LAr Filter Regeneration Procedure

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General steps:

1. Preheat the LAr filter to 175 – 180°C with ultra high purity Ar gas at 160 slpm ($\sim$6.7 scfm on the flowmeter). It takes about 3 hours. Regarding the voltage of the gas heater, you can start with 55 V at the variac and bump it to 100 – 110 V after everything is stable for 10 minutes. You can stop preheating when the bottom thermocouple reaches $\sim$145°C. Currently we are using the ultra high purity Ar from the gas port of the LAr dewar, because a huge amount of gas Ar is needed.

2. Use 1 – 2% H_2 balanced with Ar to regenerate the LAr filter. Keep the temperature between 175 and 225°C. With the flow rate of 80 slpm ($\sim$3.3 scfm on the flowmeter), we expect to use 5 gas cylinders, each takes about 1.25 hours. You can keep the voltage of the gas heater between 55 – 75 V (at the variac).

3. Cool down the system with ultra high purity Ar gas. This take $\sim$80 minutes and you will sort of uniformly ramp down the voltage of the gas heater from 65 V to 20 V.

Time to stop regeneration:

- Humidity plateaus. Better plateaus at 0.02%.
- 5 hours of 2% H_2 gas at 80 slpm ($\sim$3.3 scfm on the flowmeter).
- Temperature in the LAr filter does not rise anymore.

Safety:

- All the doors of the LNTF hut have to be open.
- The intake fan has to be turned on.
- The oxygen deficiency sensor and monitor (ODM) have to be checked.
- The pressure in the LAr filter is shown on PG3.
- The gas flow has to be greater than 2 scfm (marked on the flowmeter) to prevent the heater from getting too hot.
- The gas heater power cord is plugged into the heater engineering control (EEIP approved), which shut down the power if the temperature of the gas heater exceeds the preset value, 355°C. The shut-down temperature can be set at the detector control GUI (Ignition), and the gas heater temperature is displayed in the same page.
- When sealed, the pressure of the LAr filter and gas pipe should not exceed 150 psig. The pressure relief valve on the LAr filter will open at $\sim$150 psig, while the pressure relief valves along the pipe will open at 150 – 200 psig.

- If seeing smoke or smelling something unusual, shut down the variac power supply (heater) and investigate.

Technical notes:

