

Closed-Cycle Joule–Thomson Cryocooler for Resistance Thermometer Calibration down to 0.65 K

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Abstract A closed-cycle Joule–Thomson cryocooler for resistance thermometer calibration has been developed. It consists of a Gifford–McMahon mechanical refrigerator and a closed-cycle ^3He Joule–Thomson expansion circuit that utilizes the isenthalpic expansion of ^3He for cooling. The developed cryocooler can reach temperatures as low as 0.6 K and can operate for months with a simple procedure. The typical cooling power of the cryocooler is 1 mW at 0.65 K with a molar flow rate of $160 \mu\text{mol} \cdot \text{s}^{-1}$ through the ^3He Joule–Thomson circuit. The possible mechanical vibration level experienced by the resistance thermometers was measured with a laser vibrometer. It was confirmed that the maximum acceleration level is $0.1 \text{ m} \cdot \text{s}^{-2}$ and will not cause a problem for thermometer calibration.

Keywords Calibration · Cryocooler · Helium 3 · Joule–Thomson effect · Liquid helium free · Mechanical refrigerator · Resistance thermometer · Vibration measurement

1 Introduction

Liquid-helium-filled cryostats are common apparatus for the calibration of thermometers in the cryogenic temperature range. However, they generally require cumbersome handling procedures, particularly the need for periodic refilling of the cryostats with liquid helium. The refilling can also influence measurements because it changes the temperature distribution inside the cryostat abruptly, and this disturbs the precise

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electrical and thermal measurements. In this case, the measurements must be discontinued until the temperature distribution inside the cryostat approaches equilibrium.

Since a mechanical refrigerator can reach the cryogenic temperature range with a relatively simple operation, it is becoming more commonly used in various cryogenic experiments than the liquid-helium-filled cryostat. Several mechanical refrigerators have been successfully used for precise thermometer calibration [1–5].

On the other hand, the achievable minimum temperature of the mechanical refrigerator is around 3 K, even with the latest models in the market. This is insufficient for the calibration of thermometers down to 0.65 K, the lower limit of the International Temperature Scale of 1990, ITS-90 [6].

In this study, a closed-cycle Joule–Thomson cryocooler that uses ^3He as the working fluid for the Joule–Thomson circuit is developed for liquid-helium-free cryogenic thermometer calibration down to 0.65 K. The developed cryocooler can reach temperatures as low as 0.6 K and can operate continuously for months with a simple procedure. The construction and basic cooling characteristics of the cryocooler are presented here.

The mechanical refrigerator conveniently provides a cryogenic measurement environment. However, its intrinsic mechanical vibration due to the cyclically moving displacer and cyclic pressure variation in the refrigerator can be a matter of concern in its application to mechanically sensitive instruments such as standard resistance thermometers. The mechanical vibration level of our cryocooler is also presented.

2 Cooling Characteristics of the Cryocooler

2.1 Cooling Principle and Construction

The closed-cycle Joule–Thomson cryocooler developed for thermometer calibration down to 0.65 K consists of a Gifford–McMahon (GM) mechanical refrigerator and a closed-cycle Joule–Thomson (JT) circuit that utilizes the isenthalpic expansion of the working fluid ^3He for cooling. The cryocooler is based on a Gifford–McMahon/ ^4He -Joule–Thomson cryocooler (GM/ ^4He -JT) that uses ^4He as the working fluid of the JT circuit. Details of the construction [7] and thermodynamic cycle analysis [8] of the GM/ ^4He -JT cryocooler have been presented elsewhere. Since the vapor pressure of ^4He is very low below 1 K, cooling below 1 K with a GM/ ^4He -JT cryocooler is not practical. To lower the achievable minimum temperature, ^3He is used as the working fluid of the JT circuit because it has the highest vapor pressure among all substances in this temperature range.

