

Users guide for EPMA



Terms and Conditions for
Accessing the Electron-
Microprobe Laboratory at GFZ

The electron-microprobes laboratory (short: microprobe laboratory) is part of the section for inorganic and isotope geochemistry at the Helmholtz Centre Potsdam GFZ - German Research Centre for Geosciences. The laboratory is to conduct research by providing scientific advice and providing the laboratory infrastructure. The present user regulation is binding for all users of the microsonde laboratory.

By using our facility you agree to the following points:

1. Laboratory equipment

The task of the microprobe laboratory of the GFZ Potsdam is to enable measurements of the electron microscope. The laboratory is currently equipped with

- **Two JEOL electron microprobes - JXA 8230 and JXA 8530F+**
- **CarbonCoater Polaron CC7650**

The devices are available to users for the processing of their research questions, as is the other laboratory infrastructure. The exact equipment of the two JEOL electron microprobes as well as the performance of the other laboratory equipment is given on the GFZ webpage (www.gfz.de/en/scientific-infrastructure/laboratories/).

2. Services of the microprobe laboratory

The type of analyses that can be performed depends on the equipment available and their readiness.

3. User circle

The users of the microprobe laboratory are predominantly geoscientific or research scientists working on material sciences. This gives the possibility to carry out high-spatial resolution chemical analyzes of solids. Users of other research areas that need microanalysis are also welcome. The facility is available for GFZ employees as well as for external users / research groups.

4. Access to the microprobe laboratory

All scientists wishing to use our facility need to contact the head of the microprobe laboratory. The head of the laboratory will endeavor to provide short term measurement times. Appointment requests are processed in the order of their input.

In the case of an urgent need to carry out measurements, the laboratory staff may decide to revoke assigned measuring dates and to assign them to other users. If measurements can be carried out on both existing devices, there is no claim to the use of a particular device.

Analytical time can be canceled by the laboratory staff if the laboratory is not ready for use due to technical defects or personnel bottlenecks. The allocation of substitute dates for canceled analytical time is preferably

carried out against regular appointments. If a user cannot perceive an appointment in the laboratory, one has to cancel the appointment at least two working days beforehand. If one does not comply with this obligation, the laboratory time will still be invoiced if the appointment cannot be assigned otherwise in the short term.

5. Carrying out measurements and support for the user

The extent to which laboratory users are assisted in carrying out analyzes by the laboratory staff depends on the complexity of the analysis and the knowledge of the equipment of the users. In principle, it is desirable for users to conduct their analytical activities independently.

In order to learn how to operate the device, it is not enough to have attended a student seminar on microanalysis. A on-site personal instruction in the microprobe by the laboratory staff and several hours of on-site training are a prerequisite to run the facility independently.

Measurements for less extensive research projects, which would not justify a comprehensive training, can be carried out together with the laboratory staff. The user is supervised by the laboratory staff for a maximum of seven hours per day. Within this period, all work requiring the presence of the user and the laboratory personnel must be completed. The period of time in which users can access the care of the laboratory staff extends from Monday to Friday (working days) between 9am and 4pm.

Extended on-site analytical time might be possible but need to be discussed with the laboratory staff. A decision is case-sensitive.

These regulations are not affected by measurements for pre-stored analyses. If points, lines or maps are stored during the presence of the laboratory staff, they will be measured during the rest of the day and the night. On-site measurements during the weekend are not possible. However, previously stored points, lines or maps can be analysed during the weekend.

Valid for both cases: at the beginning of the new work day, the measurements must be completed.

6. Work safety issues

The users of the microprobe laboratory are instructed in the safety regulations before starting their work in the laboratory. The use of the laboratory may only take place after safety instruction by the laboratory staff. The user has to acknowledge the operating instructions, the hazard assessments and the user regulations of the laboratory with a signature.

7. About the general behavior in the microprobe laboratory

The user has to follow the instructions of the laboratory staff and has to deal carefully with all laboratory equipment.

The use of external storage devices is prohibited. Installing your own software on the computers of the laboratory is strictly forbidden. The copying of software from the laboratory computers is strictly prohibited. Modifying configuration files and initialization files on the laboratory computers is strictly forbidden.

Any breach of the User Regulations, Laboratory Regulations or Occupational Health and Safety Regulations may lead to permanent exclusion from the laboratory use.

8. Liability

GFZ continually strive to provide high-quality data. Nonetheless, GFZ shall not be held liable in the event that results from our facility are subject of subsequent revision. Furthermore, we will not be held responsible for samples lost in transit.

GFZ shall not be responsible to the user for any indirect or consequential loss or similar damage such as, but not limited to, loss of profit, loss of revenue or loss of contracts, provided such damage was not caused by a willful act.

If damage to the laboratory equipment is caused by faulty operation or negligence of the user, the damage must be remedied timely by the user.

9. Data protection

The users of the micro-probe laboratory committed themselves to data protection. Data from other users, which are stored on the laboratory computers, may not be evaluated, copied or published. The use of external storage devices is prohibited.

The primary data / raw data obtained in the laboratory (e.g. measurement data, calibration values) are archived on separate data media. However, this does not release users from their obligation to ensure their own data.

10. Publishing of Data

Unless otherwise agreed to in writing, the data obtained in the microprobe laboratory will be used by the user for at least one reviewed manuscript to appear in the international scientific literature.

Manuscripts which are to be submitted for publications must be sent to the laboratory staff in a timely fashion. The staff member will review the microprobe component and will recommend any points where improvements might be made. If improvements affect scientific aspects of the manuscripts, the head of the laboratory can also take the opportunity to comment on this part of the manuscript.

