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Recent status of the cryogenic sample environment at the MLF, J-PARC

Motoyuki Ishikado[1,*], Ryuta Takahashi[2,3], Yasuhiro Yamauchi[3], Masatoshi Nakamura[3], Sora Ishimaru[3], Sara Yamauchi[4,5], Seiko Ohira-Kawamura[2], Hiroshi Kira[1], Yoshifumi Sakaguchi[1], Masao Watanabe[2], and Takayuki Oku[2]

[1]*Neutron Science and Technology Center, Comprehensive Research Organization for Science and Society (CROSS), Tokai, Ibaraki 319-1106, Japan*
[2]*J-PARC Center, Japan Atomic Energy Agency (JAEA), Tokai, Ibaraki 319-1195, Japan*
[3]*NAT Corporation, Tokai, Ibaraki 319-1112, Japan*
[4]*J-PARC Center, High Energy Accelerator Research Organization (KEK), Tokai, Ibaraki 319-1106, Japan*
[5]*Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK), Tokai, Ibaraki 319-1106, Japan*

*E-mail: *m_ishikado@cross.or.jp*

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Recent status of cryogenic sample environment equipment at the Materials and Life Science Experimental Facility (MLF) of the Japan Proton Accelerator Research Complex (J-PARC) has been reported. We have reviewed the specifications and performance of cryogenic sample environment equipment and a newly installed Gifford-McMahon (GM) cryofurnace, which are mainly managed by the Cryogenic and Magnet group in the sample environment team at the MLF. Moreover, the recent maintenance and update of each piece of equipment, the improved temperature-control function, and the expansion of the usable beamline of the cryogenic sample environment equipment are described.

KEYWORDS: sample environment, Cryogenic and Magnet Group, superconducting magnet, dilution refrigerator, commissioning, J-PARC, MLF

1. Introduction

It is important to realize various sample conditions, including extreme ones such as low and high temperatures, to investigate the physical properties of samples at neutron and synchrotron radiation facilities. In particular, low-temperature conditions are often required in dynamic property measurements because the observed peaks are sharpened by eliminating thermal vibrations at low temperatures. In the Materials and Life Science Experimental Facility (MLF) of the Japan Proton Accelerator Research Complex (J-PARC), there are many closed-cycle refrigerators (CCRs) in beamline (BL) instruments to perform low-temperature experiments down to 5 K. To perform significantly low-temperature experiments, the advanced cryogenic equipment such as a ^{3}He cryostat and a dilution refrigerator (DR) is required. The equipment is shared by several BLs. Hereafter, we refer to the equipment as “BL-common” equipment. The Cryogenic and Magnet group in the sample environment (SE) team at the MLF is responsible for the care of the BL-common equipment. The activities of the Cryogenic and Magnet group started in 2013, and the progress in 2016 and 2018 was reported at the 9th and 10th international workshops on SE at scattering facilities [1, 2].

Recently, the demand for extreme environments with a combination of extremely low temperatures and high magnetic fields has increased dramatically. In response to these requests, we have been working on expanding the temperature range using a DR, and re-examining the maximum usable magnetic field at BLs where iron steel frames are used in the vacuum scattering chamber, and reducing background. Here, we report the activities of our Cryogenic and Magnet group.

2. Cryogenic SE equipment

In the Cryogenic and Magnetic group, there are five pieces of BL-common equipment, which are in operation for user experiments: a bottom-loading-type ^{3}He cryostat, top-loading-type ^{4}He cryostat, ^{3}He-^{4}He DR insert, vertical magnetic field superconducting cryomagnet (SC magnet), and newly installed Gifford-McMahon (GM) cryofurnace.

