# 3.7.4

# Present status of sample environment at J-PARC MLF

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**Abstract.** In Materials and Life Science Experimental Facility (MLF) of J-PARC, a Sample Environment (SE) team has been officially organized in a newly established section, Technology Development Section, with succeeding the previous ad hoc SE team. We, the SE team, are proceeding with the SE standardization in MLF, and working on operation of so-called beamline (BL)-common SE equipment. We currently have a vertical-field superconducting cryomagnet, a $^{3}He$-$^{4}He$ dilution refrigerator insert, a top-loading $^{4}He$ cryostat and a high-temperature furnace as the BL-common SE equipment. In order to maintain them and to prepare for users' experiments, two SE areas are equipped in the experimental halls, though their spaces are still not enough. Much larger workshop for long-time commissioning, a sample synthesis room and sample characterization rooms will be soon prepared in a new building under construction. We also contribute to safety improvement as the members of the MLF equipment safety committee.

## 1. Introduction

Sample environment (SE) is one of indispensable components to perform the neutron scattering experiment. Various environments, such as high and low temperatures, magnetic field and high pressure, are required by users depending on their research fields. Japanese neutron facilities have had a culture that each instrument individually prepares its SE equipment, where instrument scientists are responsible not only for their instruments but for SE and researchers operate the equipment for their experiment. However, such environments should be supported by facility-wide activities. In the Materials and Life Science Experimental Facility (MLF) in Japan Proton Accelerator Research Complex (J-PARC), the original SE team was organized as "SE task" in 2004, which is about four years before the first neutron beam of MLF. Members of this task were scientists working at MLF, and had discussed the strategy in our facility and standardization of SE based on their experience of neutron experiments as a user or instrument scientist. This team was reorganized as the "SE team" in 2009, almost concomitantly with an opening of the user program. Then two engineers joined, and had discussed a guideline of the SE

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standardization of SE, taking over the previous SE task, and the support system in SE. This SE team was a so-called interim team, and each member had another responsibility, though it was an advantage that the members could respond to users' requests immediately. As shown in Fig. 1, the number of proposals and users are increasing year by year. Then the variety of the required SE should also increase. Therefore, it is necessary to respond users' requests effectively and to establish the user support system.

