A Cryogen Recycler with Pulse Tube Cryocooler for Recondensing Helium and Nitrogen

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Abstract. We have developed a cryogen recycler using a 4 K pulse tube cryocooler for recondensing helium and nitrogen in a NMR magnet. The liquid helium cooled NMR magnet has a liquid nitrogen cooled radiation shield. The magnet boils off 0.84 L/day of liquid helium and 6 L/day of liquid nitrogen. The recycler is designed with both a liquid helium return tube and a liquid nitrogen return tube, which are inserted into the fill ports of liquid helium and nitrogen. Therefore the recycler forms closed loops for helium and nitrogen. A two-stage 4 K pulse tube cryocooler, Cryomech model PT407 (0.7W at 4.2 K), is selected for the recycler. The recycler was first tested with a Cryomech's test cryostat and resulted in the capacities of recondensing 8.2 L/day of nitrogen and liquefying 4 L/day of helium from room temperature gas. The recycler has been installed in the NMR magnet at University of Sydney since August, 2014 and continuously maintains a zero boil off for helium and nitrogen.

1. Introduction

Helium is a finite resource, and once released into the atmosphere, it readily escapes into space. This fact in conjunction with the multitude of helium applications that do not utilize any kind of helium recovery have resulted in a steady decrease in the world's helium resource. According to the US Geological Survey, cryogenic applications accounted for 32% of the 47 million cubic meters of helium used by the United States in 2013, more than any other category. Helium users face periodic helium shortages as well as high and continuously increasing helium prices. Therefore, it is important to recover and reuse helium.

We have developed a helium reliquefier to convert an existing open liquid helium cryostat into a closed-cycle system that does not require liquid helium refills [1]. The reliquefier retrofits into the fill port of the cryostat, recondenses the liquid helium boil-off, and then returns the liquid helium to the cryostat. The reliquefier is the most efficient way to recover boil-off from a single cryostat. So far few hundreds of liquid helium systems around the world have been installed with the reliquefier and no longer require refill of liquid helium.

Some liquid helium cooled superconducting magnets, such as NMR magnets [2], use liquid nitrogen to cool radiation shields. In these systems, liquid nitrogen is refilled weekly and liquid helium is refilled monthly by an experienced technician. The operation of these magnets is costly because of cryogen refill.
We were contacted by Richard Marsh at DMIS and The University of Sydney to develop a recycler to recondense helium and nitrogen simultaneously in their NMR magnet. The magnet is Oxford Model, 3T/150 NMR/FTMS. The recycler should provide >6 L/day recondensing rate for nitrogen and 0.84 L/day recondensing rate for helium to maintain zero boil offs of cryogen. This paper presents the design and performance of the recycler as well as its operation with the NMR magnet.

2. System design
The recycler has a similar design of the reliquefier [1] on helium liquefaction circuit. Figure 1 shows a schematic of the reliquefier with a test cryostat at Cryomech, Inc. The test cryostat has a liquid nitrogen reservoir for cooling a 77 K radiation shield and a liquid helium reservoir. Both liquid nitrogen and helium reservoirs are made of stainless steel with matching volumes of 1.8 Liters. The recycler includes the pulse tube cryocooler (1,2,7,8) and the main assembly (10). For reduced vibration Cryomech's model PT407-RM pulse tube cryocooler was chosen. The vibration reduction results from the motor/valve assembly’s (1) separation from the cold head (2).

The pulse tube cold head resides in the vacuum insulated neck (4) of the main assembly and is surrounded by helium gas. A vacuum insulated liquid helium return tube (11) with an OD of 12.7 mm is inserted into the helium fill port (b) of the cryostat (g). A flexible vapor line (3) connects the gas inlet on the top of the recycler and a helium vent of the cryostat. It forms a closed helium loop - where no helium leaves the system. A temperature sensor and a heater are installed on the 2\textsuperscript{nd} stage/condenser (8).

![Figure 1. Schematic of the recycler with a NMR magnet.](image)

1. Remote rotary valve; 2. Pulse tube cold head; 3. Vapour line; 4. Sleeve; 5. Radiation shield; 6. Nitrogen condenser; 7. Pulse tube 1\textsuperscript{st} stage; 8. Pulse tube 2\textsuperscript{nd} stage/helium condenser; 9. Liquid nitrogen return tube; 10. Main assembly of recycler; 11. Liquid helium return tube; 12. Pressure regulator; 13. Helium gas cylinder. a. liquid nitrogen fill port; b. liquid helium fill port; c. liquid nitrogen bath; d. radiation shield; e. liquid helium bath; f. liquid helium container; g. Cryostat.
A nitrogen condenser (6) is attached on the cooling station inside the vacuum chamber. The cooling station is also used for cooling the radiation shield (5). The cooling station in the vacuum space is cooled by the 1st stage (7) of the pulse tube through gaseous heat exchange. The upper end of liquid nitrogen return tube (9) is connected to the condenser and the lower portion with an OD of 9.5 mm inserted into the nitrogen fill port (a). Nitrogen vapor and condensed liquid flow reversely through the liquid nitrogen return tube. Nitrogen vapor of temperature 77 K is recondensed by the nitrogen condenser. To maintain the desired vapor pressure a heater is installed on the nitrogen condenser.

The temperatures on the 1st and 2nd stage/condenser of the pulse tube cryocooler as well as the temperatures of liquid nitrogen and liquid helium are measured. However, the temperature of the nitrogen condenser has not been measured in the present study due to the difficulty of sensor installation. The vapor pressures of nitrogen and helium are measured by two pressure transducers.