In each case of publication, the laboratory has to be mentioned:

- If a user is not able to conduct their analytical activities independently or the laboratory head contributed scientifically to the work (e.g. by developing of analytical routines or by the evaluation of data), then the head of the laboratory must be considered as a co-author.

- In the case of independent laboratory work by the user without any scientific contribution of the laboratory staff, the microprobe laboratory is to be mentioned in the acknowledgment of the publication.

A copy of the publication (in electronic or printed form) must always be made available to the laboratory staff.

In addition, users agree to follow the DFG recommendations for the publication of scientific data ('Ensuring Good Scientific Practice', Deutsche Forschungsgemeinschaft (DFG) Kodex V 1.2).

11. Charging structure

The use of the microprobe laboratory is cost-effective.

A fixed price will be purchased for coating the sample(s) with carbon, for calibrating the elements necessary for the planned analyses and for sample loading. Carbon coating needs to be done at the GFZ to make sure that the carbon layer thickness of a sample is similar to that of the standard material.

In order to cover a on-site personal instruction in the microprobe by the laboratory staff and the on-site training one day of personal costs must be purchased.

The price of each analytical day depends on the facility (JXA 8230 or JXA 8530F+) necessary to carry out the envisaged measurements and on the necessity for personal support from the laboratory staff.

We offer only daily analytical time because pre- and post-analytical work has always to be done. The amount of money charged daily can be provided upon request, please contact Dr. Franziska Wilke.

12. Preparation of the sample(s) prior to any microprobe analyses

The user is responsible for a proper preparation of his / her analytical time. This means that one need to make sure

- a) that the sample(s) fit to our sample holder (details are on the webpage)
- b) that the sample(s) are well polished (last step of polishing with 0.25µm diamond or corundum paste) and show a flat and even surface
- c) that the user knows the / their sample(s) by former reflected light microscopy studies
- d) that areas of interest in the sample are marked on the back of the sample and / or formerly taken pictures of that areas guide the user or the laboratory staff towards the areas of interest

If any of the above mentioned point cannot be provided by the user, the GFZ shall not be make responsible for any analytical day terminating without conducted measurements. The price of an analytical day is independent of the achieved goals.

→ Please see "Users guide for EPMA: Sample collection and preparation"

Potsdam,

Recognition of User Regulations

I hereby confirm that I have read and understood the terms and conditions for the use of the microprobe laboratory at the Helmholtz Centre for Geosciences (GFZ) in Potsdam. I acknowledge all rights and obligations arising out of this User Regulations by my signature.

Family name

Surname

Affiliation (Name of Institute / Company, Street, Postcode, City)

Phone

e-mail

Title of project (also draft title possible)

POF Topic relevance (if applicable)

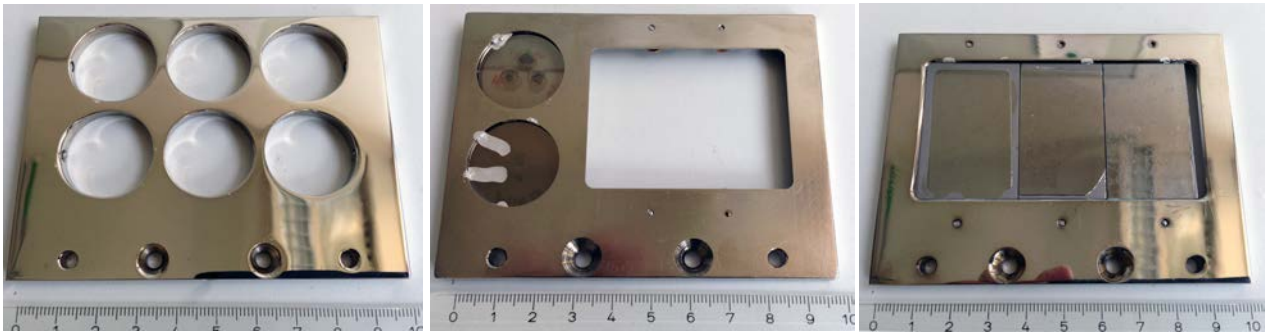
Signature.....

Sample collection and preparation

Size of samples

Nearly any solid material can be analysed. In most cases, samples are prepared as standard-size 27 x 46 mm rectangular sections, or in 1-inch round disks. Rectangular sections of rock or similar materials are most often prepared as 25 µm-thick sections without cover slips. Thick section up to 1mm are also okay.

Alternatively, 25mm (1-inch) cores can be polished. Chips or grains can be mounted in epoxy disks, then polished half way through to expose a cross-section of the material.



Polishing

The most critical step prior to analysis is giving the sample a fine polish so that surface imperfections do not interfere with electron-sample interactions. This is particularly important for samples containing minerals with different hardnesses; polishing should yield a flat surface of uniform smoothness. Final polishing should have been done using ¼µm particle size of diamond or corundum powder.

Petrographic work, marks and coating

Having the thin section or mount ready, petrographic work on a microscope should be done prior to the use of the EPMA. Overview and detailed photomicrographs using plane polar are necessary of minerals or areas of interest in a thin section. Areas of interest could be marked at the back of the thin section / mount using a permanent marker. Please do not mark anything on top of the thin section because we have to wipe and clean your sample using ethanol. This is because most minerals are electrical insulators and directing an electron beam at the sample can lead to electrical charging of the sample, which must be dissipated. Prior to analysis, samples will therefore be coated with a thin film of carbon by means of evaporative deposition. Why carbon? It is thin and light enough that interference with the electron beam and emitted X-rays is minimal.