

Radon Emanation System Notes & Procedures

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1 Introduction To SDSM&T Radon Emanation System

1.1 Radon Emanation System Components

- Class-2000 radon emanation cleanroom and HEPA filter for housing both emanation chambers.
- Small (13 L, 10" diameter, 16" height) and large (300 L, 24" diameter, ~40" height) chambers (each with pressure gauges, inlet & outlet valves to gas panel, and connections for the auxiliary pump).
- Emanation gas panel with 15 valves (V1-V15), two flow meters (F1-F2), one needle valve (NV1), and two pressure gauges (P1-P2) —all numbered —and 3 radon traps (a large and a small brass-wool trap, and an activated carbon trap between V1 and V3). although the flow meters may be throttled by turning their black knobs, usually you should leave them fully open (and the flow through F1 will be off-scale).

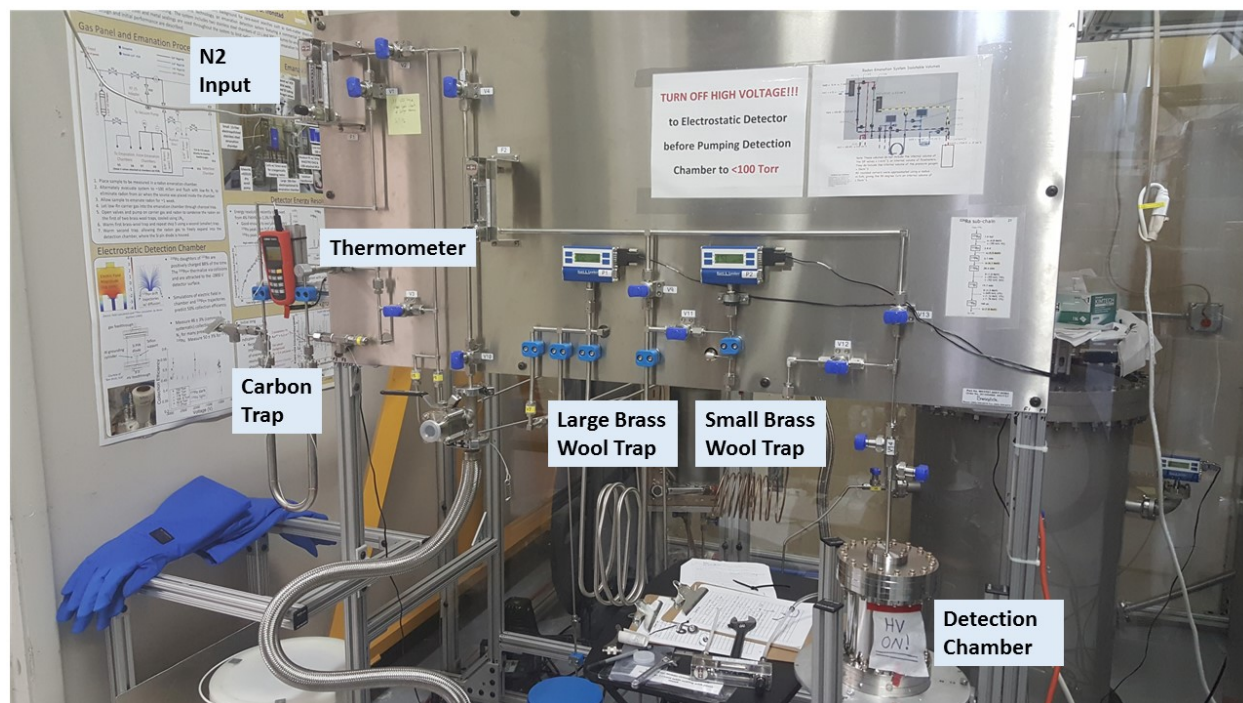


Figure 1: SDSM&T Radon Emanation Gas Panel. Class-2000 radon emanation cleanroom, small, and large emanation chambers pictured in background.

- Low-noise Keithley High Voltage supply for biasing the detector. 50 V are applied across the detector and -2000 V are applied from ground.
- Detection chamber with Si pin diode alpha detector¹.
- External vacuum pump below gas panel used for rinsing the detection chamber and panel.
- (no longer in use: Auxiliary vacuum pump next to emanation cleanroom for a faster evacuation of emanation chambers. Now we just move the main pump.)
- Small, blue dewar to cool the brasswool radon traps.
- Medium, white dewar to cool carbon trap with IPA and dry ice.
- Thermometer for monitoring temperature of IPA dry ice mixture.
- Liquid nitrogen dewar with outlets for boil-off gas and liquid extraction.
- Pylon brand Radon-222 source with a rate uncertainty $\delta R_{\text{syst}}/R \sim 4\%$.

2 General Goals, Principles, and Rules of Emanating Radon

- To make high precision measurements of the ^{222}Rn emanation rate for various components (e.g. resistors, gloves, cables, etc.) for potential use in LZ, SuperCDMS, and BetaCage low-background experiments.
- Common units for the flow rate are cubic feet per hour (cfh)(F1) and liters per minute (lpm)(F2). $1 \text{ cfh} = 0.472 \text{ lpm}$.
- After N volume exchanges given by $N = t \cdot F/V$, where t is the time flushing, F is the flow rate, and V is the volume being flushed, the number of radon atoms that have been evacuated from the original volume is given by

$$A = A_0(1 - e^{-N})$$

where A_0 is the number of radon atoms originally present in the volume, if we have perfect mixing.

2.1 Emanation System Rules of Thumb

To operate the emanation system correctly, keep these rules in mind. Irreparable damage (to either you or the system components) may occur if you do not follow these rules.

Using the Electrostatic Detector

- **DO NOT PUMP ON THE DETECTION CHAMBER WHEN HIGH VOLTAGE IS APPLIED**—Make sure the detection chamber is isolated and has at least 100 Torr of pressure (e.g. when performing a pre-transfer background run but rinsing and flushing the rest of the panel). Arcing was a major problem before a stable configuration of data taking was found.

¹**DO NOT PUMP ON DETECTION CHAMBER WHEN HIGH VOLTAGE IS APPLIED.** Wait at least 5 minutes after removal of HV before pumping on detection chamber.

- Allow the electrostatic detector to “warm-up” for ≥ 10 minutes² before starting a run.

Using the Pylon Source

- Connect the pylon source in series between the N_2 gas source and the gas panel.
- Flow gas through the pylon source at a rate ≤ 10 lpm with the nitrogen dewar regulated to less than 10 psig to prevent the desiccant from detaching.³
- Prevent pressure build-up within the pylon source by opening the outlet and then the inlet, and when closing it, closing the inlet and then the outlet.

