Helium Recovery – How we do it in Santa Cruz

Art Ramirez July 5, 2013

This advisory provides some tips to aid in the design and construction of a helium recovery and liquifaction plant system.

Liquifiers suitable for a single-investigator lab are now available from Cryomech, Quantum Design and perhaps other companies. Quantum Technology makes LN2 traps for helium purification. Many researchers have purchased cryostats with integrated liquifaction cold heads. I decided not to go this route but rather chose a central liquifaction "plant" which allows me to have different sets of cryostats cold at any one time. Such a plant produces ~18-30 liters per day into a storage dewar with capacity of up to 160 liters.

Estimate helium usage: Be sure to account for the extra helium needed for initial cooldown in your estimate of total usage. This is especially important if you want to support more than one cryostat. If contemplating one of the liquifiers that operates at 5 psi or above atmospheric pressure, take into account the time needed to depressurize the dewar as this will eat into the time-averaged liquid production.

Helium recovery: If your liquefier cold-head is not attached to the measurement dewar, as discussed here, then you will have to transfer helium. During transfer, several liters of liquid will become gas and your liquifier won't be able to keep up. This is the main reason to have a large amount of room-T low-pressure storage. I know of two different approaches to room-T helium gas storage.

PV - nRT
1 cubic ft = 28.3 liters
1 liter LHe = 757 liters gas

1) Medium-pressure storage. Metal tanks, such as propane cylinders, can store the boil-off at a medium pressure, say ~50 psi. Metal containers introduce negligible impurities, thus you might be able to get away without a purifier. I've had a few mishaps in which the helium became contaminated with air at the 1-20% level. If this happens and you don't have a purifier, then you will lose all the contaminated helium since the liquefier cold heads need 5-nines pure helium for effective operation. The companies mentioned above will offer medium-pressure set-ups.

One thing to consider – I've been told that the temperature-control mechanisms in Quantum Design measurement cryostats are predicated on the need for the annulus pump to see 1 atmosphere pressure at their exhaust. Thus, an intermediate compressor or pump that bridges the pressure difference will be needed for the medium pressure route.

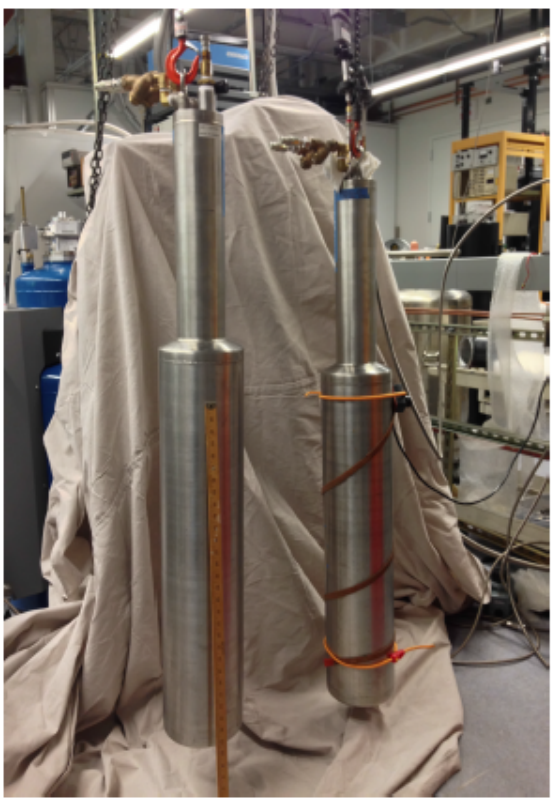
2) High-pressure storage. This is the route I took since I anticipate keeping 3-4 cryostats cold continuously with a helium production rate of 40-50 liters per day. Since the storage medium is comprised of high-pressure (DOT2400) cylinders,

which can be pressurized to above 2500 psi, I bought a 3-stage compressor (made by Bauer Compressors Inc.).

