Working instruction for the Luminescence Laboratory

Treatment of sediment samples

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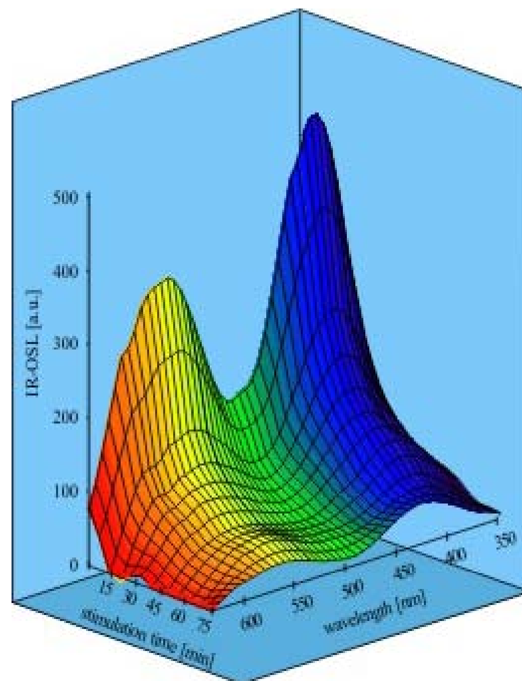


IRSL spectrum of a feldspar

1. Treatment of the fine grain fraction

The complete treatment of the samples takes place in the laboratory's dark room using red respectively green diode light. Use as much light as necessary and as little as possible.

Sieving

Required lab devices:

- Sample materials
- 63 µm, 90 µm, 200 µm sieves
- PVC-ring for the sieves
- 3 l PE beakers (2 – 3 items)
- 250 ml beakers (4 items)

- Label the beakers with the sample number and the grain fraction (<63 µm, 63-90 µm, 90-200µm, >200 µm)
- Form a set of sieves consisting of a 63 µm sieve at the bottom, a 90 µm sieve in the middle and a 200 µm sieve at the top and put it via a PVC ring on the 3 l beaker
- Unwrap the sample and, in case of steel cylinders, scrape off on both sides approximately 1 cm of the sample with a spatula and reject it (light exposed material during sample taking)
- Give a part of the sample material in the sieve and sieve it wet, until the whole sample is seven. Pay attention that the sieve doesn't block and therefore sieving residues might spill over
- Transfer the sieving residues (grain fraction 63-90 µm, 90-200 µm, > 200 µm) in a beaker, decant and dry it with max. 50°C in a drying cabinet. Pack the sieve residues after the drying process in black plastic bags and label them (with the sample number and the grain fraction).
- Transfer the < 63 µm fraction in a beaker, decant it and continue with the next treatment step

Destruction of organic material

Required lab devices:

- H_2O_2 (10% and 30%)
- Pure water in a wash bottle
- Optionally vibrating table
- Optionally PE-Erlenmeyer flask

- Dilute the sample with 10% H_2O_2 (just as much as the sample is covered) and sway it (possibly transfer the sample in a labeled PE flask, fix it on a vibrating table and switch the vibrating table on) CARE: Material with a lot of organic matter can foam over and overheat. In this case, dilute with pure water and cool it
- Repeat this process with 10% H_2O_2 until no more reaction is observed. Between the addition of new reagent, rinse the sample material once in a while with pure water and decant it
- In case of no more reaction of the sample with the reagent appears, dilute sample with 10 ml 30% H_2O_2 and repeat the treatment with 30% H_2O_2 as long as no more reaction occurs. (After use of about 100 ml H_2O_2 it can be supposed that all organic material is destroyed).

Destruction of carbonate

Required lab devices:

- HCL (10% and 30%)
- Pure water in a wash bottle
- pH – meter

- Take the pH–value of the sample. If the pH–value is low, (> 5), put a little sample material on a watch glass and test it with 30% HCL whether the sample contains carbonate at all.
- If the sample contains carbonate, dilute the sample with 10% HCL, shake it and control the pH–value. **Caution**: pH–value must not sink under 3! Repeat this process as long as no more reaction can be noticed. Alternatively to HCL one can use 20% acetic acid, which slows the reaction a little down.
- Between the adding of new reagent, rinse the sample once in a while with pure water and decant it. Occurs no more reaction of the sample with the reagent, dilute the sample with 30% HCL to test whether the carbonate is removed from the sample. If the test is negative, dilute the sample immediately with pure water and rinse it afterwards several times. If the test is positive, rinse the sample as well and treat them again with 10% HCL.
- In case of samples with a low pH–value (<3), increase the pH–value up to a neutral range by adding 0,01 n ammoniac solution.

