

LUX Xenon Sampling System Procedure

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1 Introduction

1.1 Purpose

The purpose of this document is to guide the user through the process of sampling xenon from the LUX circulation system and analyzing the xenon purity with the coldtrap/RGA method.

2 Prerequisites

2.1 Skills and Training

This work requires cryogenics training. HS5030-W "Pressure and Cryogen Safety" This work will be performed by a Sampling System Expert, to be trained by Attila Dobi.

2.2 Permits and Authorizations

Work shall only be completed as authorized by the shift manager.

2.3 Equipment

- 4 L transfer Dewar with handle
- Cooler (for storing liquid nitrogen)
- Cryo-gloves
- Face shield
- Safety Glasses
- Lab-jack for supporting cooler
- Liquid nitrogen supply with delivery hose
- Portable O₂ monitor

2.4 Hazard Mitigation

Hazard	Mitigation
High Pressure Gas	<ul style="list-style-type: none"> • All operators have completed HS5030-W, Pressure/Cryogen Safety. • Burst disk set at 41.1 psig (BDCT1) prevents dangerous overpressure in the coldtrap. • All components of the system have been pressure tested prior to operation Pressure Test Procedure • Only a fixed amount of xenon will ever be cryopumped into the coldtrap. At most a 0.5 L volume at 3 atm, the maximum pressure in the LUX xenon system, of xenon will be introduced into a 1.0L volume. Engineering controls are built in so that the maximum pressure in the coldtrap will be 1.5 atm, after warming the xenon ice. Since the LUX xenon system can not exceed 4 bar of pressure, without blowing a burst disk, the maximum amount of xenon that can be transferred to the sampling volume is 4 atm in 0.5 L. When expanded into the entire sampling system volume of 2.5 L the resulting pressure is 1.5 atm. In case of accidental overpressure a bust-disk set to 41.1 psig will relieve the pressure in the coldtrap plumbing.
Oxygen Deficiency Hazard (ODH)	<ul style="list-style-type: none"> • Oxygen monitors alert personnel to oxygen deficiencies • Inert gases used are only asphyxiants, not toxic. • Closed system reduces asphyxiation hazard; consult ODH analysis for details • Quantity of nitrogen gas and liquid-nitrogen is well below amount need to create oxygen deficiency hazard. Four Liters of Liquid nitrogen will be used to cool the coldtrap. 4 L of LN can expand suddenly to 2800 L of gaseous nitrogen at room temperature. This is less than 0.2% of the air volume of the Davis cavern. • ODH analysis completed and shown that ODH is zero.
Cryogen Safety	<ul style="list-style-type: none"> • All operators have completed HS5030-W, Pressure/Cryogen Safety. • Personal protective equipments(PPE) such as cryo-gloves, safety glasses, and face shield protect operators from exposure to cryogenic liquid. They will be located by the SRV. • The coldtrap consists of 316 S.S. (stainless steel) plumbing, with VCR and conflat connections, which have been pressure tested.
Administrative Locking	<ul style="list-style-type: none"> • For covering and locking valves in this procedure see operations procedure 36 Administrative Locking.

2.5 Lessons Learned

- RGA overpressure burnout in August 2013.

Before the sampling algorithm had completed the user clicked a button on slow control to start a new sample. Specifically, the logic had gotten to the stage where SAM is waiting to confirm that the cold trap has warmed up and that the xenon is ready to be pumped away or recovered to the SRV. The sampling system Logic began the pre sampling process in parallel to the pump out process exposing the RGA temporarily to an atmosphere of xenon causing the filament and electron multiplier to burn out. The way the slow control backend is set up multiple instances of a C script can run in parallel when a front end command is sent. The problem was fixed by modifying the logic. All user inputs for the “Sampling System Master Control” are disabled until the analysis algorithm has completed and until all reported sampling errors are cleared by the user. As long as the user follows the procedures the code will do the rest.

- Hanging hose ejecting from sampling Dewar during LN fill.

There have been two instances of the SRV vent line’s flex hose ejecting from the sampling system Dewar at the start of the LN fill. High pressure can build up in the LN fill line so when starting the fill there is a possibility that the hose will jolt initially. This has been address with CP13 version 6. Mitigated by removing the cover of the Dewar, and placing the Dewar directly under the flex hose (bend as little as possible). Also we can contain the flex hose to prevent it from swinging around. The correct response for the operator is to hit the ”Refill Stop” button, wait for the line to stop venting and place the hose back in the Dewar.

- SRV recovery procedure.

It was pointed out that when recovering the sampled xenon to the SRV the high pressure lines should first be opened to the cold SRV prior to opening the valves to the low pressure side. No incidents ever occurred because the pressure in the lines are always checked prior to shorting the low and hight pressure plumbing , this is just good practice.

3 Filling the Sampling System's Dewar in the Lower Davis

Hazard Analysis:	A cryogen is used in this procedure. Wear PPE including cryo-gloves, safety goggles, and face shield when handling cold cryo-hose fittings and pouring liquid nitrogen. Avoid cuffs and wear proper shoes that cover the toes. Before filling the 4 L transfer Dewar assemble materials, Check that SRV vent line ready to deliver LN (Liquid Nitrogen), Sampling system is ready for LN. Notify others in the area of impending LN fill. Assure vent line is adequately contained by Dewar that hose whip will not kick the hose out of the Dewar. Don PPE: Cryo-gloves, safety glasses, face shield, oxygen monitor. Do not place the Dewar lid on Dewar, leave the Dewar open to allow pressure relief. Cuffs that could trap liquid should not be worn when working with LN. Before using an oxygen monitor, the operator should test if the device is operational. A simple test is to hold breath for several seconds, then slowly (>15 seconds) exhale near the sampling port. If monitor does not alarm and reads <19.5% do not use. Assure others (non-operators) are out of the area. The SRV LN Panel is about 2 m from SRV Vent Line and 4 L Dewar. Observe filling of 4 L Dewar from a distance at least 3' away. Listen for alarms and check personal and fixed oxygen monitors. If unexpected event occurs during filling or oxygen levels drop to <19.5%: Stop Fill by pushing "Stop Fill" button on the SRV LN Panel and leave the area (it is sufficient to take the stairs up to the upper davis). While filling the 4 L transfer Dewar there is a cryogen hazards to the users and others in the lower Davis, near the SRV. LN Splashes,a spill, or overflow could cause the floor to get slippery and cause oxygen deficiency. Check that the area in lower Davis is clear before transporting the 4 L Dewar to the sampling system. Before pouring LN, check floor and area around the cold trap Dewar for equipment that might be adversely affected by an LN spill. Pour slowly at start, especially if Dewar is warm.
Operation Risk:	When starting the fill of the sampling system's Dewar there is a chance that the hanging hose of the SRV vent line could be ejected from the Dewar, due to elevated pressure in the LN fill line. If this occurs stop the flow of LN by pressing the "Refill STOP" on the SRV LN Panel. See Figure 5 for a photo of the SRV LN BOX. The SRV vent line is near an oxygen monitor by the SRV and a LN spill during the filling of the sampling system Dewar may set it off. If the oxygen alarm is set off evacuate the area. Before beginning a LN fill check that the LN percentages of both the SRV and TS are greater than 52%, displayed on the SRV LN system panel. This is done in order to ensure that the SRV vent line will be free for the next 5 minutes. If either of them are reading less than 52% then wait until the automated LN system refills them. The coldtrap is a cryopump vessel which could potentially accumulate an amount of xenon which could overpressure when the xenon ice warms up. A burst disk set to 41.1 psig will relieve a overpressure in the coldtrap system plumbing.

This section describes the process of filling the 4 L transfer dewar form the SRV vent line and then transferring its contents to the Sampling System's cold trap dewar. Note, the Dewar is to be replaced every six months.

1. Before beginning check the usage data on the sampling system's Dewar. If the last replacement was over six months ago then replace the Dewar and label the new starting date.

2. On the LN system panels check that the LN percentages of both the SRV and TS are greater than 52%. This is done in order to ensure that the SRV vent line will be free for the next 5 minutes. If either of them are reading less than 52% then wait until the automated LN system refills them.
3. Push the button labeled “Refill SmpSys” on the SRV LN Panel. This will time out the SRV vent for 600 seconds. (Note: You can either use the touch screen interface or the physical button on the LN box).
4. Don Cryogen PPE: Cryo-gloves, safety glasses, face shield. Take the clip tested, functioning oxygen monitor (stored in the LUX office) close to your breathing zone.
5. Place the 4L Dewar into the 95 L S.S. pot located at the SRV vent line.
6. Adjust the vent line such that the 4 L Dewar is directly underneath the SRV vent line. Do not place the lid on the sampling system’s Dewar, leave it open.
7. Push the button labeled “Refill SmpSys” again. If the line is warm then at first only nitrogen gas will vent from the line.
8. If the SRV vent hose is ejected from the sampling system Dewar due to an overpressure in the line press the “Stop Fill” button.” Wait for the line to stop venting and return to Step 6.
9. After 5-10 minutes, depending on the initial line temperature, the flow of LN through the SRV vent line will begin at a rate of about 3 l/min. See Figure 5 for a photo of the SRV LN BOX.
10. When the sampling system’s Dewar is nearly full (about 2 minutes), stop the fill by pushing the “Refill STOP” on the SRV LN Panel. Once the “Stop Fill” button is pressed LN will continue to flow for about 30 seconds, wait until the flow stops. See Figure 5 for a photo of the SRV LN BOX.
11. There is a 2 minute timer on the Sampling System LN fill that will shut off the flow of LN, the timer begins when LN line temperature drops below -180 C. If you still need to top off then restart the fill by pressing the button labeled “Refill SmpSys”again (press twice). And then stop the fill by by pushing the “Refill STOP”. Remember, once the “Stop Fill” button is pressed LN will continue to flow for about 30 seconds.
12. If the LN fill fails to stop, or in the event of an emergency hit the RED “Emergency Stop” button on the LN panel. Then see [LN System Procedure](#)
13. Check that the floor of the lower Davis is clear of obstacles or workers. Lift the Dewar by handle and carry over to sampling system.
14. After a sample has been drawn, pour the LN from the 4 L Dewar into the cold trap Dewar. Before pouring LN, check floor and area around the cold trap Dewar for equipment that might be adversely affected by an LN spill. Pour slowly at start, especially if Dewar is warm.
15. Completion: Return 4 L Dewar to storage near the SRV vent line and log activity in the LUG.

4 Using the Automated Sampling System

Hazard Cryogen and pressure hazards mitigated as noted in Section 2.4
 Analysis:

This section describes the process of sampling and analyzing xenon from the LUX circulation xenon system with the sampling system. Because the sampling procedure is exactly the same for each of the locations, we will henceforth refer to the valves in each sampling location as VC&VB (valve on the circulation side) and VA (the valve on the sampling system side). Additionally, we will refer to the pressure transducer nearest the sampling port as PT-A. The valves and pressure transducer corresponding to each sampling location are summarized in Table 1.

Sample Location	VA	VB	VC	Pressure to Watch
Getter Input	CV35	CV34	CV37	PT-C15
Getter Output	CV74	CV76	n/a	PT-C20
Detector Return	CV8	CV11	n/a	PT-C1
PMT Purge Line	CV7	CV9	n/a	PT-D41, PT-D4
Recirc Pump Inlet	CV60	CV61	n/a	PT-C15
To SRV	CV90	CV89	n/a	PT-S10

Table 1: Sample ports with their corresponding valves and pressure gauge to watch

Note: Refer to Table 1 for valves VA, VB and VC as they depend on the location you will be collecting a sample from.

4.1 Collecting a Xenon Sample from the Circulation System

Hazard Analysis: A cryogen is used in this procedure. Wear PPE including cryo-gloves, safety goggles, and face shield when handling cold cryo-hose fittings and pouring liquid nitrogen. Avoid cuffs and wear proper shoes that cover the toes. Use a portable O2 monitor during LN transfer to ensure the O2 concentration is above 19.5%.

Operation Risk: The coldtrap is a cryopump vessel which could potentially accumulate an amount of xenon which could overpressure when the xenon ice warms up. A burst disk set to 41.1 psig will relieve a overpressure in the coldtrap system plumbing.

This procedure is for sampling xenon from the LUX xenon gas system. A 0.5 L sampling volume will be filled with xenon from the LUX circulation system. The sample should only be taken while xenon is circulating in the LUX system for proper mixing of impurities. In this procedure, there are six different locations from which the Xenon can be sampled. The valves and pressure transducer corresponding to each sampling location are summarized in Table 1. See Figure 1 and Figure 2 for diagrams of the coldtrap plumbing and sampling port locations. Once the sample is collected in the 0.5 L sampling volume, you will cool the trap and begin the analysis process in the slow control. When done, the xenon ice in the coldtrap will warm and vaporize, you can choose to vent this xenon or to recover it to the SRV. The analysis will be done automatically, purity values will appear in the slow control database. You will need to record several quantities, do this in the LUG.

1. Check the pressure of instrumentation nitrogen. PT-GC10 should be more than 70 psig and PT-GC9 should read more than 200 psig. If not than the instrumentation nitrogen supply needs to be replaced.
2. Refer to Table 1 for valves VA, VB, VC and the pressure gauge to monitor as they will depend on the location you will be collecting a sample from.