Using Liquid Nitrogen (LN_2)

- To safely put LN_2 in the small blue dewar, use the blue cold gloves and face mask.
- Avoid letting the LN_2 touch your clothing. The potential danger is worse than a small splash on your bare skin since clothes will retain the cold liquid.
- To avoid splashes of LN_2 when topping off the blue dewar, wait until there is a steady stream of LN_2 before holding the hose in the blue dewar. While waiting for the steam of LN_2 , hold the hose above the blue dewar and point it at the emanation clean room. Note the time of the fill next to P1, P2, and F2’s range in the lab book.

Using the Carbon Trap and IPA

- Make sure to purge carbon trap (see Section 5) before a transfer.
- It takes about 5-7 lbs of dry ice to cool the dewar of IPA from room temperature to -78°C . Get this ice from Matheson Tri Gas. Get ~ 10 lbs for a large chamber transfer.
- Remove the IPA from the dewar before adding dry ice. Pouring IPA over dry ice is a faster cooling method than adding dry ice to warm IPA.
- Pour IPA over dry ice SLOWLY. If too much is poured at once, the IPA will spill onto the floor due to the violent bubbling caused by the sublimation of the dry ice.
- The dewar holds about 10 L.
- Once IPA is warm (after ~ 36 hours), siphon it back into the metal storage cylinder to reduce evaporation and avoid the need to pour IPA over dry ice.

Using the Gas Panel

- Be sure to purge the line connecting the large LN_2 dewar before putting any nitrogen into the panel. Do this by starting the vacuum pump and opening a path from the dewar to V2, to V4, and then to V10. Let air flow for approx. 10 seconds. Keep all other valves closed.
- Do not fill any part of the panel to much more than 1000 Torr⁴.
- Rinse and flush the panel and detection chamber at least five times to rid the system of potential radon (and progeny) contaminants.

²This period has an unusually high rate of noise consisting of an exponentially decreasing noise-wall.

³If this happens replace the desiccant and cotton (found in the source cabinet near the large cleanroom).

⁴This is to avoid busting the relief valves and harming the pressure gauges.

3 Panel & Chamber Rinsing/Filling and Flushing

The goal of ‘rinsing’ AKA ‘filling and flushing’ the panel is to remove any radon that may be within the panel before placing a new sample in the detection chamber. In general, the emanation chambers are NOT included as part of the panel. Therefore the phrase ‘all valves’ excludes V5, V6, V7, and V8, the emanation-chamber outlets and inlets. Emanation chambers are not rinsed unless it is necessary after placing a new item within one of the chambers.

3.1 Emanation System Panel

1. Make sure LN₂ dewar is attached to the panel by the inlet next to F1 with metal gas line and set the regulator to about 10 psig (higher pressure is harder on the pump). Use V1 and V2 to isolate the panel inlet to avoid wearing out F1.
2. Purge line connecting dewar and panel (if not already done). (See Section 2.1)
3. If this is the end of a run with the carbon trap still cold, open NV1 and leave it open to pump on it. Otherwise, leave NV1 closed.
4. Are you rinsing the detection chamber at this time?
 - (a) IF **YES** – open all valves on panel except for V1, NV1, and V2 and pump down below 10 Torr. (we will use V2 flow through the panel next).
 - (b) IF **NO** – close, or keep closed, V14 and V15 to isolate the detection chamber from the rest of the panel. Open all valves except V1, NV1, and V2 and pump down below 10 Torr.
5. Fill the panel: Close V10 and open V2 to fill panel (and, if **YES**, detection chamber) to ~ 1000 Torr as read on either P1 or P2.
6. Flush the panel: Close V2 and open V10 to evacuate panel (and, if **YES**, detection chamber) to ≤ 10 Torr as read on either P1 or P2.
7. Repeat steps 4 & 5 four (or more) times.

3.2 Small Emanation Chamber

1. Attach LN₂ dewar regulated to ~10 psig to panel inlet next to F1 with metal gas line, while V1, NV1, and V2 remain closed.
2. Purge line attaching dewar to panel. (Section 2.1)
3. Open V4, V9, V5, and V7. Keep all other valves closed.
4. Fill small emanation chamber to ~1000 Torr with LN₂ boil-off by opening V2. Once the pressure gauge on the chamber reads 1000 Torr, close V2.
5. Evacuate the chamber using either the main pump or the auxiliary pump. Both pumps may be used.
 - (a) Main Pump: Turn on pump. Open V10. Once chamber pressure is ≤ 10 Torr, close V10.

- (b) Auxiliary Pump: Connect auxiliary scroll pump to the elbow on the top of the chamber. See 3.4 for details. Turn on pump. Open the blue dial attached to the elbow of the small emanation chamber. Close this dial once the pressure of the chamber is \leq to 20 mTorr. This may take several hours.
- 6. Repeat steps 4 and 5 at least 5 times

3.3 Large Emanation Chamber

- 1. Attach LN₂ dewar regulated to \sim 10 psig to panel inlet next to F1 with metal gas line, while V1, NV1, and V2 remain closed.
- 2. Open V3, V6, and V9. Keep all other valves closed.
- 3. Fill large emanation chamber to \sim 1000 Torr with LN₂ boil-off by opening V2. Once the pressure gauge on the chamber reads 1000 Torr, close V2.
- 4. Evacuate the chamber using either the main pump or the auxiliary pump. Both pumps may be used.
 - (a) Main Pump: Turn on pump. Open V8 at chamber outlet and open V10. Once chamber pressure is \leq 10 Torr, close V10 and V8.
 - (b) Auxiliary Pump: Connect the auxiliary scroll pump to the large chamber. See 3.4 for details. Turn on pump. Open the blue dial on the side of the chamber. Close this dial once the pressure of the chamber is \leq to 30 mTorr. This will take several minutes.
- 5. Repeat steps 3 and 4 at least 5 times

3.4 Using the Auxiliary Pump (when we get a replacement)

The auxiliary pump is located on the bottom shelf of the metal shelving unit next to the emanation clean room. The pump can be connected to either of the emanation chambers by three bellows (flexible pieces of hose). In general, the bellows will stay connected to both the auxiliary pump and one of the emanation chambers by KF-25 flanges. The KF-25 flanges are located at the top of the small emanation chamber and on the side of the large emanation chamber. Next to each flange is a valve that controls flow between the bellows the emanation chamber. Make sure these valves are closed before moving the bellows. To evacuate one of the emanation chambers using this pump, follow these steps

- 1. If the bellows are attached to the desired pump already, nothing further needs to be done for this step
- 2. If the bellows are on the opposite chamber, make sure the pump is off and the valve between the pump and the chamber is closed.
- 3. Release the C-clamp holding the bellows to the chamber.
- 4. Make sure the dial between the KF-25 flange and the desired chamber is closed
- 5. Release the C-clamp on the KF-25 flange attached to the desired chamber. There should be a cover that comes off with the clamp. Place this cover on the other chamber's KF-25 flange and attach it with a C-clamp.
- 6. Attach the bellows to the desired chamber with a C-clamp.

