



Institut für
Geowissenschaften



LABORATORY GUIDE

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MINERAL SEPARATION

General information

All work must be scheduled in advance. Operation of any machine or equipment is prohibited without prior training and authorization from the lab manager. For refresher training, the lab manager must be consulted before use.

Booking a lab time slot

TO BE UPDATED SOON - UNTIL THEN GET IN TOUCH WITH ME!

Laboratory Sample Labeling and Storage Rules

All rock samples in beakers, petri dishes, or bags must be clearly labeled with the Sample ID, the separation stage, and the chemical applied. Samples are only to be temporarily stored in the laboratory in labeled storage boxes indicating the responsible person's name and the project. Unlabeled or improperly stored samples are subject to disposal.

Table 1.1 – Work flow chart for a single sample.

Work day	Crush/Grind	Sieve	WT	HAc	H ₂ O ₂	Wash/Dry	HM	Frantz	SPT	DI	Pick
1	x	x	x	x							
2					x						
3						x					
4						x					
5							x	x	x		
6										x	
7											x



Figure 1.1 – Panoramic view of room 0.41, showing (from left to right) the hammer plate, jaw crusher, and disk mill.

Lab cleaning

Once a year, a lab-wide cleaning will be scheduled, bringing together all lab users to thoroughly clean the workspace — including those hidden corners and rarely visited areas that never see the light of day.

1.1 Crush and grind

Machinery for crushing and grinding rock samples is located in room 0.41. The room is equipped with various sizes of hammers and a hammer plate, a FRITSCH Jaw Crusher PULVERISETTE 1, and a FRITSCH Disk Mill PULVERISETTE 13 (Fig. 1.1). When working in this room, you must wear **safety goggles, hearing protection, a respiratory protection mask, long trousers, and sturdy footwear**. In case dust is generated, the ventilation system must be activated using the panel located at the end of the neighboring room 0.42.

1.1.1 Hammer and hammer plate

- Before using the hammer and hammer plate make sure that the equipment to be used is clean. In any case it is recommended to wipe the tools and the plate with ethanol and a paper towel.

- Crush the samples until the desired grain size is obtained. For submission to the jaw crusher, the target grain size is approximately 5 cm in diameter, while for the disk mill it is about 1 cm in diameter.
- Between samples and at the end, use a vacuum cleaner, metal brushes, ethanol, paper towels, and compressed air to clean the work area and the hammer. Finally, take the broom and sweep the floor.

1.1.2 Jaw crusher

- Before using the jaw crusher make sure that the equipment to be used is clean. In any case it is recommended to wipe the tools and accessories with ethanol and a paper towel.
- Assemble the jaw crusher and make sure that the vacuum cleaner and funnel is properly attached.
- It is recommended to carry out several crushing steps with decreasing plate distances to avoid additional wear on the plates and the motor.
- Between samples and at the end, use a vacuum cleaner, metal brushes, ethanol, paper towels, and compressed air to clean the work area and the hammer. Finally, take the broom and sweep the floor.

1.1.3 Disk mill

- Before using the disk mill make sure that the equipment to be used is clean. In any case it is recommended to wipe the tools and accessories with ethanol and a paper towel.
- It is recommended to carry out several grinding steps with decreasing plate distances to avoid additional wear on the plates and the motor.
- Between samples and at the end, use a vacuum cleaner, metal brushes, ethanol, paper towels, and compressed air to clean the work area and the hammer. Finally, take the broom and sweep the floor.

1.2 Sieving

Sieves and sieving machines are located in room 0.42. The room is equipped with various sizes of sieves, two FRITSCH Vibratory Sieve Shaker ANALYSETTE 3 (Fig. 1.2), some tools, and an ultrasonic bath for sieve cleaning. When working with the sieves in this room, you must wear **a respiratory protection mask, long trousers, and sturdy footwear**. In case dust is generated, the ventilation system must be activated using the the panel located at the end of the room.

- Before using the sieves and sieve machines make sure that the equipment to be used is clean. In any case it is recommended to wipe the tools and accessories with ethanol and a paper towel. Also clean the work space on the table.
- Sieves of various sizes can be stacked together, starting with the sieve pan at the bottom, followed by progressively larger mesh sizes, with rubber bands placed between them as needed and the transparent lid on top. Finally, position the ventilation hood above the stack.
- Attach the sieve stack with the rubber straps on each side, switch on the sieve shaker and adjust it to achieve optimum results.



Figure 1.2 – Sieve machines (left) and water table (right) in room 0.42.

- Between samples and at the end, use a vacuum cleaner, brushes, copper brushes, picking needles, ethanol, paper towels, compressed air, and ultrasonic bath to clean the work area and the sieves. Finally, take the broom and sweep the floor.
- Alternatively, the sieves can also be used manually.

1.3 Water table

The water table is located in room 0.42. The room is equipped with a HOLMAN-WILFLEY 800 Laboratory Shaking Table and accessories (Fig. 1.2). When working with this machine, you must wear **long trousers**, and **sturdy footwear**.