3. Monitor and ensure the stability of the pressure gauge of interest (see Table 1). The pressure should be stable within 1 psig for one minute while the system is in a steady state. If not, contact the director of operations.
4. Confirm that all VA, VB and VC noted in Table 1 are closed. If not, stop and contact the director of operations. See Table 1.
5. Get liquid nitrogen (LN).
 - a. Locate the sampling system's portable dewar
 - b. Don proper PPE. Safety glasses, face shield, cryo gloves. Also, wear a portable O2 monitor.
 - c. Fill the dewar from the SRV vent line. Go to Section 3 and follow the instructions.
6. In slow control, under “Text”, the state of “Sampling System Status” should be “Idle”, the state of “Sampling System Error Status” should be “None” and the state of “SAM Run Status” should be “Idle”. If not then stop, the sampling system is not ready to draw a sample. If “Error” is displayed see Section 6. If “Ready for SRV Recovery” is displayed then see section 4.5. If “Pumping Out”, “Analyzing” or “Calibrating” is displayed then wait for those processes to complete. If more than an hour goes by without a change in state then contact the system owner (Attila Dobi).
7. Go to the LUX slow control page, select ‘Control’ in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the pencil button.
8. In the ‘Sampling Port Select’ box select the appropriate sampling port. The sampling will not start if you don’t do this. Press change. For help see figure 3.
9. In the ‘Sampling System Master Control’ box select ‘Pre Sample’ and then hit the ‘Change’ button. This will automatically prepare the system for sampling. While the pre-sample program is running the Sampling System Status box will display “PreSample” and the “SAM Run Status” will display “Wait”, they can be found under the “Text” tab. Do not change anything on the control page at this time, it could result in an error. For help see figure 3.
10. After the button is pressed wait 2 minutes and refresh the page. When the Sampling System Master Control’s current value changes to ‘Ready to Sample’ the sampling system is ready to draw the sample from the LUX circulation system. If the “Sampling System Master Control” current value changes to ‘Error’ then stop and go to Section 6.
11. Open VC and VB. Then unlock and open VA to fill the 0.5 L sample volume with xenon from the LUX system. Crack VA open a bit until PT-SAM2 reaches 700 Torr. Drawing 700 Torr is sufficient for purity analysis. Figure 4 shows which pressure gauge to watch, it is visible to the user as he/she opens the sampling ports.
12. Close the sampling valves used(VA, VB and VC) to prevent leakage of xenon from the LUX system. Place an administrative lock on VA and tag it.
13. Start a LUG entry. Log the data and time of the sample, the port used for sampling, and the pressure of PT-SAM2 after the sample was taken.
14. Go to the LUX slow control page, select “Text” in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the ‘pencil’ button.
15. The Sampling System Status’s current value should be “Ready to Sample” and the value of “SAM Run Status” should be “Idle”. If the value of “Sampling System Status” is “Error” then stop and go to Section 6. If the value of “SAM Run Status” is “Wait” then contact the system owner (Attila Dobi). For help see Figure 3.

16. Raise the cooler under the “U” of the coldtrap via a lab-jack until the bottom of the cooler touches the bottom of the “U”.
17. Fill the cooler underneath the cold trap’s “U” to the “Fill” marking with LN from the sampling system’s dewar.
18. Wait 1 minute, this is sufficient time for the coldtrap plumbing to cool down, once it has been immersed in LN.
19. Go to the LUX slow control page, select ‘Control’ in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the pencil button.
20. Before the analysis has begun check that the LN level is above the “Min LN” line, if not then add more LN.
21. In the ‘Sampling System Master Control’ box select either “Analyze and Dump” or “Analyze and Recover” and then hit the ‘Change’ button. “Analyze and Dump” will pump out the xenon sample after the measurement. “Analyze and Recover” will prompt the user when he/she can recover the xenon to the SRV. For help see Figure 3.
22. While the analysis program is running. The Sampling System Status box will display “Analyzing” and the “SAM Run Status” will display “Wait”. Do not change anything on the control page at this time, it could result in an error.
23. If the user chose “Analyze and Dump” then they can leave the system at this point if necessary, as long as they finish the rest of the steps in this section the next day. Once the LN evaporates the xenon will be pumped out automatically and the sampling system will prep for the next measurement. For help see Figure 3.
24. If the user chose “Analyze and Recover” then they should check back in 30 minutes to recover the xenon, or return the next day. Once the LN evaporates SAM will wait for the user recover the xenon to the SRV (section 4.5).
25. The measurement is completed in about 30 minutes. The value of “Sampling System Status” will change from ”Analyzing” to “Remove LN”, but the SAM Run Status will still read “Wait”.
26. The user can either let the LN evaporate or carefully lower the cooler containing LN from the coldtrap via a lab-jack.
27. Once the cold trap warms sufficiently the “Sampling System Status” will change from ”Remove LN” to “Ready for SRV Recovery” or “Pumping Out”, depending on what the user selected initially.
28. If the user wishes to recover the xenon to the SRV then go to section 4.5.
29. The user must wait until the “Sampling System Status” changes to idle before collecting another sample.
30. After the analysis is complete the purity results will be written to the Slow Control data base. Add the purity values returned for N₂, O₂, He, Ar, Kr, Kr86, CH₄ into the LUG entry that was started.

4.2 Collecting a Xenon Sample from the Calibration-Xenon Bottle

Hazard Analysis: A cryogen is used in this procedure. Wear PPE including cryo-gloves, safety goggles, and face shield when handling cold cryo-hose fittings and pouring liquid nitrogen. Avoid cuffs and wear proper shoes that cover the toes. Use a portable O₂ monitor during LN transfer to ensure the O₂ concentration is above 19.5%.

Operation Risk: The coldtrap is a cryopump vessel which could potentially accumulate an amount of xenon which could overpressure when the xenon ice warms up. A burst disk set to 41.1 psig will relieve a overpressure in the coldtrap system plumbing.

This procedure is for sampling xenon from a xenon bottle prepared with a known concentration of impurities. A 0.5 L sampling volume will be filled with xenon from a calibration bottle. Once the calibration sample is collected in the 0.5 L sampling volume, you will cool the trap and begin the calibration process in the slow control. When done, the xenon ice in the coldtrap will warm and expand. You can choose to vent this xenon or to recover it to the SRV. The calibration will be done automatically, calibration values will appear in the slow control database. You will need to record several quantities, do this in the LUG.

1. Check the pressure of instrumentation nitrogen. PT-GC10 should read more than 70 psig, and PT-GC9 should read more than 200 psig. If not than the instrumentation nitrogen supply needs to be replaced.
2. Confirm that all VA, VB and VC noted in Table 1 are closed. If not, stop and contact the shift manager. See Table 1.
3. Get liquid nitrogen (LN).
 - a. Locate the sampling system's portable dewar.
 - b. Don proper PPE. Safety glasses, face shield, cryo gloves. Also, wear a portable O₂ monitor.
 - c. Fill the dewar from the SRV vent line. Go to Section 3 and follow the instructions.
4. In slow control, under "Text", the state of "Sampling System Status" should be "Idle", the state of "Sampling System Error Status" should be "None" and the state of "SAM Run Status" should be "Idle". If not then stop, the sampling system is not ready to draw a sample. If "Error" is displayed see Section 6. If "Ready for SRV Recovery" is displayed then see section 4.5. If "Pumping Out", "Analyzing" or "Calibrating" is displayed then wait for those processes to complete. If more than an hour goes by without a change in state then contact the system owner (Attila Dobi).
5. Go to the LUX slow control page, select 'Control' in the top menu [LUX Slow Control](#). Then select 'Sampling' and hit the pencil button.
6. In the 'Sampling System Master Control' box select 'Pre Sample' and then hit the 'Change' button. This will automatically prepare the system for sampling. While the pre-sample program is running the Sampling System Status box will display "PreSample" and the "SAM Run Status" will display "Wait", they can be found under the "Text" tab. Do not change anything on the control page at this time, it could result in an error.
7. After the button is pressed wait 2 minutes and refresh the page. If the Sampling System Master Control's current value changes to 'Ready to Sample' the sampling system is ready to draw the sample from the LUX circulation system. If the "Sampling System Master Control" current value changes to 'Error' then stop and go to Section 6.

8. Minimize the regulator of the calibration bottle, SAM-R1, by twisting the handle counter clockwise until it stops.
9. Open the valve on the calibration bottle. Open SAM-V7 and SAM-V8 then increase the regulator SAM-R1(by twisting the knob clockwise) to fill the 0.5 L sample volume with 700 Torr of calibration xenon. Use PT-SAM2 to monitor the pressure at the output of the regulator. Figure 4 shows the location of PT-SAM2's display.
10. Close the valve on the calibration bottle. Close SAM-V7 and SAM-V8 and minimize SAM-R1(by twisting the handle counter clockwise until it stops).
11. start a LUG entry. Record the data and time of the sample, the port used for sampling, and the pressure of PT-SAM2 after the sample was taken.
12. Go to the LUX slow control page, select “Text” in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the ‘pencil’ button. For help see Figure 3.
13. The “Sampling System Status” current value should be “Ready to Sample” and the value of “SAM Run Status” should be “Idle”. If the value of“Sampling System Master Control” is “Error” then stop and go to Section 6. If the value of “SAM Run Status” is “Wait” then contact the system owner (Attila Dobi). For help see Figure 3.
14. Raise the cooler under the “U” of the coldtrap via a lab-jack until the bottom of the cooler touches the bottom of the “U”.
15. Fill the cooler underneath the cold trap’s “U” to the red “Fill” marking with LN from the sampling system’s dewar.
16. Wait 1 minute, this is sufficient time for the coldtrap plumbing to cool down, once it has been immersed in LN. The coldtrap should remain immersed in LN through the measurement procedure. If the LN level goes below the “Min LN” line then add more LN.
17. Go to the LUX slow control page, select ‘Control’ in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the pencil button.
18. In the ‘Sampling System Master Control’ box select “Calibrate” and then hit the ‘Change’ button. While the analysis program is running. The Sampling System Status box will display “Calibrating” and the “SAM Run Status” will display “Wait”, in slow control under “Text”. Do not change anything on the control page at this time, it could result in an error.
19. After the calibration has begun check that the LN level is above the “Min LN” line, if not then add more LN.
20. The user can leave the system at this point if necessary, as long as they finish the rest of the steps in this section the next day. Once the LN evaporates the xenon will be pumped out and the sampling system will prep for the next measurement. The automated pump out of the xenon will begin approximately five minutes after both temperature sensors on the cold trap read above 260 K.
21. The calibration measurement is completed in about 30 minutes. The value of “Sampling System Status” will change from ”Calibrating” to “Remove LN.”
22. The user can either let the LN evaporate or carefully lower the cooler containing LN from the coldtrap via a lab-jack.

23. Once the cold trap warms sufficiently the “Sampling System Status” will change from ”Remove LN” to “Pumping Out”, during this time the xenon will be pumped away.
24. After the calibration is complete the results will be written to the Slow Control data base. Add the purity values returned for Cal_N2, Cal_O2, Cal_He, Cal_Ar, Cal_Kr, Cal_Kr86, Cal_CH4 into the LUG entry that was started.
25. The user must wait until the “Sampling System Status” changes to idle before collecting another sample.

4.3 Collecting a Xenon Sample from the Xenon Storage Bottles

Hazard Analysis: A cryogen is used in this procedure. Wear PPE including cryo-gloves, safety goggles, and face shield when handling cold cryo-hose fittings and pouring liquid nitrogen. Avoid cuffs and wear proper shoes that cover the toes. Use a portable O2 monitor during LN transfer to ensure the O2 concentration is above 19.5%.

Operation Risk: The coldtrap is a cryopump vessel which could potentially accumulate an amount of xenon which could overpressure when the xenon ice warms up. A burst disk set to 41.1 psig will relieve a overpressure in the coldtrap system plumbing.

This procedure is for sampling xenon from the LUX xenon storage bottles. A 0.5 L sampling volume will be filled with xenon from the LUX circulation system. There are two bottle farms each containing four xenon storage bottles which can be sampled. The valves and pressure transducer corresponding to each xenon storage bottle are summarized in Table 2. See Figures 1, Figure 2 for diagrams of the coldtrap plumbing and sampling port locations. If sampling xenon from the storage bottles get clearance from a gas system expert. Once the sample is collected in the 0.5 L sampling volume, you will cool the trap and begin the analysis process in the slow control. When done, the xenon ice in the coldtrap will warm and vaporize, you can choose to vent this xenon or to recover it to the SRV. The analysis will be done automatically and the purity values will appear in the slow control database. You will need to record several quantities, do this in the LUG as well as the xenon inventory spreadsheet.

Target cylinders	VA	VB	VC	Pressure Gauge D
SB-1	SBV1	SBV11	SV15	PT-SB01
SB-2	SBV2	SBV12	SV15	PT-SB01
SB-3	SBV3	SBV13	SV15	PT-SB01
SB-4	SBV4	SBV14	SV15	PT-SB01
SB-5	SBV5	SBV15	SV30	PT-SB02
SB-6	SBV6	SBV16	SV30	PT-SB02
SB-7	SBV7	SBV17	SV30	PT-SB02
SB-8	SBV8	SBV18	SV30	PT-SB02

Table 2: Storage bottle and panel valves used for sampling xenon bottles and the pressure gauge to watch.

1. Check the pressure of instrumentation nitrogen. PT-GC10 should be more than 70 psig and PT-GC9 should read more than 200 psig. If not than the instrumentation nitrogen supply needs to be replaced.