4 Biasing the Detector and Starting a New Run

After a new sample is put into the detection chamber, a new run needs to be started. Follow these steps to start a new run or to bias the detector.

4.1 Biasing the Detector (On to Off)

If the power supply is on, then the light on the power supply will be illuminated and the dial at the bottom of the panel will be turned to 2. To properly turn off the power supply, follow these steps:

1. Slowly decrease the power supply from 2000 V to 0 V. This is done by turning the bottom dial on the power supply from 2 to 0 and should take about 45 seconds.
2. Flip the power switch down to finish turning off the detector. This is the rightmost switch on the panel.
3. Flip the Trip switch (directly to the left of the power switch) down so that you won't trip the power when next turning the detector on.
4. Wait at least 10 min before turning the power supply back on.

4.2 Biasing the Detector (Off to On)

If the power supply is off, then the light on the power supply will be off, and the Power switch (the right-most switch on the supply panel) will be flipped down. To turn the power supply on, follow these steps:

1. Check that the dial at the bottom of the panel is turned to 0 and the Trip switch (directly to the left of Power switch) is flipped down.
2. Flip the Power switch up. If the light on the panel does not illuminate, then check the trip switch. It must be down in order to turn on the detector.
3. Return the Trip switch to the up position.
4. Slowly turn the dial at the bottom of the panel from 0 to 2. This should take about 45 seconds. Go more slowly if the current meter on the supply is moving, or settling above zero.

4.3 Starting a New Run

After a new sample is put into the detection chamber, a new run needs to be started. Follow these steps to start a new run.

1. Bias the detector. It should currently be off.
2. Right-click on the "NewPart.bat" script icon and edit it. Increase the run number by one and change "sample = y" as needed to indicate whether the run is a sample ("y" for material or blank run) or a background run ("n"). Save the edited script.

3. double-click on the “NewPart.bat” script to run it. The script will create a folder in the Data Runs directory titled ”Run x” where x is the run number, and create subfolders titled ’part a’, ’part b’, ’part c’, etc. for sample runs and ’part 0’, ’part 1’, etc for background runs. (We typically only have one part for each run in current data-taking.) The script will wait 10 minutes before starting a run.
4. Open MAESTRO, right click, and select ’MCB Properties.’ This brings up a new window.
5. In the new window click on the second tab, change the run number, and then hit the close button. DO NOT HIT THE ’X.’ The information does not save unless you hit ’close’.
6. Close MAESTRO.
7. Record the start time of the data-taking (after the 10-minute wait) in the log book.

5 Purging the Carbon Trap

1. Let trap warm to room temperature
2. Turn on vacuum pump and open NV1 completely
3. Start flow from dewar to panel and set regulator to 10 psig. Dial back the regulator to a lower pressure if the pump complains.
4. Record start time in lab book
5. The path of the N₂ should be: V1, carbon trap, NV1, V3, V4, V10. Flow N₂ through the trap for 2 hours.
6. Record the stop time in the log book

6 Starting a Background Run

A background run should be started at least one day before any transfer. This is to make sure there is not a large (greater than 2-3 cpd) number of events in the regions of interest. Background runs will also be used to assess the background due to the detection chamber itself. These background runs will last several days.

1. Attach LN₂ dewar regulated to ~25 psig to panel inlet next to F1 with metal gas line, while V1, NV1, and V2 are closed (Section 1).
2. Purge the metal line attaching the dewar to the panel (Section 2.1).
3. Fill and flush the panel and detection chamber 5 or more times (see Section 3).
While filling and flushing, warm the small trap with the heat gun, this extra step is to help prevent blockages in the small trap that occasionally occur when transferring sample radon from the large trap to the small trap (Section 8.2).
4. Open V1, NV1, V3, V4, V14, and V12. Keep all other valves closed.
5. Fill panel to ~500 Torr as read by P1.

6. Open then quickly close V13 to let some gas into the detection chamber. Wait for the pressure to stabilize and then repeat this process until the pressure on P2 is ~ 100 Torr.
7. If the pressure of the detection chamber exceeds ~ 100 Torr, bleed off excess pressure with the main vacuum pump by opening V13 and V10.
8. Once P2 has equalized with the detection chamber, isolate the detection chamber by closing V14 and note the pressure in the lab notebook.
9. Start a new run 4

7 Emanating a Sample

Generally a sample should be emanated and measured at least twice. Before emanating a sample two things should be considered.

1. Leak Rate of Chamber and Off-Gassing

Before beginning emanation for a sample, it must be affirmed that the chamber is below the acceptable leak rate. Otherwise the lab air introduced to the chamber will interfere with the sample. Over the course of emanation, the small chamber can gain a maximum of 55 mTorr of lab air and the large chamber can gain a maximum of 2.5 mTorr. In general, a leak rate less than or equal to 9×10^{-9} torr/(1 sec) is acceptable. This rate can be determined by using the helium leak checker.

A rate of rise test may also be performed. To do this, follow these steps:

- (a) Seal Chamber.
- (b) Pump chamber to less than 10 mTorr.
- (c) Note pressure of chamber at least once an hour for 2 days.
- (d) Plot pressure vs time.

If the pressure vs time plot is linear, then there is a leak in the chamber. If it starts to asymptote, then most of the increase in pressure is due to off-gassing, and you can infer a limit on the leak from the lowest slope.

2. Emanation Pressure

Emanating a sample at different pressures leads to different types of emanation. Typically the first emanation should occur at low pressure (less than 10 mTorr). The pressure of the second emanation will depend on the type of material being emanated. If it is metal, then emanate at high pressure (greater than 40 Torr). If it is plastic, emanate at low pressure once more.

8 Transferring Radon Gas Sample

This section is comprised of the main brunt of the principles for transferring and trapping the radon sample before putting it in the detection chamber to measure.