- Before using the water table make sure that the equipment to be used is clean. In any case it is recommended to clean everything with water and brush (maybe some soap too).
- Assemble the water table. Switch on and adjust sample input, water flow rates, and sample flow controllers to obtain optimal results.
- After each sample, transfer the desired fractions into labeled steel bowls, decant excess water, and place them in the oven to dry at 50°C max.
- Between samples and at the end, use plenty of water and brush the table and its accessories clean.
- Finally, take a mop and wipe the floor.

Maintenance

- Maintain the oil level so that the eccentric shaft is just immersed in oil, allowing sufficient lubrication of the upper components through splashing (approximately **0.5 L**).
- At **monthly intervals** or after any **extended period of non-use**, remove the head motion lid and apply a small quantity of oil to the exposed moving parts.



Figure 1.3 – Water table in room 0.42 (in action).

- Visually check the head motion oil level (SAE 20W/50) by removing the head motion lid, and top up as necessary.
- If the machine is in continuous or daily frequent use, change the oil after one month of initial operation, and thereafter every six months.

1.4 Chemical cleaning

Wet chemistry sample cleaning is performed under the left fume hood in room 0.56. When working in this room, you must wear a **lab coat, long trousers, and sturdy footwear.**

- Before and after work make sure that the work space and equipment to be used is cleaned.
- Fresh 10% acetic acid (HAc) and 3% hydrogen peroxide (H₂O₂) is mixed using deionized water and stock solutions from the respective cabinets. To effectively remove/dissolve clays, organic matter, and carbonates, the heavy fraction from the water table is soaked for at least 12 hours each in 10% acetic acid (HAc) and 3% hydrogen peroxide (H₂O₂) in fitting glass beakers. Between and after treatments, samples should be thoroughly rinsed with deionized water, and finally dried in the oven at 50°C max for two days.

1.5 Hand magnet

The hand magnet is located in the middle drawer under the worktable at the end of room 0.56. When working in this room, you must wear a **lab coat, long trousers, and sturdy footwear.**

- Before and after work make sure that the work space and equipment to be used is cleaned.
- Spread some scrap paper on the table and fold scrap paper to make a paper tray.
- Fill the tray with some amount of sample and hover the magnet above the sample to remove ferromagnetic particles/minerals.



Figure 1.4 – Frantz I in operation (left) and Frantz II under dust cover (right).

- Collect the magnetic mineral fraction in a labeled bag. The label must include the Sample ID and either “HM” (for hand magnet) or “magnetic” to indicate its contents.
- Between samples and at the end, discard of the paper, and wipe table and tools with ethanol.

1.6 Frantz magnetic separator

The mineral separation labs houses two FRANTZ LB-1 Magnetic Barrier Laboratory Separators in room 0.56. When working in this room, you must wear a **lab coat**, **long trousers**, and **sturdy footwear**. It is also advisable to remove any mechanical wristwatch, as exposure to the device’s magnetic field may damage it or cause it to stop functioning. Individuals with pacemakers are not permitted in this room for the same reason.

- Remove the dust cover and check the condition of the Frantz magnetic separator and any accessories stored in the Tupper box.
- Regardless of cleanliness, use the vacuum cleaner for a thorough clean of the Frantz and its accessories.
- If you see fingerprints on the chute, wipe it clean with ethanol.
- Ensure both the forward and sideward tilt angles are set to 10°.
- Assemble the Frantz, retrieve a glass funnel from the cabinet, and connect the power cord to the 110–220 V converter.
- Verify that all knobs are set to zero, then switch on the power supply, feed, and chute vibrations.
- Adjust the settings. Commonly, we start with a current of 0.6 A.
- Add the sample to the feeder funnel, open the feeder, and readjust the settings as needed.
- When finished, turn everything off and empty the cup containing the magnetic fraction into a plastic bag labeled with the Sample ID and “magnetic”.
- Pour the non-magnetic fraction into the feeder funnel and increase the current to 0.9 A.

- Repeat the process: when finished, turn everything off and empty the cup containing the magnetic fraction into the plastic bag labeled with the Sample ID and “magnetic”.
- Pour the remaining non-magnetic fraction into the feeder funnel and increase the current to 1.2 A.
- Repeat the process: when finished, turn everything off and empty the cup containing the magnetic fraction into the plastic bag labeled with the Sample ID and “magnetic”.
- Either continue by pouring the non-magnetic fraction into the feeder funnel, setting the forward tilt to 5°, and keeping the current at 1.2 A,
- Or pack the non-magnetic fraction into a labeled bag with the Sample ID and “non-magnetic”.
- Disassemble the Frantz and thoroughly clean it, its accessories, and the surrounding workspace using the vacuum cleaner.
- Store the accessories in the Tupper box.
- Switch off the power supply, unplug the converter, and replace the dust cover.

Frantz Reminder

In case you are unsure of how to handle, clean, and/or leave the Frantz behind after your work in the lab is done, you are hereby reminded of a few major facts.

