Development of 1.5W Cryostat for a Cold Trap Application

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ABSTRACT

A laboratory cryostat for purifying gases before their analysis was developed for the Japan Agency for Marine-Earth Science and Technology (JAMSTEC) based on an industrialized version of the SRI475 cryocooler. At room temperature and 77K, SRI475 cryocooler has cooling power of about 1.5W. This type of Stirling rotary cryocooler was selected as a compact solution because a lot of cooling power was needed. A need for cooling power was significant because there was a substantial thermal mass to be cooled during a cool-down phase because the cryostat has a removable cold trap mounted on the cold tip. In addition, the cryostat uses a low active vacuum to reduce convective losses. The interface between the cold tip and the cold trap was made of nickel-plated OFHC copper. Because it is a very soft material, additional threaded parts were brazed on to it for the mounting of the cold trap. To compensate for possible displacement of the cold tip because of mounting forces and moments, the internal clearance of the displacer piston towards the cold finger wall had to be increased. Nevertheless, after the integration of the cryocooler with the cold trap, measurements showed very good results with performance of the cryostat within expected range.

INTRODUCTION

The paper presents some challenges and solutions during development of a laboratory cryostat used for purifying CO_2 gas during isotopic analysis by mass spectrometry (Sakai and Matsuda, [1]). Initial requirements were that the cryostat operates under a low active vacuum, has enough cooling power to reach the working temperature of the cold trap in 10 minutes and a cold trap can be dismantled from it.

CRYOSTAT

The cryostat consists of a vacuum vessel, a cold trap within the vessel and a cryocooler enabling condensation of impurities inside a cold trap. A development started with estimation of heat losses that had to be covered by a cryocooler. Since our experience is mostly in high-vacuum applications, convective heat losses in low vacuum were hard to estimate. On the other hand, we decided to omit radiation shields as target temperature was relatively high and it would be hard to make a robust shield that would withstand the multiple assembly and disassembly cycles which were expected with a removable cold trap set-up. Based on this, an integral Stirling rotary cryocooler SRI475 (Figure 1 and 2) was selected for integration with a cold trap.

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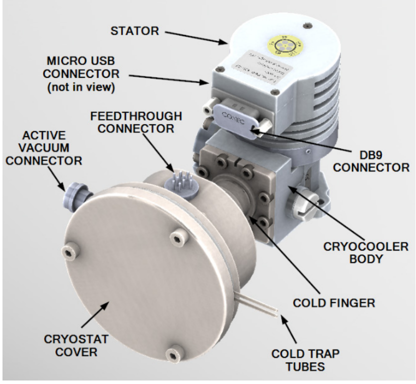

Figure 1. Cryostat with SRI475 cryocooler.

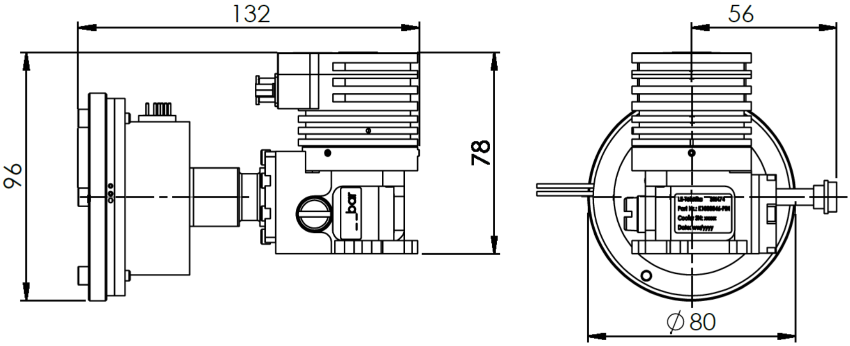

Figure 2. Cryostat dimensions (in millimeters).

To make the cryostat compact, the cold trap was mounted directly on the cold tip of the cold finger (Figure 3 and 4). Because of this, steel tubes outside the cold trap were also bent to make connection less rigid as any misalignment during assembly would result in mechanical load on the cold finger, potentially causing loss of performance and reduced lifetime.