- V3, V5, V6, V16/V17, V18/V19 isolate the LAr filter. During the regeneration, V3, V5, and V6 should be always closed. V17 and V18 are metal valves, which will be always open. We will rely on V16 and V19 to isolate the LAr filter, instead of V17 and V18. However, we close V17 after regeneration to prevent cold Ar from entering the gas heater when filling LAr.
- V16, V20, V21, V22, V24, V25 are diaphrame valves and are not adjustable. When opening them, make a full open. Use Reg1/2/3 and V19 to control the flow rate.
- The Ar gas flows from the gas port of the LAr dewar, Reg3, V20, V16, HT1, V17, into the LAr filter, and vents from V18, V19, FC1, outside the LNTF hut along the venting pipe, as shown in Fig. 2.
- The $2\%H_2+Ar$ gas flows from the gas cylinders, Reg1/2, V26/V27, V22, V16, HT1, V17, into the LAr filter, and vents from V18, V19, FC1, outside the LNTF hut along the venting pipe, as shown in Fig. 3.
- The vacuum vessel surrounding the LAr filter should be evacuated from V4 all the time during regeneration. The pressure can be read from PG6.
- Control Reg1/2/3 and V19 carefully to avoid compromising the LAr filter. We want the LAr filter to have pressure greater than the atmosphere so that the air won't diffuse in, but not too overpressurized. $16 - 35$ psia in the LAr filter is good.
- The pressure at the outlet of Reg3 and in the LAr filter should be very close to each other, because the pressure drop along the inlet line is small.
- The pressure at the outlet of Reg1/2 is a few psi higher then the pressure in the LAr filter, which corresponds to the pressure drop along the inlet line.