8.1 Sample Transfer: Emanation Chamber to Large Trap

1. Before starting, ensure you have at least 12 kg LN for a transfer from the small chamber, and 20 kg for a transfer from the large chamber (or 8 kg and 12 kg if you are willing to chance it).
2. If it has not already been purged, purge the carbon trap (see Section 5 for details).
3. Cool carbon trap with dry ice and IPA. See Section 2.1 for details.
4. Make sure that detector is off. See Section 4.1 for details.
5. Rinse the panel including the detection chamber. See Section 3.1 for details.
6. Evacuate panel to low pressure ≤ 5 Torr measured on P2, and record this pressure in the lab notebook.
7. Close all valves.
8. Place small blue LN₂ dewar on the white lifting shelf.
9. Freeze the large trap by filling the small blue dewar completely with LN₂ (6–7 kg). Use the blue cold gloves and face mask for this. Then raise the shelf with the pulley and supporting it by hand to keep it smooth (works better with two people) so the LN covers the trap, and secure by locking the shelf with the handle and tying off the rope.
10. Bring emanation chamber up to ~ 200 Torr by flowing gas through V1, Carbon Trap, NV1, V3, V5/V6 (for small/large chamber). First over-pressure the gas panel then open V5/V6. Close V5/V6 as soon as the pressure gauge on the chamber reads 200 Torr.
11. Before opening any more valves and starting the transfer, note the path to be used. Typically this path is: F1, V1, Carbon Trap, NV1, V3, V5/V6, small/large chamber, V7/V8, large trap, V9, F2, V10, pump. The order for opening these is outlined below.
12. Turn main vacuum pump on with valves V9, V10, and F2 open for exhaust, making sure that the rest of the panel is closed to the transfer gas.
13. Inside the cleanroom, open the emanation chamber valves (outlet then inlet). For the small emanation chamber open the right valve, V7, and THEN the left, V5. Record this time in lab notebook. This marks the start of the transfer and end of emanation.
14. Complete 4 volume exchanges within the chamber by swinging the pressure by a factor of 4 (typically 200 to 50 torr) 4 times. Raise the pressure in the chamber by closing V9 and then opening V1 and V3. Lower the pressure by closing V1 and V3 and then opening V9. Make sure to leave NV1 open. One volume exchange takes about 5 minutes for the small emanation chamber but at least an hour for the large emanation chamber.
15. Close V5/V6 then V7/V8 (for the small/large chamber), stopping the flow through the trap and allow the vacuum to pump the panel for several more minutes while trap remains frozen in liquid nitrogen.
16. Close V9. Make sure that both V7 and V8 are closed to avoid back-diffusion of ²²²Rn from the large trap into the emanation chambers.

8.2 Sample Transfer: Large Trap to Small Trap

1. Move dewar of liquid nitrogen to small trap by lowering the lifting shelf, placing dewar (optionally on the lab jack) on the shelf under small trap, and then raising the lifting shelf and securing it as before. Make sure the coils are fully submerged, but do not submerge the straight lengths much, to reduce chances of clogging.
2. Before starting flow, note the path of flow should be: F1, V1, carbon trap, NV1, V3, V5/V6, small/large chamber, V7/V8, large trap, V11, small trap, V12, V13, F2, V10. The order to open these is outlined below.
3. Open V12, V13, and finally V11, allowing carrier gas to flow through small trap.
4. Open V10.
5. Note pressure of large trap in lab book.
6. Heat large trap to room temperature with heat gun.
7. Again, note pressure of large trap in lab book.
8. Open the outlet (V7/V8) and then the inlet (V5/V6) of the emanation chamber.
9. Open V1 and V3 (NV1 should still be open).
10. Establish steady flow rate through large trap and into small trap, allowing exhaust to exit through V13, F2, and V10 to the main vacuum pump.
11. Allow carrier gas to flow in this manner for 20 minutes.
12. Close V11 and V13, then close V10. Leave V12 open.
13. Remove blue dewar from small trap and use heat gun to warm it to room temperature.
14. Bring P1 up to ~ 500 Torr by flowing through V1, carbon trap, NV1, V3, V5/V6, small/large chamber, and V7/V8.
15. Open V14.
16. Quickly open and then close V11. This provides a burst of gas through the small trap that pushes the sample into the detection chamber. Continue to quickly open and close V11 until the P2 reads 100 Torr. Wait for P2 to mostly stabilize between opening and closing V11.
17. Close V14.
18. Start a new run (see Section 4).
19. Evacuate the panel as per Section 3.1, including the carbon trap. Also evacuate the emanation chamber to begin another emanation period if applicable (as described under step 4 of Section 3.1).

9 Blank Rate Tests

A blank rate is a measure of the background of the transfer process. This background includes radon emanated from the emanation chambers, background due to the N₂ gas and any other contamination that occurs during the assay process. Since this is a measure of the complete system, a blank rate test is in fact done in the same way as one would transfer a radon sample.

1. Begin by rinsing the emanation chamber, panel, and detection chamber in the manner that is outlined in Section 3.
2. Allow chamber to emanate for several days.
3. Follow the sample-transfer procedure to assay how much radon was emanated from the chamber (as described in Section 8).

10 Placing Sample in Emanation Chamber

1. Turn the HVAC on high for at least one day before opening a chamber.
2. Dress in cleanroom suits, booties, gloves, hairnets, and beard covers.
3. Over pressure the panel and detection chamber.
4. Open the chamber.
5. Continue flowing N₂ out of the base of the detection chamber to help keep dust from falling inside.
6. Use the hand held UV light and black cloth to inspect the cleanliness of the emanation chamber before placing the sample. Remove any dust with the nylon brushes and cleanroom wipes.
7. Place the cleaned sample inside the cleaned emanation chamber.
8. Close the emanation chamber, tightening the bolts in a star shaped pattern. Make sure to use anti-seizing grease when tightening the bolts on the large chamber.
9. Bring emanation chamber to vacuum using the auxiliary pump. If you are not able to do this, either the bolts are not tight enough or a new copper seal is needed on the inside of the emanation chamber.
10. Once emanation chamber is at vacuum, rinse the emanation chamber several times (see section 3). For the small chamber this means increasing the pressure ≥ 1000 Torr and then evacuating it to ≤ 10 Torr. For the large chamber this means doing the same thing, only filling to 300 Torr before evacuating to sub 3 Torr.
11. Allow sample to emanate in chamber for several days while the chamber is at vacuum.
12. Follow the transfer procedures outlined above for the transferring of a sample from the emanation chamber.

11 High-Radon Sample in Detection Chamber (Pylon Flow-thru)

(this section needs to be updated!)