Cold trap

The cold trap consists of a stainless-steel tube through which CO_2 gas is led. The tube is encapsulated in aluminum plate (Figure 5) which is used to fix it on a cold tip of the cryocooler. Part of

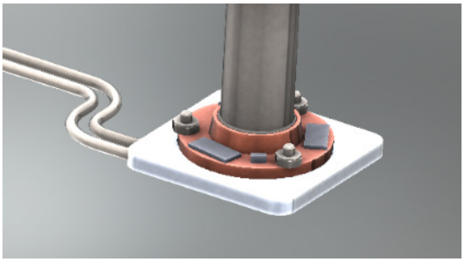

Figure 3. Cold finger with cold trap, heaters and temperature sensors (bottom view of 3D model, wires not shown).

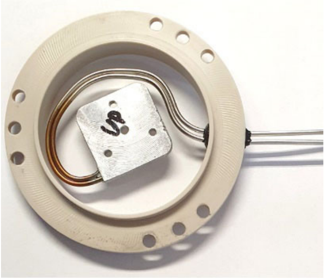

Figure 4. Cold trap integrated in a seal holder.

Figure 5. X-ray image of the cold trap before integration into the cryostat.

the tube encapsulated in aluminum has multiple bends to increase the surface area for heat transfer between gas and tube wall.

SRI475 CRYOCOOLER

SRI475 is currently the most powerful Stirling Rotary Integral cryocooler in the LE-TEHNIKA portfolio. It is operated at 24V DC and can deliver in standard version more than 1.5W of cooling power at 77K with a reject temperature of room ambient temperature (Figure 6). In high-power variant, it can deliver around 1.8W under the same conditions. Its mass without a cold finger or a dewar is around 500g.

A digital electronics module integrated into cryocooler is responsible for drive and control of DC brushless motor. The cooler can operate in two modes. The first one is with fixed motor speed and the second is with a fixed target temperature. The first mode is used in the initial operating phase when the motor operates at maximum speed and the cooler provides the highest cooling power to cool down the cryostat to its working temperature as fast as possible. The second mode is used when operating temperature is reached. This temperature is set as a target temperature for a PID controller which regulates the speed of the motor in a way that the temperature is kept stable within desired limits.

For this purpose, temperature sensors are mounted on a cold tip. They are mounted on the opposite side of a cold finger cover to which the cold trap is mounted (Figure 3). The cover is made from OFHC copper to minimize temperature difference and is fastened to the trap with several screws. In addition, a contact between the trap and the cover is filled with a thermal grease so that its thermal resistance is minimized.

Since OFHC copper is soft and has low yield strength it was necessary to reinforce the cold tip in the area where the cold trap is mounted. This was realized by brazing steel on it, in the next step, threaded holes for M2 fastening screws were made. In addition, the cold tip was equipped with temperature sensors and resistive heaters. Heaters are intended to shorten waiting time before the cold trap can be disassembled from the cold tip after operation and to prevent excessive condensation.

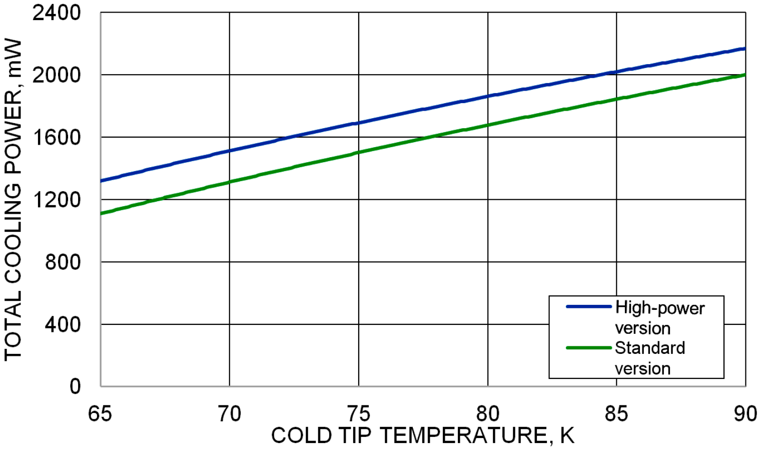

Figure 6. SRI475 cooling power at room temperature.