2. Monitor and ensure the stability of the pressure gauge of interest (see Table 1). The pressure should be stable within 1 psig for one minute while the system is in a steady state. If not, contact the director of operations.
3. Confirm that all VA, VB and VC noted in Table 1 and Table 2 are closed. If not, stop and contact the director of operations. See Table 1.
4. Get liquid nitrogen (LN).
 - a. Locate the sampling system's portable dewar
 - b. Don proper PPE. Safety glasses, face shield, cryo gloves. Also, wear a portable O2 monitor.
 - c. Fill the dewar from the SRV vent line. Go to Section 3 and follow the instructions.
5. In slow control, under “Text”, the state of “Sampling System Status” should be “Idle”, the state of “Sampling System Error Status” should be “None” and the state of “SAM Run Status” should be “Idle”. If not then stop, the sampling system is not ready to draw a sample. If “Error” is displayed see Section 6. If “Ready for SRV Recovery” is displayed then see section 4.5. If “Pumping Out”, “Analyzing” or “Calibrating” is displayed then wait for those processes to complete. If more than an hour goes by without a change in state then contact the system owner (Attila Dobi).
6. Go to the LUX slow control page, select ‘Control’ in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the pencil button.
7. In the ‘Sampling Port Select’ box select ‘XeBottle’. The sampling will not start if you don’t do this. Press change. For help see figure 3.
8. In the ‘Sampling System Master Control’ box select ‘Pre Sample’ and then hit the ‘Change’ button. This will automatically prepare the system for sampling. While the pre-sample program is running the Sampling System Status box will display “PreSample” and the “SAM Run Status” will display “Wait”, they can be found under the “Text” tab. Do not change anything on the control page at this time, it could result in an error. For help see figure 3.
9. After the button is pressed wait 2 minutes and refresh the page. When the Sampling System Master Control’s current value changes to ‘Ready to Sample’ the sampling system is ready to draw the sample. If the “Sampling System Master Control” current value changes to ‘Error’ then stop and go to Section 6.
10. Close all valves on the storage panel. SV1, SV2, SV3, SV6, SV16, SV18, SV15, SV25, SV30, SV31, SV9, SV33, SV32, SV49, SV50 (should be closed anyhow), SV12 and that Regulator SR1 is minimized.
11. Ensure that all xenon storage bottles are closed. SB01, SB02, SB03, SB04, SB05, SB06, SB07, SB08. And that their corresponding VCR hand valves are closed SBV11, SBV12, SBV13, SBV14, SBV15, SBV16, SBV17, SBV18.
12. Ensure that the following valves near the compressor are closed: SV10, SV11, SV37, SV39, SV34, SV43, SV44, SV45. Minimize regulator SR2 by turning the the knob counter clockwise, stop once you encounter resistance, do not force.
13. Note: the values of VA,VB,VC can be found in 2.
14. If PT-SB01 or PT-SB02 read more than 10 PSIA AND the xenon in the lines have been confirmed ‘clean’ within the past 30 days, then cryopump the remaining xenon from the storage panel to SRV (potentially at high pressure) by following sub steps 14.a through 14.e, else skip to 15. This is done to ensure that the xenon from the previous xenon bottle that was sampled is not mixed with the next

bottle to be sampled. Again, before cryopumping the xenon to the SRV ensure that there was no air contamination indicated by any previous sample from the bottle farm, to do so check the LUG entires for ‘Coldtrap’. (If the user wishes to only sample the xenon from the HP lines and not the xenon storage bottles then proceed from step 25, ignoring VA,VB and watching pressures PT-SB01 and PT-SB02 as Gauge D).

- 14.a Ensure that the pressure in the SRV xenon space [PT-S10] is less than 0 psig . Also ensure that SRV LN Space Temperature [TSRV02] is reading below 165K, this will ensure that the SRV is ready for cryopumping xenon. Note: PT-S10 reads in psig, and TSRV02 reads in K.
- 14.b Talk to a gas system expert and the shift manager to make sure nothing besides standard xenon circulation is happening at the time. There should be no SRV recovery from the circulation system, no ACRS alarm condition, and no compressing should be happening.
- 14.c Open (in the following order): SV2, SV9, SV10, SV15 and VC (VC may also be SV15). Cryopump into the SRV until Pressure Gauge D and PT-S10 (on the SRV) flatten out below 5 PSIA. The flattening of the pressure signal vs. time indicates the flow induced by cryo pumping has stopped. Then, empty the line to the sampling system by ensuring CV80, CV42 and MFC8 are closed, then open SV33, SV32, SV44, SV45, SV11. Cryopump into the SRV Pressure Gauge D flattens out below 5 PSIA.
- 14.d Close valves VC, SV15, SV9, SV10, SV2 and SV32, SV33, SV11, SV45, SV44 (in that order).
- 14.e Monitor pressure on Gauge D, PT-S15 and PI-S25 to confirm that you vacuumed the storage panel. Look for the pressures to be stable over a few minutes. Record the level these pressure gauges read out. Estimate the uncertainty of that reading. Remember HP gauge to 4000 psi, can't read well vacuum below 5 PSIA).
15. Double check that SV15, SV16, SV30, SV31, SV18 are closed. Double check that all eight xenon bottles in the bottle farm are closed shut.
16. Attach the turbo pumping cart via SV19 as illustrated in Figure 6 by following steps 16.a to 16.k, or skip ahead to step 17 if the turbo pumping cart is already attached via SV19.
 - 16.a Attach the pumping cart at SV-19. Ensure SV18 and SV19 closed. See Figure 6.
 - 16.b Close vales PCV1, PCV2, PCV3, PCV4 on the pumping cart.
 - 16.c Remove air using SP1 opening PCV3 until pressure drops to mbar level (1e-3 Torr).
 - 16.d Close PCV3, open PCV2.
 - 16.e Start TP1.
 - 16.f Open PCV1.
 - 16.g Pump until 10-5torr (PT-PC1 min pressure reading)
 - 16.h Start leak-checker, with PCV4 closed.
 - 16.i Close PCV1.
 - 16.j Open PCV4, Helium leak check the plumbing to SV-18.
 - 16.k Close PCV4, stop leak checker.
17. Ensure the turbo pumping cart is set up as illustrated in Figure 6 (The following steps use valve labels as indicated on the figure).
18. On the turbo pumping cart ensure that valves PCV4, PCV3 are closed.

19. Start up SP1 (the scroll pump on the turbo pumping cart attached to SV-19). If both the turbo and scroll pump are running then spin down the turbo pump, it will take about 10 minutes, this is to prevent any damage from pumping out the xenon.
20. Open SV19, SV18, SV15, VC and VB (Do Not Opened The Storage Bottle Valve!!!) . This will begin to evacuate the remaining xenon in the high pressure lines using only the scroll pump.
21. Once the pressure by the scroll pump is less than 1 Torr. Start the turbo pump, let it spin up. After the turbo has spun up let it pump on the lines for more than five minutes.
22. Close VC, SV15, SV18 and SV19 (in that order). Place an administrative control lock or tag on SV18 and SV19. [At this point the turbo pumping cart is isolated and can be shut down if the user wishes by closing all hand valves on the turbo cart then spinning down the turbo pump, it will take about 10 minutes, then shutting off the scroll pump].
23. Then open VA (the xenon storage bottle to be sampled), wait for the pressure in the lines to equalize. (Note: VB, the corresponding VCR hand valve to the bottle being sampled, should already be opened).
24. Close VA (the xenon storage bottle that was sampled), also close VB (its corresponding VCR hand valve). Monitor the pressure in the storage panel on Gauge D, this will be the supply pressure to the input of regulator SR2 in the following steps.
25. Open VC.
26. Open SV10 and SV44. Monitor the pressure on Gauge D.
27. Ensure that SR2 (the regulator at the compressor bypass) is minimized then open SV45. This will allow xenon from the high pressure side to flow through the regulator into the low pressure side.
28. Open up the regulator SR2 (by turning the knob clockwise) until the output pressure on PI-S23 reads just above 0 PSIG (or just above 1 ATM). It is ok to go up to 10 PSIG. If the pressure of PI-S23 10 PSIG then crank back one full turn (counter clockwise) on the regulator SR2.
29. Open valve SV11, this will fill the lines up to the sampling port CV89 with xenon from the output of the regulator SR2.
30. Open valve CV89 (behind the circulation panel by the SAES Getter).
31. Slowly open CV90 until PT-SAM2 reaches 700 Torr. Drawing 700 Torr is sufficient for purity analysis. Figure 4 shows which pressure gauge to watch, it is visible to the user as he/she opens the sampling ports. If more pressure is needed slowly increase the output pressure of the regulator SR2 by turning it clockwise.
32. Close the sampling valves CV89 and CV90 to prevent leakage of xenon from the LUX system. Place an administrative lock on CV90 and tag it.
33. Close SV10, SV11, SV44, SV45 and minimize SR2 (by turning the knob counter clockwise until you encounter resistance, do not force)
34. Close VC.
35. Start a LUG entry. Log the data and time of the sample, the port used for sampling, the xenon storage bottle sampled, and the pressure of PT-SAM2 after the sample was taken.
36. Go to the LUX slow control page, select “Text” in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the ‘pencil’ button.

37. The Sampling System Status's current value should be "Ready to Sample" and the value of "SAM Run Status" should be "Idle". If the value of "Sampling System Status" is "Error" then stop and go to Section 6. If the value of "SAM Run Status" is "Wait" then contact the system owner (Attila Dobi). For help see Figure 3.
38. Raise the cooler under the "U" of the coldtrap via a lab-jack until the bottom of the cooler touches the bottom of the "U".
39. Fill the cooler underneath the cold trap's "U" to the "Fill" marking with LN from the sampling system's dewar.
40. Wait 1 minute, this is sufficient time for the coldtrap plumbing to cool down, once it has been immersed in LN.
41. Go to the LUX slow control page, select 'Control' in the top menu [LUX Slow Control](#). Then select 'Sampling' and hit the pencil button.
42. Before the analysis has begun check that the LN level is above the "Min LN" line, if not then add more LN.
43. In the 'Sampling System Master Control' box select either "Analyze and Dump" or "Analyze and Recover" and then hit the 'Change' button. "Analyze and Dump" will pump out the xenon sample after the measurement. "Analyze and Recover" will prompt the user when he/she can recover the xenon to the SRV. For help see Figure 3.
44. While the analysis program is running. The Sampling System Status box will display "Analyzing" and the "SAM Run Status" will display "Wait". Do not change anything on the control page at this time, it could result in an error.
45. If the user chose "Analyze and Dump" then they can leave the system at this point if necessary, as long as they finish the rest of the steps in this section the next day. Once the LN evaporates the xenon will be pumped out automatically and the sampling system will prep for the next measurement. For help see Figure 3.
46. If the user chose "Analyze and Recover" then they should check back in 30 minutes to recover the xenon, or return the next day. Once the LN evaporates SAM will wait for the user recover the xenon to the SRV.
47. The measurement is completed in about 30 minutes. The value of "Sampling System Status" will change from "Analyzing" to "Remove LN", but the SAM Run Status will still read "Wait".
48. The user can either let the LN evaporate or carefully lower the cooler containing LN from the coldtrap via a lab-jack.
49. Once the cold trap warms sufficiently the "Sampling System Status" will change from "Remove LN" to "Ready for SRV Recovery" or "Pumping Out", depending on what the user selected initially.
50. If the user wishes to recover the xenon to the SRV then go to section 4.6 (specifically for SRV recovery when sampling from the xenon bottle farm). NOTE: [If the sample contains air contamination or elevated levels of Kr then pump out the xenon in the sampling system by using the "Pump Out" function of the sampling system. Then contact the DSCM about removing the contaminated xenon in the high pressure lines to the bottle farm.]
51. The user must wait until the "Sampling System Status" changes to idle before collecting another sample.

52. After the analysis is complete the purity results will be written to the Slow Control data base. Add the purity values returned for N2, O2, He, Ar, Kr, Kr86, CH4 into the LUG entry that was started. Also update these values in the Xe-inventory google-doc.

4.4 Collecting a Xenon Sample from the SRV (when warm)

Hazard A cryogen is used in this procedure. Wear PPE including cryo-gloves, safety goggles,

Analysis: and face shield when handling cold cryo-hose fittings and pouring liquid nitrogen.

Avoid cuffs and wear proper shoes that cover the toes. Use a portable O2 monitor during LN transfer to ensure the O2 concentration is above 19.5%.

Operation The coldtrap is a cryopump vessel which could potentially accumulate an amount of xenon which could overpressure when the xenon ice warms up. A burst disk set to

41.1 psig will relieve a overpressure in the coldtrap system plumbing.

This procedure is for sampling xenon from the LUX xenon gas system. A 0.5 L sampling volume will be filled with xenon from the SRV. When sampling from the SRV get clearance from an SRV and gas system expert. Once the sample is collected in the 0.5 L sampling volume, you will cool the trap and begin the analysis process in the slow control. When done, the xenon ice in the coldtrap will warm and vaporize, you can choose to vent this xenon or to recover it to the SRV. The analysis will be done automatically and the purity values will appear in the slow control database. You will need to record several quantities, do this in the LUG.