Fine–grain fractionation with Atterberg cylinders

Required lab devices:

- Atterberg cylinders with plugs and clamps for the dumping tube
- 0,01 n NH_3 solution
- 3 l beakers
- Watch

- Label the Atterberg–cylinder and the beakers with the sample-number and the grain fraction (> 11 µm)
- Close the dumping tubes with the clamps, transfer the samples into the Atterberg–cylinders and dilute them with 10 ml of 0,01 n NH_3 solution (shake the sample well for one time. If the sample precipitate afterwards, or does not form a uniform suspension, add once again 10 to 20 ml NH_3 solution.)
- Fill the Atterberg-cylinder with pure water up to the 19 cm mark and shake it well. Let the sample sediment for 30 minutes afterwards.
- After the 30 min. period, remove the plug from the cylinder (IMPORTANT: before the drain off) and drain off the liquid (with the grain fraction <11 µm) in the beakers and decant them if necessary.
- Repeat the process (without adding 0,01 n ml NH_3-solution) until the supernatant is clear, that means if you put two fingers behind the cylinder they should be seen clearly.
- Rinse the sample material out of the Atterberg and dry it in a beaker
- Move the material from the beakers (grain fraction <11 µm) into the Atterberg–cylinders. Fill up to the 15 cm label, shake well and let them sediment for 3 hours.
- Remove the plug after the three hour period (IMPORTANT: before the drain off) and reject the supernatant (grain fraction <4 µm).
- Repeat the process (without adding 0,01 n ml NH_3-solution) until the supernatant is clear.
- Rinse the sample material out of the Atterberg and dry it in a beaker. Add approximately 10 ml Na-oxalate to the 4-11 µm before the last drying.

Fine–grain quartz treatment

H_2SiF_6 treatment for the isolation of fine – grain quartz according to G.W. Berger et al., 1980 and M.L. Jackson et al., 1976

Required lab devices:

- 100 ml PE beaker
- PE stirring staff
- Conditioned H_2SiF_6 30% (fluorosilicic acid)
- HCL 10%
- PE bottle for the contaminated fluorosilicic acid
- pure water in a wash bottle
- apron, protective goggles, acid-proof gloves

Conditioning:

- give commercial quartz in a PE–beaker
- Add H_2SiF_6 (30%) in the weight ratio, sample : acid = 1 : 10
- Keep it three days at 4°C and stir it occasionally
- Remove quartz by centrifugation that is followed by filtration

Attention! Wear acid–proof gloves, apron and protective goggles when working with fluorosilicic acid. When skin comes into contact with fluorosilicic acid, wash it immediately with floating water.

- Transfer the samples into the labeled PE–beaker, add conditioned H_2SiF_6in the weight ratio, acid : sample = 40 : 1 and leave it for 3 days. Stir it twice a day.

- Decant the H_2SiF_6in the provided PE–bottle and rinse the samples twice with pure water and then with 10% HCL, whereas the water from the first two washings is also transferred into the PE–bottle for the used H_2SiF_6. Rinse the samples again well (at least 6 – 8 times).

- Repeat the etching once again under the same conditions and rinse again with HCL and VE water and dry the samples at 50°C in the drying cabinet.

- Remove the fraction <4 µm according to the law of Stoke (e. g. Atterberg-cylinder)

2. Pipette technique for the fine – grain measurement

Required lab devices:

- Discs to pipette
- Disc-holder
- Transfer pipette, adjusted to 200 µl
- Forceps
- Beaker
- Analytical balance

Make a suspension out of:

- 200 µl pure water/ discs
- Approximately 1,5 mg sample/ discs

According to the number of the discs to pipette the sample material is balanced with the analytic balance and dissolved afterwards in pure water.

- Charge the disc-holder with discs. Place the discs in the middle of the recess (when the discs are to near to the edge the water drop will burst).
- Take up 200 µl of the suspension with the pipette.
- Cover the discs with only one drop of the suspension. For this purpose, squeeze the pipette only to the first stop (with the second stop the air bubbles are pushed into the water drop). (you will see the cupola of the water drop in the backlight of the lamp)
- ATTENTION: For the pipette act it is the best to stay calm and base the arm as calmly as possible on the worktop.
- Afterwards put the disc-holder carefully in the vacuum desiccator. Turn on the desiccator and vacuum pump (40°C). Let it dry for some hours.