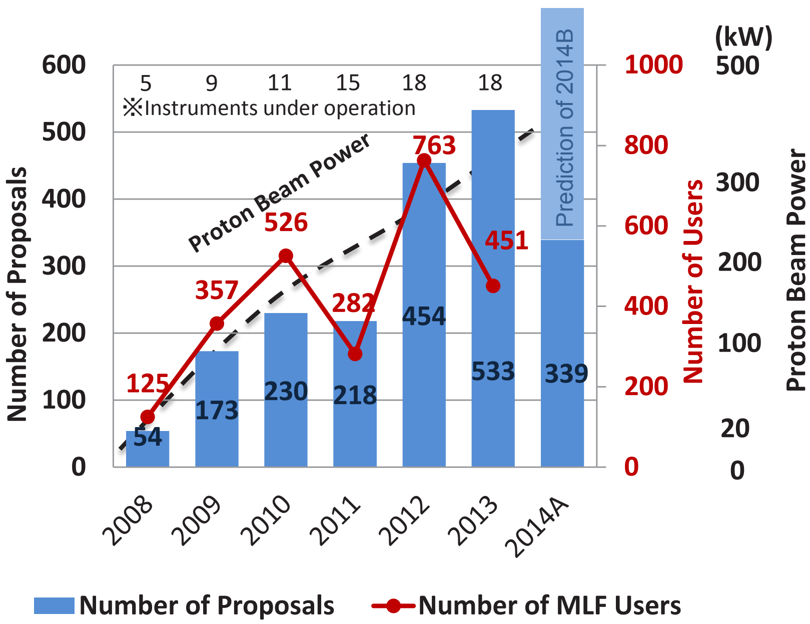


**Figure 1.** Number of proposals and users in MLF.

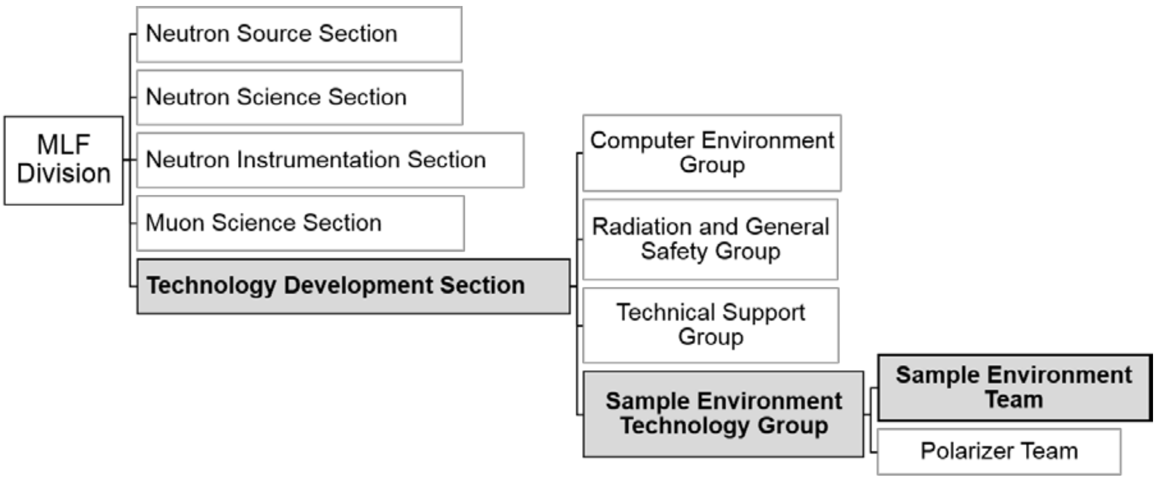


**Figure 2.** Organization of the MLF division.

In April 2013, the Materials and Life Science Division at J-PARC Center has established a new section, Technology Development Section, for the purpose of technical support of common equipment and development, operation and maintenance of common devices. The Technology Development

Section have four groups, Computer Environment Group, Radiation Safety Group, Technical Support Group and Sample Environment Technology Group as shown in Fig. 2. Then the SE team was officially organized in the Sample Environment Technology Group. The newly organized SE team consists of staff members from JAEA, KEK and CROSS so as to be able to support any beamlines (BL's) across the boundaries of institutes, no matter if it is a public BL or not. The new SE team aims to manage the overall SE in MLF, which is the first attempt in Japanese neutron scattering experimental facilities. Here we can take comprehensively safety measures on the whole equipment in MLF. In this team, we aim to establish a proper system composed of several professional sub-groups such as cryogenics and magnets, high temperature, high pressure, soft matter, and so on.

## 2. SE equipment

### 2.1 Standardization of SE in MLF

As mentioned above, we aim to prepare the SE equipment which is used commonly at each BL. Then we need to consider some compatibility among the instruments. We have discussed standardization of SE in MLF to provide a so-called "SE protocol", on the basis of the experience in practical experiments and by investigating the situation of other facilities, since the original SE team was organized. Each BL is enabled to use such BL-common SE equipment by referring to this guideline for designing the instrument. This results in sharing of technology, reducing costs and efficiency of experiments.

**Table 1.** Representative items in the SE protocol.

| Items | Specification, Type, etc. |
|---|---|
| **Dimensions for a vacuum scattering chamber** | - Flange size: 200φ, 400φ and 800φ in accordance with JIS<br>- 1000φ as an option flange.<br>- Flange to beam center: 600 mm |
| **PCD and size of bolts on a goniometer** | - PCD: 650 mm (large equipment) / 260 mm (small equipment)<br>- Size of the bolts: M12 (large equipment) / M10 (small equipment)<br>- Goniometer to beam center:<br>350 mm (large equipment) / 210 mm (small equipment) |
| **Standard closed-cycle refrigerator** | - Cold head: RDK-415D (Sumitomo)<br>- Compressor: F-50L (water-cooled) |
| **Temperature controllers and censors** | - Controllers: LakeShore 340 and 350<br>- Censor (T > 1.5 K, under zero magnetic field): Si diode censor<br>- Censor (T > 1.5 K, under magnetic field): Cernox censor |
| **Sample cells** | - Bottom of the cold tail to beam center: 50mm<br>- Dimensions at attachment: M4 x 8 screws, PCD 44 mm position<br>- Dimensions at attachment (room temperature, 1 atm):<br>M6 x 1 screw for powder samples |
| Pulse motor controller | PM16C-04XD(L) (16ch, Tsujicon) |