2.1 Current status of cryogenic SE equipment

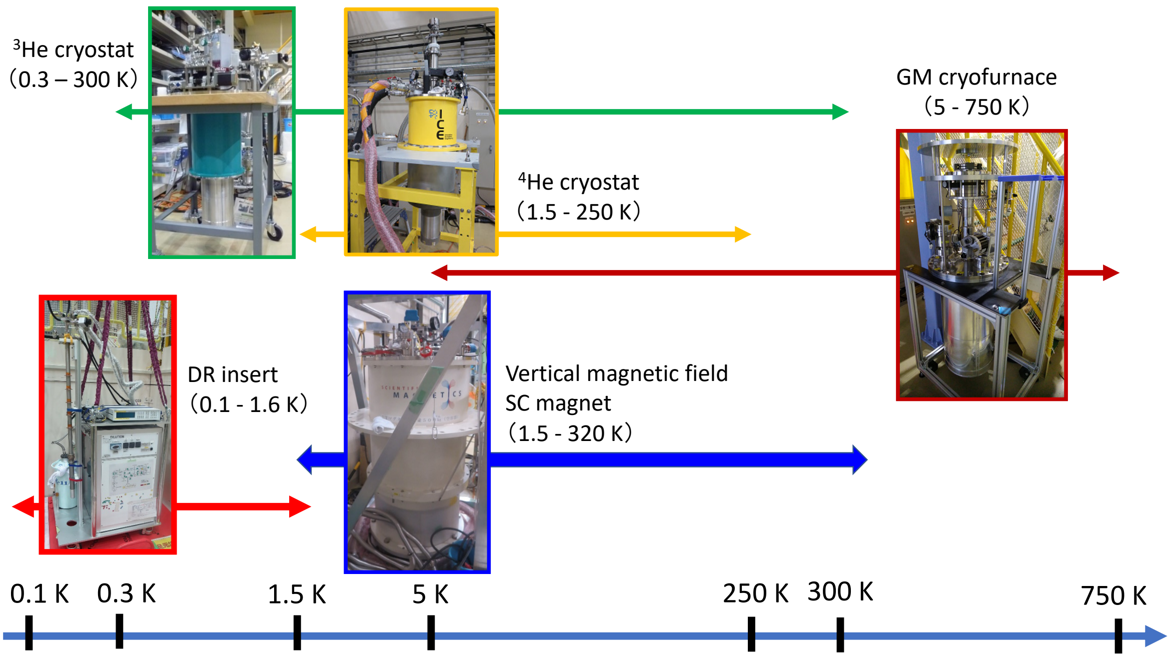

Fig. 1. BL-common cryogenic SE equipment at the MLF. The controllable temperature range of each piece of equipment is indicated by arrows in each color.

The available temperature range of the cryogenic equipment is shown in Fig. 1. The ^{4}He cryostat covers the temperature range from $\sim$ 1.5 K to 250 K. The ^{3}He cryostat covers a lower temperature range down to 0.3 K. The DR covers an even lower temperature range (0.1 K) and can be used in combination with the SC magnet to perform high magnetic field and low-temperature experiments.

Table I lists the overall specifications of BL-common sample environment equipment updated since ref. [1, 2]. The DR insert is used in combination with the SC magnet or ^{4}He cryostat. Here, RT and LT are the room temperature and the lowest temperature, respectively. As for "Available BLs", BL01 (4SEASONS), BL02 (DNA) and BL14 (AMATERAS) are inelastic neutron scattering BLs. BL15 (TAIKAN) is small angle neutron scattering BL. BL17 (SHARAKU) is polarized neutron reflectometer BL. BL18 (SENJU) and BL21 (NOVA) are neutron diffraction BLs. BL22 (RADEN) is neutron imaging BL.

Table I. Specifications of BL-common SE equipment

Specifications	Vertical magnetic field SC magnet	Bottom-loading ^{3}He cryostat	DR insert	Top-loading ^{4}He cryostat	GM cryofurnace
Manufacturer	Scientific Magnetics	NIKI Glass	TAIYO NIPPON SANSO	ICEoxford	Suzuki Shokan
Magnetic field	Max. ±6.8 T	- - -	- - -	- - -	- - -
Wet or dry	wet	dry		wet	dry
Sample space (Max.)	Dia. 48 mm Φ Height 100 mm	Dia. 100 mm Φ Height 130 mm	Dia. 34mm Φ Height 75 mm	Dia. 54 mm Φ Height 100 mm	Dia. 54 mm Φ Height 100 mm
Temp. range	~ 1.5 - 320 K	~ 0.3 - 300 K	~ 0.1 K - 1.6 K	~ 1.5 -250 K	~ 5 - 750 K
Aperture angle	± 10° (vertical) 330° (horizontal)				
Vac. chamber	JIS 800 Flange	JIS 400 Flange		JIS 400 Flange	JIS 400 Flange
Gonio stage	700 mm Φ	300 mm Φ		300 mm Φ	
Beam window	Al window	Al window	Al window	Al window	Al window
Gonio angle	± 180°	± 135°		± 180°	± 180°
Setup time	2 days from RT	~19 hrs from RT	~18 hrs from RT	~4 hrs from RT	~7 hrs from RT
Keeping time (Max.)	~ 45 hrs (Liq. He)	~ 150 hrs at LT (one shot)	- - -	~ 65 hrs (Liq. He)	- - -
Combination	DR insert		SC magnet ^{4}He cryostat	DR insert	
Available BLs	BL01,14,15,17,18,22 Since Nov. 2012 -	BL01,02,14,18,21 Since Nov. 2016 -	BL02,14,15,17,18,22 Since Nov. 2016 -	BL01,02,14,18,21 Since Nov. 2016 -	BL02,14, 18 Since Jun. 2017 -