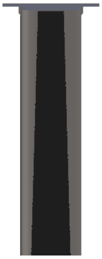

Figure 7. Cross-section of the cold finger showing part of conical displacer piston (exaggerated).

To improve the cryostat sensitivity to the deflection of the cold finger which can occur because of misalignment of the cold trap on the cold finger or through positioning of a sealing ring with steel tubes on a vacuum vessel and finally even because of thermal dilatations, a displacer piston with a regenerator was made conical. In this way, spacing between the cold finger wall and the piston increased towards the cold tip in a similar manner as deflection. Conical shape (Figure 7) was achieved by reducing wall thickness of the regenerator container while the internal matrix of the regenerator was kept the same. In this way thermal conductivity along the wall was reduced towards the tip and in the same way also losses because of shuttle heat transfer while dead volume increased by about 2%.

MEASUREMENTS AND RESULTS

Heat losses were measured at 77K/room temperature with liquid nitrogen based on the volume flow rate of nitrogen evaporating from the cold finger (Table 1 and Table 2).

Additional losses because of cold trap tubes are in range of 70-80mW and are low compared to the heat losses of a cryostat itself (Table 3).

Results show that heat losses increase significantly (more than 3x) when low vacuum is used instead of high vacuum (Figure 8).

Table 1. Heat losses under high vacuum for a cryostat without a cold trap.

Vacuum [mbar]	Heat losses [mW]
1,4e-5	258
1,9e-5	262
3,0e-5	281

Table 2. Heat losses under high vacuum for a cryostat with a cold trap.

Vacuum [mbar]	Heat losses [mW]
1,4e-5	320
1,6e-5	320
1,9e-5	343

Table 3. Heat losses under low vacuum for a cryostat with a cold trap.

Vacuum [mbar]	Heat losses [mW]
1,2e-2	1059
4,6e0	1097

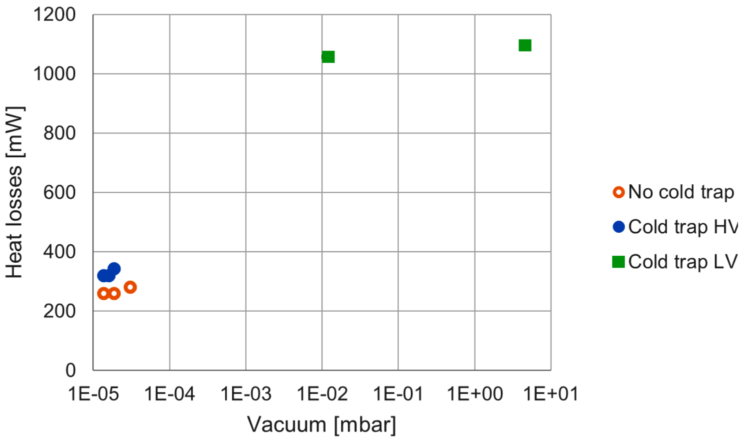

Figure 8. Heat losses

CONCLUSIONS

A very compact cryostat was made around SRI475 cryocooler. Compact size made it ideal for laboratory work where ample cooling power of SRI475 cooler ensured high availability of purifying system for spectroscopy measurements and stable operation under various vacuum levels. Results also show that heat losses are well within cooling capability of SRI475 cooler.

REFERENCES

1. Sakai, S., Matsuda, S., “A Practical Cryogen-Free CO2 Purification and Freezing Technique for Stable Isotope Analysis,” *Analytical Chemistry*, Vol. 89, No. 8 (2017), pp. 4409-4412.