- **Assembly:** Nothing special to take care of except for: “Take care of the small parts”. The Frantz is a costly toy and this includes its accessories. Is something missing or broken? Do you hear funny noises when running the machine? Stuff like that.
- **Cleaning:** Every once in a while you may notice that your predecessor at the Frantz left it at various stages between clean and dirty. Therefore, try to keep the place as clean as possible and notify each other, if you notice something odd. The Frantz is cleaned solely without water; Use the vacuum cleaner and its various heads to remove dust and grains and don’t forget the surrounding work space; By doing so, try not to scratch the slide, it already looks terrible. If necessary, wipe the Frantz and its parts clean using Ethanol. After cleaning carefully, put the small parts in the corresponding box, which may once in while also be cleaned.
- **Final steps:** Loosely re-install the slide using one screw; unplug the transformer, set all knobs to zero, and cover the Frantz. Done!
- **Important:** Parts may break or disappear. This happens to all of us. However, nothing is worse than leaving behind the broken machine for the next user to notice. So please, come forward, so we can order replacement parts if necessary.

1.7 Heavy liquid density separation

Heavy liquid density separation is performed under the right fume hood in room 0.56. When working in this room, you must wear a **lab coat**, **long trousers**, and **sturdy footwear**.

1.7.1 SPT

When working with Sodium-Polytungsten (SPT), you must wear a **lab coat**, **long trousers**, and **sturdy footwear**. The fume hood may be open while working with SPT.

Gather the following items:

- Large vacuum bottle labeled **SPTonly** for SPT retrieval.
- Large vacuum bottle labeled **SPTwash** for collection of rinse solution.
- Spray bottles filled with deionized water (VE-Wasser) for washing samples and glassware.
- Large glass funnel for heavy-mineral-containing sink fraction.
- Polypropylene (PP) Büchner funnel for float mineral fraction.
- Round filter cut from regular coffee filters.
- Basket filter for the float fraction.
- Separation funnel with a suitable valve.
- Suitable stirring rod.

The separation process:

- Assemble the separation station.
- Pour SPT solution from the SPT-cylinder, adjusted to a density of 2.7–2.9 g/cm³, into the separation funnel.
- Add the sample, followed by additional SPT on top.
- Mix the suspension thoroughly using the stirring device.
- Reduce mixing speed to a slow rotation, keeping the floating fraction in motion while the sink fraction remains still.
- At the end, turn off the stirrer and wait for a clear separation.
- Open the valve and collect the heavy minerals in a filter/funnel over the **SPTonly** bottle.
- Turn on the vacuum system, connect the hose to the **SPTonly** bottle, and extract as much SPT solution as possible.
- Swap the filter/funnel from the **SPTonly** bottle to the **SPTwash** bottle, and wash thoroughly several times with deionized water, using the vacuum system to speed up the process.
- Carefully remove and fold the filter paper containing the heavy mineral fraction, place it in a labeled petri dish (Sample ID and SPT↓), and dry it in the oven at a maximum temperature of 50°C.

- Release the remaining suspension into the basket filter placed over the **SPTonly** bottle and extract as much SPT solution as possible. It is good practice to briefly initiate rotation to re-suspend the sample in SPT solution before opening the valve.
- Swap the basket filter/funnel to the **SPTwash** bottle and wash the sample several times with deionized water, using the vacuum system to speed up the process.
- Place the basket filter in a labeled petri dish (Sample ID and SPT↑), and dry it in the oven at a maximum temperature of 50°C.

Cleanup and SPT recovery:

- Thoroughly wash all equipment with deionized water, collecting the wash water in the **SPTwash** bottle.
- Clean all equipment in the ultrasonic bath for 15 minutes, then place it on a rack to dry.
- Filter the SPT solution from the **SPTonly** bottle back into the SPT cylinder.
- Drain the diluted SPT solution from the **SPTwash** bottle into the PTFE beaker located in the large oven beneath Frantz II.
- Wipe down the workspace with a damp cloth.

1.7.2 DI

When working with Diiodomethane (DI), you must wear **disposable nitrile gloves**, a **lab coat**, **long trousers**, and **sturdy footwear**. The fume hood must be shut and operational while working with DI.

DI heavy liquid separation is similar to mineral separation using SPT, with important differences:

DI is classified as having unspecified toxicity and must be handled with appropriate safety measures and care. The solvent is **ACETONE** not water.

Gather the following items:

- Small vacuum bottle labeled **Dionly** for DI retrieval.
- Small vacuum bottle labeled **DIwash** for collection of rinse solution.
- Spray bottles filled with acetone for washing samples and glassware.
- Two small glass round filters (Sartorius 3hw).
- Separation funnel with a suitable valve.

The separation process:

- Assemble the separation station.
- Pour DI from the DI-bottle into the separation funnel.

