# Operation of Cryomech LHeP22 in Low Temperature Physics Lab at Cornell

D. Lotnyk, E.N. Smith, J.M. Parpia

Dept of Physics & LASSP, Clark Hall, Cornell University

Abstract: We describe the operation of a Cryomech LHeP22 helium recovery and liquefaction system sited in our low temperature lab alongside our nuclear demagnetization refrigerator.

**Introduction:** With liquid helium becoming ever more expensive and supplies being rendered more tenuous, the viability of ultralow temperature physics research – defined as access to temperatures in the mK range is called into question. Low vibration environments are needed for nuclear demagnetization apparatus like ours as well as apparatus designed to achieve low temperatures and high magnetic fields. Both these applications are affected by heat leaks from eddy currents that are generated when electrically conducting components are placed in magnetic fields. Thus cryocoolers integrated with dilution refrigerators (so-called dry fridges) with their attendant vibrations can degrade the ultimate performance. With a working ultra-low temperature apparatus consuming, (in our case) approximately 500 – 600 cc of liquid helium per hour, the only solution involves a liquefier.

**The System:** The LHeP22 was installed in October of 2017. It is sited 15 feet (~5m) from the $PrNi_5$ stage equipped wet dilution refrigerator (SHE420). The nominal liquefaction capacity of the liquid helium plant is 20 liters/day, while the boil off from the cryostat is around 12 liters/day.

The system consists of a dewar containing the cryomodule and plumbing, a control panel (mounted alongside the dewar), a purifier (consisting of a getter that is cooled with liquid nitrogen), a triaxial transfer line to transfer directly from the cryomech dewar into the research cryostat. The recovery system -- a 300 CuFt capacity gas bag (roughly 11 liters worth of gas storage when at capacity) and the recovery compressor that pumps the helium vapor from the gas bag into 400 psi storage are sited in the sub-basement along with the compressor for the liquefier (split system). The storage consists of 8 storage vessels, each capable (at 400 psi) of holding 16 liters worth of gas. The minimum pressure for the operation of the liquefier is 34 psi, so approximately 10% of the gas is tied up in storage.

**Operation:**

Every day or two it is recommended that the water condensate valve on the recovery compressor is blown down manually. The drain valve button is depressed to drain any water and the recovery compressor is cycled to working pressure and then turned off allowing water to be extracted from the helium gas into the condensate drain from which it is ejected after depressing the drain valve. Every time we conduct the blow down, we look at the “dot” on the water trap on the recovery compressor and verify that it is blue (clean) and not lilac/violet (intermediate) or pink (saturated). If it is violet we swap the old trap for a replacement and then regenerate the trap. This is dealt with later.

Every 4 days (we have the 8 inch dewar) we refill the Purifier liquid nitrogen trap (used to extract air from the incoming gas to be liquefied). This requires ~25 liters of nitrogen.

There is also a water trap on the purifier but we have not had to ever clean it in 2 years of operation. The air trap on the purifier has to be cleaned every 5-6 weeks; the water trap depends on ambient humidity - every 3 weeks in the summer and 2 months in the winter.

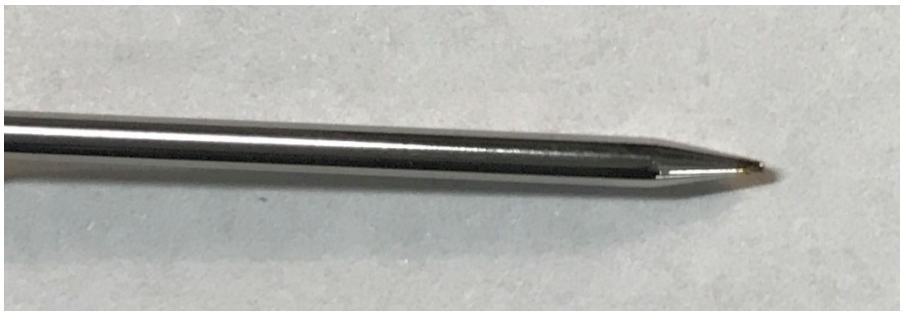

Fig 1 Shows discoloration at the tip of discharge electrode.

A discharge gauge monitors the oxygen content in the recovered gas after purification on its way to the liquefier. If there is too much oxygen, registered an alarm sounds and shuts down the purifier signaling that the purifier should be replaced and regenerated. The discharge tip gets contaminated and has to be cleaned from time to time (see manual). A dirty tip is shown here for reference – (Fig 1) the contamination is on the end.


Fig 2 Image of water trap in heater jacket showing lilac colored "dot".

