

Safety instruction for the sample preparation laboratory LR 022

This laboratory is intended for sample preparation using x-rays, cutting devices and polishers. The use of these instruments and the handling of samples can bear health risks. Here we give rules for safe work to avoid risks that arise specifically for this laboratory. The general safety instructions at HZB are not superseded by the assessment here and remain fully valid (<https://www.helmholtz-berlin.de/bin/unterweisungen>).

Eating, drinking, and smoking in any laboratory is not allowed to avoid any possible incorporation of potentially dangerous materials.

Short guidelines for the use of the Laue diffractometer, the wire saw, and polisher are available at <http://www.helmholtz-berlin.de/quellen/corelabs/quantum-materials/samplecharaclab/>. Users MUST follow these guidelines. In case this is not possible they MUST contact an instrument responsible to seek alternative routes for their experiment.

1. Handling of samples:
Samples can be dangerous e.g. toxic, or have sharp edges etc.
There are gloves to be used, as well as protective eye glasses.
A glove box is available for materials that must be kept confined. For handling of such materials, the lab-responsible must be contacted.
2. The use of the x-ray equipment (Laue, Bruker) is subject to the safety rules given in the x-ray training course (<https://www.helmholtz-berlin.de/bin/unterweisungen>). The users of these instruments MUST undergo this training which is provided by the "Strahlenschutz" department.
3. The Laue apparatus contains a Laser class 2 which can be dangerous for the eye. Protective laser glasses are available and must be used. The users MUST undergo the "Lasertraining" which is provided under "<https://www.helmholtz-berlin.de/bin/unterweisungen>".
4. Wire saw: The wire saw is interlocked. Risks arise from the fast moving wire that potentially can break, from loose sample pieces that fly around. The saw may only be operated with the door locked. Samples must be fixed well with a proper glue.
The cooling water is contaminated with the various materials treated on this saw. Skin contact with the cooling water of this saw should be avoided.
5. Polisher: The polisher is operated manually. It must run slow (<50turns/min). Samples are mounted on a support and must be fixed well. The rotation must start with unloaded polisher plate.

I have read and understood the safety instructions for Laboratory LR 022

Date, signature

Safety instruction for the PPMS /squid laboratory P007/P008:

This laboratory is equipped with two instruments (PPMS) to measure physical properties like specific heat, electrical conductivity etc. and a Squid magnetometer (MPMS). The use of these instruments, their regular cryogenic service and the handling of samples can bear health risks. Here we give rules for safe work to avoid such risks that arise specifically for this laboratory. The general safety instructions at HZB are not superseded by the assessment here and remain fully valid (<https://www.helmholtz-berlin.de//bin/unterweisungen>)

Eating, drinking smoking in this laboratory is not allowed to avoid any possible incorporation of potentially dangerous materials.

1. Handling of samples:
Samples can be dangerous e.g. toxic, or have sharp edges etc. . Also some types of (quartz) sample holders can have sharp edges. There are gloves to be used, **as well as** protective eye glasses. If toxic samples are handled an instrument responsible person or the lab-responsible must be contacted.
2. There can be high magnetic fields present in the rooms. Persons with pacemakers are not allowed to work in that room.
3. Special risks arise from the handling of cryogenic liquids and gases:
The laboratory has a “low oxygen warning system”. On that alarm – which cannot be overheard – the laboratory must be vented (windows fully open) and left immediately. The fire brigade or the HZB entrance gate must be informed.
In regular intervals, liq. Helium and Nitrogen must be refilled to the systems. These cryogenic liquids cause heavy burns on the skin, also the cold gas causes burns. Burns can also arise when cold tubes are touched without protection.
For any transfer of cryogenic liquids, protective gloves and a head protection must be used. Persons that have not been trained for such transfers must contact an instrument or lab responsible for assistance. For this action, they are not allowed to work alone.
In the lab and on the website (link) there is an instruction how to transfer cryogenic liquids.
4. The instrument software usually prevents sample changes at low or high temperature with the risk of skin burns. However, parts can still be cold or hot in part. Protective gloves must be used if uncertain.
5. When cryogenic ports to the cryostats or to storage vessels are opened the pressure on the container must be checked. Overpressure of more than 50 mb is dangerous and can cause skip burns. Gloves and head protection must be used. If uncertain, an instrument or lab responsible must be present.
6. The gas lines in the laboratory must always be kept closed, except when in use, e.g. for He transfers. In case permanent connections are needed for the experiment, the lab responsible must be contacted before setting up the connections.

7. If additional external pumps (leak testers etc.) are installed, the exhaust must be connected to the vacuum system. Mechanical pumps create toxic radicals that contaminate the laboratory air. The lab responsible must be contacted. These radicals also lead to the "strange" smell of an open cryostat or sample port. You should stay away from that and vent the laboratory (open windows).

How to transfer liquid Nitrogen

This gives a brief description for N2 transfers. If you observe a deviation from this description, contact an instrument or lab responsible person.

- Open the N2 filling ports on the cryostat. Wear gloves because there could be overpressure.
- Check the N2 vessel. Moving it a bit will tell if it is full or empty – just feel the mass of the liquid. Make sure that the filling line is tightly connected on the vessel (M24 key). Check the overpressure on the N2 vessel. A pressure of 0.5 Bar is perfect.
- Insert the end of the filling line into one filling port of the cryostat. Tighten the connection.
- Open the filling valve on the vessel. You should now see cold gas coming from the other open slot, you may also hear a sound from the backstream valve. That should stop after a minute or less.
- Observe the filling procedure in a distance. Cold gas will come out of the open port. Do not leave the room. If the pressure on the vessel drops a lot, there is a valve on the vessel to increase the pressure. Use it with care.
- When completely full, liquid will come out of the open port. Close the filling valve immediately. If the pressure regulation valve on the vessel is open, close it. After that, there may still be liquid coming out of the open port for a minute. Wait for that to stop.
- Use a fan to heat up the (likely) frozen port with the filling line. Wear gloves. Remove the filling line when it is unfrozen. Wear gloves, because parts of the filling line may still be cold.
- Close both filling lines with the safety valve plugs. Use the fan if needed. Do not overheat the surrounding. Make sure that everything is warm and tight.

Make sure that the N2 can pressure valve is closed and put it to the storage place.