Helium recovery and purification at CHMFL

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Helium recovery and purification at CHMFL

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Abstract. Currently, rising demand and declining reserves of helium have led to dramatic increases in the helium price. The High Magnetic Field Laboratory of Chinese Academy of Sciences (CHMFL) has made efforts since its foundation to increase the percentage of helium recovered. The piping network connects all the helium experimental facilities to the recovery system, and even exhaust ports of pressure relief valves and vacuum pumps are also connected. In each year, about 30,000 cubic meters helium gas is recovered. The recovery gas is purified, liquefied and supplied to the users again. This paper will provide details about the helium recovery and purification system at CHMFL, including system flowchart, components, problems and solutions.

1. Introduction
As an important research tool, the magnetic field has been extensively used in such diverse disciplines as biology, chemistry, engineering, metallurgy, and physics. High magnetic fields may aid in new discoveries and contribute much to the fundamental knowledge and understanding of matters. The High Magnetic Field Laboratory of Chinese Academy of Sciences (CHMFL) is being funded for the production and use of high magnetic fields in all areas of research. The lab is open to all qualified scientists and engineers worldwide [1].

In the lab, there is a LHe plant which was purchased from Air Liquide in 2011 [2]. The plant is used to supply liquid helium for all superconducting magnets and cryogenic experimental facilities. Each year, more than 40,000 liters of liquid helium need to be supplied, and the quantity is increasing (see Figure 1). In order to decrease the operating budget, a helium recovery system has been built. The evaporated helium is recovered by the helium recovery system, then purified to 99.999% and filled to storage tanks. The pure gas is re-liquefied by the LHe plant. The system has been in service for more than three years since its commissioning in 2012. In this paper details about the helium recovery and purification system at CHMFL, including system flowchart, components, problems and solutions are presented. Methods to improve recovery percent and reduce influence of recovery system on the performance of the user’s experimental facilities are also mentioned.

2. Main components

2.1. LHe plant
The LHe plant is based upon a Claude cycle with static gas bearing turbo expanders. It consists of an oil injected screw compressor, oil removal system, gas management panel and coldbox. It can produce 110 liters of liquid helium per hour in liquefaction mode, or provide 360 W cooling capacities at 4.5 K in refrigeration mode (with LN2 pre-cooling).
Figure 1. Annual liquid helium consumption of the twelve experimental facilities: Crystal Growth System (CGS), Optical Magnetic System (OMS), STM-MFM-AFM Combo System (SMA), Fourier Transform Infrared Spectroscopy (FTIR), RAMAN Spectroscopy, JANIS_9T Magnet, Electron Spin Resonance Spectrometer (ESR), Physical Property Measurement System (PPMS), Magnetic Property Measurement System (MPMS), Magnetic Resonance Imaging (MRI), Ultra Low Temperature Experiment System (ULTES) and Superconducting Magnet Technology Research (SM_TECH).

As shown in Figure 2, pure gaseous helium (99.999%) compressed at ambient temperature enters the coldbox through high pressure (HP) pipe. In the first step, the HP helium is cooled down in the first counter-flow plate fin heat exchangers by the returning low pressure stream of helium and the pre-cooling liquid nitrogen. The HP gas is further cooled down to about 80 K before it is transferred into the 80 K adsorber. Almost all contaminants are removed by the activated carbon in the adsorber. Then the HP stream is divided into the turbine and the main J-T stream. In the turbo expanders the HP helium is expanded to LP helium before joining the returning stream. At the same time, the main stream is liquefied into liquid helium by the J-T valves, and the liquid helium is transferred to the main 1000 L dewar via a transfer line. The transfer line is made of two coaxial lines in which liquid flows in the inner pipe from the coldbox toward the dewar and the cold vapour from the dewar returns via the annular channel to minimize the heat load to liquid helium.

2.2. Liquid helium dewars
The main dewar which is fixed beside the coldbox is equipped with a superconducting gauge for level measurement and a heater for necessary dewar pressurization.

Since the experimental facilities are dispersedly located in a separate building, we need to transfer liquid helium from the main dewar to mobile dewars and distribute to the users. We have 23 mobile dewars manufactured by CRYOFAB with different capacities of 60 L, 100 L, 200 L and 500 L. The maximum possible inventory of liquid helium is about 8000 L, including the capacity of the main dewar.
2.3. Gas bag

The 50 m³ gas bag hangs on the roof at a height of 12 m. It is equipped with a level transmitter which allows starting or stopping the recovery compressor according to its filling (see Figure 3). For safety considerations, a safety device sealed by oil column is installed on the gas bag for emergency venting. The set point of the safety device is 3000 Pa.
2.4. Recovery compressors
There are two 4-stage-piston compressors for recovery helium compression. One compressor with a capacity of 25 m$^3$/h is manufactured by SAUER; another one with a capacity of 50 m$^3$/h is manufactured by BAUER. To reduce the gas losses, gas from safety valves and automatic condensate drain valves is gathered to a condensate tank. On the top of the condensate tank, there is an interface connected to the intake buffer. Two filters for the removal of residual humidity, particles and oil vapours from the compressed gas are serially installed at the exhaust side of the compressors.