**Regenerating the Traps:**

Regeneration of the water requires placing the heater jacket (Fig. 2) onto the trap and heating. When the water trap is saturated it contains ~350 gm of water (We weigh the trap before and after regeneration). With heater on, we run dry nitrogen gas through the trap at a flow that is just enough to register on the pressure tank regulator. This will empty a 300 CuFt cylinder in about 4 days. We run the nitrogen through the trap overnight and this extracts something like 100 gm to 150 gm of water. The dot (see Fig. 2) goes blue. We then pump on the heated water trap through a custom coaxial flow trap (Fig. 3a) that passes the gas evolving from the heated trap gas past liquid nitrogen cooled surfaces as it flows toward a dry piston pump. In this way overnight we extract a further 200 gm of water (Fig. 3a) before our trap plugs with ice. The trap is regenerated with a heat gun, and after a further day of pumping (Fig. 3c) we are now assured that the trap is back to its original empty weight. In our case the empty weight is about 9 Kg and it takes on 0.350 kg water. We always use these traps and use the gas ballast to preclude water contamination of pumps.


Fig 3 a) Liquid nitrogen trap to condense water b) with ~200 gm extracted c) remaining 50 gm trapped.

The air trap is easier to deal with. We first vent down the gas (it will be at high pressure from storage) to recovery while the trap is at nitrogen temperature. Then we vent the contents to air while heating the trap (the trap is pulled out from liquid nitrogen at this point and some of the air that was adsorbed will be liberated). After the contents are down to atmospheric pressure, we pump on the trap (no LN2 trap is needed) using the same piston pump while the system is heated.

**Transferring the Helium Liquid with the triaxial line.**

The triaxial line is needed to allow us to transfer helium liquid at high efficiency into the research dewar separated from the Cryomech by about 3.5m. The separation is essential for vibration isolation. The cryomech dewar is located on the Clark Hall basement floor, while the research dewar is on a concrete slab over the subbasement. The research dewar is also isolated on air legs. There is about a 1 m height difference between the Cryomech dewar and the research dewar fill port, with the research dewar being higher. Thus additional pressure is needed to effect the transfer.

Ahead of our usual refill procedure, we ensure that there is at least 2.5 psi in the Cryomech dewar. We time the transfer so that it is in the middle of a liquefaction cycle. [Since the liquefier's capacity is greater than the cryostat's gas evolution rate, the liquefier will often be running when called for to maintain the dewar pressure between 0.5 and 8.0 psi]. Transfer when the gas pressure is lower than 2.5 is slow since the dewar pressure will be depleted, resulting in a slow transfer unless heat is applied to the liquefier to maintain the pressure at 2.5 psi. We don't like to use this feature. Ideally, we start the transfer when the liquefier is running and drawing in helium from storage. There will be an initial excess pressure generated in the research dewar as hot gas flows in. The research dewar pressure may rise by 2 psi but it rapidly falls. By trial and error, we apportion the recovered gas so that about half goes through the regular boil off line to recovery from the cryostat, and about half goes back to recovery through the triaxial line. This leads to significant frost on the 3-4 meter line from the transfer line to recovery (Fig. 4). We ensure that the manifold does not get too cold (frosty).

Fig 4 Image of the LHEP while transferring. You can see that the frost on the recovery line carrying return gas from the transfer line extends quite far

We maintain a USB camera to observe the gas bag during the transfer and control the rate so that the recovery compressor keeps up with the evolving gas. In our system we open the valve on the transfer line from closed position by about 200 degrees. We transfer at a rate about between 1 and 1.5 liters a minute. By trial and error we set (and then do not adjust) the two valves that control the relative impedance of the gas through the direct path and through the transfer line, and then open the valve on the dewar line recovery to the same degree every transfer. Then as long as the purifier and liquefier are running, we can transfer with the Cryomech dewar at 2.5 psi, and the transfer proceeds smoothly. After the transfer we wait for about 10 minutes before withdrawing the transfer line. We cap off the transfer line with an o-ring sealed end cap. This ensures that if the valve at the end of the transfer line leaks a bit, any gas being "transferred" inadvertently will return to the storage via the gas bag. It takes about 5 hours before the system starts liquefying after the transfer is complete. Typically for this system in

operation we have 2 tanks (30 liters gas capacity) open for storage. The other tanks are at minimum pressure (34 psi) and are kept sealed off. They will only be used if we elect to warm up the dewar at some point. It also helps to have a digital pressure gauge on the recovery from the research dewar. The pressure reveals how fast the transfer is proceeding also ensuring that the impedance in the line is the same every time.