The representative items in this protocol are listed in Table 1. For the instruments having a vacuum scattering chamber, such as the total diffractometer and chopper spectrometer, were recommended to refer to the standard dimensions of the flange shown in Table 1. An optional flange of 1000φ is expected to be used for additional devices such as a radial collimator and polarizer. A symmetric line including two bolts should be located on the neutron beam. For the instruments having a goniometer, such as the small-angle scattering instrument, PCD and size of the bolts to set the SE equipment on the goniometer are standardized for two kinds of equipment, large and small ones, as shown Table 1. Cryomagnets and some cryostats and furnaces are classified in the large equipment, while other compact systems are classified in the small equipment. The instruments can use any BL-common SE equipment designed based on these two kinds of dimensions by preparing an appropriate adapter. The standard closed-cycle refrigerator (CCR) is available for the measurement at temperatures above ~4 K. Some instruments have a top-loading type of this CCR, and other have a bottom-loading type. The insert of the top-loading type CCR is not standardized at present. The recommended compressor, F-50, is a water-cooling type. The SE team possesses not only its spare but air-cooling type compressors (CSA-71A), so that one can carry out test operation of the CCR even in the period when the cooling water is not supplied. The standard temperature controller has been decided to be LakeShore 340. However, we currently recommend LakeShore 350 as a new standard because manufacturing LakeShore 340 has already been stopped. Dimension around the sample cell is standardized by entirely referring to that at the Research Reactor JRR-3, since some users relatively use the reactor (JRR-3) and spallation source (MLF). To control the rotary stage on the SE equipment, the SE team recommends to introduce a pulse motor controller listed in Table 1 (Tsujicon), which can be controlled on a software for data taking. Note that we have also discussed other items such as coupling for cooling water and helium recovery lines and data logger

**Table 2.** Typical SE equipment prepared by each BL.

| BL | SE equipment |
|---|---|
| BL01 | 4K top-loading cryostat, high-temp. insert |
| BL02 | Top-loading cryofurnace |
| BL03 | Gas-flow cryostat |
| BL04 | Auto sample changer |
| BL08 | 4K cryostat, 1K cryostat, 10K top-loading cryostat, V-furnace, Auto sample changer |
| BL09 | 10-700K top-loading hot stage, 4K top-loading cryostat, 1000K furnace, Auto sample changer for installing V-Ni holder, Auto sample changer for in-situ measurement, Storage battery cell |
| BL10 | Goniometer (5-axes) |
| BL11 | Multi-anvil press (500 t x 6 axes), Paris Edinburgh press (200 t), Palm cubic press (100 t), Low-temperature press (100 t) |
| BL12 | 4K cryostat, 6K cryostat, $^3$He cryostat, 14T vertical-field magnet |
| BL14 | 4K bottom-loading cryostat, Top-loading cryofurnace |
| BL15 | Auto sample changer, Loading machine, 10T vertical-field magnet, 4T horizontal-field magnet Laser furnace, 4K bottom-loading cryostat, Air core electromagnet, |
| BL16 | Auto sample changer, High-temperature cell, Low-temperature cell, Humidity controlled cell |
| BL17 | Electromagnet, 4K cryocooler |
| BL18 | 4K cryostat, 2-axis goniometer, 1-axis goniometer, High-pressure cell |
| BL19 | Loading machine, Furnace system for high temperature loading, Dilatometer, Cryogenic loading machine, Eurelian cradle, Gandolfi goniometer + heater, Fatigue machine, High temperature loading machine for small sample, 100K cooling chamber for loading experiment |
| BL20 | Auto sample changer, 30 sample changer, V-furnace, Atmosphere furnace, Cryofurnace with sample changer, Goniometer |
| BL21 | Auto sample changer, in-situ meas, V foil heater, Cryofurnace, Impedance measurement system, Temperature-controlled sample changer, 2 dimensional element spectroscopy system |

2.2 SE equipment

In MLF, some SE equipment, which is dedicated for an instrument, strongly required by users or used frequently as a standard one, has been managed and operated by the instrument staff at each BL. Representative SE equipment prepared at BL's is shown in Table 2. Some of them are designed based on the SE protocol, in order to enable us to use them even at another instrument. On the other hand, the SE team has introduced and managed the BL-common SE equipment, which may not be frequently used but is necessary. At present, we possess four pieces of the BL-common SE equipment, which are in operation or under commissioning: a vertical-field superconducting cryomagnet, a $^{3}$He-$^{4}$He dilution refrigerator insert, a top-loading-type $^{4}$He cryostat and a high-temperature furnace with an Nb heater. We have managed them and support their operation during users' experiments. Note that we plan to introduce other equipment in response to users' demands in the future.


