

Performance Analysis of Pulse Tube/³He Joule–Thomson Cryocooler for Thermometer Calibration

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ABSTRACT

Joule–Thomson (JT) circuits enhance the cooling capability of mechanical refrigerators. A pulse tube/³He JT cryocooler for precise thermometer calibration between 0.65 K and 25 K is described in this paper. It comprises a commercial 4 K two-stage pulse tube refrigerator and a JT circuit, which is designed and built at the National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology.

Owing to the nature of thermometer calibration, the cryocooler requires a wide operational temperature range with a high temperature control stability rather than high cooling power. The cryocooler for the thermometer calibration uses ³He as the working fluid for the JT circuit to reduce the minimum temperature to 0.5 K. This cryocooler utilizes a needle valve that can vary the flow impedance for JT expansion. An oil rotary pump, a mechanical booster pump, and a compressor were used for ³He circulation. Using this, the temperature control stability whose standard deviation is less than 0.07 mK can be obtained. The temperature instability observed near 2 K due to the two-phase flow in the JT circuit is also discussed.

INTRODUCTION

Accurate thermometry is indispensable in many experiments. In principle, the temperature can be measured either using a primary thermometer, which can measure the thermodynamic temperature without calibration or a secondary thermometer which approximates the thermodynamic temperature but requires calibration for the temperature measurement. The primary thermometer performs thermometry based on a well-understood physical system such as a constant volume gas thermometer and a Johnson noise thermometer.¹ The constant volume gas thermometer relies on the relationship between the properties of an ideal gas and thermodynamic temperature. The Johnson noise thermometer relies on the relationship between the thermally induced voltage fluctuations which appear across an electrical resistor and thermodynamic temperature. However, precision primary thermometry usually requires a large and complex measurement system and long measurement time. Applicable temperature ranges for the said methods are limited. The range depends on their physical principles and apparatus settings. These drawbacks make primary thermometry impractical for most experiments except for some experiments relate to temperature standard work. Although secondary thermometers such as

resistance thermometers require calibration, they are compact, stable, easy-to-handle, and widely used as practical thermometry devices for many experiments.¹

The International Temperature Scale of 1990 (ITS-90) consists of defining temperature fixed points and interpolation instruments that provide approximated thermodynamic temperature between fixed points.² ITS-90 defines the temperature scale from 0.65 K to the highest temperature practically measurable in terms of the Planck radiation law using monochromatic radiation. The ITS-90 is designed to be the reference temperature scale for the calibration of secondary thermometers with highest accuracy.

Our institute realized, maintained, and disseminated the abovementioned temperature scale.³ For the cryogenic temperature standard work, a cryocooler that can control the temperature of a thermometer comparison block between 0.65 K and 25 K with sub-millikelvin stability is needed. We have been developing JT cryocoolers for cryogenic thermometer calibration for more than ten years.³⁻⁶ The latest version of the cryocooler consists of a 4 K two-stage pulse tube refrigerator and a Joule–Thomson (JT) circuit. The detailed construction of the JT cryocooler and cooling characteristics, including the observed temperature instability are presented in this paper.

The cryocoolers for cryogenic detectors and instruments are usually designed to be operated at a specific target temperature with sufficient cooling power that are determined by the detectors and instruments.⁷⁻¹⁰ On the other hand, the cryocoolers for the cryogenic temperature standard work are required to be operated at relatively wide temperature range and high temperature control stability. The requirement for the cooling power is usually not very high.

APPARATUS

Cryocooler design

The cryocooler for the thermometer calibration consists of a commercial 4 K two-stage pulse tube refrigerator (SHI SPR-052A) and a JT circuit developed at National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology. Figure 1 shows the exterior view of the cryocooler. Figure 2 shows a schematic view of the cryocooler. The diameter of the vacuum chamber is 416 mm, and the height of the chamber is 940 mm. The cooling power of the first and second stages of the pulse tube cryocooler is 10 W at 45 K and 0.5W at 4.2 K, respectively. The first stage of the pulse tube refrigerator is thermally connected to the first radiation shield with a copper thermal link and cools the shield. The second stage of the pulse tube refrigerator cools the second radiation shield. The second radiation shield is designed to be an inner vacuum-tight chamber. A heat exchange gas can be introduced through a tube (not shown in Figure 2), which connects the second radiation shield and a heat exchange gas reservoir set outside the vacuum chamber. The heat exchange gas is evacuated by a turbo molecular pumping system at an appropriate timing that is just before the completion of the precooling. The heat exchange gas accelerates the precooling of the components inside the shield from room temperature to cryogenic temperature, which typically takes 30 h.