1. Check the pressure of instrumentation nitrogen. PT-GC10 should be more than 70 psig and PT-GC9 should read more than 200 psig. If not than the instrumentation nitrogen supply needs to be replaced.
2. Monitor and ensure the stability of the pressure gauge of interest (see Table 1). The pressure should be stable within 1 psig for one minute while the system is in a steady state. If not, contact the director of operations.
3. Confirm that all VA, VB and VC noted in Table 1 are closed. If not, stop and contact the director of operations. See Table 1.
4. Get liquid nitrogen (LN).
 - a. Locate the sampling system's portable dewar
 - b. Don proper PPE. Safety glasses, face shield, cryo gloves. Also, wear a portable O2 monitor.
 - c. Fill the dewar from the SRV vent line. Go to Section 3 and follow the instructions.
5. In slow control, under "Text", the state of "Sampling System Status" should be "Idle", the state of "Sampling System Error Status" should be "None" and the state of "SAM Run Status" should be "Idle". If not then stop, the sampling system is not ready to draw a sample. If "Error" is displayed see Section 6. If "Ready for SRV Recovery" is displayed then see section 4.5. If "Pumping Out", "Analyzing" or "Calibrating" is displayed then wait for those processes to complete. If more than an hour goes by without a change in state then contact the system owner (Attila Dobi).
6. Go to the LUX slow control page, select 'Control' in the top menu [LUX Slow Control](#). Then select 'Sampling' and hit the pencil button.

7. In the ‘Sampling Port Select’ box select ‘XeBottle’. The sampling will not start if you don’t do this. Press change. For help see figure [3](#).
8. In the ‘Sampling System Master Control’ box select ‘Pre Sample’ and then hit the ‘Change’ button. This will automatically prepare the system for sampling. While the pre-sample program is running the Sampling System Status box will display “PreSample” and the “SAM Run Status” will display “Wait”, they can be found under the “Text” tab. Do not change anything on the control page at this time, it could result in an error. For help see figure [3](#).
9. After the button is pressed wait 2 minutes and refresh the page. When the Sampling System Master Control’s current value changes to ‘Ready to Sample’ the sampling system is ready to draw the sample from the LUX circulation system. If the “Sampling System Master Control” current value changes to ‘Error’ then stop and go to Section [6](#).
10. Sampling from the warm SRV:
 11. Ensure that the SRV is warm (no xenon ice) and ready for sampling. Check that the temperature of the LN space, TSRV02 is greater than 240 K and that the pressure inside the xenon space, PT-S10, is between 0 and 1000 PSIG. (This is to ensure that the xenon has fully vaporized and there is no longer residual ice).
 12. Close all valves on the storage panel. SV1, SV2, SV3, SV6, SV16, SV18, SV19, SV15, SV25, SV30, SV31, SV9, SV33, SV32, SV49, SV50 (should be closed anyhow), SV12 and that Regulator SR1 is minimized.
 13. Ensure that the following valves on the SRV are close, if not close them. SRVV1, SRVV2, SRVV3.
 14. Ensure that the following valves near the compressor are closed : SV10, SV11, SV37, SV39, SV34, SV43, SV44, SV45. Minimize regulator SR2 by turning the the knob counter clockwise, stop once you encounter resistance, do not force.
 15. Monitor the pressure in the SRV xenon space, PT-S10, while sampling. If the reading shows signs of continuous decline to atmospheric pressure it is an indication of a burst disk failure.
 16. Open SRVV2, SV2, SV9
 17. Open SV10 and SV44.
 18. Ensure that SR2 (the regulator at the compressor bypass) is minimized then open SV45. This will allow xenon from the high pressure side to flow through the regulator into the low pressure side.
 19. Open up the regulator SR2 (by turning the knob clockwise) until the output pressure on PI-S23 reads just above 0 PSIG (or just above 1 ATM). It is ok to go up to 10 PSIG. If the pressure of PI-S23 10 PSIG then crank back one full turn (counter clockwise) on the regulator SR2.
 20. Open valve SV11, this will fill the lines up to the sampling port CV89 with xenon from the output of the regulator SR2.
 21. Open valve CV89 (behind the circulation panel by the SAES Getter).
 22. Slowly open CV90 until PT-SAM2 reaches 700 Torr. Drawing 700 Torr is sufficient for purity analysis. Figure [4](#) shows which pressure gauge to watch, it is visible to the user as he/she opens the sampling ports. If more pressure is needed slowly increase the output pressure of the regulator SR2 by turning it clockwise.

23. Close the sampling valves CV89 and CV90 to prevent leakage of xenon from the LUX system. Place an administrative lock on CV90 and tag it.
24. Close SV10, SV11, SV44, SV45 and minimize SR2 (by turning the knob counter clockwise until you encounter resistance, do not force)
25. Close SV2, SV9 and SRVV2 (unless SRVV2 is intended to be in a locked open state, ask the current Detector Operator Manager).
26. Start a LUG entry. Log the data and time of the sample, the port used for sampling, the xenon storage bottle sampled, and the pressure of PT-SAM2 after the sample was taken.
27. Go to the LUX slow control page, select “Text” in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the ‘pencil’ button.
28. The Sampling System Status’s current value should be “Ready to Sample” and the value of “SAM Run Status” should be “Idle”. If the value of “Sampling System Status” is “Error” then stop and go to Section 6. If the value of “SAM Run Status” is “Wait” then contact the system owner (Attila Dobi). For help see Figure 3.
29. Raise the cooler under the “U” of the coldtrap via a lab-jack until the bottom of the cooler touches the bottom of the “U”.
30. Fill the cooler underneath the cold trap’s “U” to the “Fill” marking with LN from the sampling system’s dewar.
31. Wait 1 minute, this is sufficient time for the coldtrap plumbing to cool down, once it has been immersed in LN.
32. Go to the LUX slow control page, select ‘Control’ in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the pencil button.
33. Before the analysis has begun check that the LN level is above the “Min LN” line, if not then add more LN.
34. In the ‘Sampling System Master Control’ box select “Analyze and Dump” and then hit the ‘Change’ button. “Analyze and Dump” will pump out the xenon sample after the measurement. Do not use “Analyze and Recover” since the SRV is warm and not ready for xenon recovery. For help see Figure 3.
35. While the analysis program is running. The Sampling System Status box will display “Analyzing” and the “SAM Run Status” will display “Wait”. Do not change anything on the control page at this time, it could result in an error.
36. If the user chose “Analyze and Dump” then they can leave the system at this point if necessary, as long as they finish the rest of the steps in this section the next day. Once the LN evaporates the xenon will be pumped out automatically and the sampling system will prep for the next measurement. For help see Figure 3.
37. If the user chose “Analyze and Recover” then they should check back in 30 minutes to recover the xenon, or return the next day. Once the LN evaporates SAM will wait for the user recover the xenon to the SRV (section 4.5).
38. The measurement is completed in about 30 minutes. The value of “Sampling System Status” will change from ”Analyzing” to “Remove LN”, but the SAM Run Status will still read “Wait”.

39. The user can either let the LN evaporate or carefully lower the cooler containing LN from the coldtrap via a lab-jack.
40. Once the cold trap warms sufficiently the “Sampling System Status” will change from ”Remove LN” to “Ready for SRV Recovery” or “Pumping Out”, depending on what the user selected initially.
41. If the user wishes to recover the xenon to the SRV then go to section [4.5](#).
42. The user must wait until the “Sampling System Status” changes to idle before collecting another sample.
43. After the analysis is complete the purity results will be written to the Slow Control data base. Add the purity values returned for N2, O2, He, Ar, Kr, Kr86, CH4 into the LUG entry that was started.

4.5 Recovering Sampled Xenon to the SRV

Hazard Cryogen and pressure hazards mitigated as noted in Section 2.4
 Analysis:

1. This section should only be used when the value of “Sampling System Status”, in slow control, is “Ready for SRV Recovery.” For help see Figure [3](#).
2. If the user wishes to cancel the SRV recovery and wants to pump out the xenon instead then skip ahead to step [12](#).
3. Ensure that the pressure in the SRV xenon space [PT-S10] is less than 0 psig . Also ensure that SRV LN Space Temperature [TSRV02] is reading below 165K, this will ensure that the SRV is ready for cryopumping xenon. Note: PT-S10 reads in psig, and TSRV02 reads in K.
4. Talk to a gas system expert and the shift manager to make sure nothing besides standard xenon circulation is happening at the time. There should be no SRV recovery from the circulation system, no ACRS alarm condition, and no compressing should be happening.
5. Ensure that CV11, CV37, CV61, CV76, CV80, CV9 and CV42 are closed.
6. Ensure that SV1, SV10, SV11, SV30, SV25, SV3, SV49, SV15, SV33, SRVV1, and SRVV3 are closed.
7. Open SRVV2 (SRVV2 is locked open and is the path into the SRV). Then open SV2, SV9.
8. Wait until there is no high pressure in the line by checking that the SRV xenon space pressure PT-S10 has leveled off at less than 0 psig.
9. Open SV33 and then SV32, this will connect the high pressure side of the plumbing to the low pressure side.
10. Open CV89 and CV90. This will begin the process of cryopumping the xenon from the sampling system into the SRV.
11. Once PT-SAM2 reads less than 10 Torr a sufficient amount of xenon has been recovered into the SRV. Figure [4](#) shows the location of PT-SAM2’s display. Note: PT-SAM2 reads in Torr.
12. Close SV32, SV33, SV9, SV2, CV89 and CV90.

13. In “Sampling System Master Control” select “Pump Out” and hit change. This will evacuate the remaining xenon remaining in the sampling system. The the system will automatically prep for the next sample.
14. The value of “Sampling System Status” will change to “Pumping Out” while the remaining xenon vents, and then to “Idle” once the process is complete.

4.6 Recovering Sampled Xenon to the SRV when Sampling From the Xenon Storage Bottles

Hazard Cryogen and pressure hazards mitigated as noted in Section 2.4
 Analysis:

1. This section should only be used when the value of “Sampling System Status”, in slow control, is “Ready for SRV Recovery” AND when sampling xenon from the bottle farm. For help see Figure 3.
2. Ensure that the pressure in the SRV xenon space [PT-S10] is less than 0 psig . Also ensure that SRV LN Space Temperature [TSRV02] is reading below 165K, this will ensure that the SRV is ready for cryopumping xenon. Note: PT-S10 reads in psig, and TSRV02 reads in K.
3. Talk to a gas system expert and the shift manager to make sure nothing besides standard xenon circulation is happening at the time. There should be no SRV recovery from the circulation system, no ACRS alarm condition, and no compressing should be happening.
4. Ensure that CV11, CV37, CV61, CV76, CV80, CV9 and CV42 are closed.
5. Ensure that SV1, SV10, SV11, SV30, SV25, SV3, SV49, SV15, SV33, SV44, SV37, SV34, SRVV1, and SRVV3 are closed.
6. Open SRVV2 (SRVV2 is locked open and is the path into the SRV). Then open SV2, SV9, SV10, SV15 and SV30.
7. Wait until there is no high pressure in the line by checking the SRV xenon space pressure PT-S10, PT-SB01 and PT-SB02. Wait for the readings to level off below 5 PSIA to ensure there is no high pressure remaining in the line.
8. Open SV33, SV44 and then SV32, SV45 and SV11, this will connect the high pressure side of the plumbing to the low pressure side.
9. Open CV89 and CV90. This will begin the process of cryopumping the xenon from the sampling system into the SRV.
10. Once PT-SAM2 reads less than 10 Torr a sufficient amount of xenon has been recovered into the SRV. Figure 4 shows the location of PT-SAM2’s display. Note: PT-SAM2 reads in Torr.
11. Close CV89, CV90, SV11, SV32, SV33, SV45, SV44, SV10, SV15, SV30, SV9, SV2.
12. In “Sampling System Master Control” select “Pump Out” and hit change. This will evacuate the remaining xenon remaining in the sampling system in preperation for the next sample.
13. The value of “Sampling System Status” will change to “Pumping Out” while the remaining xenon vents, and then to “Idle” once the process is complete.

5 Manual Sampling and Analysis

Hazard Cryogen and pressure hazards mitigated as noted in Section 2.4
Analysis:

This section describes how to manually perform the xenon sampling and purity analysis. This section is to be used in the case that the slow control is not working, or the sampling system logic is malfunctioning.

5.1 Manually Collecting a Xenon Sample from the Circulation System

This procedure is for sampling xenon from the LUX xenon gas system. A 0.5 L sampling volume will be filled with xenon from the LUX system. The valves and pressure transducer corresponding to each sampling location are summarized in Table 1. See Figure 1 and Figure 2 for diagrams of the coldtrap plumbing and sampling port locations.

1. Refer to Table 1 for valves VA, VB and VC and the pressure gauge to monitor as they will depend on the location you will be collecting a sample from.
2. Monitor and ensure the stability of the pressure gauge of interest (see Table 1). The pressure should be stable within 1 psig for one minute while the system is in a steady state. If not stop and contact the shift manager.
3. Confirm that all VA, VB and VC noted in Table 1 are closed. If not, stop and contact the shift manager. See Table 1.
4. Check the value of PT-SAM2 is less than 5 Torr. This is to ensure that there is no xenon or air in the sampling lines. Note, there is up to a 5 Torr offset of PT-SAM2. Figure 4 shows the location of PT-SAM2's display.
5. If PT-SAM2 is greater than 5 Torr then Turbo Pump 2 will have to be turned off before pumping out the sampling lines. If PT-SAM2 is less than 5 Torr then go to step 13. Pumping on the plumbing when it contains more than 5 Torr could damage the turbo pump.
6. Go to the LUX slow control page, select 'Control' in the top menu [LUX Slow Control](#). Then select 'Sampling' and hit the 'pencil' button.
7. Set the value of "SAM Vacuum Gauge 1 Control" and "SAM Vacuum Gauge 2 Control" to OFF. For help see Figure 3.
8. Ensure that the valves of SAM-V[1-6] are set to Closed. If not then set them all to Closed.
9. Ensure that SAM-V7 is closed (manual valve with a green handle on the sampling system)-
10. Set the value of "SAM Turbo 2 Control" to OFF and wait 10 minutes for the turbo to spin down.
11. Open SAM-V1, SAM-V2 and SAM-V[4-6]. This will begin to pump out the remaining pressure in the sampling volume. The valve is fully open when the valve status reads 5 V in slow control, if there is a reading of less than 5 volts than the valve has not full opened. The valve status will read -10 V in the closed state.
12. Open VB and VC.
13. Wait until PT-SAM2 goes below 5 Torr.