**Figure 3.** BL-common SE equipment managed by the SE team. (a) Vertical-field superconducting cryomagnet, (b) $^{3}$He-$^{4}$He dilution refrigerator insert, (c) top-loading-type $^{4}$He cryostat and (d) High-temperature furnace with a Nb heater.

Figure 3(a) shows the vertical-field superconducting cryomagnet (Scientific Magnetics). This equipment has an asymmetric-coil pair so that it can be used even in the polarized neutron experiment, and its maximum magnetic field is 7 T. The magnet coils are coated with $B_4C$, which is a neutron absorber. The magnet was designed supposing that an open angle in the vertical direction is ±10º for 20 mm of the sample height. In the horizontal direction, the magnet has 30º of a blind angle for pipes and wires, the direction of which was determined by investigating the detector positions at most instruments. The system has a liquid helium bath without any re-condense system. The sample stick is rotated approximately 180º, and is cooled down to 1.5 K. This cryomagnet can be set both on a vacuum scattering chamber with the 800φ flange and on a goniometer. As the system has aluminum windows, it is suitable for the single-crystal diffractometers, chopper spectrometer, small-angle neutron scattering (SANS) instrument etc. rather than the powder diffractometer. On-beam commissioning works have been carried out at cold-neutron disk-chopper spectrometer, AMATERAS (BL14), polarized neutron reflectometer, SHARAKU (BL17) and extreme environment single crystal neutron diffractometer, SENJU (BL18) [1].

Other than the conventional sample stick, a $^{3}$He-$^{4}$He dilution refrigerator insert (Taiyo-Nippon Sanso co.), shown in Fig. 3(b), can be also set into the cryomagnet. The lowest temperature is below 100 mK, and we have achieved 50 mK with no sample during the first test operation. The inner diameter of the sample space is 40 mm, and the distance from the bottom surface of the cold head to the beam center is 40 mm, which is shorter than the standard value in MLF. This insert is currently under commissioning. In order to use this dilution refrigerator insert in the measurement under zero magnetic field, we have prepared a top-loading-type $^{4}$He cryostat, which was manufactured by Suzuki Shokan co. (Fig. 3(c)). This cryostat is a wet system having liquid helium and nitrogen baths, and has a rotary stage of the insert. Temperature range is from 2K to the room temperature. As the system also has aluminum tails and windows as well as the cryomagnet. The system has a 400φ flange to be set on the vacuum scattering chamber, and also can be set onto a goniometer as the small-size equipment. This equipment is still under commissioning.

