Technology

Development of Helium Recycling System for NMR

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1. Introduction

The Helium Recycling System is a device designed to recondense (i.e., reliquefy) evaporated liquid helium (LHe). In its operation, helium gas (GHe) that would otherwise be vented to the atmosphere due to evaporation is recovered and recondensed within the system, thereby minimizing gas losses. Our company offers a range of recondensation capacities from 1 L to 18 L per day (in liquid helium equivalent). This device can be retrofitted to many systems utilizing LHe, thereby reducing the helium management burden during experiments and operational processes. Moreover, with several hundred units already sold, the system has earned a high degree of trust in many research institutions and industrial applications.

uclear Magnetic Resonance (NMR) systems, which are capable of probing molecular structures and dynamic states, are widely employed in fields such as condensed matter physics, polymer chemistry, biochemistry, and medicine—with several thousand units in operation domestically. In NMR systems, superconducting coils are used to generate strong magnetic fields, and LHe is employed to Maintain the superconducting state. In recent years, due to an increase in LHe demand coupled with supply uncertainties leading to severe shortages and price hikes, the demand for helium recycling systems employing mechanical refrigerators has growestically, NMR systems are primarily provided by two major companies, one of which is Bruker Japan. In collaboration with Bruker Japan, our company has been working toward the commercialization of a helium recycling system specifically designed for NMR applications. Meanwhile, a similar product is already available from the other major supplier. Our newly developed helium recycling system for NMR has undergone demonstration and evaluation tests in cooperation with Bruker Japan, confirming that its performance meets the practical requirements for commercialization. This paper presents an overview of the NMR helium recycling system and its distinctive features.

2. System overview

In designing the NMR helium recycling system, special attention was given to two issues based on the characteristics of NMR equipment. The first is the elimination of the effects of mechanical vibrations. Since external mechanical vibrations can introduce significant measurement noise in NMR systems, the design must mitigate these disturbances. The second issue is the stability of the LHe dewar pressure. Pressure fluctuations within the NMR apparatus can cause magnetic field instabilities that may affect spectral quality; therefore, a

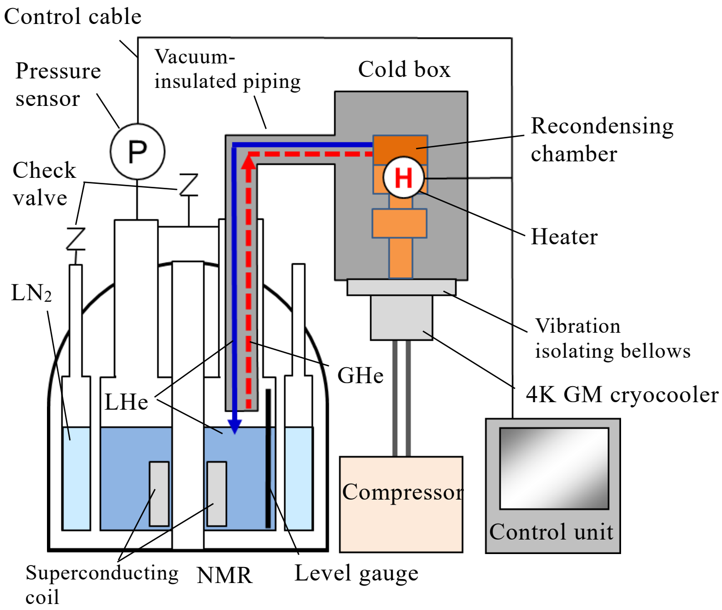

Figure 1 Main configuration of NMR system and helium recycling system

control mechanism to maintain a constant internal pressure of the LHe dewar is required.

Table 1 System specifications

Recondensing capacity (specification value)	1 liter per day (LHe equivalent)
Cold box dimensions	ϕ190 x H 660
Control unit dimensions	W 321 x H 295 x D 330
Compressor dimensions	W 450 x H 485 x D 591
Power consumption	6.5/7.5 kW (50/60 Hz)
Maintenance cycle	Every 10000 hours

Figure 1 illustrates the main configuration of the NMR system and the helium recycling system, while Table 1 presents the specifications of the helium recycling system for NMR applications. The NMR system is equipped with an LHe dewar, in which the superconducting coil is installed, and an LN2 dewar designed to reduce heat ingress. Both the LHe and LN2 dewars are fitted with non-return valves to prevent pressure surges. In the absence of the helium recycling system, the evaporated gas is typically discharged to the atmosphere through these valves. An LHe level meter is integrated into the LHe dewar, which triggers LHe replenishment when the liquid level falls.