Figure 2 shows the 3-D modeling of the recycler installed on the NMR magnet. The geometries of two liquid return tubes are designed to fit into both nitrogen and helium fill ports of the magnet. The remote motor/rotary valve assembly is mounted on the wall.

3. Experimental results and performance

3.1. Experimental tests and results

The cooling load map of the pulse tube cryocooler was measured before assembling the recycler, see figure 3. It provides 0.74 W at 4.15 K on the 2nd stage and 27 W at 55 K or 37 W at 65 K on the 1st stage while consuming an electrical power of 6.5 kW. Based on the estimation of the heat transfer for nitrogen condensation, the 1st stage will operate at ~65 K to build a large enough temperature differential for recondensing > 6 L/d of nitrogen.

The recycler equipped with the PT407RM is first tested with the test cryostat at Cryomech. The test system with the recycler in the cryostat has separate closed-loops for nitrogen and helium. The liquid nitrogen reservoir is filled with liquid nitrogen before the recycler is turned on while the liquid helium reservoir is still at room temperature. The liquid helium reservoir is first evacuated and then
filled with helium gas through the pressure regulator (12) in figure 1. The system is ready to be cooled down by the recycler.

![Figure 3. Measured cooling load map of the PT407RM.](image)

Figure 4 shows cool down curves of the recycler and the test cryostat. The PT407 cold head reaches the liquefaction temperature of 4.2 K in 2 hours. The recycler internal components including the liquid return tube are cooled to liquid helium temperature in 2.5 hours. The liquid helium then starts to drop into the helium reservoir and rapidly decreases the temperature of the reservoir. It takes ~19 hours for the helium reservoir to reach liquid helium temperature and build up liquid helium.

![Figure 4. Cooling down curve of the system.](image)
The flow rate of helium gas from the gas cylinder is recorded to measure the liquefaction capacity in the liquefaction process. The flow rate of gas indicates the helium liquefaction rate of the recycler from room temperature gas. They are both shown in figure 5. The liquefaction rate is increasing during the test because some components of the system are still cooling down. The gas flow rate can be as high as ~1.97 SLPM, corresponding to a liquefaction rate of 4 L/d.

Once the liquid helium reservoir accumulates enough liquid for a test of zero boil off, helium supply from the gas cylinder is shut off. The heater inside the liquid helium reservoir is turned on to maintain a positive vapour pressure for the helium circuit. Figure 6 shows stable temperatures of the recycler condenser and liquid helium in the reservoir during the test of zero boil off. A heat load of 0.31 W is applied to the liquid helium reservoir to maintain an absolute vapour pressure of 105 kPa. The heat load of 0.31 W in the liquid helium indicates a liquid helium boil off rate of 10.2 L/d. The recondensing/reliquefaction capacity is much higher than the liquefaction rate of 4 L/d for the room temperature gas. Explanations of the performances have been presented in reference [1].

The nitrogen circuit has liquid nitrogen continuously during the testing. In the beginning, a small portion of liquid nitrogen boils off and releases through a 5 psig pressure relief valve because of the higher temperature of the cryocooler’s 1st stage. When the 1st stage of the cryocooler reaches a temperature below 77 K, the nitrogen condenser starts to recondense the nitrogen boil off. The condensed liquid can then drop back to the liquid nitrogen reservoir.

Figure 7 shows operating parameters of the nitrogen circuit during stable operation. A heat load of 15.2 W is applied to the nitrogen condenser for maintaining an absolute vapour pressure of 105 kPa. This heat load indicates the recycler has a nitrogen recondensing rate of 8.2 L/day. The liquid nitrogen temperature is at 78.8 K.
3.2. Installation and performance

The recycler tested at Cryomech Inc. meets the design specifications of recondensing capacity for the NMR magnet. It was installed in the NMR magnet at the University of Sydney. Figure 8 shows a photo of the recycler installation on the magnet. The system has been operating smoothly since August, 2014.

The operating parameters of the recycler in the magnet are listed in table 1. The vapour pressures of nitrogen and helium in the system are controlled by the heaters on the nitrogen and helium condensers. The nitrogen vapour pressure is maintained at 106 kPa by applying a heat load of 5.7 W to the nitrogen condenser. A heat load of 0.45 W is applied to the helium condenser to keep a vapour pressure of 108 kPa. The recycler demonstrates abilities to maintain zero boil for the nitrogen and helium with a large margin of extra capacity.
Table 1. Operating parameters of the reliquefier in the NMR magnet

| Nitrogen circuit | Helium circuit |
|------------------|----------------|
| $T_1$ | $T_2$ | $Q_N$ | $Q_{He}$ | $P_N$ | $P_{He}$ |
| 65.1 K | 3.9 K | 5.7 W | 0.45 W | 106 kPa | 108 kPa |

Note: $T_1$ is the 1st stage temperature of the pulse tube cryocooler; $T_2$ is the 2nd stage temperature of cryocooler; $Q_N$ is the heat load applied to the nitrogen condenser; $P_N$ is the absolute vapor pressure of nitrogen; $Q_{He}$ is the heat load applied to the helium condenser; $P_{He}$ is the absolute vapor pressure of helium.

4. Conclusions
A recycler, employing a 4 K pulse tube cryocooler, has been developed for recondensing nitrogen and helium simultaneously in a NMR magnet. It has a recondensing rate of 8.2 L/day for nitrogen vapour and liquefaction rate of 4 L/day for helium gas from room temperature. The recycler installed in a NMR magnet at The University of Sydney has been maintaining zero boil of nitrogen and helium with a large margin of extra capacity since August, 2014.

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References
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