14. Once PT-SAM2 is less than 5 Torr, turn on Turbo Pump 1 by setting “SAM Turbo 1 CTRL” to ON in the slow control.
15. After 5 minutes turn on PT-SAM3 by setting “SAM Vacuum Gauge Control” to ON.
16. Wait until PT-SAM3 reads less than 1e-7 Torr. Then open SAM-V3 and close SAM-V2 in slow control.
17. Ensure that SAM-V3 is open, then wait until PT-SAM1 reads less than 1e-7 Torr. To ensure that a sufficient vacuum is reached before drawing a xenon sample.
18. Close SAM-V1 and SAM-V[4-6].
19. Unlock and open VA to fill the 0.5 L sample volume with xenon from the LUX circulation system. After about 5 seconds the bottle should be full and at system pressure. PT-SAM2 should read a similar pressure PT-A (see Table 1). You can also choose to just crack VA open to draw a smaller sample. Drawing 700 Torr, read by PT-SAM2, is sufficient for analysis. Figure 4 shows the location of PT-SAM2’s display, it is visible to the user as he/she opens the sampling valves.
20. Close the sampling valves used(VA, VB and VC) to prevent leakage of xenon from the LUX system. Place an administrative lock on VA and tag it.
21. The sample is taken.
22. Start a LUG entry. Log the data and time of the sample, the port used for sampling, and the pressure of PT-SAM2 after the sample was taken.

5.2 Collecting a Xenon Sample from the Calibration-Xenon Bottle

This procedure is for sampling xenon from a xenon bottle prepared with a known concentration of impurities. A 0.5 L sampling volume will be filled with xenon from a calibration bottle.

1. Confirm that all VA, VB and VC noted in Table 1 are closed. See Table 1. If not, stop and contact the shift manager.
2. Go to the LUX slow control page, select ‘Control’ in the top menu [LUX Slow Control](#). Then select ‘Sampling’ and hit the ‘pencil’ button.
3. Set the value of “SAM Vacuum Gauge 1 Control” and “SAM Vacuum Gauge 2 Control” to OFF.
4. Ensure that the valves of SAM-V[1-6] are set to Closed. If not then set them all to Closed.
5. Ensure that SAM-V7 is closed (manual valve with a green handle on the sampling system)-
6. Set the value of “SAM Turbo 1 Control” to OFF and wait 10 minutes for the turbo to spin down.
7. Ensure that the calibration xenon bottle’s valve is closed (closes clockwise)
8. Open SAM-V1 and SAM-V[3-6]. Also open SAM-V7, SAM-V8 and increase SAM-R1 by turning the handle clockwise.
9. Wait until PT-SAM2 goes below 5 Torr.
10. Once PT-SAM2 is less than 5 Torr, turn on Turbo Pump 1 by setting “SAM Turbo 1 CTRL” to ON in the slow control.
11. After 5 minutes turn on PT-SAM1 by setting “SAM Vacuum Gauge Control” to ON.

12. Wait until PT-SAM1 reads less than 1e-7 Torr. To ensure that a sufficient vacuum is reached before drawing a calibration xenon sample.
13. Close SAM-V1 and SAM-V[3-6]. Also minimize SAM-R1 by turning the regulator knob counter-clockwise.
14. Increase the regulator SAM-R1(by twisting the handle clockwise) to fill the 0.5 L sample volume with 700 Torr of calibration xenon. Use PT-SAM2 to monitor the pressure at the output of the regulator.
15. Close SAM-V7, SAM-V8 and minimize SAM-R1(by twisting the handle counter clockwise until it stops). Also, close the cylinder valve on the calibration xenon bottle.
16. The calibration sample is taken.

5.3 Manual Purity Analysis

Hazard Analysis: A cryogen is used in this procedure. Wear PPE including cryo-gloves, safety goggles, and face shield when handling cold cryohose fittings and pouring liquid nitrogen. Avoid cuffs and wear proper shoes that cover the toes. Use a portable O2 monitor during LN transfer to ensure the O2 concentration is above 19.5%.

Operation Risk: The coldtrap is a cryopump vessel which could potentially accumulate an amount of xenon which could overpressure when the xenon ice warms up. A burst disk set to 41.1 psig will relieve a overpressure in the coldtrap system plumbing.

This procedure describes how to use the coldtrap to measure the purity of a sample of xenon. Once the sample is collected in the 0.5 L sampling volume, you will cool the trap and introduce the sampled xenon. Then you will measure and record the impurities seen at the RGA. When done, you will allow the xenon in the coldtrap to warm and expand. After analysis the xenon in the trap will be pumped out by scroll-pump. You will need to record several quantities, do this in the LUG.

1. Close the following valves: SAM-V1, SAM-V2, SAM-V3, SAM-V4, SAM-V5, SAM-V6, SAM-V7. (SAM-V1 though 6 are settable in the slow control).
2. Open SAM-V3
3. Using Turbo Pump 1, pump the system down until PT-SAM1 reads below 5e-8 Torr.
4. Turn on the RGA's (Residual Gas Analyzer) filament and the CEM (Electron Multiplier). The RGA is controlled in the slow control under 'Sampling'.
5. If the RGA was previously off, run the RGA for 30 minutes to get rid of spurious peaks while the filament initially degases.
6. Get liquid nitrogen (LN).
 1. Locate the sampling system's portable dewar
 2. Don proper PPE. Safety glasses, face shield, cryo gloves and jacket. Also, wear a portable O2 monitor.
 3. Fill the dewar from the SRV vent line. Go to Section 3 and follow the instructions.

4. Raise the cooler under the “U” of the coldtrap via a lab-jack until the bottom of the cooler touches the bottom of the “U”.
4. Fill the cooler underneath the cold trap’s “U” to the “Fill” marking with LN from the sampling system’s dewar.
6. Wait 1 minute, this is sufficient time for the coldtrap plumbing to cool down, once it has been immersed in LN. The coldtrap should remain immersed in LN through the measurement procedure. If the LN level goes below the “Min LN” line then add more LN.
7. Ensure SAM-V4, SAM-V5 and SAM-V6 are closed. (Settable in slow control under ‘Sampling’)
8. Record the pressure on PT-SAM2 in the LUG entry. This measurement is very important, because it will be needed in the data analysis.
9. Record the pressure on the vacuum gauge (PT-SAM1) in the LUG entry.
10. In the slow control Sensor Setup increase the sampling rates of SAM_RGA_N2, SAM_RGA_O2, SAM_RGA_He, SAM_RGA_Ar, SAM_RGA_Kr, SAM_RGA_Kr86, SAM_RGA_CH4 and SAM_RGA_Xe to sample every 3 seconds.
11. The next step is to open the leak-valve SAM-V4 to introduce some xenon into the coldtrap and allow xenon ice to form. We use a leak rate that is small enough that no significant change in pressure should be seen on PT-SAM2 during ice formation. If the pressure on PT-SAM2 starts dropping noticeably (by more than 5 torr), then the leak valve is incorrectly set. In that case, close the shut-off valve SAM-V4 immediately. (Also note that the PT-SAM2 pressure may fluctuate by a few tenths of a Torr due to changes in the room temperature.)
12. The Vacuum gauge, PT-SAM1, should rise to just under 1e-5 Torr. This indicates the presence of xenon ice in the trap. The RGA should respond, as well, with higher pressures. The xenon partial pressure should quickly plateau Record its value in the LUG entry.
13. Wait at least 20 minutes for RGA partial pressures to stabilize.
14. Record the stable pressure read by PT-SAM2 in the LUG entry.
15. In the next step, we will open a leak-valve with a higher leak setting to make the purity measurement. The data analysis is simpler if the measurement time is always five minutes. Please time this carefully with a stop watch.
16. Noting the time carefully, open the leak-valve SAM-V5 for 3 minutes. (Keep SAM-V4 open)
17. The pressure as read by PT-SAM2 will decline as xenon is drawn from the sampling volume into the cold trap. RGA readings will rise for a minute or so, then fall slowly for the rest of the measurement.
18. Record the peak RGA reading for O2 (Mass 32), Kr (Mass 84), Ar (Mass 40) and N2 (Mass 28). These will be needed for later analysis.
19. Record the pressure on the vacuum gauge PT-SAM1.
20. After 3 minutes, close the leak-valves SAM-V4 and SAM-V5.
21. Wait 5 minutes. (To get RGA background levels)
22. Noting the time carefully, open the leak-valve SAM-V6 for 5 minutes.

23. The pressure in the sampling system will drop. RGA readings will rise for a minute or so, then fall slowly for the rest of the measurement.
24. Record the peak RGA reading for O2 (Mass 32), Kr (Mass 84), Ar (Mass 40) and N2 (Mass 28). These will be needed for later analysis.
25. Record the pressure on the vacuum gauge PT-SAM1.
26. After 5 minutes, close the leak-valve by closing SAM-V6.
27. Record the final pressure reading on PT-SAM2.
28. In the slow control Sensor Setup change the sampling rates of SAM_RGA_N2, SAM_RGA_O2, SAM_RGA_He, SAM_RGA_Ar, SAM_RGA_Kr, SAM_RGA_Kr86, SAM_RGA_CH4 and SAM_RGA_Xe to sample every 60 seconds.
29. When you are done with the measurement warm up the coldtrap by removing the LN Dewar from the “U”.
 1. Close SAM-V3
 2. Carefully lower the cooler containing LN from the coldtrap via a lab-jack.
 3. Place a bucket underneath the coldtrap, to catch any condensation that may drip from the cold plumbing.
 4. Wait 5-10 minutes for the trap to warm up. Watch the analog pressure gauge(PI-SAM1) on the coldtrap. Once the pressure stops rising all the xenon is back to gas phase.
30. Turn off Turbo-Pump 2(settable in slow control) , and wait until the turbo spins down. Keep the scroll-pump running (play a game of foosball!). DO NOT TURN OFF TURBO PUMP 1! (Turbo Pump 1 is the one with the RGA)
31. When Turbo-Pump 2 has spun down, open SAM-V1, SAM-V2, SAM-V4, SAM-V5, SAM-V6. This will pump down the coldtrap volume with the scroll-pump.
32. Turn on PT-SAM2. Once the pressure on PT-SAM2 is less than 5 Torr, turn on Turbo Pump 2 (settable in slow control).
33. Once PT-SAM3 pressure is less than 1e-5Torr, open SAM-V3.
34. Close SAM-V2 and then turn off PT-SAM3, and Turbo Pump 2.
35. The sampling system is once again being pumped to vacuum.
36. Contact the system owner for information on how to derive a purity result (Attila Dobi). Or See Appendix: [C](#)

6 Debugging Sampling System Errors

This section is meant to guide the user in the even that the “Sampling System Status” displays “Error.” The following error conditions are possible and can be debugged. 1) Xenon is too dirty. 2) Input pressure did not decline. 3) Valve Failure. 4) Low Sample Pressure. Do not start debugging while the status of ““SAM Run Status””, in slow control, reads “Wait”. It is important to let the code finish before changing values on the Control page. Once the status of ““SAM Run Status”” reads “Idle” then it is okay to proceed. If ““SAM Run Status”” does not change to “Idle” within 30 minutes after an error is displayed then contact the system owner (Attila Dobi).

6.1 Poor Vacuum

This message is displayed when the value of vacuum gauge PT-SAM1 or the RGA partial pressure of N₂, O₂ is to high to begin sampling. These parameters indicate that the sampling system plumbing has not yet been sufficiently pumped out.

To clear the error follow these steps:

1. In “Sampling System Master Control” select “Idle” and hit the “change” button.
2. Ensure that PT-SAM2 reads less than 5 Torr (the sampling volume is in vacuum). If not then contact the system owner (Attila Dobi).
3. In slow control turn off “SAM Vacuum Gauge 2” [PT-SAM3].
4. Open SAM-V1 and SAM-V2. If “SAM Turbo 2” is not on, then turn it on in slow control and wait 2 minutes. Then turn on “SAM Vacuum Gauge 2” [PT-SAM3].
5. Once “SAM Vauum Gauge 2” [PT-SAM3] reads less than 1e-6 Torr. Open SAM-V3.
6. Wait until PT-SAM1 reads less than 1e-6 Torr. Then resume section 4.1.

6.2 Xenon is too dirty

If this message is displayed then the sampled xenon contains to much of an impurity for the analysis to continue. To find out which species caused the overpressure check plots of all SAM_RGA partial pressures from the time the analysis was begun until the time of the Error condition.

To clear the error follow these steps:

1. Remove the LN from the cold trap’s U, by lowering the cooler with the lab jack.
2. Wait until SAM-TC1 and SAM-TC2 read more than 265 K. This is sufficient temperature for all the xenon to have vaporized.
3. In “Sampling System Master Control” select “Pump Out” and hit the “change” button.
4. The sampled xenon will be evacuated and the sampling system will prep for the next sample.
5. Once the xenon has pumped away the “Sampling System Status” will display “Idle”.