- We should always keep the gas flow (Ar or $2\%H_2+Ar$) between 2 and 6.7 scfm (marked on the flowmeter).
- Maintain the catalyst temperature between about $175°C$ and $225°C$.
- Do NOT exceed $225°C$, even though $250°C$ may be tolerated.
- The temperature of the hot gas and heater body may increase while the voltage from the variac power supply is fixed. It is owing to the drop of the gas flow. We can adjust Reg1/2 to cope with this situation. This happens all the time with the $2\%H_2+Ar$ cylinders and we should keep an eye on these parameters.
- The humidity sensor operates between -20°C and 50°C. With the Ar flow rate of 160 slpm (∼6.7 scfm on the flowmeter), the hot Ar gas likely still has higher temperature than 50°C when it arrives the humidity sensor. The humidity sensor will show 0% all the time in this case. It will start functioning when the temperature drops. We can turn off the gas heater or lower the flow rate to obtain colder gas when we need to check the humidity. (Modification is being planned.)
- The LED light on the gas heater control may be broken. The controlling part should be functional, and you just have to follow the labels for the toggle switch.
- The torque for V17 is 4 foot-pound, 3/4" socket.
- The torque for V18 is 21.7 foot-pound, 3/4" socket (not needed, just FYI). Need different torque wrenches for V17 and V18 typically.
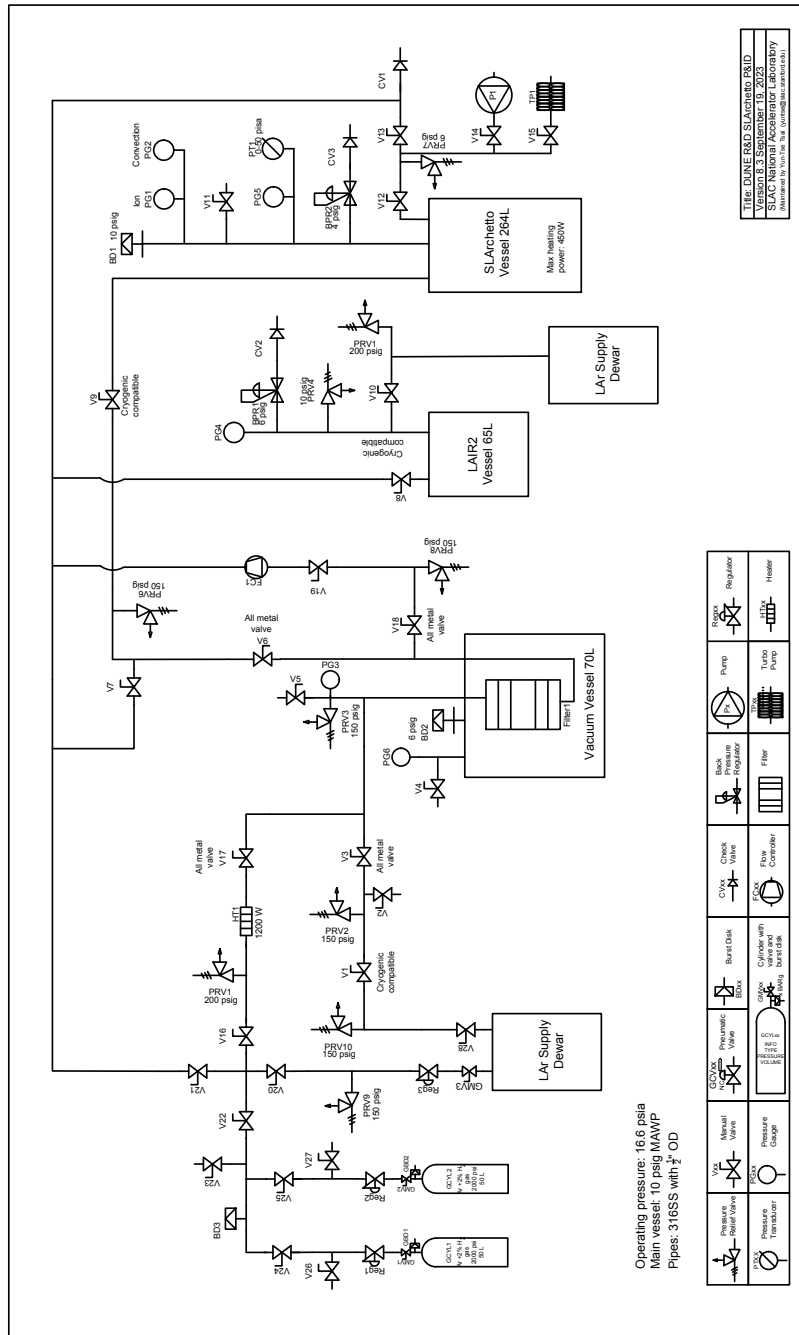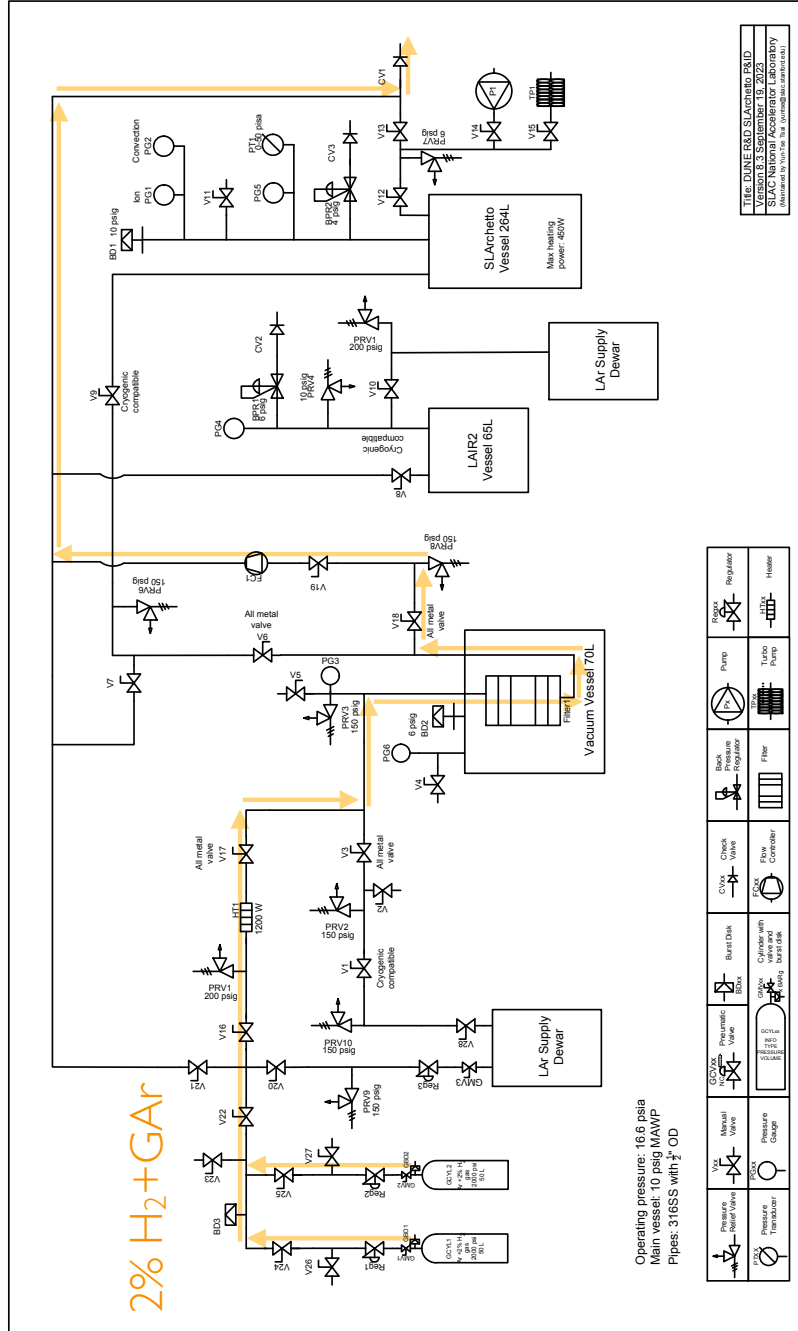
Figure 1: P&ID