We will install a remote start for the recovery compressor. It is often prudent to start the compressor before the transfer, so that the gas bag is never overfull. It is essential that the bag be monitored to ensure it doesn't get overfull (if the transfer is too fast) leading to loss of helium.

**Transfer efficiency:**

The readings during a typical transfer are reported here.

| Time | Cryomech Liters | Cryostat % | Cryomech dewar PSI | Cryomech Coldplate K | Storage Pressure (PSI) | Bag % | Recovery line pressure Torr |
|---|---|---|---|---|---|---|---|
| 9:05 | 69.4 | 40.0 | 2.55 | 4.16 | 79 | 25% | 741.5 |
| 9:10 | 66.7 | 40.5 | 2.11 | 8.04 | 78 | 50% | 746.3 |
| 9:15 | 62.7 | 44.0 | 1.87 | 12 | 76 | 67% | 751.3 |
| 9:20 | 56.0 | 51.1 | 1.98 | 17 | 73 | 89% | 745.9 |
| 9:25 | 49.8 | 58.6 | 2.08 | 19 | 76 | 92% | 745.6 |
| 9:31 | 44.1 | 64.8 | 2.12 | 17 | 97 | 84% | 745.6 |
| 9:35 | 39.1 | 70.6 | 2.14 | 17 | 114 | 75% | 745.6 |
| 9:40 | 33.9 | 77.3 | 2.14 | 17 | 133 | 67% | 745.5 |
| 9:45 | 28.2 | 84.6 | 2.13 | 17 | 154 | 56% | 745.5 |
| 9:50 | 22.8 | 90.9 | 2.12 | 17 | 172 | 48% | 745.4 |
| 9:55 | 17.5 | 97.6 | 2.14 | 20 | 192 | 34% | 745.6 |
| 9:58 | 16.7 | 97.8 | 2.18 | 14.4 | 202 | 25% | 742.0 |

We started with the research dewar at 40% full and 69.4 liters in the Cryomech dewar. At the end, the research cryostat was at 97.8% and the Cryomech system was at 16.7 liters. 1% of the research dewar capacity corresponds to 0.768 liters so the transfer efficiency was 44.4 l/52.7 l = 84.3%.

**Keeping track of Helium Inventory**

This is an essential task to check for losses. We set up a google spread sheet to inventory the total amount of liquid equivalents in the system. Helium resides in 6 different volumes listed below and an additional column tracks the number of storage tanks.

1. Cryomech dewar: Units: [Liters]
2. Cryomech dewar gas above Liquid: Units [Liters]
3. Research Dewar: Units [%] + volume below level stick
4. Research Dewar gas above liquid: Units [Liters]
5. Gas Bag: Units [%]
6. Storage Cylinders: Units [PSI]
7. Number of storage cylinders.

| Time | Date | Dewar % | Cryo Level liters | Stor Cyl Press (psi) | #Cyl open | Gas Bag % | Liq Eq. Dewar liters | Liq Eq in Dewar gas [liters] | Liq eq In Cryom Gas [liters] | Total Inventory |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | |
| | | | | | | | | | | |

The calculations proceed as follows:

Liquid in Cryomech (liters) = Liquid reading *0.97. The liquid in the Cryomech is at a higher temperature than the helium in the dewar. The 0.97 compensates for the lower density of higher temperature liquid helium.

Liquid equivalent in gas in Cryomech dewar: = (150 - Cryomech liquid)*0.152 *0.97. This compensates for the higher density of the gas under pressure.

Dewar Liquid (liters)= Static helium below stick + % * Conversion factor (there is some helium in the research dewar below our 60 cm long level-meter "stick").

Dewar gas (liquid) = (100-% reading) * 0.768 *0.13. The 0.768 coming from the conversion of 1% to a volume in liters and the 0.13 being the density of gas relative to liquid.

Gas bag liquid: Gas Bag % *7.5/100. We assume that the gas bag between its 0 and 100 % levels holds 7.5 liters

Storage Tank Helium Liquid: # of tanks * (Cyl Pressure/400) *16 We take that each tank at 400 psi would convert to 16 liters.

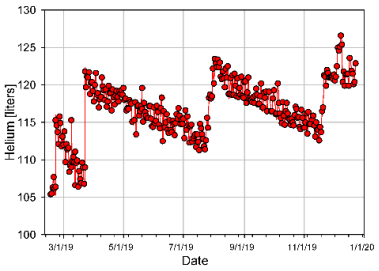


Fig 5 Helium in Cryomech system over a 9 month period. Losses were higher in Feb – March due to a leak in transfer stick. Rises in helium are due to addition of gas and from surplus helium liquid acquired in the department.