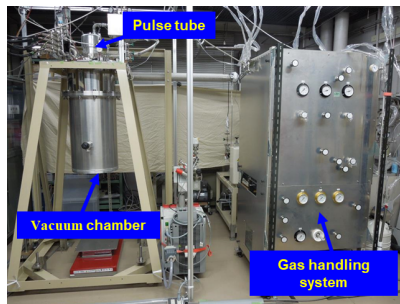


Figure 1. Exterior view of the pulse tube / ^3He JT cryocooler for thermometer calibration.

The JT circuit uses ³He as the working gas. The gas handling system (GHS) supplies room temperature ³He to the high-pressure side of the JT circuit and evacuates the ³He pot through the low-pressure side of the circuit. The details of the GHS are also presented in a separate section. The room temperature ³He supplied to the high-pressure side of the circuit is cooled at the first and second stages of the pulse tube refrigerator when it flows through the copper tubes coiled around the copper blocks attached to the first and second stages. The ³He is further cooled by the counterflow heat exchanger in the second radiation shield. This JT circuit has only one counterflow heat exchanger. This configuration helps to suppress the pressure drop across the low-pressure side of the JT circuit from the ³He pot to the GHS set at room temperature to achieve low vapor pressure at the ³He pot. The drawback of the single counterflow heat exchanger configuration is that precooling of the ³He before the JT expansion takes place only immaturely and a reduction in the cooling power of the JT circuit. Because the required cooling power to the JT circuit for thermometer calibrations is relatively small (order of mW), the reduction in the cooling power is acceptable.

The ³He precooled by the counterflow heat exchanger expands as it flows through a needle valve. This is an isenthalpic expansion. The impedance of the needle valve can be varied by operating a handle set on the top flange of the vacuum chamber. The pressure drop across the needle valve can be varied by changing the impedance of the needle valve. This means that the temperature at the ³He pot can be coarsely set by changing the intensity of the JT expansion effect. Part of the expanded ³He condenses into the ³He pot as liquid under appropriate conditions. The vacuum pumps in the GHS evacuate the ³He pot through the low-pressure side of the counter heat exchanger. The evacuated ³He circulated the JT circuit. The temperatures at the first and second stages of the pulse tube refrigerator in normal operation with the JT circuit operated are approximately 43 K and 3.1 K, respectively.

Counterflow heat exchanger

The construction of the counterflow heat exchanger is a coiled capillary in a tube type. This type of counterflow heat exchanges has been successfully applied for dilution refrigerators.¹¹⁻¹³ The capillary in the counterflow heat exchanger for the present JT cryocooler is coiled in a twisted spiral shape.⁴ High-pressure gas flows in the twisted spiral copper-nickel capillary of 0.3 mm inner diameter, 0.5 mm outer diameter, and 4 m length. The capillary of 4 m can be

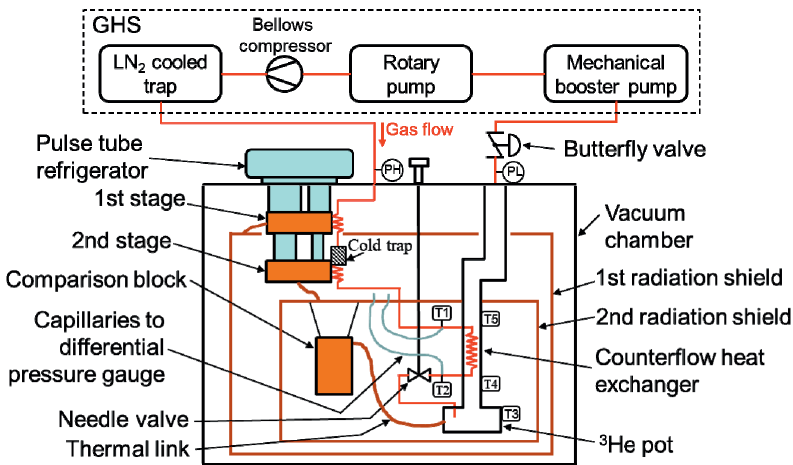


Figure 2. Schematic representation of the pulse tube / ³He JT cryocooler for thermometer calibration.

coiled approximately 100 mm in length and is inserted in a stainless-steel outer tube of 14.4 mm inner diameter, 15 mm outer diameter, and 200 mm length. The low-pressure gas flows in the space between the twisted spiral capillary and the outer tube. For confirmation purposes, the pressure drop across the low-pressure side of the counter flow exchanger is measured before the installation of the JT circuit. It is 0.3 Pa for 300 $\mu\text{mol/s}$ of ^4He flow rate at room temperature. The typical ^3He flow rate in actual operations ranges from 100 $\mu\text{mol/s}$ to 200 $\mu\text{mol/s}$.