3. The treatment of the coarse grain fraction

The complete treatment of the samples takes place in the laboratory's dark room using red respectively green diode light. Use as much light as necessary and as little as possible.

Sieving (manually,- by hand)

For a small amount of sample material

Required lab devices:

- Sample materials
- 90 µm, 200 µm sieves (if necessary use other mesh size)
- PVC-ring for the sieves
- 3 l PE-beakers

- ➢ Label the beakers with the sample number and the grain fraction (90-200µm, >200µm, < 90 µm)
- ➢ Form a set of sieves consisting of a 90 µm sieve at the bottom and a 200 µm sieve at the top and put it via a PVC – ring on the 3 l beaker
- ➢ Unwrap the sample and, in case of steel cylinders, scrape off on both sides approximately 1 cm of the sample with the spatula and reject it (light exposed material during sample taking)
- ➢ Give a part of the sample material in the sieve and sieve it wet, until the whole sample is seven. Pay attention that the 90 µm sieve doesn't block and therefore sieving residues might spill over
- ➢ Transfer the grain fractions > 200 µm and < 90 µm in a beaker and dry them with max. 50°C in a drying cabinet. Pack the sieve residues after the drying process in black plastic bags and label them.
- ➢ Transfer the 90 - 200 µm fraction in a beaker, decant it and continue with the next treatment step

Sieving (mechanically, with the sieving machine)

For large amounts of sample material

Required lab devices:

- Sieving machine with sputter lid, collection basement with drain lug
- Sample materials
- 90 µm, 200 µm sieves (according to the grain fraction spectrum also 1 mm and 125 µm sieves)
- 10 l black plastic bucket for the material < 90 µm
- 250 – 400 ml beakers

- ➢ Label the beakers with the sample number and the grain fraction (90-200 µm, >200µm, < 90 µm)
- ➢ Form a set of sieves consisting of a 90 µm sieve at the bottom and a 200 µm sieve at the top and set it onto the sieve bottom. Insert seals between the sieves and the bottom.
- ➢ Put the tube of the drain lug into the bucket (labeling < 90 µm)
- ➢ Unwrap the sample and depending to the clay content suspend it some days in water.
- ➢ Put part of the sample material into the sieve, fit the sputter lid and sieve it wet. Sieve as long as the whole sample material is seven, and the water which runs through the drain lug is clear. Be careful that the 90 µm sieve doesn’t block and therefore sieving residues spill over.
- ➢ Transfer the grain fraction > 200 µm into a beaker and dry it at 50°C maximum in the drying cabinet. Put a lid on the bucket with the material <90µµm and let the suspension sediment for one day. Decant the supernatant water afterwards, transfer the material into a PE-beaker and dry it in a drying cabinet at 50°C. Pack the sieve residues after the drying process in black plastic bags and label them. Transfer the 90 - 200 µm fraction into a beaker, decant it and continue with the next treatment step.

Destruction of carbonate

Required lab devices:

- HCL (10% and 30%)
- Pure water in a wash bottle

- Dilute the sample with 10% HCl and shake it. Repeat this process as long as no more reaction can be noticed. Alternatively to HCL one can use 20% acetic acid, which slows the reaction a little bit down.
- Between the adding of new reagent, rinse the sample once in a while with pure water and decant it. If no more reaction of the sample with the reagent will occur, dilute the sample with 30% HCL to test whether the carbonate is removed from the sample. If the test is negative, dilute the sample immediately with pure water and rinse it for several times. If the test is positive, rinse the sample as well and treat them again with 10% HCL.

Destruction of organic material

Required lab devices:

- H_2O_2 (10% and 30%)
- Pure water in a wash bottle

- Dilute the sample with 10% H_2O_2 and shake.
- Between the addition of new reagent, rinse the sample material with pure water.
- If no more reaction of the sample with the reagent appears, dilute the sample with 30% H_2O_2 and shake it. Rinse the sample with pure water and dilute it again with 30% H_2O_2. When no further reaction can be noticed, rinse the sample well. Then dry it in the drying cabinet at 50°C maximum. While adding H_2O_2, take care that the sample does not overheat. If necessary cool it down by adding of pure water.