Figure 1 shows schematically the cryocooler. A vacuum chamber of 420 mm diameter and 1,200 mm length encloses all the cold parts of the cryocooler. A two-stage GM refrigerator is used for the system. The cooling powers of the first and second stages of the refrigerator are 4 W at 50 K and 1.6 W at 10 K, respectively. The minimum temperature of 5.8 K is obtained at the second stage with no heat load. The first and second stages of the refrigerator cool the first and second radiation shields, respectively. The first stage of the refrigerator and the first radiation shield are thermally connected by flexible copper braids. The second stage of the refrigerator and the second radiation

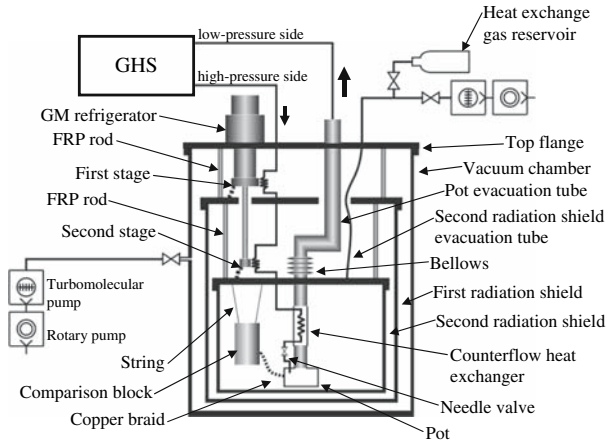


Fig. 1 Schematic of the closed-cycle ^3He Joule–Thomson cryocooler

shield are thermally connected as well. The first radiation shield, which is made of copper and aluminum, is 333 mm in diameter, 730 mm in height, and 11 kg in mass. It is suspended by four fiber-reinforced plastic (FRP) rods from the top of the vacuum chamber. The second radiation shield is made of copper and is a vacuum-tight chamber. The vacuum-tight second radiation shield enables the introduction of a heat exchange gas during the initial cooling stage. It is 192 mm in diameter, 342 mm in height, and 10 kg in mass. It is suspended by four FRP rods from the top of the first radiation shield. The cryocooler is designed to have a relatively large experimental space to ensure flexibility in the experimental configuration.

The GM refrigerator is also used as a pre-cooler for the JT circuit through which ^3He circulates. The JT circuit utilizes the isenthalpic expansion (Joule–Thomson effect) of the ^3He for refrigeration. A gas handling system (GHS) set outside the vacuum chamber consists of an oil rotary pump, a compressor, a reservoir tank for ^3He , and a liquid-nitrogen cold trap for purification of the circulating ^3He . The GHS provides pressurized ^3He gas at room temperature to the JT circuit. The ^3He enters the chamber through the high-pressure side of the JT circuit and is cooled to approximately 8 K by the heat exchangers attached to the first and second stages of the refrigerator. The cooled ^3He is further cooled by the counterflow heat exchanger and then expands when it flows through the needle valve. The impedance of the NMIJ-fabricated needle valve can be varied by operating the handle (not shown in the figure for simplicity) on the top flange of the vacuum chamber. Due to the Joule–Thomson effect, the temperature of the pre-cooled ^3He decreases as it expands, and part of it condenses into a pot under appropriate conditions. The pot is pumped by a vacuum pump in the GHS through an evacuation tube, which corresponds to the low-pressure side of the JT circuit. The temperature of the pot decreases as the pumping lowers the vapor pressure of the condensed ^3He in the pot. The bellows on the top of the second radiation shield reduce the heat load and mechanical vibration from the top of the vacuum chamber to the second radiation shield. The minimum temperature of 0.6 K is attained at the pot.

2.2 Operation of the Cryocooler

Only the GM refrigerator is used to cool the cryocooler from room temperature to the cryogenic range in the initial stage of cooling. A small amount of ^4He exchange gas is introduced within the second radiation shield to enhance thermal contact between the parts inside the second radiation shield and the refrigerator. The pot usually takes approximately 60 h to reach $\sim 8\text{ K}$, which is the minimum temperature of the initial stage of cooling. The ^4He exchange gas is evacuated from inside the second radiation shield after completion of the initial cooling. Then, the circulation of the ^3He through the JT circuit is started and the minimum temperature of 0.6 K at the pot can be achieved through this ^3He circulation.