6.3 Input Pressure Did Not Decline

If this message is displayed then the sampling valves VA, VB and VC (to the circulation panel) might have been left open by the operator. The system will stop the sampling process to prevent too much xenon from being drawn out of the circulation panels. This error might also be displayed if pneumatic pressure was lost during the analysis process. The user should do the following:

1. Confirm that all VA, VB and VC noted in Table 1 are closed.
2. Check that there is between 60-100 psig being supplied by the instrumentation nitrogen feed. The pressure is read out on sensor PT-GC10.
3. Once these have been checked the user can re-try the analysis.
4. In “Sampling System Master Control” select “Analyze&Dump” or “Analyze&Recover” and hit the “change” button. The user can chose to pump out the xenon after analysis or recover it to the SRV.
5. The user should continue at Section 4.3

6.4 Valve Failure

If this message is displayed then the sampling valves have failed to open, potential due to loss of pneumatic pressure. The user should do the following:

1. Check that there is between 80-100 psig being supplied by the instrumentation nitrogen feed. The pressure is read out on sensor PT-GC10, and the nitrogen supply bottle is PT-GC9.
2. Once this has been checked the user can re-try the analysis.
3. In “Sampling System Master Control” select “Analyze&Dump” or “Analyze&Recover” and hit the “change” button. The user can chose to pump out the xenon after analysis or recover it to the SRV.
4. The user should continue at Section 4.3

6.5 Low Sample Pressure

If this message is displayed then the initial pressure of the xenon sample is insufficient for analysis. The user should do the following:

1. There needs to be at least 400 Torr of pressure read by PT-SAM2.
2. Go to section 4.2, this will guide the user to drawing the xenon sample. Ensure that PT-SAM2 reaches 400 Torr.
3. Once this has been checked the user can re-try the analysis.
4. In “Sampling System Master Control” select “Analyze&Dump” or “Analyze&Recover” and hit the “change” button. The user can chose to pump out the xenon after analysis or recover it to the SRV.
5. The user should continue at Section 4.3

6.6 Low LN

If this message is displayed when there is insufficient LN to continue.

1. Follow the steps in section 3 to get more LN. And fill the cooler underneath the cold trap’s “U” with LN.
2. Wait 1 minute after adding more LN.
3. Under Sampling System Master Control, Select ‘Idle’ then hit Change. This is necessary to clear the Sampling System Error message.
4. Under ‘Sampling Port Select’, Select the current sampling location and hit ‘Change’.
5. Under Sampling System Master Control, Select ‘PreSample’ then hit ‘Change’. The analysis can not be restarted with out doing the PreSample step again, there is no ill effect in re-running ‘PreSample’ once the sample has already been collected.
6. Restart the purity analysis or calibration process. Section 4.1 and 4.2.

6.7 PumpOutErr

If this message is displayed when the pump out function in SAM logic returns an error.

1. Check that there is between 80-100 psig being supplied by the instrumentation nitrogen feed. The pressure is read out on sensor PT-GC10, and the nitrogen supply bottle is PT-GC9.
2. Ensure that the cooler containing LN has been lowered from the U of the cold trap. And that SAM-TC1 and SAM-TC2 read more than 265 K.
3. If the status of “SAM Run Status” reads “Idle” then go to the Control page under “Sampling” and in “SAM Master Control” select “PumpOut” and hit change. This will restart the pump out process.
4. If the problem persists, or if the value of “SAM Run Status” remains at “Wait” for more than 5 minutes, then contact the system owner (Attila Dobi)

6.8 NetErr

If this message is displayed when a network time out occurred, or if a value state or an instrument control command failed.

1. Check that there is between 80-100 psig being supplied by the instrumentation nitrogen feed. The pressure is read out on sensor PT-GC10, and the nitrogen supply bottle is PT-GC9.
2. Check that all ethernet and power cables on SAM are plugged in and that the Sampling System instrument values are updating in slow control.
3. Contact the system owner (Attila Dobi)

A Sampling System Logic

This section explains the decision tree that is made when the following is selected “Idle”, “Pre Sample”, “Analyze&Dump”, “Analyze&Recover”, “Calibrate”, “PumpOut” and “Error”. The code is contained in the SC_Backend/slow_control_code/LUX_SAM/SAM.c

Note: At each step the code will check the time stamp of the value from the MySQL data base. If it is less than 120 seconds then there is a network connection error. SAM will go to its Error condition and display NetErr.

A.1 Sampling System Control: Error

The ‘Error’ status is displayed if one of the parameters required to continue along the logic tree failed. All valves will be shut by SAM.c

1. Close SAM V1, V2, V3, V4, V5, V6.
2. Set all sampling system update period values to 60 seconds.
3. Display the appropriate error in “Sampling System Master Error” text box.
4. Display “Error” in “Sampling System Status” text box.

A.2 Sampling System Control: Idle

When this value is selected the sampling system logic does nothing.

A.3 Sampling System Control: PreSample

1. NewSetValue=1;
2. Check N2PP,O2PP and PT-SAM1 < 1e-7 and timestamp is < 5min. If not then Error-poor vacuum.
3. Close SAM V1, V2, V4, V5, V6. Open SAM V3. Check status. If valve status not confirmed then Error-valve failure.
4. If OK then display "Ready to sample".

A.4 Sampling System Control: Analyze&Dump/Analyze&Recover/Calibrate

Note: the option of "Calibrate" is identical to "Analyze&Dump", except purity values will not be calculated and written to the slow control table.

1. Analyze and dump the sample when new_set_val == 2. Analyze and wait for the user to recover the sample to the SRV when new_set_val == 3. Run Calibration when new_set_val==4.
2. Check TP1 current >0.2 Amp and <1.0 Amp //Turbo pump on?
3. Check RGA on
4. Check N2&O2&PT-SAM1 <1e-7 //no air leak, plumbing is sufficiently clean. If not then Error-poor vacuum.
5. Check P3>400 Torr. // Sufficient pressure for a xenon sample. If not then Error-low sample pressure
6. Assign a dummy variable to the initial pressure, check that after sampling the value has decreased
7. Check V1,V2,V4,V5,V6 are closed & V3 open. If not then stop! Error-valve failure.
8. Check TC1&TC2 < 85 K //make sure the cold trap is immersed in LN. If not then Error-low LN.
9. Open V4 // to form ice
10. Check V4 is open? If not then Error-valve failure.
11. Check XePP, N2PP, O2PP and PT-SAM1 over pressure condition every 1 min. If overpressure then close V4 and display Error-Dirty Xe.
12. wait 5 min
13. Close V4
14. Ensure V4 is closed. If not then Error-valve failure.
15. wait 5 min
16. Check Xe PP before moving on
17. Check that V4, V5, V6, V1, V2 are still closed! & V3 is open. If not then Error-valve.
18. Open V5 // for first measurement
19. Check V5 is open? If not then Error-valve failure.
20. wait 3 min

21. Check XePP, N2PP, O2PP and PT-SAM1 every 1 min. If overpressure then close V4 and display Error-Dirty Xe.
22. Close V5
23. Ensure V5 is closed. If not then Error-valve failure.
24. wait 5 min, In the last 3 min define RGA backgrounds
25. Check that the pressure remaining in the sampling system is less than it was before flowing. If not then shut all valves! Error-input pressure did not decline.
26. Check TC gauge < 85 K
27. Open V6 // for high flow measurement
28. Check V6 is open? If not then Error-valve failure.
29. wait 5 min. Average the values of N2, O2, He, Ar, Kr, CH4 and Flow for the first 3 minutes // will be used to calculate purity
30. Check XePP, N2PP, O2PP, and PT-SAM1 every 1 min. If overpressure then close V4 and display Error-Dirty Xe.
31. Close V6
32. Ensure V6 is closed. If not then Error-valve failure.
33. wait 5 min, In the last 3 min define new backgrounds
34. Close V3.
35. Check V3 closed? If not then Error-valve failure.
36. Measurement is done
37. Run Function to write purity values to a table. Either a Calibration(new_set_val==4) or Sample Analysis(new_set_val==2—3). // First calculate the background subtracted leak rate normalized values then divide by the latest calibration.
38. Begin Clean Up.
39. Check TC1 > 270 K //LN has evaporated? If not then wait. The value of “Sampling System Status” will change to “Remove LN” and will remain there until the cold trap has sufficiently warmed.
40. Check V1, V2, V3, V4, V5, V6 are closed. If not then close.
41. Open V4, V5, V6 and Check status. If not then Error-valve failure.
42. Check PT-SAM2 Pressure >100 Torr. If not then wait.
43. At this point the logic will only continue if either “Analyze&Dump” or “Calibrate” was chosen as the initial condition. If “Analyze&Recover” was chosen then SAM will display “Ready to Recover to the SRV” once the system is ready for manual SRV recovery.
44. Check that SAM Turbo Pump 2 is off (if not, turn off and wait 10 min)
45. Check V3 is closed

46. Open V1 and V2, Check status // This will begin to evacuate the coldtrap and sampling volume
47. Wait until PT-SAM2 is < 5 Torr
48. Turn on SAM Turbo Pump 2
49. wait 5 min, then turn on PT-SAM3 (vacuum gauge of TP2) (Keep repeating until the CCG turns on)
50. wait until PT-SAM3 < 1e-6 Torr then Open V3 (check V3) // This will expose the RGA to all of the plumbing
51. Close V2 (and check) //This will isolate TP2 from the sampling system. If not then Error-valve failure.
52. Turn off TP2 and check.
53. clean up complete (The sampling system should be being pumped to vacuum by TP1)

A.5 Sampling System Control: Pump Out

1. NewSetValue=5;
2. Check TC1 > 270 K //LN has evaporated? If not then wait. The value of “Sampling System Status” will change to “Remove LN” and will remain there until the cold trap has sufficiently warmed.
3. Check V1, V2, V3, V4, V5, V6 are closed. If not then close.
4. Open V4, V5, V6 and Check status. If not then Error-valve failure.
5. Check PT-SAM2 Pressure >100 Torr. If not then wait.
6. Check that SAM Turbo Pump 2 is off (if not, turn off and wait 10 min)
7. Check V3 is closed
8. Open V1 and V2, Check status // This will begin to evacuate the coldtrap and sampling volume
9. Wait until PT-SAM2 is < 5 Torr
10. Turn on SAM Turbo Pump 2
11. wait 5 min, then turn on PT-SAM3 (CCG of TP2) (Keep repeating until the CCG turns on)
12. wait until PT-SAM3 < 1e-6 Torr then Open V3 (check V3) // This will expose the RGA to all of the plumbing
13. Close V2 (and check) //This will isolate TP2 from the sampling system. If not then Error-valve failure.
14. Turn off TP2 and check.
15. clean up complete (The sampling system should be being pumped to vacuum by TP1)

B Collecting a Xenon Sample From the LUX System with a 0.5 L Sampling Bottle

This procedure is for sampling xenon from the LUX xenon system. A sample bottle is attached to a pump-out port of the xenon system, a slug of air will be pumped out via a turbo pumping cart, and then the sample bottle is filled with xenon from the system, and removed. Because the sampling procedure is exactly the same for each of the six locations, we will henceforth refer to the valves in each sampling location as VB (the valve closest to the sampling port) and VA (the valve immediately after VB). Additionally, we will refer to the pressure transducer nearest the sampling port as PT-A. The valves and pressure transducer corresponding to each sampling location are summarized in Table 1.

Note: Refer to Table 1 for valves VA, VB and VC as they depend on the location you will be collecting a sample from.

1. Find a green all-metal valve (AMV). There is a dedicated valve (SS-4UW-V51) for the Turbo-Pump cart. Find a 1/4" VCR bellows. Also find a 1/4" all-male VCR Tee and 1/4" gaskets. Prepare a portable pump cart.
2. Monitor and ensure the stability of the pressure gauge of interest (see Table 1). Ensure that the pressure is stable to within 1 psig for one minute, if not contact the shift manager.
3. Confirm that the sampling valves of interest (VA, VB and VC) are closed.
4. Remove the lock and cap from VB. Note: Make sure the sampling valve, or at least the hose connecting to it, is securely restrained. If it is not, you may inadvertently loosen a VCR connection and open a major leak directly to the xenon system.
5. Set up the sampling plumbing according to Figure 7 and Figure 8.
 1. Attach the 1/4" VCR bellows to the sampling valve
 2. Attach the VCR Tee to VB.
 3. Attach the AMV to one end of the Tee.
 4. Open the AMV.
 5. Attach the 0.5 L sample-bottle to the other end of the Tee.
 6. Attach the pump station to the free end of the AMV. Note: Strain relieve any hoses you use so you avoid torquing any of the VCR connections.
6. Confirm VA, VB and VC are closed.
7. With the scroll-pump on the pumping cart, pump up to the sampling valve and the sample bottle.
8. Open the valve on the sample bottle (BV) to pump out the bottle.
9. After the pressure on PT-PC1 reaches 1e-1 Torr open VB and pump it with the scroll-pump until the PT-PC1 is below 1e-1 Torr (to pump out the space between VA, VB and VC).
10. Ensure that PT-PC1 is less than 1e-1 Torr then start the turbo-pump.
11. Once the pressure gauge on PT-PC1 reaches 1e-5Torr(it's minimum value), helium leak check the new connections using the RGA on the turbo-cart.
12. Turn off the RGA's filament once the leak checking is complete.