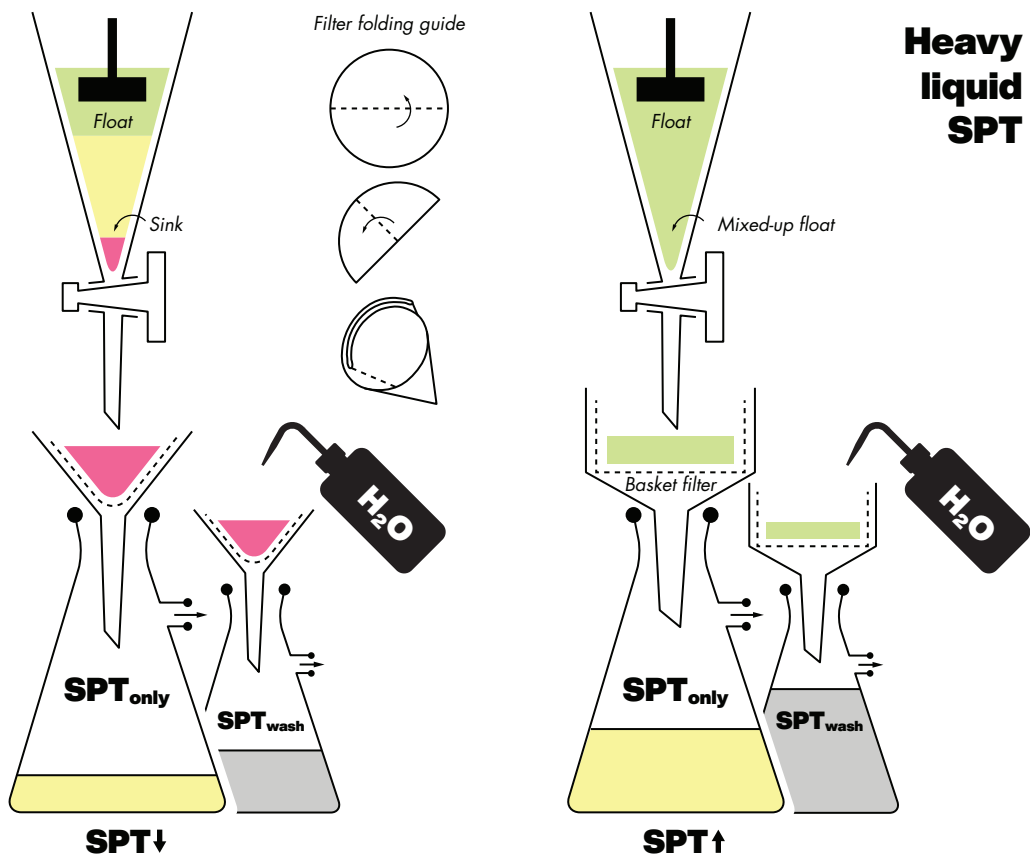


Figure 1.5 – Schematic setup of SPT heavy liquid separation.

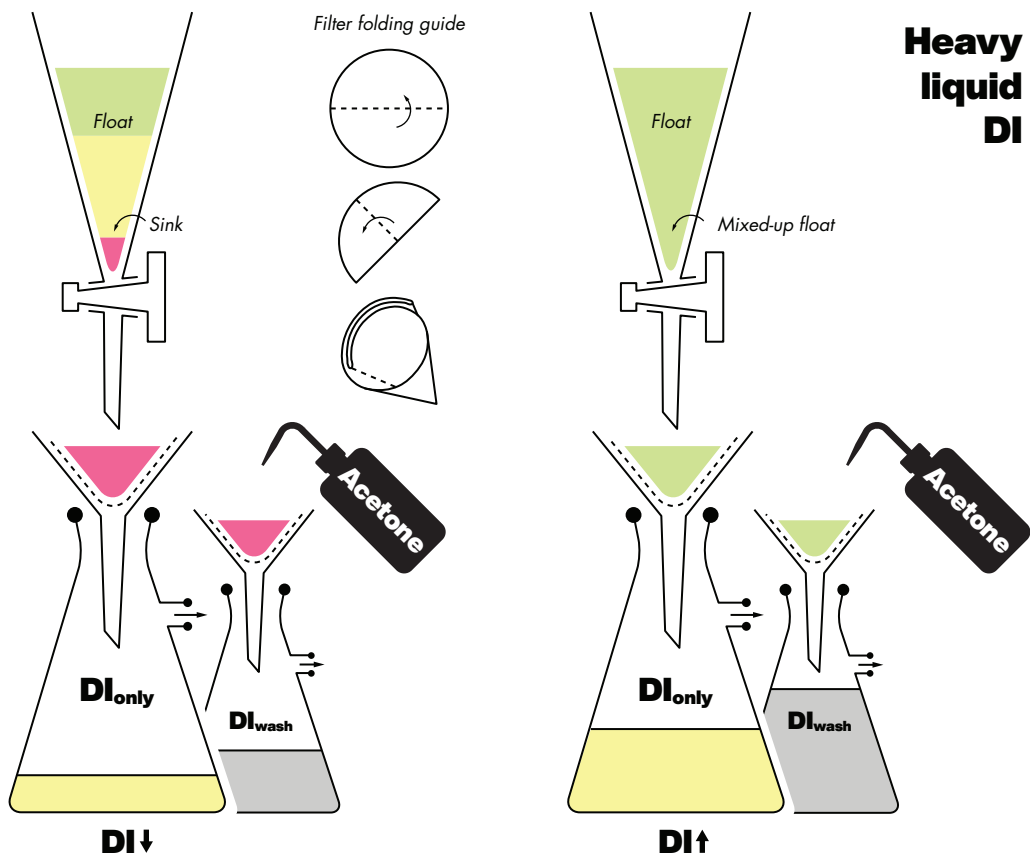


Figure 1.6 – Schematic setup of DI heavy liquid separation.