1. Fill and flush the panel and detection chamber.
2. When detection chamber is evacuated to a low pressure record the initial pressure in the lab notebook.
3. Close V4, V12, V11 & V9.
4. Regulate LN₂ dewar to ~ 10 psig. Hook the dewar to the bottom inlet of the flow meter (on the left leg of the panel) using long plastic tubing and cap the ends of the metal gas line.
5. Open LN₂ valve and flow through the flow meter, regulating the flow to a maximum of 10 lpm or ~ 20 cfl.
6. Remove the plastic cap off of the output of the pylon source.
7. Regulate flow through the flow meter and connect its output tube to the pylon source input by the desiccant drying column.⁵
8. Open the pylon source outlet then inlet to prevent pressure build-up within the source.
9. Increase the regulation on the LN₂ dewar until the flow rate through the pylon source is ≤ 10 lpm and purge the pylon source of built-up radon into the lab air for ≥ 15 minutes.
10. Connect pylon source into the panel inlet near F1 and establish flow through F1, V2, V4, & V10, with V1, F2, V5, V6, & V3 closed.
11. Place a small plastic bag over the panel exhaust (near V10) to prevent dust from entering the panel. Once the flow drops to zero cfl, exhaust the radon source into the lab for an additional ≥ 30 minutes.
12. Flow the Pylon source (to flow at ??? lpm for ??? minutes) through: [why is this done?] V2 (keep V4 closed) \rightarrow V3 \rightarrow V15 \rightarrow V14 \rightarrow V13 \rightarrow F2 \rightarrow V10 \rightarrow exhausted to lab
13. For each volume exchange through the detection chamber ???% of the original gas in replaced. As a result, after ??? minutes, \sim ??? volume exchanges have taken place and the corresponding uncertainty on the amount of Radon inside the chamber is $\sim 15\%$?
14. Close V15 to stop the flow through and record the time as the t_0 of the sample⁶.
15. Close V13 (with V11 still closed) to begin to evacuate the rest of the panel. Open V12 to use P2 to measure the pressure of the detection chamber. Once the pressures have equalized, record P2 in the lab notebook as the final pressure of the detection chamber.
16. Close V14 to isolate the detection chamber from the rest of the panel.
17. Bias the High Voltage Supply, note this time in the lab notebook, allow the HV Supply to warm up for ≥ 10 minutes and after noting this time, start the run.

⁵Natural impedance through the pylon source and desiccant impede gas flow will reduce the flow rate.

⁶This time is important for the activity transfer efficiency code once the run finally starts

12 High-Radon Sample in Emanation Chamber (Pylon Build-up)

In order to calibrate the detector and assess transfer efficiency, a high radon sample is transferred to one of the emanation chambers and then the detection chamber. This source is typically from Rn build up in the lab's pylon source. The pylon source is found in the radiation file cabinet.

To build up Rn in the pylon source for use in a calibration run or transfer efficiency test, follow the below instructions.

1. Using V1 and V10 flush & rinse the panel and emanation chamber. 3
2. Check that descant attached to pylon is blue and not purple.
3. With all valves closed. Attach pylon source to LN₂ dewar with nylon tubing.
4. Flush long-term radon build-up in pylon source.
 - (a) Place the end of tubing attached to the outlet of the pylon source in a plastic bag
 - (b) Open outlet then inlet of pylon
 - (c) Start flow through pylon with the regulator set to ~ 20 cfh
 - (d) Flow through for at least 20 min.
5. Stop flow by closing regulator, inlet, and then outlet of pylon.
6. Connect pylon source to gas panel
7. Continue to purge pylon and gas lines by flowing through pylon, V2, V4, V10 for ≥ 20 minutes.
8. Have a stopwatch on hand.
9. Close regulator and then pylon source inlet and outlet. Hit start on stopwatch. This marks the start of the build-up time
10. Close V2
11. Pump on the part of the panel exposed to radon during the flow-through to rid the panel of leftover contaminant.
12. Since it will take $t_{\text{fill}} \sim 2$ minutes for the small emanation chamber ($t_{\text{fill}} \sim 2:45$ for the large chamber) use $\geq 100\times$ larger build up time so that any additional emanation during the transfer is $\leq 1\%$ ⁷ of the total radon in the emanation chamber. (i.e. $t_{\text{trans}} \sim 4$ hours.)
13. Open a path to the desired emanation chamber. This is typically V2 to V5 if using the small chamber and V2 to V7 if using the large chamber.
14. Hit "lap" on the build-up timer and open V2, then the outlet and inlet of the Pylon source. This marks the end of the build-up time
15. Allow sample to transfer for 2 minutes (small chamber), or 2:30 seconds (large chamber). Hit stop on the build-up timer and close the inlet of the emanation chamber.

⁷This contributes to a systematic uncertainty $\sim 1\% = t_{\text{fill}}/t_{\text{trans}}$ on the expected rate that gets to the detection chamber

16. Close V2 and open V3, V4, & V10 to evacuate the part of the panel that was just exposed to the high radon sample. Make sure to rinse this area several times before beginning the transfer to reduce extra emanation.
17. Transfer sample as normal. 8

13 New Panel, Cold Emanation Procedure

13.1 Place sample inside the cold emanation chamber

Reworking this section...

1. Bring emanation chamber up to atmospheric pressure with nitrogen boil-off via the cold emanation plumbing.
 - It is not necessary to fill through the emanation gas panel as the carbon trap is not being used to filter radon from the, already low radon (~ 0.5 mBq/m³), liquid nitrogen boil-off.
 - This has the added bonus of being more straight forward and a lower risk to expose the emanation gas panel and extraction chamber to lab air.
 - If desired, it is possible to bring the emanation panel up to atmosphere via the emanation gas panel.
- (a) Confirm that all cryogenic valves (cryo-valves) are closed before proceeding.
- (b) Connect the gas regulator to the “gas” port of the high-pressure (HP) liquid nitrogen (LN) cylinder.
- (c) Connect the Swagelok 0.25-in flex hose to the gas regulator output.
 - Warning: it is easy to cross thread the Swagelok fittings if you are not careful.
 - Turn the nut in the direction of loosening and wait until you feel threads fall into place, then tighten.
 - Do not force it if you are getting resistance.
- (d) Open the “gas” valve of the high-pressure liquid nitrogen cylinder.
- (e) Adjust the regulator pressure to ~ 15 psig, without any gas flowing. If there is an output valve on the regulator, open it now.
- (f) Open CV-1 to allow N₂ flow into the liquid nitrogen plumbing up to the cryo-valve “trident” (where CV-2, CV-3, and CV-4 are located).
- (g) Open CV-3 to begin filling the emanation chamber with N₂ boil-off gas.
- (h) When the emanation chamber is slightly above atmospheric pressure (approx. 700 Torr), close CV-3 and CV-1.
- (i) Open V_{bypass} and V_{flush} on the emanation gas panel.
 - This is done for leak checking reasons since it will be easier to access the emanation gas panel valves before the clean tent walls go up.
 - Note that the entire path from emanation chamber to the pump/leak checker: CV-5, V_{Em-out} , V_{bypass} , V_{flush} , V_{vac} .
 - CV-5 can be accessed from inside the clean space once the walls are up.