The temperature of the pot can be controlled by several parameters, for example, the supply pressure of ^3He from the GHS, the impedance setting of the needle valve, and the pumping speed. Since the JT circuit of the cryocooler is a closed circuit, these parameters are not completely independent. Practically, the temperature of the pot is coarsely controlled by the supply pressure of ^3He and the impedance setting of the needle valve. Then, the temperature of the pot is finely controlled by the pumping speed. In addition, the temperature can be finely controlled with a heater wound around the pot.

Figure 2a shows an example of the temperature control results at the pot by regulating the pumping speed. The temperature setting of the pot is changed in a stepwise manner from 0.6 K to 1.7 K in this figure. Figure 2b shows the detail of one of the steps near 1 K. Figure 3a shows an example of the temperature control results at the pot with a heater when the temperature setting is changed from 13 K to 14 K. Figure 3b shows the detail of one of the steps at 13 K. The temperature measurement resolutions of the temperature controller used here are 0.1 mK below 10 K and 1 mK above 10 K. The cooling power of the cryocooler at the pot is measured by changing the amount of power introduced by a heater wound around the pot. Figure 4 shows typical results measured near the minimum temperature. The figure also shows the corresponding molar flow rate of ^3He . A cooling power of approximately 1 mW at 0.65 K with a molar flow rate of $160\ \mu\text{mol}\cdot\text{s}^{-1}$ is obtained.

3 Vibration Characteristics of Cryocooler

3.1 Mechanical Vibration of Cryocooler and Measurement Apparatus

The GM refrigerator is a convenient apparatus for cooling objects down to cryogenic temperature without using cumbersome cryogens such as liquid helium. However, the refrigerator exhibits an intrinsic mechanical vibration [9] and has a potential risk of disturbing precise measurements. Since capsule-type standard rhodium-iron resistance thermometers (CSRIRTs) that are sensitive to mechanical vibration are used for high-precision thermometry at low temperatures, special attention should be paid to the vibration level of the CSRIRTs mounted on the cryocooler.

A comparison block made of copper is installed in the cryocooler to hold several CSRIRTs for calibration. The comparison block is 30 mm in diameter, 70 mm in height,

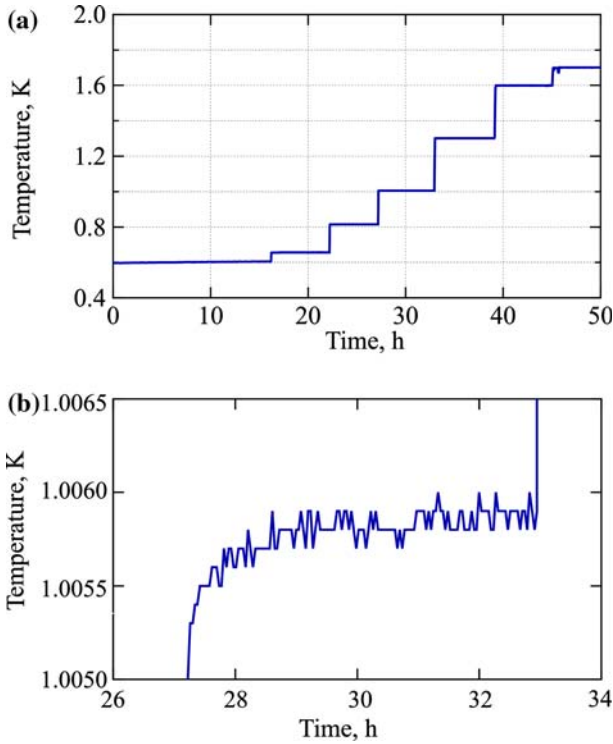


Fig. 2 (a) Temperature of the pot is controlled by regulating the pumping speed; temperature setting of the pot is changed in a stepwise manner approximately from 0.6 K to 1.7 K and (b) temperature control result at the pot near 1 K

and 0.48 kg in mass and is suspended by four strings from the top of the second radiation shield. Flexible copper braids connect the comparison block and the pot to establish thermal contact between them. The structural coupling between the comparison block and the refrigerator should be weakened because of the suspending configuration.