2.2 Newly installed GM cryofurnace

The GM cryofurnace was newly introduced in June 2017 (Fig. 2) which is listed in Table I. This is dry-typed cryostat equipped with GM cryocooler as shown in Fig. 2 (a) and (b). The available temperature range is ~ 5 - 750 K. The entire temperature range is covered using one sample stick. Two types of temperature sensors are attached to the tip of the sample stick. The Pt sensor is used for the high-temperature region (14 - 750 K), and RhFe sensor covers the low-temperature region (5 - 300 K). To obtain temperatures above 300 K, a heater and aluminum radiation shields are mounted on the tip of the sample stick (Fig. 2(c)). When the sample was heated over 300 K, the refrigerator was running and kept at 300 K using the inner vacuum chamber (IVC) heater to prevent the increase of the cold head temperature, and the IVC is evacuated to a high vacuum of ~ 10^{-5} Pa using a turbo-molecular pump.

3. Improvement of the SE equipment

Here, we describe the update of the performance of each SE equipment by performing maintenance and commissioning in response to the request or needs arising from user experiments.

3.1 Vertical magnetic field SC magnet

A vertical magnetic field SC magnet has been used in user experiments since November 2012. The specifications are listed in Table I; it is a wet-type cryomagnet. The liquid ^{4}He level gauge and power supply has been replaced due to aging. Although the maximum magnetic field is 7 T on the specifications, this does not always mean that the magnetic field will reach 7 T. To find the actual maximum magnetic field, a quench was intentionally performed. In other words, the magnetic field was slowly increased until it was quenched. In the test experiment, quenching occurred at 6.94 T. In the user experiments, the SC magnet was used within ± 6.8 T. In addition, the consumption rate of

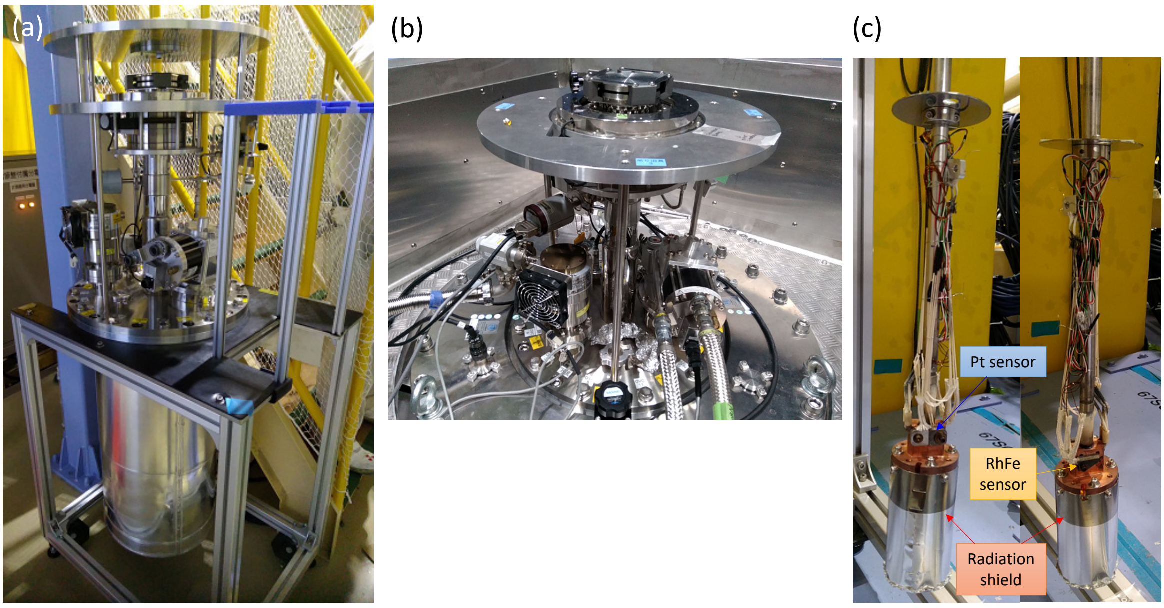

Fig. 2. (a) Photograph of the overall view of the GM cryofurnace. (b) GM cryofurnace installed on the vacuum scattering chamber. (c) Photograph of sample stick tip with radiation shield viewed from both front and rear sides. Pt sensor, RhFe sensor, and radiation shield are indicated by the arrows.

liquid helium was optimized by adjusting the degree of opening and closing of the needle valve and the flow rate of variable temperature insert (VTI) pump such that the SC magnet could be operated for 45 hrs continuously without refilling the liquid helium with no load.