- Need to be able to access V_{vac} and V_{Em-out} from the backside of the emanation panel.
 - The pump, as well as V_{vac} and V_{Em-out} , are currently inaccessible from the backside of the panel, blocked by the metal shelf and the storage cabinet.
 - However, V_{vac} and V_{Em-out} are both accessible from the backside of the panel (with some rearranging of either the metal shelf or storage cabinet)
 - * Need to decide how we want to setup the emanation computer, this could drive the decision of which to move.
 - * Originally had planned to mount the computer monitor and use the top of the shelf for the keyboard and mouse
 - * A slimmer shelf could be designed, the metal shelf moved and problem solved.
 - * Or, the metal storage cabinet could be moved (more difficult but easy enough if empty) into the store room or “somewhere” in the lab.
 - * Need to consider that we will need to wheel the leak checker behind the emanation gas panel and connect it too.
 - * Kind of leaning to moving the metal storage cabinet and leaving that space for the leak checker and pump/valve access.
 - (j) Connect the scroll pump below the vacuum port on the gas panel and connect it via a KF25 bellows.
 - The scroll pump should always have the hand valve mounted directly above the inlet port. Ideally the leak checker would have one too.
 - If the plan is to set up the clean tent on the first day and then move on to open the emanation chamber the following day (or days later) the pumps do not need to be running until the second day, after the clean tent walls are already up.
2. Disconnect the LN level sensor cable
 - Located on the top flange of the emanation chamber
 - Coil up the readout cable and place it where it will not be damaged (e.g. on top of the LN sensor display inside the HV rack)
 3. Disconnect emanation chamber pressure gauge power cable
 4. Mount clean room walls; this step requires two people
 - Easier to leave the north wall and ceiling attached at all times. They can be tricky to attach and are not in the way of anything, so this just avoids unnecessary work.
 - Mount the west wall first, the section as it is the most difficult due to the gas panel, then mount the south and the east walls.
 - Use double sided-tape to create a seal between the CR frame and vinyl curtains
 - 1/4” double-sided tape is used to seal the vinyl curtains to the CR frame
 - 3/4” double-sided tape is used to seal the overlapping vinyl sections
 - Check that everything is making a good seal and replace with fresh tape as needed (not yet clear if this needs to be redone every time)
 - McMaster product numbers 75975A655 & 75975A657, respectively
 5. Turn on the HEPA unit

- (a) Plug in the power cord to an extension cord
 - (b) Flip the switch of HEPA unit to ON, check fan speed setting
 - (c) Record time in logbook (for maintenance tracking)
6. Move shelf with CR garb next to the entrance (SW corner)
 7. Pull up top layer of the blue sticky mat so a fresh layer is exposed
 8. One person gowns up and enters the CR while the second person assists from outside
 - (a) The person on the inside will start by cleaning the inner surfaces (ceiling, walls, floor, and any other surface)
 - Clean working from top to bottom
 - Use the clean Swiffer sweeper for the walls; Berkshire wipes and IPA
 - Use the dirty Swiffer on floor; wet and dry Swiffer wipes
 - (b) The person on the outside will gather the tools and materials needed to open the emanation chamber and pass them to the person inside (when ready).
 Note: everything entering the CR should be wiped down with IPA
 9. Tool list:
 - Isopropyl alcohol (IPA)
 - Berkshire wipes
 - Swiffer sweepers (clean and dirty)
 - Swiffer wipes (wet and dry)
 - Collapsible metal table
 - Step ladder
 - UV light and safety glasses
 Find a bag or container to transport light back and forth between CRs
 - Torque wrench with extension
 - Small container for bolts
 - IPA bottle and cleanroom wipes
 - Nylon bag roll
 - Bag sealer
 - Dust counter (Dylos)
 Store in container when not being used to avoid dust accumulation
 - Blackout curtain
- Setup the dust counter as soon as possible after wiping down the clean tent surfaces and floor.
 - Once clean tent surfaces have been wiped down, the dust counter is set up, and the required tools/items are brought inside, exit the clean tent.
 - Allow any dust generated during the cleaning process to settle before opening the emanation chamber.

- This may be a natural stopping point for the day.
10. Open emanation chamber
 - Carefully remove bolts/washers from the upper flange and place bolts in a bag or container for later use; set off to the side.
 - Change outer pair of gloves after handling the flange bolts.
 - Use pulley system to lift flange and secure to top of CR frame w/ carabiner
 - Be careful not to damage the LN level sensor
 - Use a piece of acrylic to cover the external dewar to prevent dust from entering the, now open, emanation chamber.
 - The acrylic cover should be in place whenever the emanation chamber is open and not being accessed.
 11. Remove the blackout curtain from its storage container and mount it above the emanation chamber
 - The mounting design of the blackout curtain still needs to be finalized
 - Was planning on a similar method as the other cleanroom.
 12. Remove sample from emanation chamber
 - (a) Take photo of sample in the emanation chamber
 - (b) Lift the basket from the emanation chamber and remove previous sample
 - (c) Bag and label previous sample for storage
 - Use nylon bags, cutting tool and, bag sealer to store the removed sample
 13. Inspect chamber for dust with UV light
 - (a) Plug in and turn on the UV light, it takes a few minutes to warm up
Use UV safety glasses when using the UV light
 - (b) Cover the emanation chamber with the blackout curtain
 - (c) Use UV light to inspect chamber for dust
 - Take before and after photos
 - Attempt to remove dust with IPA and wipes
 - (d) Wipe down the wire-mesh basket with IPA and wipes
 14. Unbag the new sample and place it inside the emanation chamber
 - (a) Cut open the bag containing the new sample
 - (b) Wipe down the sample with IPA and place in wire-mesh basket
 - (c) Reinsert the basket into the emanation chamber
 - Rotate the basket so the LN sensor will not interfere with the sample
 - (d) Check once more with UV light
 - (e) Take photo of sample in the emanation chamber

15. Take down the blackout curtain and place inside its storage container
16. Close the emanation chamber
 - If the 8? copper gasket needs to be replaced, do so now.
 - Best if replaced every time
 - Can typically be reused once or twice
 - Unhook carabiner securing the emanation chamber flange
 - One person gets on the step ladder while the other person mans the rope
 - Slowly lower into place, be careful the liquid nitrogen level sensor does not interfere with sample
 - Replace the flange bolts, hand tight
17. Use torque wrench to seal the emanation chamber
 - (a) Check that the wrench is set to 142 in-lbs
 - (b) Make at least three passes, following the numbers on the top flange
18. Remove the CR walls
 - (a) Remove and roll up the sections of vinyl
 - (b) Store section in designated location
 - i. Above double sided-cabinet separating the alpha duo from the utility sink
 - ii. Ledge across from storage room doors, by the old emanation system cleanroom entrance
19. Reconnect LN level sensor readout cable
20. Reconnect emanation chamber pressure gauge
21. Pump down the emanation chamber and check that it is properly sealed
 - (a) Turn on scroll pump connected to the emanation gas panel
 - (b) Open V_{vac} , and V_{Em-out} , to pump down the extraction chamber.
 - (c) Open CV-5 to pump on the emanation chamber
 - (d) Once the pressure of the chambers and panel is a few Torr, close V_{Vac}
 - (e) Close the scroll pump hand valve and turn off the pump
 - (f) Break the vacuum in the KF25 bellow above the scroll pump
 - (g) Switch out the scroll pump for the leak checker
 - (h) Turn on leak checker and let it warm up and auto-calibrate
 - (i) Take leak checker off of standby mode to evacuate the KF25 bellows section
 - (j) Put leak checker back into standby mode
22. Open V_{vac} and take leak checker off standby to get a baseline leak rate
23. Move helium bottle near the emanation chamber