Figure 2: Gas flow direction: Ar gas

Figure 3: Gas flow direction: 2%H$_2$+Ar gas

Checklist	What to Do and Detailed Description
Readiness – Before the Day	
1 ultra high purity LAr dewar	We use the gas port of the LAr because we need a lot amount of gas Ar.
The LAr dewar lifted in the LNTF hut	
5 cylinders of Ar+2%H$_2$ gas	
The GAS port of the ultra high purity LAr dewar connected to Reg3 and then V20 with a copper tube	
Two Ar+2%H$_2$ gas cylinders connected to Reg1/Reg2 and V24/V25 line	
The cold insulation foam from the tubes close to the LAr filter regeneration line removed	
Heater, tubes connecting the heater and the LAr filter wrapped with a few layers of aluminum foils	For thermal insulation
Variac AC power supply and the gas heater engineering control ready	
V4 connected to the scroll pump	Prepare to evacuate the vacuum vessel
V4 opened	
V4 opened, scroll pump on	Evacuate the vacuum vessel
V3, V5, V6, V7, V8, V9, V10, V11, V12, v18 closed	
All the temperature and humidity sensors connected	
Detector control (Ignition) set up	Instruction: `https://docs.google.com/document/d/17dsjQEY3hDOYmxKYikNqeVWEoBOqyarqYrbijNPSBfg/edit?usp=sharing`
All sensors in the "Filter Regeneration" page online	
Safety Checks – Beginning of the Day	
All the doors of the LNTF hut opened	
Intake fan on	The emergency button is yellow
Oxygen deficiency sensor in place, oxygen deficiency monitor green	
Heat warning signs posted on the clean tent and the frame	

Gas heater shutdown temperature set to 355°C	In the detector control system, click "LAr Filter," set the value in "Gas heater switch off temperature"
Gas heater temperature alarm set to 400°C	In the detector control system, click "LAr Filter," choose "TC1: Gas heater", enable the alarm and set the value

Preheating with Ar gas

V3, V5, V6, V16, V19, V20, V21, V22, V23, V24, V25, V26, V27 closed	
V17, V18 fully opened	Not going to use these two valves to isolate the LAr filter; keeping them open
V4 opened, the connected scroll pump on	
PG6 at 0 psia	
Variac power supply off. Voltage set at 0	
Gas heater (HT1) plugged in to the heater engineering control, and the engineering control plugged into the variac power supply	
The GAS port of the ultra high purity LAr dewar connected to Reg3 and then V20	
Flowmeter (FC1) set to the maximum	Not using the flowmeter to control the flow
Purge the air: GMV3 opened, Reg3 increased, V20, V21 opened	Purge the air in the connection tube
V21, GMV3 closed	Finish purging
V16 opened	
GMV3 opened, Reg3 increased	Start flowing Ar gas to the LAr filter
PG3 at 5 – 15 psig (20 – 30 psia), V19 opened	
Gas flow ∼6.7 scfm, PG3 at 20 – 35 psia, stable	
Variac power supply on, the voltage increased to 55V	Turn on the heater
10 minutes of stable conditions reached	
Variac power supply set to 100 – 110V	
Humidity plateaued at 0.02% for > 10 minutes	Molecular sieves regenerated
Preheated for > 2 hours	
TC0, 1, 2, 3 at 175 – 180°C, or TC3 > 145°C	
Variac power supply off. Voltage set at 0	Turn off the heater
V16, V19, V20 closed	
GMV3 and Reg3 closed	