The amount of exchange gas in the IVC is an issue to consider when measuring inelastic neutron scattering. In the inelastic neutron scattering BLs, the exchange gas can be used with only ∼ 50 mbar at 300 K to prevent the observation of helium gas. However, the lowest temperature at the sample position is ∼ 4 K, which may be due to the heat inflow or other factors, although the cause is unknown. If we use an exchange gas (^{4}He) over 1.5 atm at 300 K, the sample position in the IVC cools below 2 K. The method of using exchange gas (^{4}He) over 1.5 atm at 300 K was used in other BLs. Large amount of ^{4}He exchange gas may have reached a superfluid state that cooled the walls of the IVC below 2 K and cooled the sample location by radiative heat flow to the walls. To lower the lowest temperature with a small amount of exchange gas, we fabricated a sample stick with a thermal contact at the coolest location of the VTI. The original sample stick is made of resin, but the parts from the thermal contacts to the sample position of the new sample stick are made of copper, and the sample is cooled by heat conduction through copper. This cooling by thermal contact from VTI works well and allows us to successfully perform experiments involving cooling temperatures around ∼ 3.5 K with small amount of He gas.

We have evaluated the influence of the high magnetic field generated by the SC magnet on the neutron instrument at the MLF. Some BLs at the MLF, such as BL01 and BL14, have the vacuum scattering chamber that contains iron steel frames. Application of a high magnetic field in the vacuum scattering chamber induces magnetization of iron in the frames, which yields magnetic forces between the coils and the frames. This applies stress to the struts in the SC magnet, which support the coils inside, which can cause serious damage to the SC magnet. To avoid such troubles, we evaluate the stress under high magnetic field on the instrument by finite element calculations. The SC magnet is guaranteed for the operation of tilting at 5 degrees. In this condition, the horizontal-axis force F is described as $F = Mg \times \sin 5^\circ$, where M is the total mass of the coils and g is the gravitational acceleration. This indicates that the magnetic force up to F on the coils in the horizontal direction is acceptable to keep the performance of the SC magnet without causing damage. The calculation results revealed the magnetic force on the coils is nearly same as F at 7 T. We should also consider the influence of the magnetic stray field on the surrounding devices. At BL01, a turbo-molecular pump is settled below the vacuum scattering chamber, which is expected to be the most seriously affected by the stray field of the magnet. According to the finite element calculation, the magnetic stray field at this pump will be in the acceptable working range when the magnetic field is applied up to 5.8 T. Based on these calculation results, the maximum applicable field is evaluated to be 5.8 T on BL01. We currently use the SC magnet on BL01 limiting the applied field up to 3.5 T for safety's sake.

3.2 Dilution Refrigerator Insert

The DR insert has been available for user experiments since November 2016. The temperature can be controlled from the lowest temperature, ~ 0.1 K, to 1.6 K using the heater in the DR insert while the DR is running for cooling. The temperature ranging from 1.7 to 5 K is controlled by using the heater in the VTI of the cryostat while the DR operation is stopped. A much higher temperature can be achieved by introducing a exchange gas into the vacuum-insulating chamber (VIC). Using this method, the temperature could reach 80 K for the SC magnet and 250 K for the ^{4}He cryostat. Cyclic temperature control, from the lowest temperature to high temperatures, and then to the lowest temperature, was achieved in response to user's requests. Many trial-and-error test experiments were required to change the DR heater control, DR cooling operation, VTI heater control, and the pressure in the VTI. Throughout the test experiments, it had become possible to control the temperature from the lowest temperature to 50 K, maintained for 24 h, and then to the lowest temperature within 4.5 hours.

While we measure the inelastic neutron scattering, it is desirable to reduce the amount of He gas in the IVC of the SC magnet as little as possible. According to the test experiments, it was found that the amount of the exchange gas could be reduced to be 150 mbar at 300 K.