- Add the sample, followed by additional DI on top.
- Gently mix the suspension by moving the separation funnel in a circular motion with your hand, ensuring thorough mixing without creating splashes or bubbles.
- Now wait for a clear separation.
- Open the valve and collect the heavy minerals in a filter/funnel over the **DIonly** bottle.
- Turn on the vacuum system, connect the hose to the **DIonly** bottle, and extract as much DI as possible.
- Swap the filter/funnel from the **DIonly** bottle to the **DIwash** bottle, and wash thoroughly several times with acetone, using the vacuum system to speed up the process.
- Carefully remove and fold the filter paper containing the heavy mineral fraction, place it in a labeled petri dish (Sample ID and DI↓), and dry it in the oven at a maximum temperature of 50°C.
- Release the remaining suspension into the second filter/funnel placed over the **DIonly** bottle and extract as much DI as possible.
- Swab filter/funnel to the **DIwash** bottle and wash the sample several times with acetone, using the vacuum system to speed up the process.
- Place the filter in a labeled petri dish (Sample ID and DI↑), and dry it in the oven at a maximum temperature of 50°C.

Cleanup and DI recovery:

- Thoroughly wash all equipment with acetone, collecting the rinse solution in the **DIwash** bottle.
- Clean all equipment in the ultrasonic bath for 15 minutes, then place it on a rack to dry.
- Filter the DI from the **DIonly** bottle back into the DI bottle using the folded Sartorius filter paper.
- Drain the diluted DI solution from the **DIwash** bottle into the DI waste container beneath the fume hood.
- Wipe down the workspace with a damp cloth.

1.8 Hand picking

Depending on the analytical procedures required for the minerals of interest, hand-picking is performed either in the Thermochemistry Lab (Room 2.43) for apatite and zircon U-Th/He analyses, or in the Mineral Separation Lab (Room 0.56) for all other purposes. When working in these rooms, you must wear a **lab coat, long trousers, and sturdy footwear**. Below, I describe the hand picking procedures performed in room 0.56 to obtain pure mineral picks from the heavy mineral fraction following heavy liquid separation.

- Gather the necessary materials: glass slides, needles, a razor blade, plastic vials, sieves, scrap paper, and wipes.
- Clean the workspace, microscope, and tools with ethanol.
- Examine the grain size of the sample under the binocular microscope to determine if sieving is required.

- If sieving is needed, select an appropriate sieve size and proceed.
- Place a glass slide under the microscope and transfer a small portion of the sample onto the slide (using either a microspatula or by gently pouring).
- Use the razor blade to spread the sample into a thin line.
- With a needle, move unwanted grains upward and away, while shifting the grains of interest downward toward yourself.
- Using the razor blade, collect the selected grains and transfer them over the edge of the glass slide into a labeled plastic vial (including both the sample ID and mineral name).

1.9 Other methods

1.9.1 Paper method

The paper method is a simple, low-tech mineral separation technique that exploits the physical shape—specifically the platy habit—of minerals such as muscovite and biotite to separate them from denser or more equant grains (e.g., quartz, feldspar, magnetite). When gently poured onto a slightly inclined sheet of paper (standard writing paper or filter paper), platy minerals tend to lie flat and may stick to the surface, whereas more equant grains roll or tumble quickly to the bottom.

- To apply the method, you'll need several sheets of clean paper and a folded paper tray.
- Pour a small amount of the dry, sieved sample onto a sheet held at a gentle angle in your hands.
- Carefully shake or tap the paper to allow the grains to move.
- Observe where different grain types accumulate: platy minerals will tend to remain higher on the sheet, while denser or rounded grains will tumble downward.
- If the desired grains are not yet sufficiently concentrated, repeat the process using the upper fraction on a fresh sheet of paper.

1.9.2 HCl and HF cleaning of volcanic ash samples

Hydrochloric (HCl) acid cleaning of volcanic glass samples is performed in room 0.56. When working in this room, you must wear **safety goggles, long trousers, sturdy footwear, a lab coat, and nitrile gloves**. All acid related work is to be done under the fume hood!

- Find and clean PE bottles, fill with some ash sample and label the bottle and lid.
- Rinse with deionized water, decant the dust fraction, and repeat until supernate is clear.
- Remove all excess water.
- Fill bottle with 10% HCl and place open bottles in the ultrasonic bath for 15 minutes.
- Decant HCl into waste container and "extinguish" the acid with the addition of deionized water.

- Rinse with deionized water until pH-strip shows neutral values.
- Do not dry the samples, but continue with the following HF treatment in room 1.51.

Hydrofluoric (HF) acid cleaning of volcanic glass samples may only be performed in room 1.51 after contacting Antje Musiol (chemistry lab manager). When working in this room, you must wear **safety goggles, long trousers, sturdy footwear, a lab coat, protective sleeves, and nitrile gloves**. All acid related work is to be done under the fume hood!

- Fill bottle with 5% HF and carefully stir the open bottle for ca. 30 sec to keep sample and acid in motion.
- Decant HF into waste beaker and "extinguish" the acid with the addition of deionized water.
- Decant this first flush also into the waste beaker.
- Rinse with deionized water until pH-strip shows neutral values—excess water goes into the drain.
- Place open bottles in the oven in room 0.56 to dry at 50°C max.
- Proceed with mineral separation optimized for extraction of volcanic glass (i.e. magnetic and heavy liquid separation).