The helium recycling system mainly comprises a cold box, a compressor, and a control unit. The cold box houses a 4K GM (Gifford-McMahon) refrigerator that produces the low temperatures required for recondensation, a recondensation chamber, and an integrated heater. Furthermore, a vacuum-insulated transfer line is attached to connect the recondensation chamber with the NMR system's helium dewar port. An anti-vibration bellows is installed between the cold box and the 4K GM refrigerator to minimize the transmission of mechanical vibrations generated by moving parts within the refrigerator. The compressor circulates the refrigerant gas for the 4K GM refrigerator and is connected to it via two flexible tubes.

The control unit operates the start/stop functions of both the 4K GM refrigerator and the compressor, and controls the heater in the recondensation chamber based on the signal from a pressure sensor mounted on the top of the NMR system, thereby maintaining a constant pressure in the LHe dewar. The recondensation process involves the upward flow of evaporated GHe through the vacuum-insulated transfer line into the recondensation chamber, where it is maintained at the liquefaction temperature by the 4K GM refrigerator, resulting in the formation of LHe. The recondensed LHe flows back through the transfer line, ensuring a stable liquid level in the dewar. As described, the heater in the recondensation chamber stabilizes the dewar pressure, balancing the amount of GHe entering the chamber with the quantity of LHe generated, thereby maintaining a constant LHe level. The control pressure is set to prevent gas discharge through the non-return valve and to avoid negative pressure conditions in the NMR system. These operational measures enable a zero-boil-off mode with no external gas loss.

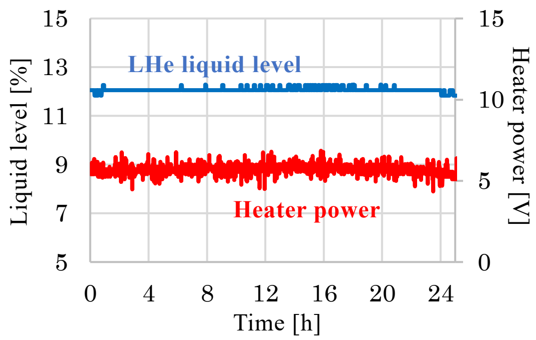

Figure 2 Liquid level maintenance operation of the recondensation system

3. Evaluation of helium recycling system operation

3.1 Evaluation with in-house equipment

Figure 2 shows the behavior of the LHe level and heater load during zero-boil-off operation of the helium recycling system. Prior to demonstration testing on an NMR system, operational characteristics were evaluated using an in-house low-evaporation cryostat. The