Can probably leave chained up and run a longer line over to the emanation chamber

24. Connect gas regulator and helium spray gun to the bottle
25. Spray small amounts of helium around the cold emanation (upper) flange and monitor leak rate.
 - If the leak rate $\leq 1\text{E}^{-10}$ torr.L/s the chamber passes.
 - If the leak rate $\geq 1\text{E}^{-10}$ torr.L/s make a few more passes on the flange with the torque wrench and check again.
 - If the copper gasket is not new, the torque may have to be increased (slightly) to seal properly. Consider replacing next time if this is the case.
 - Note: don't spray too much helium because if there is a "large" leak the baseline rate will go up and could take several minutes to drop back down to the desired sensitivity. A little helium goes a long way.
26. After leak check is complete
 - (a) Close all valves on the gas panel
 - (b) Close all cryo-valves
 - (c) Reconnect the scroll pump (leave off)
 - (d) Put helium bottle back and secure with chains (if needed)
 - (e) Cover and store the leak checker
 - Blank flange the inlet KF25 port and secure with clamp
 - Do not just cover with plastic cap

13.2 Fill emanation chamber external dewar with liquid nitrogen

Before filling the emanation chamber with liquid nitrogen it is pre-cooled by filing the external dewar with liquid nitrogen. Some time may have passed since the sample being assayed was placed and sealed inside the emanation chamber, so it is purged using nitrogen boil-off via the emanation gas panel. These steps are done in parallel to minimize the time the sample is warm prior to filling the emanation chamber with liquid nitrogen. The high-pressure (HP) liquid nitrogen cylinder will be used for nitrogen gas, while a rental low-pressure (LP) liquid nitrogen cylinder is used for filling the emanation chamber and external dewar with liquid nitrogen (any time that LN flows through the cold emanation plumbing).

1. Confirm that all cryo-valves are closed.
2. Open CV-2, then open the "liquid" valve on the LP LN cylinder to start the external dewar fill. Move on to following step (to be done in parallel).
3. Fill/flush the emanation chamber with nitrogen gas via the emanation gas panel.
 - (a) Set the gas regulator on the HP LN cylinder to 15 psig.
 - (b) Turn on the scroll pump connected to the emanation gas panel.
 - (c) Open F_{purge} for approximately 10 seconds to purge any lab air from the N_2 line.
 - (d) Close F_{purge} , then open $V_{\text{pylon bypass}}$.
 - (e) Open V_{vac} , V_{flush} , V_{bypass} , and $V_{\text{Em-out}}$ to pump the cold emanation extraction vessel.

- (f) Open CV-5 to pump on the emanation chamber.
 - (g) Close V_{Em-out} and V_{flush} when pressure of the emanation chambers are emanation pressure ≤ 10 Torr (use the extraction chamber pressure gauge).
 - (h) Decrease the pressure regulator, P_{reg} , until it is effectively closed, then open $V_{C-trap\ bypass}$ and V_{Em-in} .
 - (i) Slowly increase P_{reg} and fill the emanation chamber to approximately 600 Torr in the period of a few minutes (confirm this pressure).
 - (j) Close V_{Em-in} when emanation chamber pressure ≥ 600 Torr (again, use the extraction chamber pressure gauge).
 - (k) Close $V_{C-trap\ bypass}$, $V_{pylon\ bypass}$, and close the “gas” valve on the HP LN cylinder.
 - (l) Open V_{flush} and V_{Em-out} to pump on the emanation chamber.
 - (m) Continue to pump on the on the emanation chamber(s) for the remainder of the external dewar LN fill.
4. When the liquid nitrogen level of the external dewar reaches the desired fill level (e.g. the bottom of the upper emanation chamber flange; TBD)
 - (a) Close the HP LN cylinder “liquid” valve (note that CV-2 remains open).
 - (b) Record time in logbook.

13.3 Fill the emanation chamber with liquid nitrogen

1. Close V_{vac} and record emanation start time in the logbook
 - Note CV-5, V_{Em-out} , V_{bypass} , and V_{flush} remain open for this step (final gas panel configuration of step 2).
 - The goal is to prevent lab air from back flowing into the gas panel plumbing.
2. Close hand valve directly above scroll pump and turn off pump
3. Disconnect the vacuum bellows at the KF25 port below V_{vac} on the gas panel
4. Confirm that CV-2 is open, then open the “liquid” valve on the low-pressure liquid nitrogen cylinder
5. Once liquid nitrogen is observed flowing out of the dewar fill line, open CV-3 and close CV-2 to start filling the emanation chamber.
6. Record fill start time. Move on immediately to the next sub-step (3.6)
7. Monitor the pressure gauge P_{in}
 - (a) When $P_{in} \leq 750$ Torr (slightly above local atmospheric pressure) open V_{vac}
 - (b) Leave V_{vac} open for the remainder of the liquid nitrogen fill
8. Monitor the LN level sensor during the fill
 - (a) When liquid nitrogen is at the desired level, close the low-pressure liquid nitrogen cylinder ?liquid? valve

- This actual level is TBD
 - 100% is just below the top flange
 - 0% is a few millimeters from the bottom
- (b) Record liquid nitrogen level in logbook
9. Pump panel down and isolate
- (a) Close V_{bypass} , then open V_{exh} and reconnect the scroll pump
 - (b) Turn on and open hand valve (directly above scroll pump) to evacuate panel
 - (c) When $P_{out} \leq 10$ Torr, close V_{exh} , V_{flush} , V_{vac} , and turn off the scroll pump
- Note: At this time, all panel valves except V_{Em-out} should be closed

13.4 Liquid nitrogen emanation period

At this point the sample submerged in liquid nitrogen and allowed to emanate for some period of time TBD; assuming on the order of a week or two. During the emanation period there should not be much to do other than monitoring the system. There could be a situation where the external dewar liquid nitrogen level may need to be topped off. Important notes on pressure relief during the emanation period:

Do not close CV-3, CV-5, or V_{Em-out} at any point during the emanation period!