1.9.3 SSM–Scheibenschwingmühle (Shatter box)

- Crush sample rocks using hammer in room 0.41 to fist size;
- Further crush those slabs with the jaw crusher using size setting 1;
- Select ca. 200 g of the the crushed sample material avoiding rim sections and bag up the rest;
- Wash and dry selected sample material;
- Use jaw crusher at size setting 5 or any other device to fine crush the sample material;
- Select ca. 50 g of sample material for grinding and bag up up the rest;
- Load material into grind-chamber:
 - Use Agate grinding set for samples destined to be measured by ICP-MS or whenever Co and W should be measured (15-30 min);
 - Else, use the Tungstencarbide grinding set (1-5 min);
- Samples to be measured by ICP-MS need to be sieved using 62 µm PE-gaze sieves;
- Larger fragments need to be ground using mortar and pestle (agate);
- Repeat if needed;
- Combine and homogenize sample material and pack up in a labeled glass vial;
- Each sample needs a written documentation of Sample ID, Sample material, Grinding set used, Time required for grinding, Type of analyses required, and the name of the commissioning person;
- To clean the grinding set: (1) wipe the set with wet paper; (2) run it using quartz sand; (3) wipe the set with wet paper, compressed air, and ethanol;

1.10 Lab maintenance

1.10.1 SPT density calibration

To achieve optimal mineral separation using SPT, it is essential to adjust its density to a precise target value (typically between 2.70–2.85 g/cm³). Density calibration is done by diluting or concentrating the solution through the controlled addition of either deionized water or solid SPT granule.

- Determine the current density ($\rho_{\text{SPT, measured}}$) of the SPT solution in the glass cylinder using a hydrometer.
- Choose your target density ($\rho_{\text{SPT, target}}$) based on the minerals you want to separate (e.g., 2.85 g/cm³ to float quartz and feldspar and sink apatite and zircon).
- If the solution is too dense, dilute it by adding distilled water (Eq. 1.1).
- Shake the solution well to ensure homogeneous mixing. Allow any air bubbles to dissipate before re-measuring density.
- If the solution is too dilute, evaporate water at low temperature (e.g., in a drying oven at 50°C) or add pre-concentrated SPT.

$$V_{\text{H}_2\text{O}} = V_{\text{SPT}} \cdot \frac{\rho_{\text{SPT, measured}} - \rho_{\text{SPT, target}}}{\rho_{\text{SPT, target}} - \rho_{\text{H}_2\text{O}}} \quad (1.1)$$

Where:

- $V_{\text{H}_2\text{O}}$ = volume of water to add
- V_{SPT} = current volume of the SPT solution
- $\rho_{\text{SPT, measured}}$ = current density of the SPT solution
- $\rho_{\text{SPT, target}}$ = target density of the SPT solution
- $\rho_{\text{H}_2\text{O}}$ = density of water (typically 1 g/cm³)

1.10.2 SPT-recovery

- Decant SPT wash solution (stored in a PE bottle or directly from the SPT wash vacuum flask) into a beaker and wait for the precipitate to sink (1 day).
- Decant the supernate into a second beaker and wait for the precipitate to sink (1 day).
- Decant the supernate into a 3L PP beaker and store it in the drying oven at 50°C until SPT is fully dried (several days).
- Break the SPT and pour it into the PE bottle labeled...XXX and add some water
- As long as some SPT remains as a solid at the bottom of the container, the liquid above is close to the commonly desired density. After filtration and calibration, it can be reused.

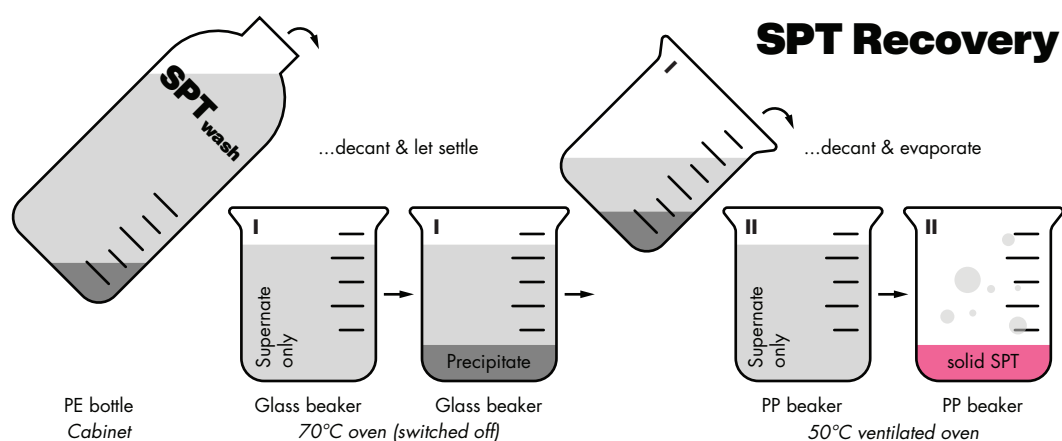


Figure 1.7 – Schematic setup of the SPT recovery.

1.10.3 DI-recovery

To separate and reuse DI from Acetone, we use a Heidolph VV2000/WB2000 rotary evaporator (Fig. 1.8).