Helium recovery rubber bag, 500 cubic ft, partially inflated

Obviously, the cryostat cannot boil off directly into these cylinders, and you don't want to run the compressor continuously so you will need an inter-mediate, low-pressure storage vessel – a bag. Large rubber bags are made for use as liners in oil storage tanks and I bought mine from Flexi-Liner Corp.

Hint – I bought the high quality rubber, which turns out to be lighter in weight than the lower quality, less expensive rubber. Unfortunately, because this bag is so light it only triggers my compressor when it's very full and wouldn't pump out completely, thus, I made a frame from PVC tubing that rests on top of the bag to add extra pressure.



Two QuantumTechnology LN2 helium purification traps. The right-hand one has a heat tape wrapped around it and is being pumped.

Regarding the tubing ID and length for routing the helium from the cryostats to the bag, bag to Bauer, and Bauer to liquefier, I use 1.5"/20', 1"/100', and 3/8"/80' respectively, where the IDs are the nominal dimensions for US copper tubing. Most of the tubing and fittings were purchased from a local big box hardware supply and the joints are sweat fit (soft soldered).

Helium Purification. The main drawback of the rubber bag storage buffer is it's porosity to air, which is large enough to matter for the liquefaction process. Thus I believe that all recovery systems using a bag need to incorporate helium purification. LN2-cooled purifiers that are essentially a large can full of high-surface-area zeolite pellets, work well, but you will need to keep the LN2 dewar filled, which is an added expense.

Be sure to make the trap big enough. My first (home-made) trap had a volume of about 3.5 liters of zeolite and it required regeneration every 1.5 days. My new trap, made by Quantum Technology, has a zeolite volume of 30 liters and thus needs regeneration every other week. Regeneration amounts to removing the trap from the LN2 bath, allowing it several hours to reach room-T and then doing a ~10 hour heated pump out, using a 5-ft heat tape. Since this takes about 1-2 days, if this is your only means of regeneration, then it might make sense to have two such traps, so that a clean trap can be put into action as soon as the dirty trap is removed from the LN2 bath.

In my lab, to purify 30 liters (gas) per minute continuously, I need roughly one 240 liter tank of LN2 per week, which includes the extra LN2 needed for regeneration.

Quantum Design is manufacturing a cold-head cooled purifier, the ATP-30, eliminating the need for LN2.

Helium Liquifaction I use two different systems, Cryomech's 18 liter/day system and Quantum Design's ATL-160.

Quantum Design's ATL-160 (left) and the ATP-30 (right)

The QD system uses the Gifford-McMahon cooling cycle and produces between 18 and 30 liters per day, depending on the pressure in the dewar, which has a capacity of 160 liters. The highest rates are achieved with a 10 psi operating pressure. Note it takes about 5 hours for the pressure to decrease from 10 psi to 5 psi, a pressure at which I've been able to fill without too much boil-off. The boil-off comes from the evaporative cooling needed to bring 5 psi liquid at 4.55K down to 0 psi (=1 atm) liquid at 4.2K. The ATL-160's air-cooled compressor can be located 20 meters away from the dewar and cold head, which allows the heat created by the refrigeration process to be dumped far away from the dewar. This system is easy to use and remotely controllable.

Cryomech's 18 liter per day system with 160 liter storage dewar

The Cryomech system uses the pulse tube refrigeration cycle and also comes with a 160 liter dewar. This system has a single working pressure around 1.5 psi and thus no depressurization time is needed prior to a transfer. The Cryomech's compressor is mounted on a cart with the dewar and cold head. The version I have is water-cooled and thus, the heat generated is dumped into a water chiller and ultimately into the air around the chiller.

One drawback of both systems is the noise generated at the cold head. The noise doesn't present a health safety issue, but it's loud enough to make a conversation across the lab impossible.

Once the entire recovery and liquefaction system is set up, running it doesn't take up too much more time than using purchased liquid helium and venting to the atmosphere, which I've been doing for over 30 yrs!