Heavy liquid separation: (for separation of quartzes)

Density of:

Quartz:	2,65 g/cm³	
Feldspar (orthoclase)	2,5 -2,6 g/cm³	(potash feldspar)
Feldspar (plagioclase)	2,6 -2,8 g/cm³	(Calcium carbonate soda feldspar)

Required lab devices:

- Heavy liquid (LST or NST) with a density of $\rho = 2{,}75$ g/cm³ and $\rho = 2{,}62$ g/cm³
- Aerometer
- Falcon tubes with a supporting stand
- 250 – 400 ml beakers
- Wash bottle
- Depending on the sample amount, one beaker for undiluted LST
- Depending on the sample amount, a 2 l (or larger) beaker for the heavy liquid diluted with water

Advice: Before using the heavy liquid always test the density with an aerometer

Separation of heavy minerals with a density of $\rho > 2,\ 75$ g/cm³:

- Fill the dried samples into the labeled falcon tubes up to the cone. Dispense it with heavy liquid (2, 75 g/cm³) and shake it as long as the material is mixed with the liquid. Put the falcon tubes afterwards in the supporting stand and let it rest over night.
- Label some beakers with the sample number and $\rho < 2,\ 75$. Transfer the samples into a beaker with a slightly circular motion while decanting. As soon as the sample material (in the form of a plug) is transferred, stop decanting and set the tube down. Rinse the sample material well and dry it afterwards at 50°C at maximum in the drying cabinet.
- Transfer the material at the bottom of the tube and the remaining heavy liquid in a separated beaker. The contaminated heavy liquid is after filtering ready for a new use.
- Clean the tubes, bins and cones via the PE wash bottle in a 2 l beaker with a filter attachment. The diluted heavy liquid is steamed at 85°C maximum until the wanted density is reached.

Separation of heavy minerals with a density of ρ < 2, 62 g/cm³:

- Transfer the samples with a density of ρ < 2, 75 g/cm³ in the falcon tubes, labeled with the sample number and ρ = 2,62 g/cm³. Dispense it with heavy liquid (2, 62 g/cm³) and shake it as long as the material is mixed with the liquid. Put the falcon tubes afterwards in the supporting stand and let it rest over night.
- Label some beakers with the sample number and ρ < 2, 62. Transfer the sample material ρ < 2, 62 g/cm³ which is floating at the top in a beaker with a slightly circular motion while decanting until the sample material (in the form of a plug) is transferred. Transfer the remaining heavy liquid in an extra beaker (the contaminated heavy liquid is after filtering ready for a new use) until only material with the density of ρ > 2,62 g/cm³ (quartz) is left in the tube. Wipe the tube with a non–fluffing cellulose cloth, so that no material ρ < 2,62 g/cm³ is brought in.
- Transfer the remaining material of the tube by using pure water in a labeled beaker. Rinse the sample well and dry it afterwards at 50°C at maximum in the drying cabinet.
- Clean the tubes and beakers via the PE wash bottle in a 2 l beaker with a filter attachment. The diluted heavy liquid is steamed at 85°C maximum until the wanted density is reached.

Heavy liquid separation: (for separation of feldspars)

Required lab devices:

- Heavy liquid (LST or NST) with a density of ρ = 2,75 g/cm³ and ρ = 2,62 g/cm³
- Aerometer
- Falcon tubes with a supporting stand
- 250 – 400 ml beakers
- Wash bottle
- Depending on the sample amount one beaker for undiluted LST
- Depending on the sample amount a 2 l(or larger) beaker for the heavy liquid diluted with water