The velocity amplitude v of the vibration at the pot and the block at room temperature are measured with two laser vibrometers from two directions from 1 Hz to 1 kHz by laser interferometry. Since they measure the vibration with laser beams scattered by the measuring object, they can measure the vibration without physically touching or attaching anything to the object. The acceleration amplitude a of the vibration is given by

$$a = 2\pi f v$$

The vibration at the pot is measured simultaneously from two directions with the two vibrometers. One is used to measure the vibration along the vertical direction, and the other is used to measure it along the horizontal direction. The vibration at the block is measured in the same manner in a different measurement run.

The vibration measurements are performed within 20 min after the GM refrigerator is turned on while the vacuum chamber and two radiation shields are opened. Note

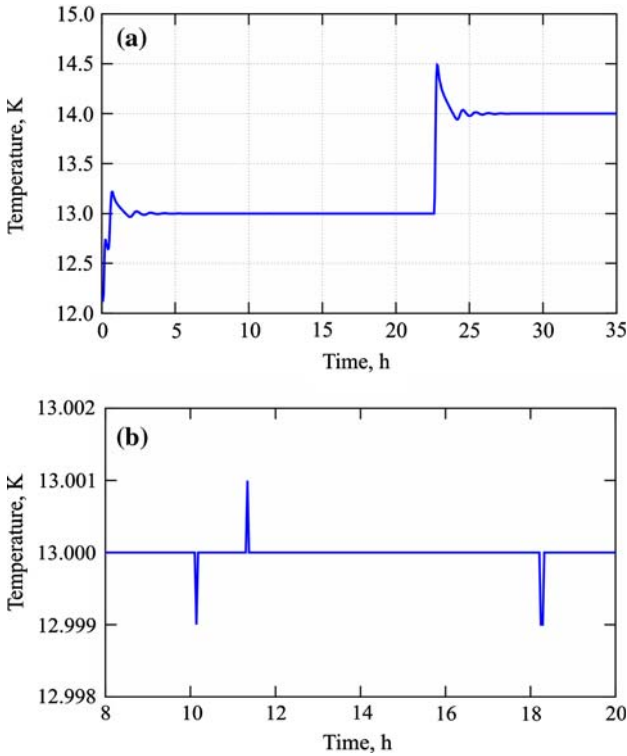


Fig. 3 (a) Temperature of the pot is controlled with a heater wound around the pot; temperature setting is changed from 13 K to 14 K and (b) temperature control result at 13 K

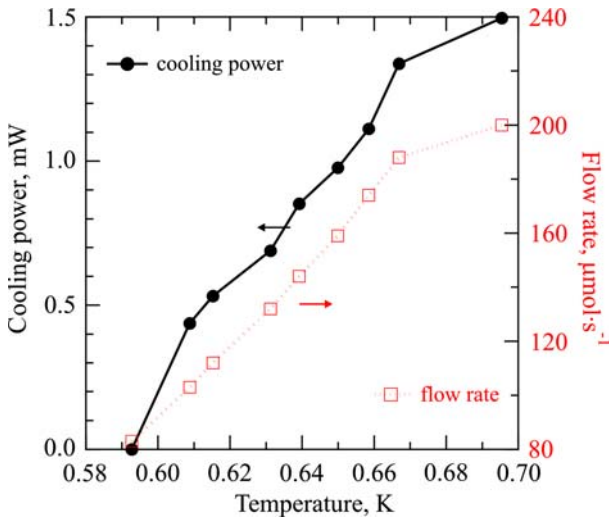


Fig. 4 Cooling power at the pot near the minimum temperature. Corresponding molar flow rate of ^3He is also shown

that these measurement conditions may lead to the measurement of a vibration level higher than that obtained under normal operation of the cryocooler. When the vacuum chamber and radiation shields are installed for normal operation, the large mass of the chamber and two shields is expected to provide an additional damping effect on the vibration [2].