The temperature distribution around the counterflow heat exchanger is measured using thermometers (Cernox temperature sensor calibrated by manufacture¹⁴) numbered from T1 to T5. T1 and T2 are inserted in the copper blocks soldered to the copper-nickel capillary. T3 is inserted in a hole drilled to the ^3He pot. T4 and T5 are inserted in the copper blocks clamped to the stainless outer tube. The estimated temperature distribution measurement uncertainties are better than 50 mK. The thermometer number corresponds to the location number on the JT circuit. Thermometer T1 is attached to the location 1, thermometer T2 attached to location 2, and so on. Location 1 is located before the inlet of the high-pressure side of the counterflow heat exchanger. Location 2 is located between the outlet of the high-pressure side of the heat exchanger and the needle valve. Location 3 corresponds the ^3He pot. Location 4 and 5 are located at inlet and outlet of the low-pressure side of the counterflow heat exchanger, respectively. The pressure on the high-pressure side of the JT circuit is measured with an absolute pressure gauge labeled PH, and that of the evacuated ^3He gas is measured with an absolute pressure gauge labeled PL. The pressure drop across the high-pressure side of the counterflow heat exchanger is measured with a differential pressure gauge. The estimated pressure measurement uncertainties are better than 5 % of readings.

Thermometer comparison block and thermometer calibration

A thermometer comparison block is suspended in the second radiation shield with four strings to improve thermal and mechanical isolation from the second radiation shield. The comparison block and the ^3He pot are thermally connected by a flexible copper thermal link. The comparison block is equipped with a resistive heater and thermometer for temperature control. The ^3He pot also equips a heater and a thermometer.

The comparison block made of oxygen-free high-conductivity copper has wells for thermometer insertion and can hold up to 10 capsule-type standard resistance thermometers. Capsule-type standard resistance thermometers are specifically designed for precise and high stability cryogenic thermometry of millikelvin order or better measurement uncertainty. They have been used for precision thermometry, such as temperature standard work for several decades worldwide. A coil of wire made of a dilute alloy of iron in rhodium¹⁵ (RhFe) or cobalt in platinum¹⁶ (PtCo) is used as a sensing element. Both thermometers are used for the present work. Figure 3 shows examples of commercial capsule-type standard resistance thermometers. The coiled sensing element is sealed in a platinum capsule with helium heat exchange gas below atmospheric pressure. The typical size of the capsule is 5 mm in diameter and 60 mm in length. Four platinum lead wires were attached to the sensing element for four-wire resistance

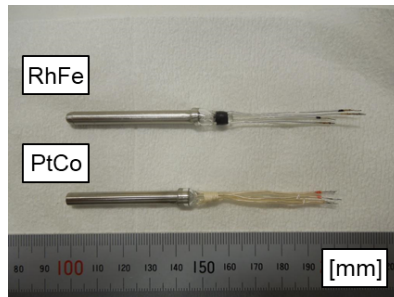


Figure 3. Two types of commercial capsule-type standard resistance thermometers

measurements. The present cryocooler is primarily designed for the calibration of this type of the thermometer.

In a thermometer calibration procedure, two to three capsule-type standard resistance thermometers which have already been calibrated are installed in the comparison block as reference and confirmation thermometers. These thermometers are used to determine and confirm the temperature of the comparison block. The thermometers to be calibrated are also installed in the same comparison block. First, the temperature of the comparison block is stabilized at a target calibration temperature, and then the resistances of the pre-calibrated and non-calibrated thermometers are measured consecutively. The resistance of the thermometers to be calibrated at the calibration temperature is then obtained. This measurement is repeated at all calibration temperatures.