Using this spreadsheet on Google sheets we track our inventory daily. In Fig. 5 we show the helium tally. There is scatter amounting to a few liters, accumulating due to temperature variations in the Cryomech dewar and reading errors in gas bag height/volume. The loss rate of He seems to be consistent with diffusion out of the gas bag and a corresponding diffusion inward of air and water vapor. The helium loss rate is 8 liters (liquid equivalent) over a 4 month period, so 2 liters a month or about 80 moles/month. The water accumulation is around 350 gm or about 20 moles a month. We measured the air accumulation in the trap (470 l-atm of gas -21 moles to saturate it) over a 1-2 month period.

We state that 2 tanks is optimum in the gas-storage circuit. This is true for our liquefaction cycle and usage. There are competing needs. It is desirable to maintain the maximum amount of liquid on hand, or the smallest amount of gas in storage, thus minimizes the external volume. However, a single tank implies 15 liquid liters of gas can be recovered before the system starts to vent – this is not ideal. So two tanks gives us a storage capacity of about 30 liquid liters. Too much gas in storage means that the system may not be ready to start liquefying when you want to transfer. This is a system and application-specific need, so individual users will have to optimize for their own needs.

**Hydrogen**

There is evidence that even small amounts of hydrogen can plug impedances especially in long running helium three experiments[1]. Here is one solution.

First the symptoms. When we started running with the liquefier we purchased standard purity (0.5 ppm) $^4$He from Airgas. About 2 months into the run the 1 K pot which draws helium from the bath plugged and we were able to get it to run again by heating the impedance while overpressuring the pot. It ran for another month then plugged again, and so on. After about 6 months it gave out altogether.

We then bought a hydrogen getter from Cryomech[2]. It is placed in series just after the purifier air trap (Fig 6). In a year's continuous running we have no problem with hydrogen in the 1 K pot fill line. We also tried some fine (sub micron) filters on the inlet and outlet of the transfer line and these did not help at all. A problem down the road is that we have no idea when the getter is saturated.

Fig 6 Image of the hydrogen getter panel attached to purifier

Another solution might be to buy ultrapure $^4$He and circulate it in a closed system like you do the dilution fridge mixture. You would need a nitrogen trap but LN2 is cheap.

**Initial Transfer**

The initial transfer is especially tricky given that users are likely not used to the transfer system. It is essential to maintain visual contact with the gas bag so it is not overfilled. Also, the valve on the transfer line is quite non-linear so changes in opening the valve need to be small and allow several minutes for settling time.

We made a special termination for the transfer line (Fig. 7). Ours ends in a nozzle that terminates in the transfer stick extension used only for the initial transfer (and to extract nitrogen (precooling liquid) from the very bottom of the dewar). That extension is screwed to the end of the Cryomech transfer stick. The helium liquid that emerges from the central line will blow back into the triaxial line, so external valving has to be used to maintain flow of cold gas down to the bottom of the dewar, while still diverting some back through the transfer line. The settings used for the initial transfer should be noted for future use.

Fig 7. Top image is of the transfer stick end that we capped off and now use a Teflon O-ring to seal. The bottom piece mates with our transfer stick extension. It is only used during the initial transfer.

After removing any liquid nitrogen used to precool the system we start by opening the transfer stick valve gently. A thermometer at the bottom of the dewar verifies that cold gas flows to the bottom of the dewar. The pressure at recovery is monitored together with the gas bag height (so as to not overfill it and lose helium). It is easy to send much of

the liquid through the return path in the transfer line, so the relative impedances of the recovery from the research dewar and the transfer line need to be controlled. We have 1 inch ball valves in the line from the research dewar recovery and from the transfer line recovery that can be adjusted independently.

**Conclusion:**

This system is an efficient and cost-effective solution to ensuring supply of helium. The operation of the cryostat was not impacted by the location of the liquid helium plant in close proximity to the cryostat.

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[1] *Hydrogen-Free Liquid-Helium Recovery Plants: The Solution for Low-Temperature Flow Impedance Blocking,* M. Gabal, A. Arauzo, A. Camón, M. Castrillo, E. Guerrero, M. P. Lozano, M. P. Pina, J. Sesé, S. Spagna, J. Diederichs, G. Rayner, J. Sloan, F. Galli, W. van der Geest, C. Haberstroh, N. Dittmar, A. Oca, F. Grau, A. Fernandes, and C. Rillo, Phys. Rev. Applied **6**, 024017 (2016). https://journals.aps.org/prapplied/abstract/10.1103/PhysRevApplied.6.024017

[2] Entegris GateKeeper IX series GPUS2500FIX08R00CA, Entegris, 11700 Willow Creek Road, San Diego, CA 92131, USA.