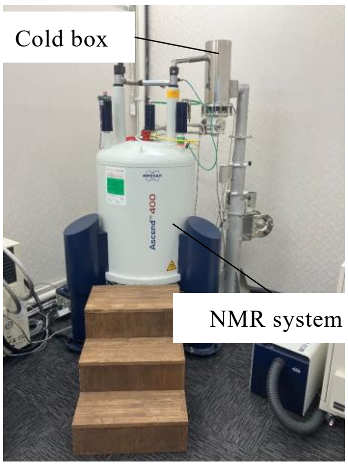

Figure 3 Installation status on the NMR system

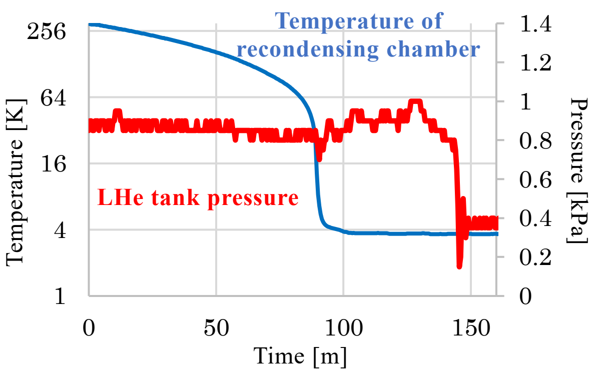

Figure 4 Cool-down characteristics of helium recycling system

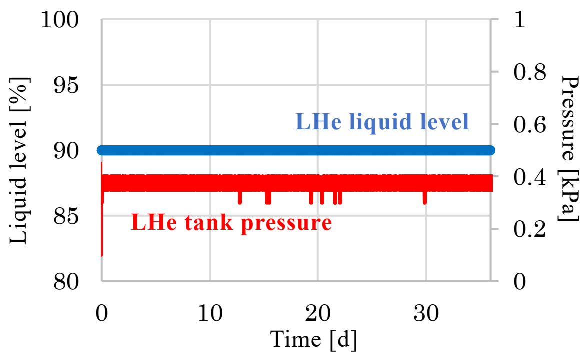

Figure 5 Long-term zero-boil-off operational results

measured helium evaporation rate in the cryostat was approximately 0.54 L per day, which is comparable to typical evaporation rates in NMR systems. The operating results confirmed that the heater output was controlled at around 5 V to maintain the pressure inside the cryostat, thereby ensuring a stable LHe level.

These results confirm that both the recondensation capacity and the stability of zero-boil-off operation for integration with an NMR system are sufficient.

3.2 Demonstration evaluation with NMR device

Figure 3 illustrates the installation of the helium recycling system on an NMR system (Ascend400), with the vacuum-insulated transfer line inserted into the LHe filling port located on the right side. Figure 4 shows the cooldown characteristics of the helium recycling system. A temperature sensor for evaluation was mounted on the exterior surface of the recondensation chamber. Upon starting the system, the temperature in the recondensation chamber decreased, reaching 4.2 K in approximately 90 minutes. Although the pressure control setting was 0.4 kPa, the system initially discharged GHe at approximately 0.8 kPa—the setting pressure of the non-return valve. After about 140 minutes, a rapid pressure drop was observed, and subsequent stabilization of the LHe dewar pressure via the control system indicated the establishment of zero-boil-off operation.

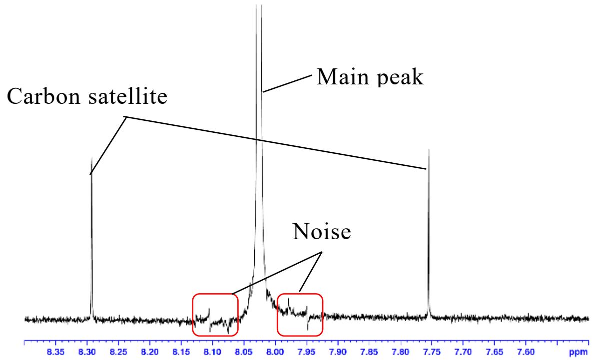

Figure 6 Spectral data

Figure 5 presents the long-term zero-boil-off operation results over 35 days. During over one month of continuous operation, the LHe level was stably maintained at 90%, with no detectable evaporation loss from the LHe dewar. Although there were concerns regarding possible pressure instabilities due to slight differences between the recondensation chamber (where the heater is installed) and the pressure measurement section of the LHe dewar (both connected by the vacuum-insulated transfer line), adjustments in the control characteristics maintained a stable pressure at the set value of 0.4 kPa, thereby confirming the feasibility of long-term zero-boil-off operation.

3.3 Spectrum evaluation

While the spectral data depend on the installation environment and parameter settings of the NMR system, Figure 6 shows example spectral data for a standard sample ($CHCl_3$). The spectrum was accumulated 16 times. In the acquired spectrum, aside from the central main peak, two short peaks (carbon satellites) were symmetrically observed at both ends. It is known that the carbon satellites typically exhibit an intensity of 0.55% relative to the main peak, and this served as a baseline for evaluating the noise introduced by the operation of the helium recycling system. Noise tended to appear on both sides of the main peak; although it was pronounced when fewer accumulations were performed, the noise reduced to below approximately 0.1% of the main peak level when accumulated 16 times.

4. Conclusion

A helium recycling system suitable for integration with NMR systems was developed and commercialized in collaboration with Bruker Japan. The evaluations confirmed that the system meets the practical requirements in terms of pressure and liquid level stability within the NMR system, as well as minimal impact on spectral quality. While the system has received high evaluations from NMR users, issues related to running costs have been identified. To address the primary cost driver—namely, the electricity expense—we are investigating the optimization of compressor selection to reduce power consumption while maintaining the necessary recondensation capacity. If successful, it is expected that power consumption could be reduced to approximately two-thirds of its current level.

Future efforts will focus on the development of a low-power model and further noise reduction measures, thereby contributing to the efficient utilization of helium resources and energy conservation through broader adoption of this technology.