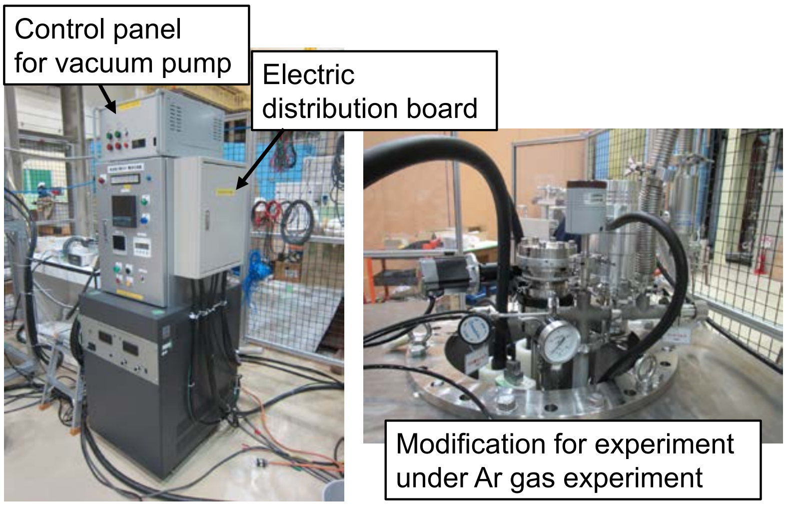


**Figure 4.** High-temperature furnace after the improvements.

Figure 3(d) shows a high-temperature furnace with a niobium heater, manufactured by AS Scientific Products. The maximum operating temperature is 1600 ºC in a vacuum and 1300 ºC in an argon gas. The sample stick can be rotated. The heater part and surrounding radiation shields are made of niobium, which is known to make up few background due to incoherent scattering. Therefore, this furnace is mainly used at quasielastic and inelastic scattering instruments. However, the elastic scattering from many niobium foils on the direct beam causes serious background in the nonzero region of energy transfer because the flight paths for neutrons scattered at a niobium foils are slightly shorter or longer than that for neutrons scattered at the sample. Thus, an oscillating radial collimator have been developed by some instruments to be used together with the furnace [2]. This equipment is now in operation and has ever been used for the experiments at AMATERAS (BL14) and small and wide angle neutron scattering instrument, TAIKAN (BL15). We note that some improvements have been made by the SE team, as shown in Fig. 4. A control panel for vacuum pumps and an electric distribution board were installed for users' convenience and safety. A gas inlet line was equipped in order to conduct a high-temperature experiment under argon atmosphere. Taking into account the heating up of the outer case of the furnace, we decided on a rule that an experiment under argon atmosphere must be performed below 1300 ºC.

## 3. Infrastructure for SE

It is essential to keep the infrastructure, which is a so-called "SE area", to prepare the SE equipment for users' experiments and to maintain its performance inside or near the experimental hall. There was no such an area when the MLF started operation. We prepared the SE areas in the 1st and 2nd experimental halls in MLF in a few recent years (Fig.5). Although the experimental hall was already occupied by instruments and their cabins, we could prepare the SE area with a space of ~30 $m^2$ in the 1st experimental hall. On the other hand, there was less space in the 2nd experimental hall. We can use a space of ~20 $m^2$ as the SE area in the 2nd experimental hall for the duration of the beam operation, while its area is reduced to half so that the rest area is used for other purposes for the construction works during the shutdown period. Utilities such as the power supply, cooling water line, compressed air line, helium gas recovery line and the exhaust line have been introduced in both SE areas, and then we started the commissioning and maintenance of the SE equipment, repair and replacement of parts, and so on. Taking into account that the number of the SE equipment increases in near future, the current SE areas in the experimental halls are definitely insufficient. Thus we need to discuss a solution to expand the area, though there is no available space inside the both experimental halls.

