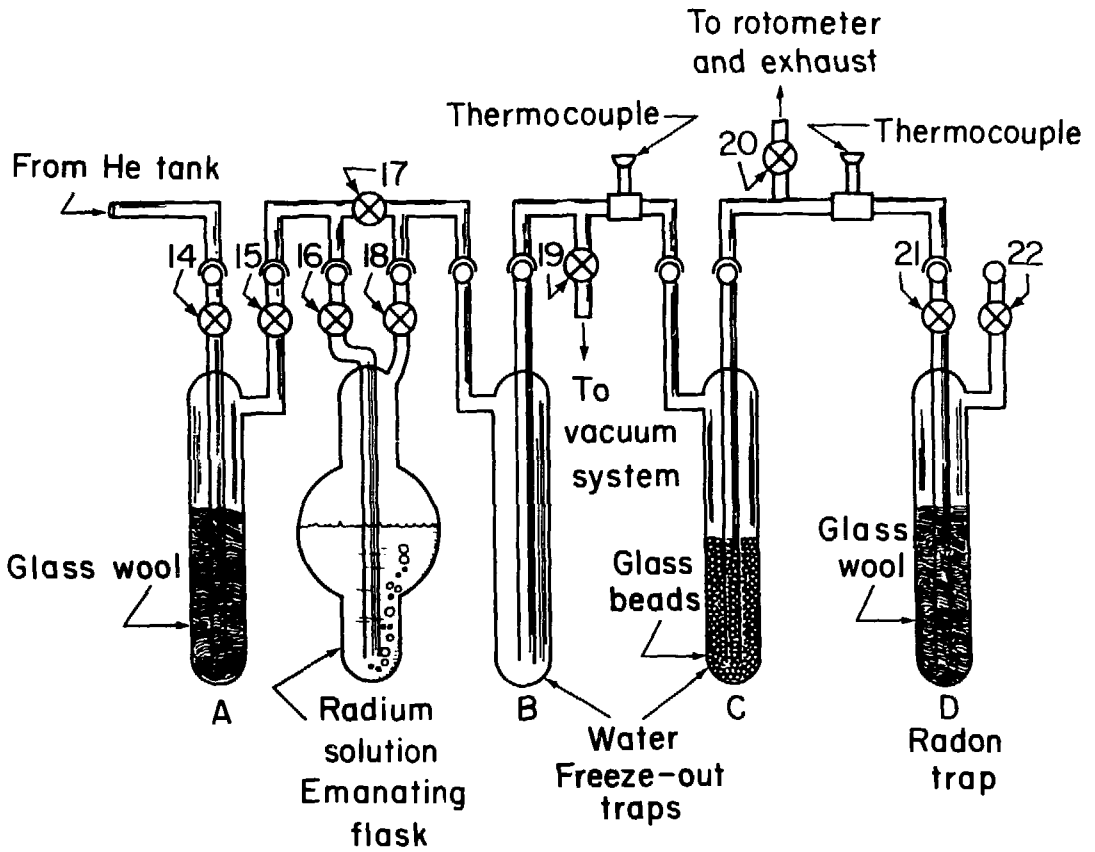


Fig. 4. Calibration System. XBL 801 - 65

7. Set the flow of helium (controlled by the regulator valve on the helium tank) at 1 l/min, using a rotameter on which the calibration is marked.
8. Open stopcocks (16) and (18) simultaneously. Close stopcock (17). Record the time on the record sheet under "Start Flush." Flush helium through the bubbler for about 20 minutes, checking periodically to keep the flow at approximately 1 l/min.
9. At the end of 20 minutes, open stopcock (17), then close stopcocks (16) and (18) simultaneously. Start the stopwatch. Record the clock time on the record sheet under "End Flush."
10. Let the helium continue to flush through the system for about 2 minutes. Close stopcock (15) and valve (20).
11. Evacuate system by opening valve (19). Evacuate to less than 100 millitorr.
12. Close valve (19), then let helium into the system by opening stopcock (15) slowly. Close stopcock (15).
13. Repeat steps 11 and 12 twice so as to remove all of the radon remaining in the lines after flushing.
14. Open stopcock (15) and valve (20). Set the helium flow at 1 l/min.