13. Pump on the plumbing for 1 hour. PT-PC1 only reads down to 1e-5Torr, so the ultimate pressure reached will not be known.
14. When done pumping, close the AMV. This will isolate the turbo from the sample volume.
15. Continue to monitor the pressure in the region to be sampled (PT-C15 if sampling the getter input, PT-C20 if sampling the getter output, PT-C1 if sampling the detector return line, PTD41 if sampling the PMT purge line). Insure that the pressure is stable to within 1 psig.
16. Record the pressure, before sampling, from the pressure gauge in the region which was sampled. PT-C15 if sampling the getter input, PT-C20 if sampling the getter output, PT-C1 if sampling the detector return line, PTD41 if sampling the PMT purge line.
17. Open VA(see Table 1) to fill the 0.5 L sample bottle with xenon from the LUX system. After 5 seconds the bottle should be full and at system pressure. The sample bottle volume is small enough so that taking a sample should not impact the system pressure. If desired, crack VA open a bit until PT-SAM2 reaches 700 Torr. Drawing 700 Torr if sufficient for purity analysis.
18. Close VA, VB and VC.
19. Close, cover and tag VA
20. Record the pressure, after sampling, from the pressure gauge in the region which was sampled. PT-C15 if sampling the getter input, PT-C20 if sampling the getter output, PT-C1 if sampling the detector return line, PTD41 if sampling the PMT purge line.
21. Close the sample-bottle's valve (BV).
22. The next step will vent the Tee, so it is crucial that the sampling valve, the sample bottle, and the AMV are closed.
23. Disconnect the sample bottle from the Tee.
24. Remove the sampling manifold from the sampling valve.
25. Cap VB and label that air is behind the cap.
26. Label the sampling bottle with the date, pressure and sample location.

C How to Analyze the Data

C.1 Overview

The principle of the RGA/coldtrap method is simple. The partial pressures of O₂, N₂, Ar, and Kr observed by the RGA are proportional to two things: the leak rate into the cold trap, and the concentration of the impurity species in the xenon. Therefore we can measure the partial pressure, account for the leak rate, and infer the impurity concentration. The proportionality constant can be measured using the calibration xenon bottle, which is known to contain 1 ppm of O₂. (The O₂ concentration in the calibration bottle is known because we prepared the gas using a known volume and pressure of O₂ and a known amount of xenon.)

Several other factors can also affect the partial pressure reading on the RGA: the conductance of the plumbing between the leak valve and the RGA, the speed of the turbo pump, the response and gain of the RGA, and non-ideal effects such as RGA saturation. However these factors are all mitigated and accounted for as follows: The conductance and pumping speed are expected to be the same from measurement to measurement, and the RGA response and gain can be monitored using the calibration xenon. Saturation

can be avoided by following the procedures carefully and by monitoring the total pressure seen by the RGA, as read by the PT- multi-purpose vacuum gauge.

If an identical and constant leak rate is used in every measurement, then the impurity partial pressure reading is directly proportional to the impurity concentration. At UMD we typically collect data in this mode. However, in the LUX coldtrap system, the pressure at the input to the leak valve decreases exponentially during the five-minute measurement period, because a finite amount of gas is contained in the sample bottle. This means that the leak rate and RGA partial pressure measurements also decrease exponentially, and we must account for this time-dependence in the data analysis.

C.2 Measuring the dynamic leak rate

We infer the leak rate by measuring the amount of gas remaining in the sample bottle as a function of time with PT-SAM2. The amount of gas is given by the pressure times the volume, and is measured in units of torr*liters. The leak rate is then the time derivative of this function, and is given in units of torr*liters/minute.

The leak rate which will be obtained in any given experiment is determined by two factors: the leak valve setting and the input pressure. By default, we always use a leak valve setting of 1 turn + 28 ticks. The input pressure may vary from measurement to measurement, depending on the pressure in the LUX xenon system. Calibration data shows how the leak rate depends in the initial pressure. (Note that if a non-standard leak valve setting is used by accident, the data can still be used, as long as the Baratron data is recorded so that we can calculate the leak rate which was obtained.)

C.3 Accounting for the dynamic leak rate

To account for the changing leak rate, we divide the RGA partial-pressure data by the dynamic leak rate as a function of time. Unfortunately, the RGA measurement and the Baratron measurement are not synchronous, because the Baratron(PT-SAM2) is sampled by the LUX slow control system, while the RGA is not. This means that an exponential function should be fit to the Baratron data, and the derivative of this function can then be used to normalize the RGA data. Fortunately, the exponential form of the Baratron(PT-SAM2) data can, in most cases, be determined simply from the initial input pressure, because we have measured the dynamic leak rates for a variety of initial input pressures at the standard leak valve setting. (Note that in order to do the normalization properly, it is important that the two datasets have accurate time stamps which can be compared to each other).

The RGA partial pressure divided by the leak rate should be independent of time, and this ratio is proportional to the impurity concentration in the xenon. In practice, however, it takes two minutes for the partial pressure data to stabilize, after which a constant plateau value is reached. Therefore we use the plateau value to infer the impurity concentration.

Ideally the plateau value would be completely independent of the initial input pressure, once the leak rate normalization has been done. In practice, we find a small linear dependence of the plateau value on the initial input pressure. The reason for this remaining correlation is not known. We can either account for this dependence by comparing to the calibration data, or we can assign a modest systematic error to the final measurement.

C.4 Peak pressure method

A second, simpler technique can be used to quickly eyeball the purity measurement directly from the RGA data. In this method, the peak value of the RGA partial pressure data is used to infer the purity measurement. This method is probably accurate only to a factor of two or three, but it gives a reasonable estimate of the order-of-magnitude of the xenon purity.

D Figures

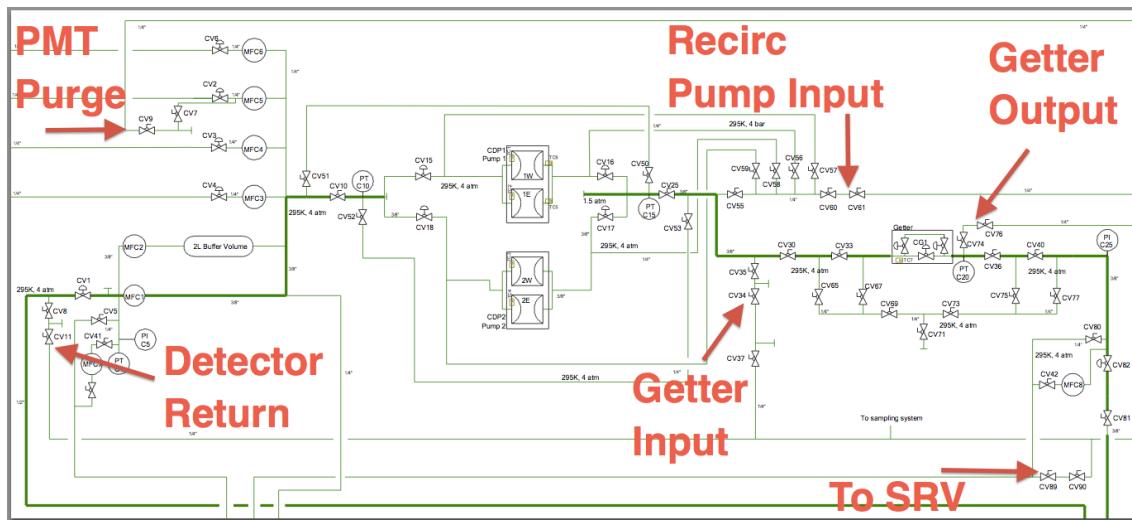


Figure 1: Sampling Locations

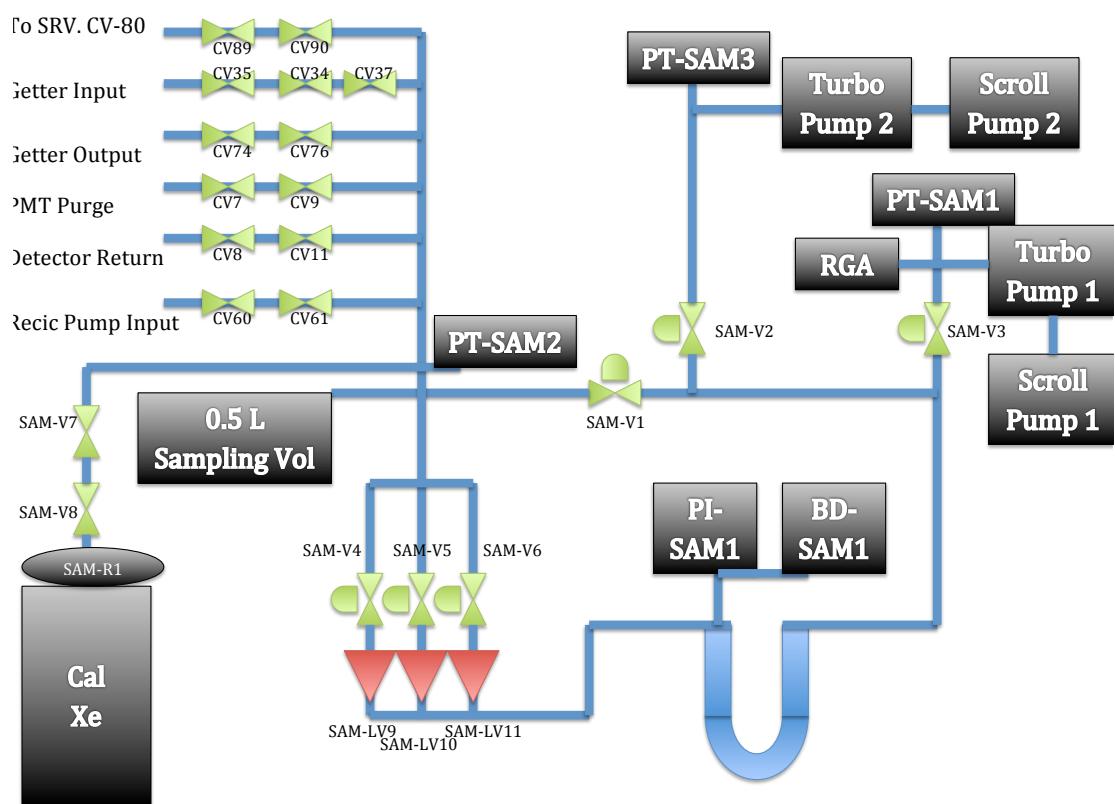
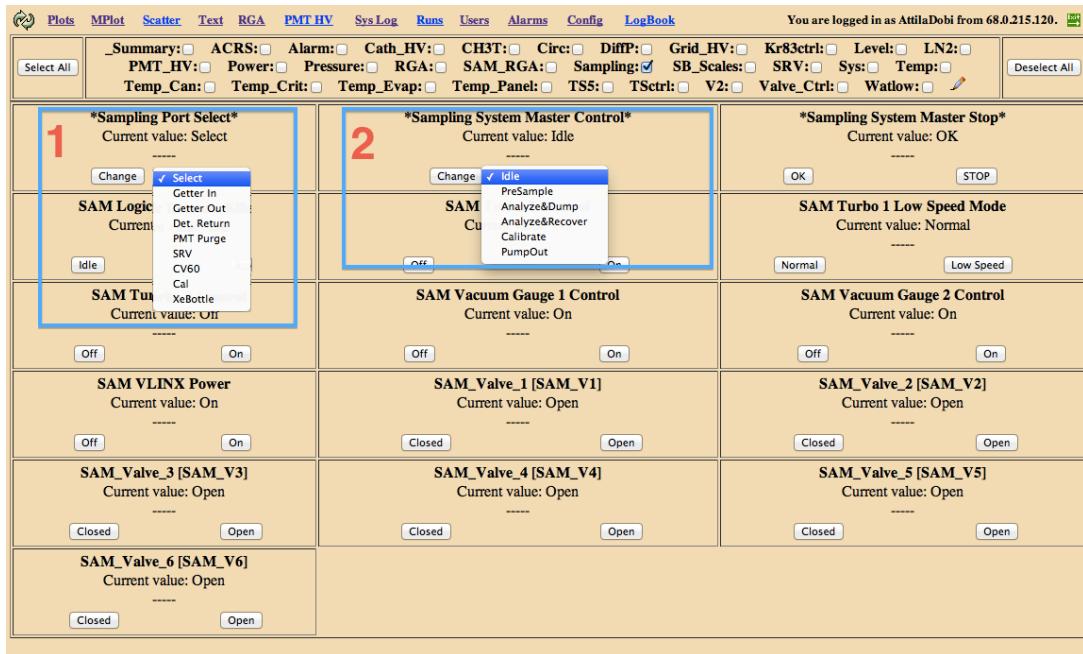