- Fill the rotary evaporator water bath with **deionized water**, switch it on, and set the temperature to **50°C**.
- Retrieve some **DIwash** from the DI waste container located under the right-hand fume hood.
- Pour the **DIwash** into a round-bottom flask (DI-flask) until the level is just above the rectangular marking. Record the exact volume.
- Carefully attach and secure the DI-flask to the rotary evaporator using a metal clamp.
- Lower the flask into the water bath and start rotation at approximately **120 rpm** and turn on the WCF cooling water set to **0.5 bar**.
- Close the vacuum valve located under the fume hood, then switch on the vacuum pump (control panel located opposite the fume hood).
- Gradually open the valve to reduce the pressure in the evaporator, then close it again. Avoid rapid pressure changes that may cause the DIwash to boil violently.
- Acetone should now begin condensing and dripping into the Acetone-flask.
- If the distillation rate slows significantly, repeat the pressure reduction step.
- Once the process is nearly complete, open the vacuum valve to remove any remaining acetone. Then stop the rotation, lift the flask from the bath, and switch off the vacuum.
- Detach the DI-flask and filter the recovered **DI** into a clean collection bottle.
- Detach the Acetone-flask and transfer the collected acetone into the bottle labeled “**Recycled acetone for DI heavy mineral separation only.**”
- Check the collection flasks attached to the vacuum pump (inside the cabinet beneath the Frantz II magnetic separator) and empty them if necessary.



Figure 1.8 – Heidolph VV2000/WB2000 rotary evaporator in action.

- Re-attach all flasks to the rotary evaporator, power down all equipment, and clean the workspace thoroughly.

MOUNT PREPARATION

General information

This section briefly outlines the materials and steps for preparing epoxy mounts with standardized 2.5 mm diameter dimensions, commonly used in radiometric dating methods such as U-Pb zircon dating (Fig. 2.1). Preparation varies by application: provenance studies require the full range of age populations, while near-depositional dating of volcanic rocks and sandstones benefits from mounts with fresh, idiomorphic grains—preferably with adhering glass or pumice. Thus, careful handpicking is essential to target the youngest grains and minimize contamination from reworked zircons.

2.1 Material and equipment

- Big microscope slide
- Double-sided adhesive tape (ASLAN company)
- 2.5 mm diameter mounting cup
- Laboratory tweezers (type 5)
- Ethanol for cleaning
- Binocular with transmitted light
- Permanent pen
- Play dough
- Aluminum cross – a self-made tool for quadrant separation
- Microbeads or other material as markers

2.2 Mount preparation: multi-sample procedure (Fig. 2.2A–C)

1. Cut a 50 × 50 mm piece of adhesive tape and clean the glass slide with ethanol.
2. Remove the paper side from the adhesive tape and carefully adhere the exposed side to the slide, avoiding air bubbles.
3. Remove the colored protective foil from the top of the adhesive tape.

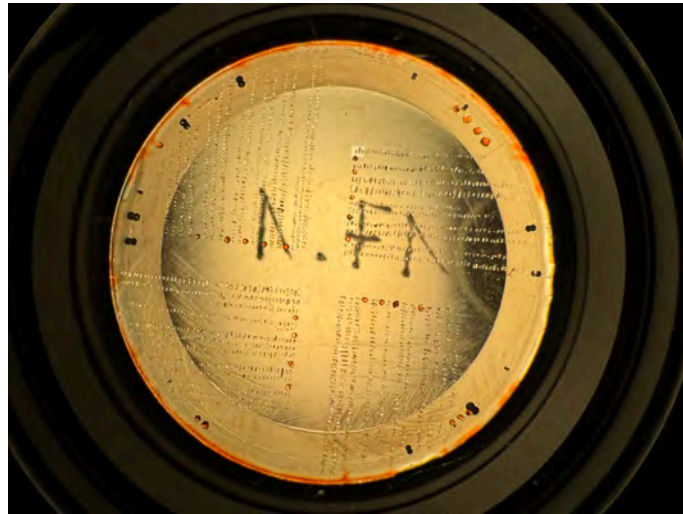


Figure 2.1 – Polished hyper-sample epoxy mount containing zircons from 22 samples.

4. Clean the mounting cup cylinder and place it centrally onto the center of the adhesive tape.
5. Trace the inner ring of the cup onto the adhesive tape using a pen. Remove the cup and clean it again for later use.
6. Draw a 15.5 × 15.5 mm square centered within the traced circle.
7. Place microbeads in the space between the circle and the square to indicate the upright orientation of the mount.
8. Pour Sample I into a designated area outside the traced circle.
9. Using tweezers, pick individual grains and carefully place them in rows within the square on the adhesive tape. Avoid pressing them in too deeply, and maintain a grain's distance between each.
10. Once Sample I is placed, add a microbead to separate it from the next sample.
11. Repeat the procedure until all samples have been emplaced.