- CV-3 must remain open to provide a path for pressure relief
 - CV-5 (and V_{Em-out}) must remain open to provide a path for pressure relief
 - The top flange of the emanation chamber has a PRV for the same reason
 - The unused lower flange port on the emanation chamber also has a PRV (may still need to be installed)
1. Visually monitor and top-off dewar with LN as needed with cryo-hose.
 2. Check the LN level sensor (should not change) and monitor the pressure of the emanation chamber.
 - (a) Attach cryo-hose to the LP liquid nitrogen cylinder
 - (b) Open the “liquid” valve on the liquid nitrogen cylinder. Be careful not to spray the emanation chamber pressure gauge with liquid nitrogen.
 - (c)

13.5 Harvest radon from the cold emanation chamber

1. Confirm that all panel valves, except V_{Em-out} , are closed.
 - Note: sections of the gas panel will be under vacuum from step 3
2. Confirm that cryo-valves CV-1, CV-2, and CV-4 are closed
3. Purge the BW trap with N_2 gas before the transfer

- (a) Open “gas” valve on high-pressure liquid nitrogen cylinder and set the regulator pressure to ~ 15 psig
 - (b) Connect scroll pump to KF25 port below V_{vac}
 - (c) Turn on pump and open valve above inlet port (record time in logbook)
 - (d) Open V_{vac} , V_{exh} , and $V_{trap-out}$ to pump on BW trap
 - (e) Open F_{purge} for approximately 10 seconds to purge any lab air from the N_2 line
 - (f) Close F_{purge} , then open $V_{Pylon\ bypass}$ and $V_{C-trap\ bypass}$
 - (g) Close V_{vac} , then open V_{flush} to fill the trap with N_2
 - (h) Close V_{flush} , then open V_{vac} to pump down the trap
 - (i) Repeat the fill/flush a total of five times
 - (j) On the final pump down, when $P_{in} \leq 1$ Torr, prepare to cool down the copper-wool trap
4. Cool copper-wool trap by submerging it in liquid nitrogen
 - (a) Place (empty) white cryo-bucket around the BW trap and slide the labjack underneath the bucket
 - (b) Raise the cryo-bucket with the labjack until the trap touches the bottom of the bucket (bottoms out)
 - Connect the 10’ long cryo-hose to LN cylinder and don the appropriate PPE
 - Note: for this step a high-pressure liquid nitrogen cylinder can be used (not using the cold emanation LN lines)
 - However, if there is still liquid nitrogen available in the low-pressure (rental) cylinder use that up first
 - (c) Fill white cryo-bucket with LN until the BW trap coils are submerged a few inches
 5. Fill extraction chamber HDPE bucket with the cryo-hose (actual height TBD)
 - (a) Place the end of the cryo hose into the yellow HDPE bucket
 - (b) Open “liquid” valve on liquid nitrogen cylinder
 - (c) When LN height reaches the desired level, close the LN cylinder “liquid” valve
 - E.g. bottom of the extraction chamber flange
 - Actual fill level TBD
 6. Transfer LN from emanation chamber to extraction chamber
 - (a) Open $V_{trap-in}$ (**Transfer start time**)
 - (b) Open CV-1 to flow N_2 into the emanation chamber to begin transfer of (emanated) liquid nitrogen from the emanation chamber to the extraction chamber.
 - (c) Monitor the liquid nitrogen level, via the level sensor, during the transfer
 - (d) Once the LN level sensor reads 0%, close CV-1 and CV-5
 - (e) Pump on the extraction vessel, via the cold copper-wool trap, until all the LN has been removed.
 - This step is tricky. We want to flow the entire volume of emanated LN through the BW trap without breakthrough and loss of sample radon.

- Will likely need to remake the BW trap to accomplish this or come up with some way to sample a known amount of LN?
 - Don't exactly know when the chamber is empty other than it being warm (no external LN bath) and at low pressure.
 - Can monitor the pressure of the extraction vessel (via P_{in})
7. When $P_{in} \leq 200$ Torr, close V_{Em-out} and $V_{trap-in}$
 8. Continue to pump on the copper-wool trap (still submerged in liquid nitrogen)
 9. When $P_{out} \leq 10$ Torr, close $V_{trap-out}$ and V_{exh}
 10. Remove copper-wool trap from liquid nitrogen and warm with heat gun to room temperature.

13.6 Transfer radon from the cold trap to the detection chamber for counting

1. Stop current run and debias the HV detector
2. Confirm that the scroll pump is still on (if pump is off, then turn it on)
3. Confirm the gas panel valve configuration is the same as the end of step 5
 - The N_2 line should already be purged (if there is any doubt purge the line again)
4. Open the valves (input/output) directly above the detection chamber
5. Open $V_{det-out}$ to fill the detection chamber with boil-off LN
6. Close $V_{det-out}$
7. Open V_{det-in} and V_{exh} to pump on the detection chamber
8. Fill/flush detection chamber a total of five times (toggling $V_{det-out}/V_{det-in}$)
9. On final pressure swing, pump detection chamber (P_{out}) to ~ 1 Torr, then close V_{exh}
10. Open V_{bypass} to build up pressure on the backside of the copper-wool trap (monitor P_{in})
11. Open $V_{trap-out}$ to expose the sample radon in the trap to detection chamber volume
12. Quickly open and close $V_{trap-in}$ to “inject” the radon sample into the detection chamber. Repeat this step until the detection chamber pressure, P_{out} , is ~ 100 Torr.
13. Close all panel valves and valves above the detection chamber
14. Close the “gas” valve on the HP LN cylinder
15. Turn off scroll pump
16. Bias the HV detector and start the run
 - At this point both the emanation and extraction vessel are at a pressure of ~ 200 Torr
 - Do we want to leave this as is, pump down, or fill to atmospheric pressure with N_2 ?

- For now, assume we will close all valves and leave everything is the current state, until all of the LN in the external dewar has evaporated and both chambers are at room temperature.
- Evaporate LN with heat gun
- Bring chambers up to atmosphere w/ boil-off nitrogen via the gas panel
- Note with only the cold emanation vessels connected to the panel, there is really no reason to use the carbon trap, CT, unless you plan to do a room temperature emanation where you will fill/flush to harvest the emanated radon gas (not just a constant flow through the cold BW trap until all the LN has boiled-off)
- After the transfer step:
 - Leave CV-5 and CV-3 open to allow LN in emanation chamber to vent as outer dewar LN level decreases
 - No longer needed with a pressure relief valve mounted on the cold emanation chamber (top) flange