The heat dissipation from a capsule-type standard resistance thermometer is only several μW or less. The heat dissipations from the resistive heaters attached to the comparison block and the ³He pot for temperature control are less than 0.1 mW in the temperature range at which the JT circuit is running. The cooling power requirement to the JT circuit is not very high for the thermometer evaluation and calibration. The cooling power of the JT circuit depends on the operating condition; it is typically measured to be 1 mW at 0.65 K. On the other hand, requirements for the control temperature stability and operational temperature range are relatively high.

Gas handling system

The GHS circulates ³He through the JT circuit. The GHS supplies room temperature ³He to the high-pressure side of the JT circuit, evacuates ³He from the ³He pot, purifies and, pressurizes the ³He, and then returns ³He to the high-pressure side of the JT circuit. A bellows compressor in the GHS compresses room temperature ³He. Typically, a pressure ratio of 3.6 can be obtained with this compressor. The JT circuit can be operated in various ways. The supply pressure of ³He ranges from approximately 40 kPa to 120 kPa and varies depending on the settings and working conditions.

For example, ³He of up to 120 kPa is temporarily supplied to the JT circuit for initial liquefaction. The supply pressure is reduced to a range of approximately 50 kPa in normal operation. Because the JT circuit is a closed circuit, the supply pressure cannot be set completely independent of other parameters. Practically, it is set by changing several controllable parameters such as openings of valves in the circuit, including the JT needle valve and the amount of circulating gas. The bellows compressor always operates at a constant frequency. It is not regarded as a controllable parameter in the system. A mechanical booster pump and an oil rotary vacuum pump evacuate the ³He pot. According to manufacture's specifications, the pumping speeds of the pumps are 505 m³/h and 35 m³/h, respectively. The evacuation speed on the ³He pot can be regulated with a butterfly valve, pressure gauge, and a proportional-integral-derivative (PID) controller. The pressure at the low-pressure side of the JT circuit typically ranges from 8 Pa to 15 kPa. A liquid-nitrogen cooled cold trap and a 25 l gas reservoir tank (not shown in Figure 2) are included in the GHS. The reservoir tank stores the ³He gas when the JT circuit is not in operation and is also used as a buffering volume to regulate the amount of the circulating gas. A cold trap cooled by the second stage of the pulse tube refrigerator is placed in the vacuum chamber.

Temperature control

The cryocooler is used for the thermometer calibrations between 0.65 K and 25 K. The thermometer comparison block needs to be controlled in this temperature range with sub-millikelvin stability. The temperature of the comparison block is set in two steps. First, the temperature of the ³He pot is controlled at a certain temperature, and then the temperature of the comparison block is set and controlled at several mK higher than that of the ³He pot.

Table 1. ^3He pot temperature control methods.

Calibration temperature	Cooling source	Control method
High(25 K–5.5 K)	Pulse tube refrigerator	Heater on ^3He pot
Middle(5.5 K–1.8 K)	Pulse tube refrigerator and JT	Heater on ^3He pot
Low(1.8 K–0.65 K)	Pulse tube refrigerator and JT	Butterfly valve (Vapor pressure in ^3He pot)

Table 1 shows the three methods employed for the ^3He pot temperature control depending on the calibration temperature. The first method is employed in the highest calibration temperature range from 25 K to 5.5 K. The ^3He pot is only cooled by the pulse tube refrigerator and the temperature is controlled with a heater attached to the ^3He pot. The current to the heater is fed by a commercial temperature controller for cryogenic thermometry and temperature control.

For a middle–temperature range from 5.5 K to 1.8 K, the JT circuit is operated in addition to the pulse tube refrigerator to cool the ^3He pot. In this temperature range except near 2 K, liquid ^3He does not appear in the ^3He pot. The heater attached to the ^3He pot is used for temperature control. It is found that, near 2 K, the temperature control turns relatively unstable. This temperature instability may be attributed to the intermittent appearance of the liquid phase at a certain point in the JT circuit. The data are shown in the next section.

For the lowest temperature range from 1.8 K to 0.65 K, the pulse tube refrigerator and JT circuit were used for cooling, but the butterfly valve is used for the ^3He pot temperature control. This is because liquid is stable in the ^3He pot in this temperature range.

On the other hand, the temperature control of the comparison block is straightforward. It is controlled with a heater attached to the comparison block for the entire temperature range. A bridge for precision thermometry is utilized for temperature control.¹⁷ This control method can result in sub–millikelvin temperature stability. The resistance of the thermometer set in the comparison block for temperature control is measured with the bridge, and then a signal related to the deviation from a pre–assigned resistance value that relates to a target calibration temperature can be obtained. The deviation signal is fed to a PID controller to drive the heater of the comparison block to eliminate the deviation.