2.3 Mount preparation: hyper-sample procedure (Fig. 2.2D–F)

1. Cut a 50 × 50 mm piece of adhesive tape and clean the glass slide with ethanol.
2. Remove the paper side from the adhesive tape and carefully adhere the exposed side to the slide, avoiding air bubbles.
3. Remove the colored protective foil from the top of the adhesive tape.
4. Clean the mounting cup cylinder and place it centrally onto the center of the adhesive tape.
5. Trace the inner ring of the cup onto the adhesive tape using a pen. Remove the cup and clean it again for later use.
6. Place aluminum cross in the middle of the traced circle;

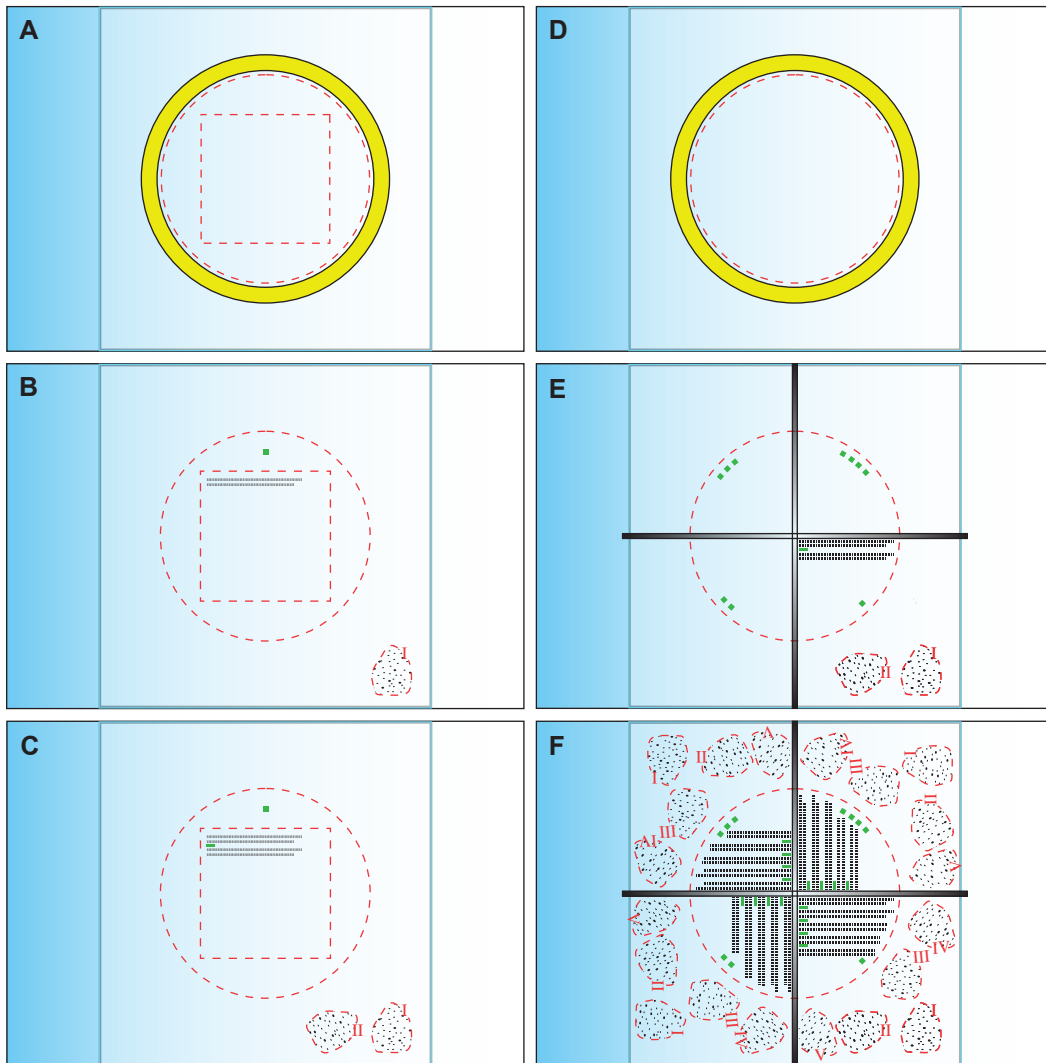


Figure 2.2 – Schematic representation of the steps for preparing a zircon mount. (A-C) Multi-sample procedure: Slide with adhesive tape, yellow mounting cup and markings of the inner rim and area of interest (AOI, square); top mark (green) and first sample poured into area I and placed in the first rows; second sample added to area II and grains placed in the following rows separated by a marker. (D-F) Hyper-sample procedure: Slide with adhesive tape, yellow mounting cup and markings of the inner rim; Aluminum cross placed in the center of the circle area and colored markers as indicators of quadrants with first two samples placed in rows from repositories poured outside the AOI separated by markers; (F) Mounting is finished.

7. Place microbeads near the margin of the traced circle to mark each quadrant (1–4 beads).
8. Pour Sample I into a designated area outside the traced circle.
9. Using tweezers, pick individual grains and carefully place them in rows within the given quadrant on the adhesive tape. Avoid pressing them in too deeply, and maintain a grain's distance between each.
10. Once Sample I is placed, add a microbead to separate it from the next sample.
11. Repeat the procedure until all samples have been emplaced.

2.4 Final steps

- It is essential to record the sample location using the format: Mount-ID – Quadrant – Sector – Sample-ID.
- Once finished, remove the aluminum cross if present, and place a clean mounting cup precisely over the traced circle.
- To prevent epoxy spills later, seal the junction between the adhesive tape and the mounting cup using play dough.
- The prepared sample slide is then submitted to the thin section laboratory (Christine Fischer), where all necessary steps will be performed to produce a polished epoxy mount suitable for further analysis.