2.5. Gas storage tanks
The high pressure storage which is used to store impure recovery gas consists of 400 high pressure cylinders with a total volume of 16 m$^3$. The maximum operating pressure is 150 bar.

As medium pressure storage, there are two tanks for storing pure helium with a capacity of 100 m$^3$ each. The maximum operating pressures are 20 bar and 16 bar respectively.

2.6. Purifier
Since the helium in the liquefaction process must fulfil very high purity standards, the recovered helium needs to be purified before being re-liquefied. The helium purifier which is manufactured by Air Liquide removes the impurities through a dryer and a bed of cryogenic adsorbent (see Figure 4). The dryer is composed of 80% of Alumina for moisture trapping and an additional 20% of molecular sieve dedicated to the adsorption of CO$_2$. The cryogenic adsorber filled with carbon molecular sieve (CMS) traps nitrogen and oxygen when cooled down close to liquid nitrogen temperature. The technical performance of the purifier is shown in Table 1.

![Figure 4. Flowchart of the purifier [4].](image-url)

The adsorption property of the CMS filled in the adsorber tank may be damaged when residual oil is mixed into the feed helium. Also the heat transfer performance of the heat exchanger may be affected if oil freezes on its surface. So a multi-stage oil filter was developed by our lab to removes oil before it passes through the purifier. The filter contains four serially connected cylinders with a
The first three cylinders which are filled of stainless steel fiber are used to retain oil droplets, and the last one with activated carbon is used to adsorb oil vapors. The results show that the residual oil content of the helium can be reduced by the filter cylinders from 75 ppb to 10 ppb. Operation time of the helium purifier is substantially extended due to reduced oil absorption. Figure 5 shows the residual oil content range in helium after being purified by several types of oil filters.

Table 1. Technical performance of the purifier [4].

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Manufacturer</td>
<td>-</td>
<td>Air Liquide</td>
</tr>
<tr>
<td>Operating pressure</td>
<td>bar abs</td>
<td>20</td>
</tr>
<tr>
<td>Flow rate range</td>
<td>Nm³/hr</td>
<td>10-40</td>
</tr>
<tr>
<td>Inlet helium impurity</td>
<td>N₂ + O₂</td>
<td>ppm ≤2000</td>
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<tr>
<td></td>
<td>H₂O</td>
<td>ppm ≤500</td>
</tr>
<tr>
<td>Outlet helium impurity</td>
<td>N₂ + O₂</td>
<td>ppm &lt;1</td>
</tr>
<tr>
<td></td>
<td>H₂O</td>
<td>ppm &lt;1</td>
</tr>
<tr>
<td>Working capacity</td>
<td>Nm³</td>
<td>960 (99% helium feed)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>520 (98% helium feed)</td>
</tr>
</tbody>
</table>

Figure 5. The residual oil content range in helium after being purified by several types of oil filter.

2.7. Piping network and interface

The piping network which is almost exclusively made of stainless steel connects all the experimental facilities to the helium recovery system. The experimental facilities are located in another building which is about 150 m away from the compressor station and gasbag. Up to now, there are totally 12 superconducting magnets and cryogenic experimental facilities.

In each facility cell, there are four quick-connect interfaces for helium receiving. The first is connected to a mobile dewar to receive gas from static evaporation of liquid and dewar decompression; the second is used to recover helium pumped from chamber with working temperature of below 4.2 K, but a prerequisite is that oil content in the helium must be removed by oil filters; the third is connected to the venting port of the experimental facility’s cryostat through a piece of rubber hose which can isolate oscillation and electricity between the experimental facility and the recovery system; the fourth interface is for spare. The four streams are collected to one stream and flow successively through an ambient heat exchanger, a diaphragm natural-gas meter and a check valve to the main recovery line.
Additionally to the experimental facilities, safety valves and venting lines of the coldbox, purifier, compressors and storage tanks are connected to the main recovery line.

3. Operation and management

In the past three years, efforts as follows have been done to improve the performance of the recovery and purification system. Up to now, the total helium recovery rate is improved to 90% (see Figure 6).

1) Adding oil filters to pumps’ exhaust port and introducing pumping helium gas to the recovery line contribute to 10% increasing of helium recovery rate.

2) Between the main recovery line and the gas bag, a pressure maintaining valve whose design set pressure is only 400 Pa is developed and installed. It can prevent recovery helium from air penetration; moreover, daily leakage detection can be easily done.

3) Quenching of superconducting magnets or sudden vacuum break of cryostats can cause a high gas pressure on the piping network, and it may damage the recovery system and other experimental facilities linked. So a safety valve with big venting area and low set pressure is installed to response these conditions.

4) For the purifier, a developed oil filter was added to its inlet to reduce oil introduction. Consequently, the operation time with the same helium feed flow rate increased.

5) It is very important to make users aware to save helium. The helium recovery rate for each experimental facility is usually supervised.

![Figure 6. Annual liquid helium distributed (blue) and recovered (red) at CHMFL](image)

4. Summary

The recovery and purification system at CHMFL has been normally running for a long period. In the future we will make the system friendlier and easier to use, and we hope to involve all the helium users in the recovery effort. The helium loss shall furthermore be minimised.

References