PPERFORMANCE ANALYSIS

Temperature Control Stability

The temperature control stability at the comparison block measured with a capsule-type standard resistance thermometer is shown in Figure 4. The abscissa represents the comparison block temperature. The ordinate represents the standard deviation of the measured temperature variation at each comparison block temperature, which ranges from 0.65 K to 25 K for a

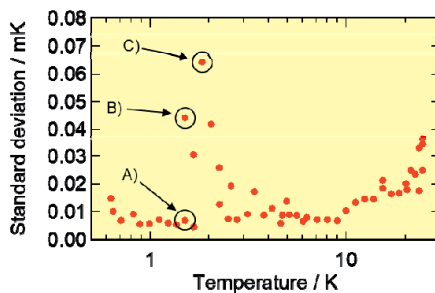


Figure 4. Temperature control stability at the thermometer comparison block between 0.65 K and 25 K. Data A) corresponds to the comparison block temperature at $T_{\text{Block}} = 1.50$ K. Data B) also corresponds to $T_{\text{Block}} = 1.50$ K but obtained with different flow setting from that of Data A). Data C) corresponds to $T_{\text{Block}} = 1.84$ K.

measurement duration of 15 min. Before measurement at each block temperature, the temperature of the comparison block is set at a target temperature and wait for stabilization for hours, typically it takes 8 to 10 h. The standard deviation varied depending on the block temperature. However, sub-millikelvin stability (less than 0.07 mK) is achieved. The stability is sufficient for the thermometer calibrations. However, notable instability is observed near 2 K. This or similar instabilities have been pointed out by researchers to relate to the two-phase flow in the JT circuit.^{3,9}

The data labeled A) corresponds to the comparison block temperature at $T_{\text{Block}} = 1.50$ K. Data B) also corresponds to $T_{\text{Block}} = 1.50$ K. Data C) corresponds to $T_{\text{Block}} = 1.84$ K. The difference in the measurement conditions between data A) and B) is the needle valve setting. The needle valve is set at a high impedance (4×10^{12} cm⁻³) position so that the pressure before the expansion sufficiently increased and liquid stably exists in the ³He pot for data A). On the other hand, the needle valve is set at a low impedance (7×10^9 cm⁻³) position for data B), and therefore, the pressure before the expansion decreased and the liquid does not stably exist in the ³He pot under this condition. For data C), the needle valve is set at the same low impedance position.

Enthalpy–temperature diagram

Figure 5 shows enthalpy–temperature (h - T) diagrams¹⁸ of the JT circuit for different temperature stability conditions. Estimated states of ³He at numbered locations in the JT circuit as indicated in Figure 2 are shown in these diagrams. The properties of ³He are obtained by