Figure 2: Diagram of the Coldtrap Sampling System



Last db update: Jun 01, 2013 @ 14:06:01		
#SAM Run Status# (SAM_wait)	#Sampling System Error Status# (Analysis_Error)	#Sampling System Status# (Analysis_Status)
OK	None	Idle
\$ Last Sampling Location \$ (SAM_Port) Getter Out	* Sample Number * (SAM_Number) 151.000 (Number)	*Ar Concentration* (Purity_Ar) 98.231 (ppb g/g)
CH4 Concentration (Purity_CH4) 6.100e-3 (ppb g/g)	*He Concentration* (Purity_He) 117.310 (ppb g/g)	*Kr Concentration (82+84+86)* (Purity_Kr_Sum) 0.013 (ppb g/g)
Kr Concentration (from 82) (Purity_Kr82) -0.019 (ppb g/g)	*Kr Concentration (from 84)* (Purity_Kr) 0.020 (ppb g/g)	*Kr Concentration (from 86)* (Purity_Kr86) 0.011 (ppb g/g)
N2 Concentration (Purity_N2) 0.884 (ppb g/g)	*O2 Concentration* (Purity_O2) 6.300e-3 (ppb g/g)	*Sum Xe Mass Pumped to SRV* (SAM_Mass_SRV) 330.841 (grams)
Sum Xenon Mass Pumped Out (SAM_Mass_Out) 247.869 (grams)	< Last Analysis [hours ago] > (Last_Analysis_t) 26.807 (hours)	< Last Calibration [hours ago] > (Last_CAL_time) 1297.387 (hours)

40

Figure 3: Top: The Sampling System Master Control box in LUX slow control. Bottom: The Sampling System status display in LUX slow control

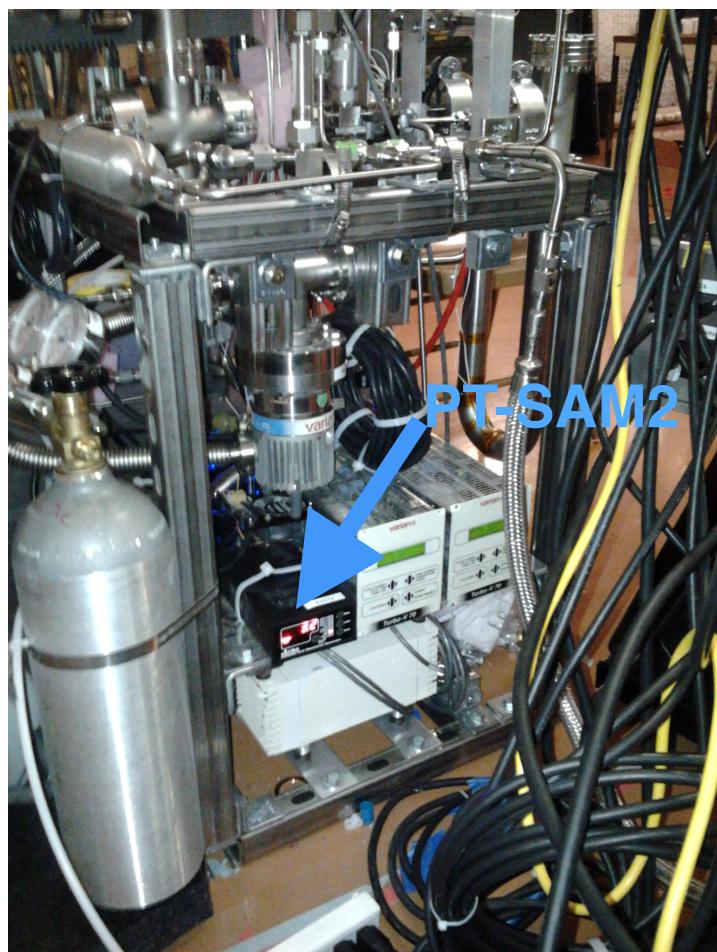


Figure 4: The view of the sampling system from behind the circulation panel, where the xenon samples will be drawn. The user should watch PT-SAM2 when drawing a xenon sample.

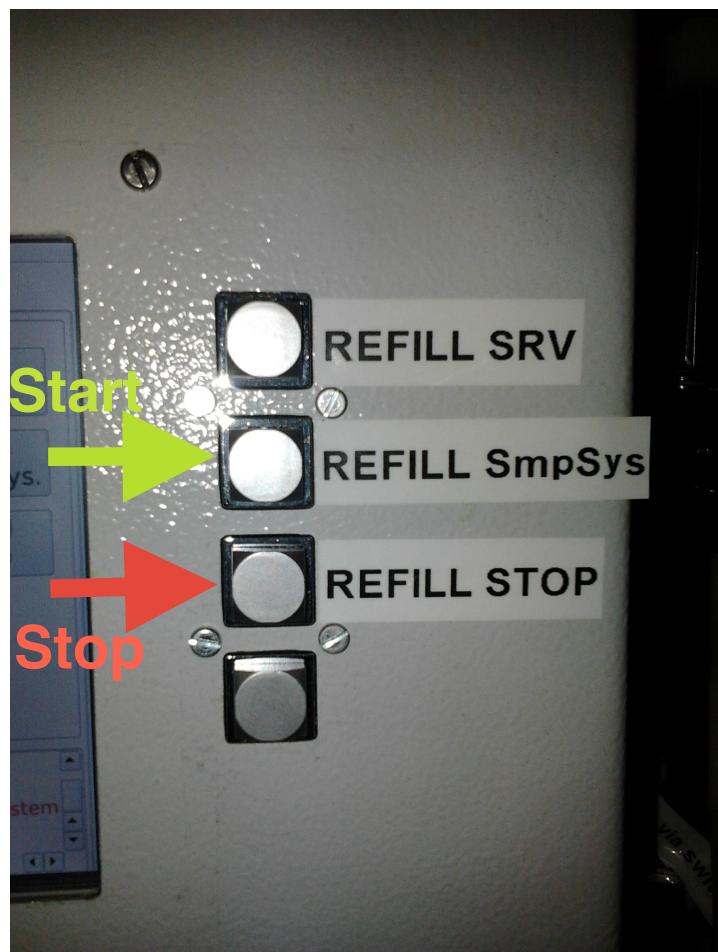


Figure 5: SRV LN system control box. To fill the 4 L transfer dewar push “Refill SmsSys” to begin the flow of LN through the SRV pre-cool line. Once the dewar is full hit the “Refill STOP” button.

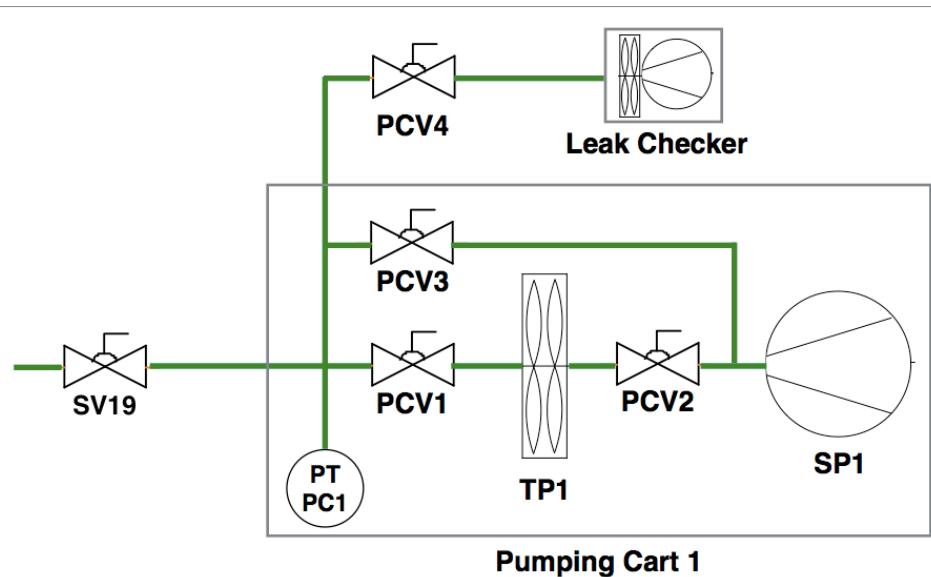


Figure 6: Diagram of the turbo pumping cart to be used when sampling LUX storage bottles in the bottle farm via SV19.

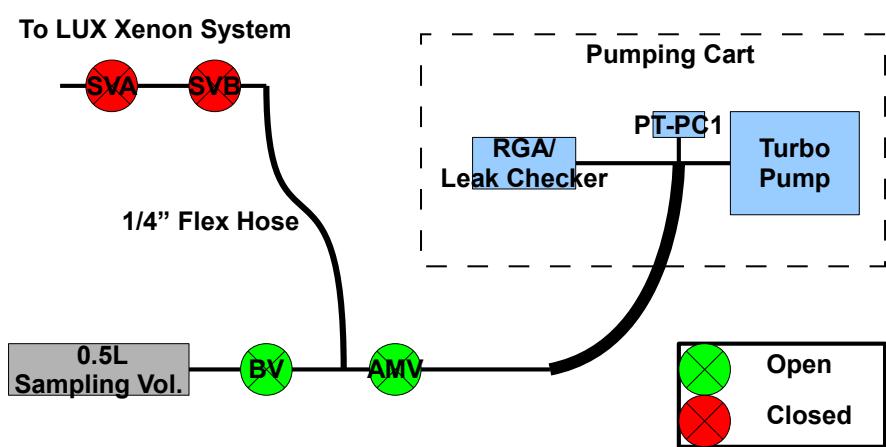


Figure 7: Illustrated procedure for attaching the sampling manifold to the LUX xenon system

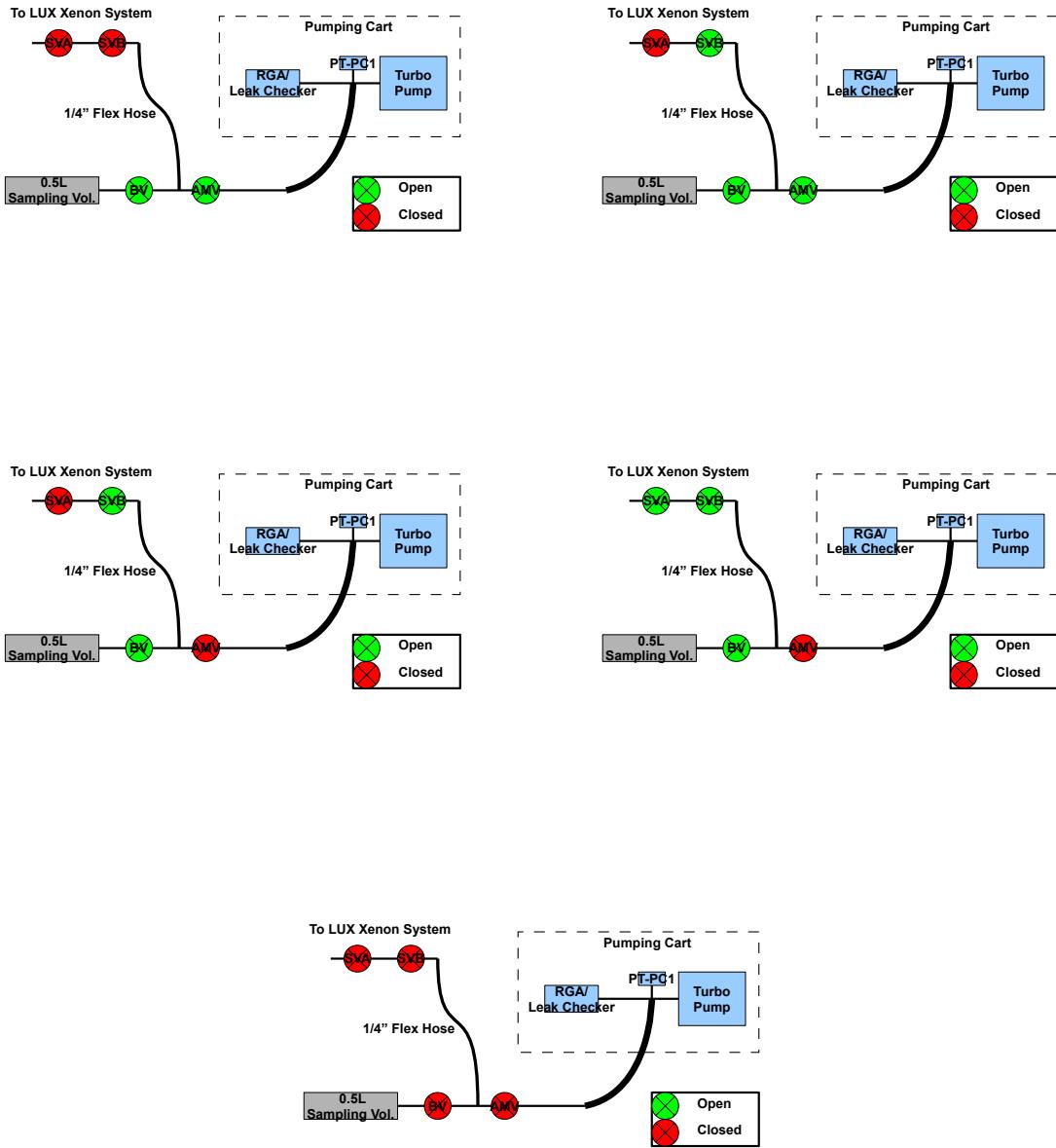


Figure 8: Illustrated procedure for filling the sample bottle. First (top left), the sampling plumbing is attached to VB and then pumped to vacuum with the scroll-pump on the pumping cart. Second (top right), once the pressure on PT-PC1 reaches less than 0.1Torr VB is opened. The plumbing up to VA is then pumped to vacuum with the turbo-pump for one hour. Third (middle left), after pumping to vacuum for one hour the turbo-pump cart is isolated by closing AMV. Fourth (middle right), VA is opened filling the 0.5 L sample bottle with xenon at system pressure. Fifth (bottom), the VA, VB and the sample-bottle's valve (BV) are closed.