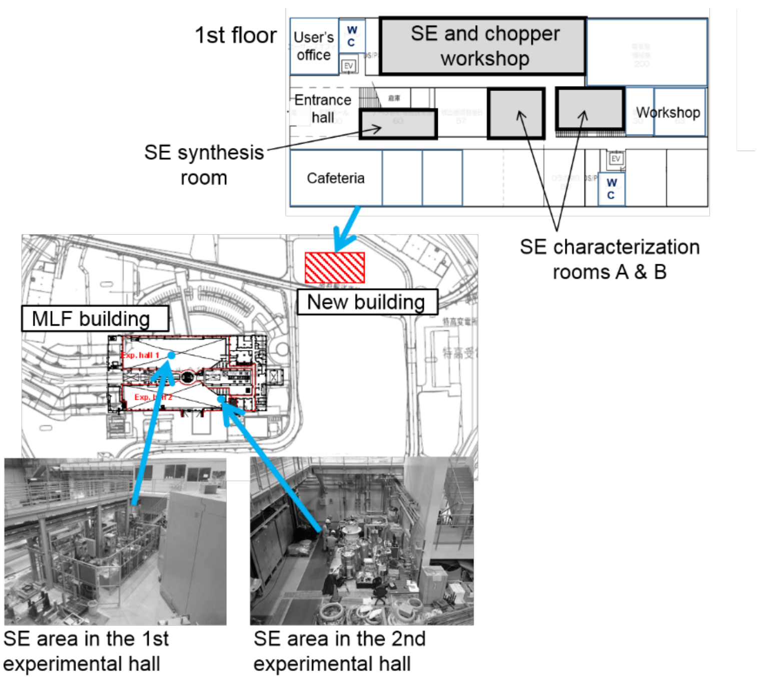


**Figure 5.** Working areas related to SE in the MLF building and the new building.

A new building for the MLF user support, which is being constructed near the MLF building, is about to be completed in the end of March 2015. This building is used for the things related to the user support, such as sample preparation, data analysis, development and maintenance of devices, and so on. As far as SE is concerned, it is planned to prepare an SE and chopper workshop, which is dedicated for maintenance of the SE equipment and choppers, a sample synthesis room, sample characterization rooms for various research field and a chemical laboratory on the first floor, as shown in Fig. 5. In the SE and chopper workshop, long-term commissioning and maintenance of the SE equipment will be carried out. In addition, the development of the new SE equipment will also be conducted here. This room is shared with the chopper development group, which works on development and commissioning of choppers. In the sample synthesis room, users can prepare the samples before bringing them into MLF. In the characterization rooms, various measurements, such as structure analysis and magnetic susceptibility, specific-heat and adsorption measurements, can be performed before the experiment.

## 4. Safety

Figure 6 shows the safety examination system for users' equipment and devices in MLF. Some of us (W.K., T.O., Y.S. and K.A.) are the regular members and one of us (Y.Y.) is a cooperative member of the MLF equipment safety committee, and we contribute to the improvement on the safety environment at MLF. This system is also applied for the SE equipment and devices, including those prepared by the BL groups. The safety examination is conducted by the committee, and the equipment is allowed to be used at MLF, on the basis of the result.

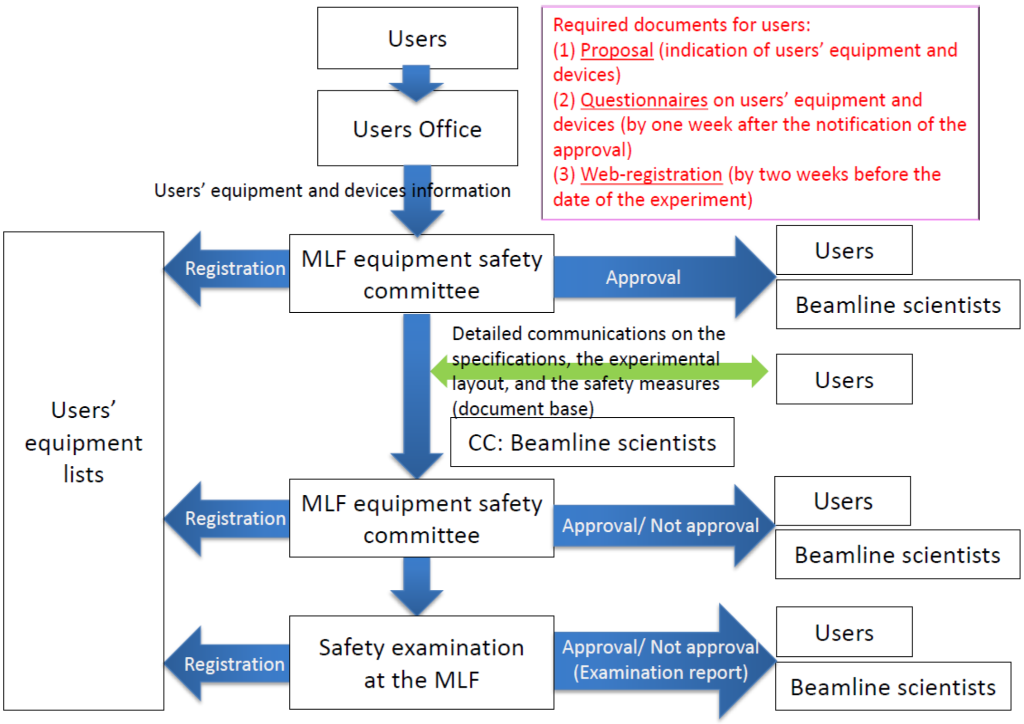


**Figure 6.** Safety examination system for users' equipment and devices at the MLF

The SE team contributes to the improvement on safety related to the SE equipment at MLF. Here we show the safety measures on the top-loading-type cryostat we took as an example. The top-loading-type cryostat generally contains a risk caused by inappropriate setting of a sample stick. Namely, the air could be inhaled inside the sample space continuously from a small leakage at the O-ring to be condensed there, when the sample stick is cooled down. As the result, the sample stick may be ejected suddenly when changing the sample, due to rapid evaporation of the accidentally condensed air. In fact, MLF has ever experienced this incident during a user's experiment. Responding to this problem, we have taken the following steps to prevent such incidents, and have strongly recommended each BL that possesses a top-loading cryostat irrespective of whether the system is wet or dry to take these safety measures.