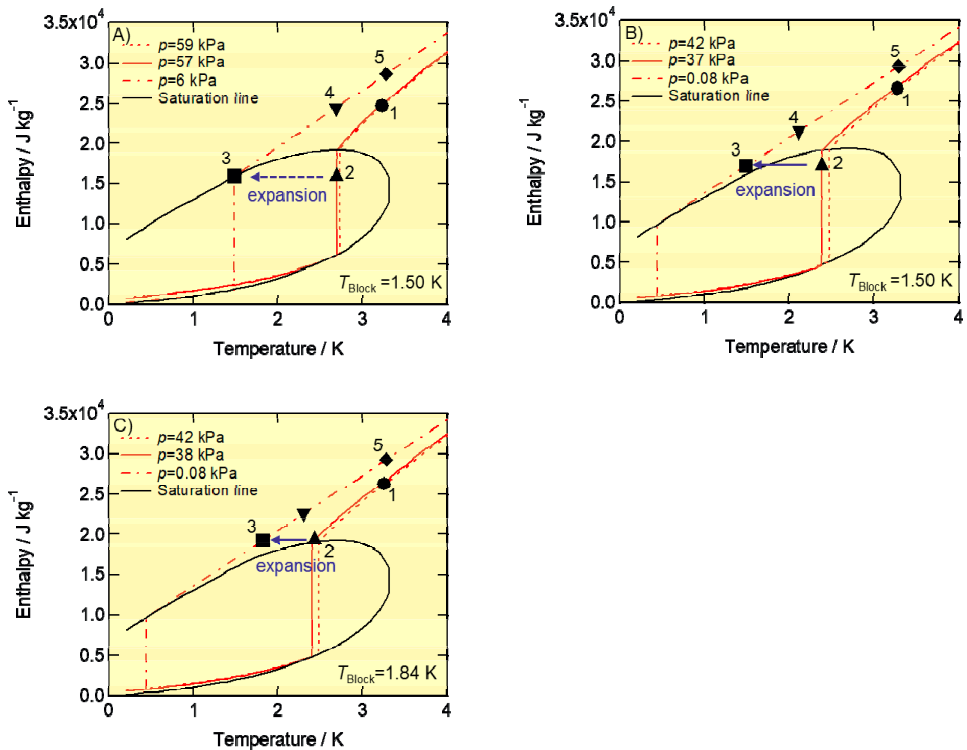


Figure 5. Enthalpy–temperature (h - T) diagrams of the JT circuit for different temperature stability conditions. Estimated states of ³He at numbered locations in the JT circuit as indicated in Figure 2 are shown. The saturated liquid and vapor lines, and isobars corresponded to the pressures at numbered locations are also shown.

utilizing the He3Pak Ver. 1.2 Software.^{19,20} The saturated liquid and vapor lines and isobars corresponding to pressures at numbered locations are also shown in the diagrams. The pressure at location 1 is represented by a dashed isobar line. Location 2 which corresponds the pressure before the JT expansion is represented by a solid isobar line. The pressures at the ³He pot, location 4 and location 5 are represented by the dashed-and-dotted isobar line.

The h - T diagram A) corresponds to data A) of $T_{\text{Block}} = 1.50$ K shown in the Figure 4. The h - T diagrams B) and C) show the h - T diagrams for the data B) of $T_{\text{Block}} = 1.50$ K and data C) of $T_{\text{Block}} = 1.84$ K shown in the Figure 4, respectively. Diagram A) corresponds to the data obtained when the temperature of the comparison block is stably controlled. As already stated in the former subsection, liquid stably existed in the ³He pot in this flow condition. The pressure in the ³He pot is controlled at the saturated vapor pressure. h - T diagrams B) and C) correspond to the data obtained when the comparison block temperature is relatively unstable. Because a liquid phase could not exist in the ³He pot in the flow conditions, the pressure at the ³He pot is evacuated at 0.08 kPa, which is lower than the saturated vapor pressure.

In the two diagrams for unstable temperature control conditions B) and C), the JT expansions start from the wet region or very close to the saturated vapor line to outside the wet region, which is indicated by solid arrows from point 2 to point 3 in the h - T diagrams.

In other words, when JT expansion crosses the saturated vapor line from the wet region, the temperature becomes unstable. In this expansion condition, liquid may appear intermittently. This can lead to temperature instability. In the diagrams for stable condition A), the JT expansion starts and ends within the wet region, as indicated by a dashed arrow from point 2 to point 3 in the h - T diagram.

During the temperature distribution measurements, it is observed that the temperature at point 5 is slightly higher (by a few tens mK) than that at point 1. The temperature difference is almost the same magnitude as the measurement uncertainty. If the counterflow heat exchanger is perfectly thermally isolated from the surroundings and thermally balanced, the temperature at point 5 cannot exceed that at point 1. However, the actual counterflow heat exchanger of the present JT cryocooler is not thermally isolated. There is certain amount of conducting heat flow through the walls of the inner capillary and outer tube that form the counterflow heat exchanger. There are differences in material and diameter between those of the inner capillary and the outer tube, and they lead the difference in the heat flow. These heat flow may affect the temperature distribution measurement. However, further investigation is needed.

It is interesting to note that the flow impedance of the high-pressure side of the present counterflow heat exchange is relatively high because a fine and long capillary is used. If pressure of the high-pressure side increased, frictional pressure drop increase and distributed Joule-Thomson effect^{18,21-23} may more become noticeable. This also needs further investigation.

CONCLUSION

The Pulse tube/³He JT cryocooler is successfully used for precise thermometer calibration. A temperature control stability of better than 0.07 mK in standard deviation at the thermometer comparison block is obtained. The temperature instability is observed near 2 K. This can be attributed to the two-phase flow in the JT circuit. When JT expansion crosses the saturated vapor line from the wet region, the temperature becomes unstable.

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