Previously, we did not monitor the temperature at the sample position but monitored the temperature at the cold head. Therefore, we measured these two temperatures and found that the temperature at the cold head was almost the same as that at the sample position within a few mK range.

There were some user experiments in which the temperature did not reach the lowest value. We doubted if the performance of the DR system was maintained. Therefore, the cooling power of the DR system was examined. We measured output value of the DR heater while it was heated from a low temperature. Using this method, the cooling power of the DR system was determined to be ~15 - 30 μW at 100 mK and ~ 200 ± 100 μW at 300 mK. This is comparable to the value provided by the manufacturing company, which is approximately 20 μW at 100 mK. This means that there is no problem with the cooling power of the DR system. Therefore, the temperature did not reach the lowest value probably because the sample has a great heat capacity or bad thermal conductivity. We are considering to introduce DR system with higher cooling power in future.

While the DR insert is used with the SC magnet on some BLs (BL15, BL17, BL22), there is no space to place the large gas-handling system of the DR system in the shielding hutch of the instrument. In such a case, the gas-handling system must be placed on the shielding hutch, and the flexible tube that connects the DR insert with the gas handling system for the circulation of the ^{3}He-^{4}He mixed gas must be extended. However, an excessive extension may limit the lowest attainable temperature. When the flexible tube was extended from 3 m to 10 m in length, the lowest temperature was 80 mK at mixing chamber (Initial lowest temperature is ~ 56 mK). When we use an extended flexible tube with a length of 5 m, the lowest temperature reached 60 mK. After the test cooling, the length of 5m flexible tube is used on all BLs.

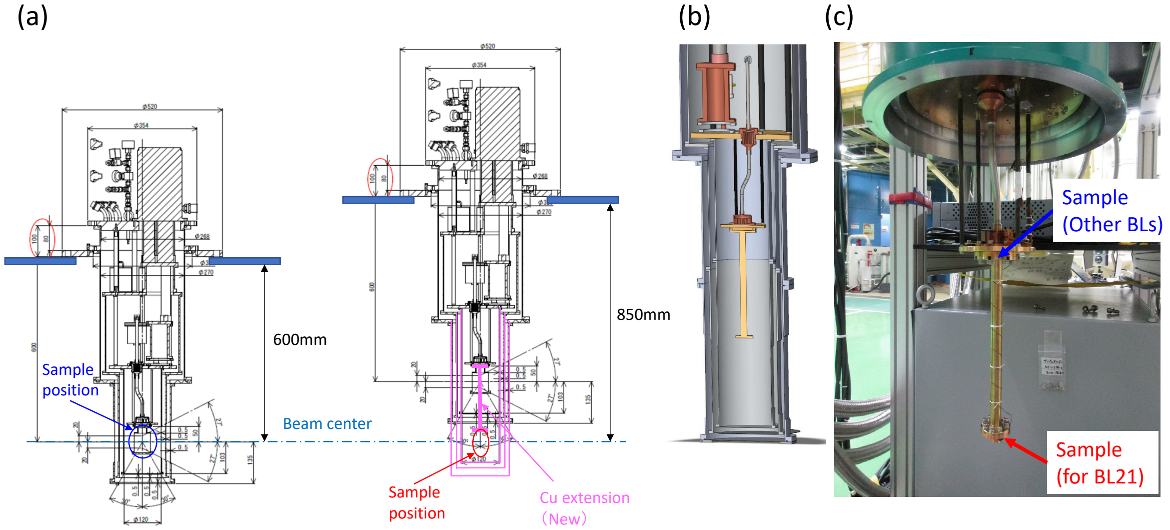

Fig. 3. (a) ^{3}He cryostat attached to the flange of the vacuum scattering chamber (blue). The right panel shows the case for BL21, and left panel shows the case for other BLs. (b) Inner structure of ^{3}He cryostat with extension adaptors. (c) Photograph of ^{3}He cryostat with an extension adaptor.

3.3 ^{3}He cryostat

The pulse tube refrigerator in the ^{3}He cryostat occasionally stopped working unexpectedly, probably because of the electric noise generated in the facility. To prevent this interruption, we installed an automatic recovery program in the Programmable Logic Controller (PLC), which we previously installed in the ^{3}He cryostat [2]. The PLC regularly reads the status of the ^{3}He cryostat. When it detects a fault, it sends the commands of a reset and a start for the re-operation of the refrigerator. This program is suitable for the smooth operation of the ^{3}He cryostat.