3.2 Measurement Results of Vibration

Figure 5a, b shows the acceleration amplitude spectra of the vertical vibration of the pot and the block, respectively. Thin lines represent the spectra obtained with the GM refrigerator turned on. Thick lines represent the background noise spectra obtained with the refrigerator turned off. The driving frequency of the refrigerator is 2 Hz. Peaks at 2 Hz and its higher harmonic frequencies are easily distinguished in the vertical vibration spectra of the pot as shown in Fig. 5a. Acceleration amplitudes of approximately $0.01 \text{ m} \cdot \text{s}^{-2}$ to $0.1 \text{ m} \cdot \text{s}^{-2}$ are observed up to 700 Hz. The vertical acceleration

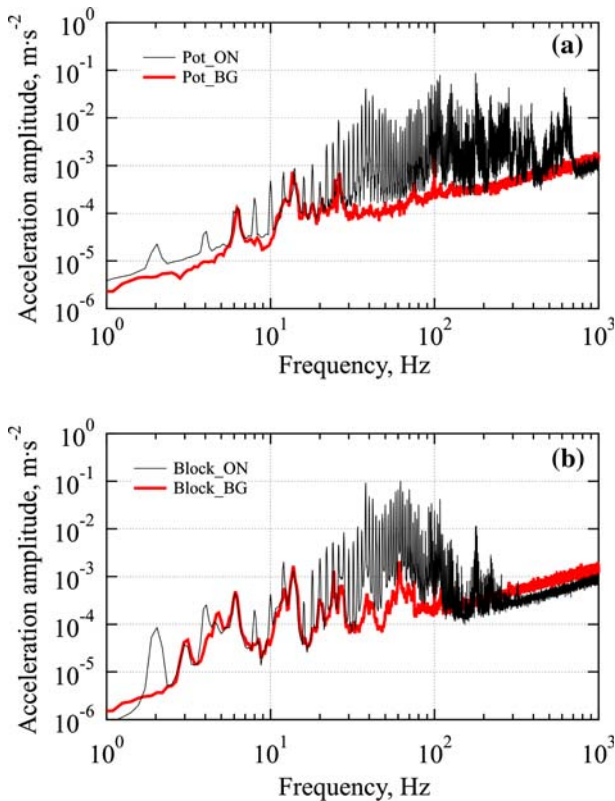


Fig. 5 Acceleration amplitude spectra of the vertical vibration of (a) the pot and (b) the block. Thin lines represent the spectra obtained with the refrigerator turned on. Thick lines represent the background noise spectra obtained with the refrigerator turned off

amplitude at the block (Fig. 5b) shows almost the same maximum acceleration of $0.1 \text{ m} \cdot \text{s}^{-2}$. Note that the acceleration amplitude at the block above approximately 100 Hz is suppressed in comparison with that at the pot. On the other hand, the acceleration amplitude at the block below 100 Hz is slightly higher than that at the pot. This may be related to the resonances of the suspension configuration and of the flexible copper braids.

Figure 6a, b shows the acceleration amplitude spectra of the horizontal vibration of the pot and the block, respectively. Generally speaking, the horizontal acceleration amplitude at the block is lower than that at the pot. The maximum horizontal acceleration amplitude at the block is $0.03 \text{ m} \cdot \text{s}^{-2}$.

It has been reported that no changes larger than 0.5 ppm in the resistance at the triple point of water are observed for a capsule-type standard platinum resistance thermometer (CSPRT) that is subjected to a vibration of, at most, $0.5 \text{ m} \cdot \text{s}^{-2}$ for 2 years [1]. Another report indicates that a CSPRT may survive an acceleration level as high as $30 \text{ m} \cdot \text{s}^{-2}$ at approximately 600 Hz [2]. The acceleration level of a fixed-point device is reported to be $0.5 \text{ m} \cdot \text{s}^{-2}$ [4], and in this case, the device holding several CSPRTs