Regenerating copper sieves

Variac power supply off. Voltage set at 0	
V26, V27, V24, V25, V23, V22, V20, V21, V16, V19 closed	
V17, V18 fully opened	Not going to use these two valves to isolate the LAr filter; keeping them open
Two Ar+2%H_2 gas cylinders connected to Reg1/Reg2 and V24/V25 line	
Purge the air: GMV1 opened, Reg1 increased, V24, V23 opened	Purge the air in the connection tube until V22 for the first time
GMV1, V23 closed	Finish purging
V22, V16 opened	
GMV1 opened, Reg1 increased PG3 at 5 – 15 psig (20 – 30 psia), V19 opened	
Gas flow between 50 and 160 slpm (Ar), or between 2.2 and 6.7 scfm (marked as Air). Preferably at 3.5 scfm Air. PG3 (LAr filter) at 5 – 20 psig (20 – 35 psia). The outlet of Reg1/2 at 20 – 40 psig.	Usually V19 is not widely open.
Variac power supply on, the voltage increased to 55 – 75 V	Turn on the heater
Temperature in the LAr filter kept at 175 – 225°C	Should the temperature exceed 225°C anywhere in the bed, switch to H_2-free gas and turn off the gas heater power supply until the hot zone cools back down to 200 – 210°C, then resume feeding the H_2 gas mixture and turn on the gas heater power supply

Gas cylinder transition

The other gas cylinder (GCYL2) connected before the operating one (GCYL1) finishes	or vice versa
Purge the connection line: GMV2 open, Reg2 open, V27 open	Purge the air until V25. Can also evacuate from V27 instead of purging. When connecting to a pump, need to install a PRV (can be a KF cap) so that the pump will not risk to encounter high pressure.
GMV2, V27 closed	Finish purging
V22, V16 opened	
GMV2 opened, Reg2 increased PG3 at 5 – 15 psig (20 – 30 psia), V19 opened	
Switching the cylinders (when the operating one has the pressure of ∼300 psig): V24 closed and V25 opened	Transition swiftly

Finishing the copper seive regeneration

The temperature of the all catalyst bed stable or subsiding	
Humidity plateaued at 0.02% for > 10 minutes	Copper sieves regenerated
Variac power supply off. Voltage set at 0	Turn off the gas heater
V22, V16, V19 closed	
GMV1 and Reg1 closed, V24/V25 closed	

Completion; cooling down

Variac power supply off. Voltage set at 65 V	
V16, V19, V20, V21, V22, V23, V24, V25, V26, V27 closed	
V17, V18 fully opened	Not going to use these two valves to isolate the LAr filter; keeping them open
The GAS port of the ultra high purity LAr dewar connected to Reg3 and then V20	
Purge the air: GMV3 opened, Reg3 increased, V20, V21 opened	Purge the air in the connection tube
V21, GMV3 closed	Finish purging
V16 opened	
GMV3 opened, Reg3 increased	Start flowing Ar gas to the LAr filter
PG3 at 5 – 15 psig (20 – 30 psia), V19 opened	
Gas flow ~6.7 scfm, stable	
Variac power supply on, the voltage slowly decreased to 20 V	Turn on the heater
Temperature in the LAr filter decreased to ~35°C	
Variac power supply off. Voltage set at 0	Turn off the heater
V16, V19 closed	
GMV1 and Reg1 closed, V20 closed	
V4 closed, scroll pump off	

Power disconnection, cleanup

V17 closed	Use a torque wrench with a 3/4" socket. The torque is 4 foot-pound.
The gas heater power supply disconnected and stored	
The power of the scroll pump disconnected	
Intake fan off	The emergency button is red
LNTF doors closed	

The empty gas cylinders disconnected, moved to the empty cylinder rack and chained appropriately

The heat warning signs removed (after the system cools down)