So far, a low-temperature experimental environment in BL21 was achieved using a ^{4}He cryostat, and a lower-temperature experimental environment was desired. Although a ^{3}He cryostat is desirable for low-temperature equipment from the viewpoint of installation feasibility, the height of the top flange of the vacuum chamber from the beam center is 250 mm higher than that of the MLF standard (Fig. 3 (a)). Therefore, the extension was added to the outer vacuum chamber, radiation shields, and sample stage of the ^{3}He cryostat to adjust its height (Fig. 3 (b), (c)). There was no change in the lowest temperature with extension, although the cooling time from the room temperature (~ 300 K) to the lowest temperature was extended from 19 to 25 h. In this way, it had become possible to perform experiments at ~ 0.3 K in BL21.

4. Frequency of use of the SE equipment

Fig. 4 (a) shows the development of the frequency of use of SE equipment in a year, which was updated based on our previous report [2]. One use is defined as one experiment in which one piece of SE equipment is used. Namely, when both the SC magnet and the DR insert are used in one experiment, the number of uses is regarded as two. This frequency started to increased in 2016. The reason for this is that the SC magnet was the only low-temperature SE equipment until 2015, and some pieces, such as the ^{3}He cryostat and DR insert, became available in 2016. The number of uses decreased from 2019 to 2022, since the beam stopped in November 2019 and November 2020 due to issues related to the neutron source and the decrease in the number of uses occurred from April to May 2020 owing to COVID-19. Fig. 4 (b) shows the number of uses of individual low-temperature SE equipment. Each piece of equipment was stably used after its introduction.

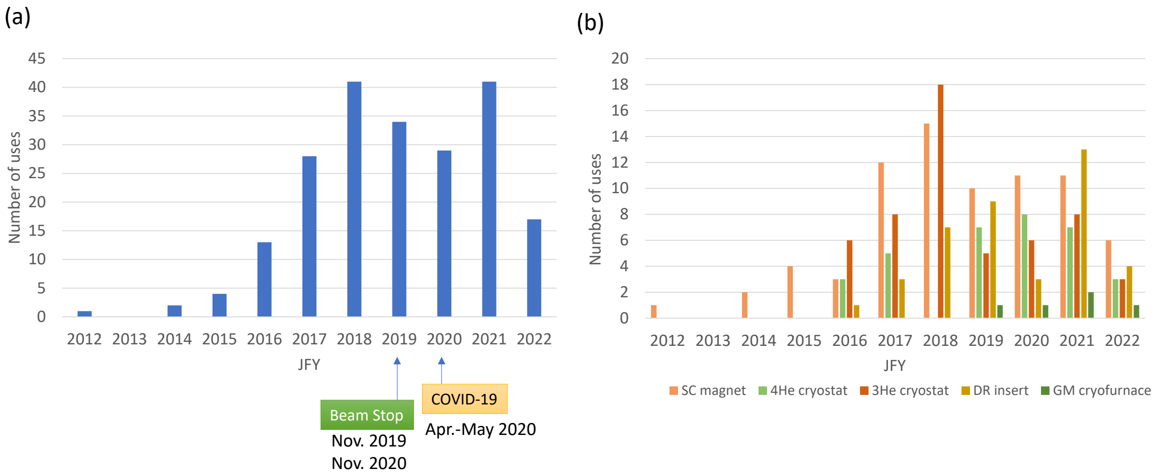

Fig. 4. (a) Development of the number of uses for low-temperature SE equipment. (b) Number of uses for individual low-temperature SE equipment. Here, the number of uses for 2022 is only 2022A (first half of JFY2022).

5. Summary

In this report, we described the specifications, update and commissioning of BL-common cryogenic SE equipment, and status of user support performed by the Cryogenic and Magnet group in the SE team at J-PARC, MLF.

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References

[1] S. Ohira-Kawamura, T. Oku, M. Watanabe, R. Takahashi, K. Munakata, S. Takata, Y. Sakaguchi, M. Ishikado, K. Ohuchi, T. Hattori, H. Kira, K. Sakai, T. Aso, Y. Yamauchi and S. Isomae, Sample environment at the J-PARC MLF, J. Neutron Research **19,** 15 (2017).

[2] S. Ohira-Kawamura, R. Takahashi, M. Ishikado, Y. Yamauchi, M. Nakamura, K. Ohuchi, H. Kira, W. Kambara, K. Aoyama, Y. Sakaguchi, M. Watanabe and T. Oku, J. Neutron Res. **21,** 17 (2019).