The safety measures taken on the top-loading cryostat at MLF are displayed in Fig. 7. Firstly, we modified the sample stick to have an open end and added a relief valve on the tube just below the top flange so that the excess pressure is released through the end hole, the relief valve on the center stick and the original relief valve on the cryostat even when the bottom baffle of the sample space is sealed by an ice block. As a further step, we applied "D-cuts" to the baffles to prevent for a baffle to be sealed entirely by ice. In addition, especially for the inserts with no hollow tubes such as dilution refrigerator, a short detachable stainless wire is necessarily connected to the cryostat and its insert when the insert is pulled up for the sample exchange, to avoid ejection of the insert. It is also recommended to adopt the O-ring with a center ring to place the O-ring surely.

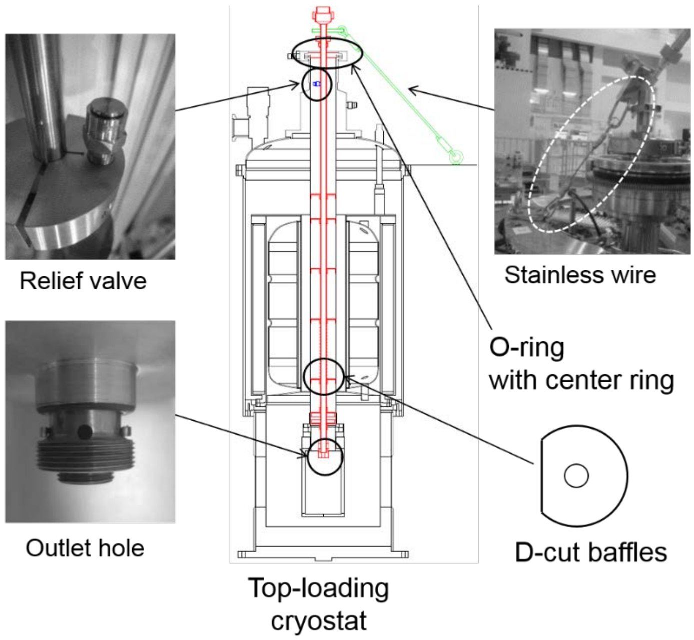


**Figure 7.** The recommended safety measures for top-loading cryostat.

## 5. Summary

The SE team has been officially organized in the Technology Development Section newly established in MLF. We are working on management and operation of BL-common SE equipment and development of SE devices. The SE team has discussed standardization of SE in MLF since the previous SE team was organized, based on our experience in practical experiments and by investigating the situation of other facilities. This guideline is recommended to be referred by each instrument. We have prepared some pieces of the versatile BL-common SE equipment, which are a vertical-field superconducting cryomagnet, dilution refrigerator insert, $^{4}$He cryostat and high-temperature furnace. They are now in operation or under commissioning. The SE areas were prepared in the 1st and 2nd experimental halls, and have been used for short-term commissioning on the SE equipment and preparation of it for users' experiments. We will also equip more areas related to SE, such as the SE workshop and sample preparation room, in the new building for user support. Furthermore, some of us contribute to the improvement on safety environment in MLF as the members of the MLF equipment safety committee. Then we take comprehensively safety measures even on the user's carry-in devices and BL-dedicated SE equipment in MLF. We note that we have just started preparation of some new environments as follows. Responding to users' request, we have just started preparing high pressure system, aiming to finally develop low-temperature and high-pressure environment for inelastic neutron scattering. We are also planning to generalize the pulsed magnet system for neutron scattering, which H. Nojiri and co-workers have developed at Spallation Neutron Source (SNS) in Oak Ridge National Laboratory and MLF [3], by cooperating with them, in order to introduce it for the general users.

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