15. Open stopcock (21) and wait a few seconds for pressures to equilibrate. Then open stopcock (22) and close valve (20).
16. Connect the exhaust of glass-wool trap (D) to the rotameter, and check to see that the flow of helium is still at 1 l/min.
17. Simultaneously open stopcocks (16) and (18). Close stopcock (17). Start stopwatch. Record this time on the record sheet as "Start Collection." No more than 5 or 6 minutes should have elapsed since "End Flush."
18. Let the helium flow at 1 l/min for about 20 minutes, checking periodically to make sure that this rate is maintained.
19. Open stopcock (17); then close stopcocks (16) and (18) simultaneously. Stop the stopwatch and record the elapsed time under "Collection Time." Record the clock time under "End Collection."
20. Let the helium flow for another 5 minutes. Then close first stopcock (22) and then stopcock (21). Shut off the helium, and remove glass-wool trap (D) from the calibration system, keeping it in the liquid-nitrogen trap.
21. Connect the offset leg of glass-wool trap (D) to the vacuum system (using valve near U-trap in carrier line). Open stopcock (22) and pump pressure in the trap down to below 50 millitorr.

22. Close stopcock (22), remove glass-wool trap from vacuum system and liquid-nitrogen Dewar. Allow trap to warm up to room temperature. A heat gun may be used to speed up the process.

23. Then follow the procedures outlined above for the transfer and counting systems.

Part 3

MAINTENANCE AND TROUBLESHOOTING

I. Maintenance

A. Water freeze-out traps

1. Glass-bead traps

All water freeze-out traps must be cleaned, dried, and reassembled after two runs. It is best to disassemble and clean them at the end of the day so that they can dry overnight.

To disassemble a trap, pull the two sections apart and wipe all traces of grease off both sections. It is important that the glass beads never touch the stopcock grease. Pour the beads into a shallow tray lined with tissue paper.

To assemble a trap, pour about 3 inches of glass beads into the bottom of the trap. Grease the fitting in the top section. (See Amos Newton in room 70-274A if you have questions about how to grease ground-glass joints.) Place the top section into the bottom section, shaking the trap gently so that the beads will settle evenly around the center tube of the trap. Do not force the sections together since they may break. After the two sections have joined smoothly, place rubber bands over the small hooks on the sides of the trap to hold it

firmly together.

2. Empty trap

Treat the same as glass-bead traps, except do not add beads.

B. Glass-wool traps

1. The only parts of these traps that need attention are the ground-glass stopcocks. They need to be taken apart and cleaned periodically.

2. You should clean a stopcock when:

The bulb on the stopcock will not hold a vacuum for at least 2 hours.

The stopcock becomes too stiff to turn safely.

3. Before you use a trap, evacuate the bulb in each stopcock. When the black mark on the back of the handle of the stopcock is turned away from the trap, the trap is vented to the outside. When the black mark is turned toward the trap, the bulb on the stopcock is vented. Turning the handle of the stopcock at a right angle to the trap closes the stopcock.

C. Transfer pump

1. A new hose should be inserted in the pump the morning of each day that the pump is to be used. The hose is easier to insert if it is first connected to the system and evacuated.
2. Smear a couple of drops of light oil (e.g. sewing-machine oil) on the tubing before inserting it.

D. Shutdown at night

1. Transfer all Freon dry-ice mixtures to a large Dewar and cover it to prevent water from accumulating in the Freon. If quite a bit of ice has collected in the Freon, pour the Freon into the large Dewar through a fine-mesh wire screen in order to filter out most of the larger chunks of ice.
2. Turn off all compressed gas tanks and shut off the gas from the regulators.
3. Check to make sure that vacuum system is shut down, and that heater on the transfer system is turned off.

II. Troubleshooting

Failure to achieve the desired pressure in any portion of the system may be caused by:

1. Leaks. Use compressed-helium tank to pressurize part of system in question to about 10 psig. Don't try to pressurize with vacuum pump in place. Disconnect pump, and plug off the end of the hose. Use a leak-detecting fluid (Snoop) at the joints to look for leaks. Fluid will bubble up where helium is escaping through it.
2. Off-gassing. Oil or water may accumulate in any part of the system (particularly in U-traps). If it does,

Disconnect part of system to be cleaned.

Using a peristaltic pump, flush acetone in a closed loop through area being cleaned. If acetone becomes yellow or brown, change it and flush again. Flush until acetone remains clear. Drain out acetone.

Repeat flushing process, this time with ethyl alcohol, which flushes out the acetone and has a higher vapor pressure.

Dry out the cleaned piece by blowing helium through it, while heating it with the heat gun until it is too hot to touch. CAUTION: Do not heat epoxied glass-copper tube junctions. The glass will shatter.

Reassemble the system and again bake it out under vacuum with the heat gun. This removes the last trace of alcohol.

If you suspect that water is the only contaminant, you can try to remove it simply by baking out the piece under vacuum with the heat gun.

3. Leaky valves. Nupro valves sometimes fail, so check them separately from the rest of the system.
4. Dirty oil. If the carrier lines will not maintain a vacuum, the oil in the pump may be old and dirty. Have the maintenance machinists change the oil. (Contact Bob Fisher, Building 70, Extension 5339.)