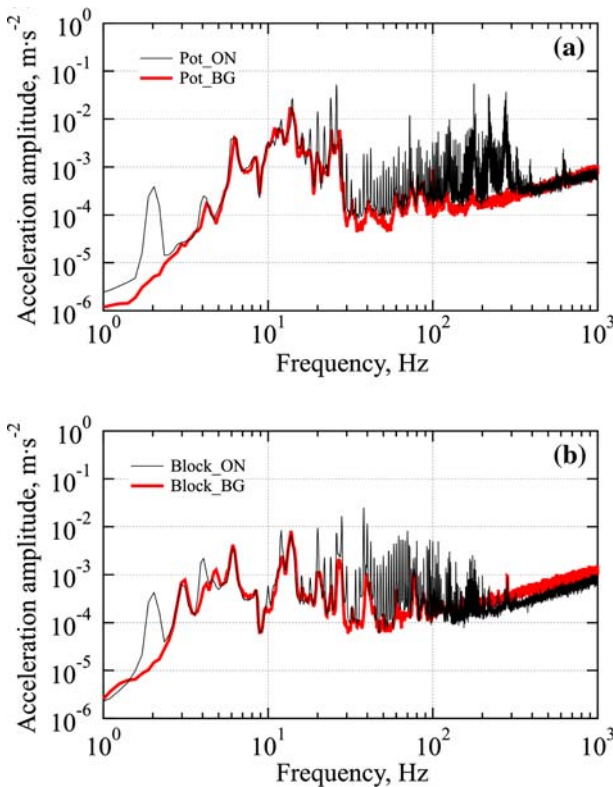


Fig. 6 Acceleration amplitude spectra of the horizontal vibration of (a) the pot and (b) the block. Thin lines represent the spectra obtained with the refrigerator turned on. Thick lines represent the background noise spectra obtained with the refrigerator turned off

for calibration is suspended from the adiabatic shield attached to the second stage of a GM refrigerator. The measured acceleration amplitude at the comparison block in the present study is a factor of 5–100 times smaller than these values. It seems reasonable to assume that such a low-acceleration level will not disturb the CSRIRT because it is similar in construction and has a harder sensing element compared to the CSPRT.

4 Conclusion

A liquid-helium-free cryocooler that can reach temperatures as low as 0.6 K was developed for thermometer calibration. It was demonstrated that the developed cryocooler has good temperature control stability and sufficient cooling power for thermometer calibration. The acceleration level of the mechanical vibration of the cryocooler was measured at the block on which the thermometers will be mounted. It was confirmed that the vibration level is sufficiently low for thermometer calibration. The developed cryocooler can be used for not only thermometer calibration but also for other measurements and with devices that require low-temperature cooling for long duration with low vibration levels.

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References

1. H. Sakurai, O. Tamura, in *Pre-prints of TEMPMEKO 90 - 4th. International Symposium on Temperature and Thermal Measurement in Industry and Science*, ed. by H.K. Graubner (Finnish Society of Automatic Control, Helsinki, 1990), pp. 112–117
2. A.G. Steele, in *Proceedings of International Seminar on Low Temperature Thermometry and Dynamic Temperature Measurement*, ed. by A. Szymrka-Grzebyk (DRUK-Zakłady Poligraficzne FP KGHM, Wrocław, 1997), pp. L48–L53
3. K.D. Hill, A.G. Steele, in *Temperature: Its Measurement and Control in Science and Industry*, vol. 7, Part 1, ed. by D.C. Ripple (AIP, New York, 2003), pp. 53–58
4. D. Ferri, D. Ichim, F. Pavese, I. Peroni, F. Sparasci, P.P.M. Steur, in *Proceedings of TEMPMEKO 2004, 9th International Symposium on Temperature and Thermal Measurements in Industry and Science*, ed. by D. Zvizdić, L.G. Bermanec, T. Veliki, T. Stašić (FSB/LPM, Zagreb, Croatia, 2004), pp. 165–170
5. T. Nakano, O. Tamura, H. Sakurai, in *Temperature: Its Measurement and Control in Science and Industry*, vol. 7, Part 1, ed. by D.C. Ripple (AIP, New York, 2003), pp. 185–190
6. H. Preston-Thomas, *Metrologia* **27**, 3, 107 (1990)
7. T. Shimazaki, K. Toyoda, O. Tamura, *Rev. Sci. Instrum.* **77**, 034902 (2006)
8. T. Shimazaki, K. Toyoda, O. Tamura, in *Proceedings of the Twenty First International Cryogenic Engineering Conference (ICEC 21)*, vol. 1, ed. by G.G. Bagnier, R.S. Safrata, V. Chrz (Icaris Ltd., Conference Management, Praha, 2007), pp. 629–632
9. T. Tomaru, T. Suzuki, T. Haruyama, T. Shintomi, A. Yamamoto, T. Koyama, R. Li, *Cryogenics* **44**, 309 (2004)