Advice: Before using the heavy liquid always test the density with an aerometer

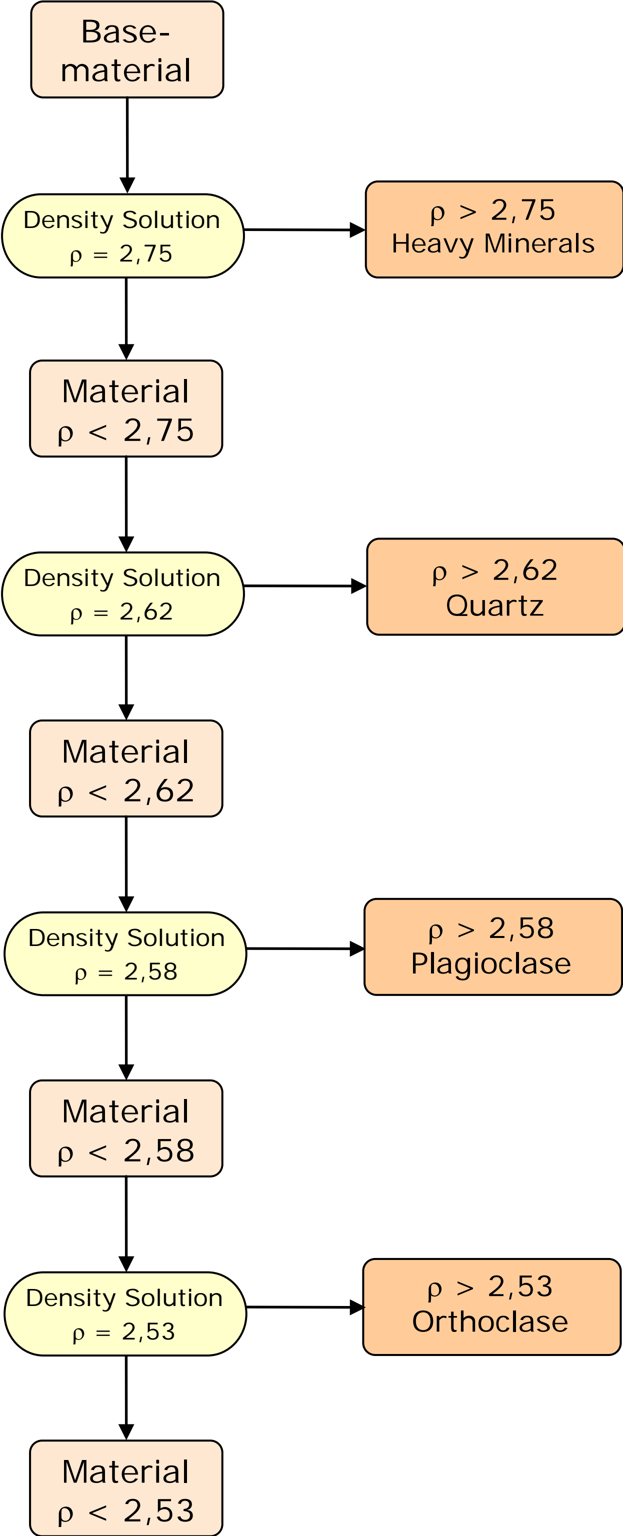
Separation of heavy minerals with a density of ρ > 2, 58 g/cm³:

- ➢ Fill the dried samples into the labeled falcon tubes up to the cone. Dispense it with heavy liquid (2, 58 g/cm³) and shake it as long as the material is mixed with the liquid. Put the falcon tubes afterwards in the supporting stand and let it rest over night.
- ➢ Label some beakers with the sample number and ρ < 2, 58. Transfer the sample material ρ < 2, 58 g/cm³ which is floating at the top in a beaker with a slightly circular motion while decanting. As soon as the sample material (in form of a plug) is transferred, stop decanting and set the tube down.Transfer the remaining heavy liquid in a separated beaker (the contaminated heavy liquid is after filtering through a sieving cloth ready for a new use) until only material with the density of ρ > 2, 58 g/cm³ (quartz) is left in the bin. Wipe the tube with a non–fluffing cellulose cloth, so that no material ρ < 2, 58 g/cm³ is brought in.
- ➢ Transfer the remaining material of the tube by using VE water in a labeled beaker. Rinse the sample well and dry it afterwards at 50°C at maximum in the drying cabinet.
- ➢ Clean the tubes, bins and cones via the PE wash bottle in a 2 l beaker with a filter attachment. The diluted heavy liquid is steamed at 85°C maximum until the wanted density is reached.

Separation of heavy minerals with a density of 2, 53 g/cm³ < ρ > 2, 58 g/cm³:

- Fill the dried samples into the labeled falcon tubes up to the cone. Dispense it with heavy liquid (2, 53 g/cm³) and shake it as long as the material is mixed with the liquid. Put the falcon tubes afterwards in the supporting stand and let it rest over night.
- Label some beakers with the sample number and ρ < 2, 53. Transfer the sample material ρ < 2, 53 g/cm³ which is floating at the top in a beaker with a slightly circular motion while decanting until the sample material (in form of a plug) is transferred. Transfer the remaining heavy liquid in a separated beaker (the contaminated heavy liquid is after filtering ready for a new use) until only material with the density of ρ > 2, 53 g/cm³ is left in the bin. Wipe the tube with a non–fluffing cellulose cloth, so that no material ρ < 2, 53 g/cm³ is brought in.
- Transfer the remaining material of the tube by using VE water in a labeled beaker. Rinse the sample well and dry it afterwards at 50°C at maximum in the drying cabinet.
- Clean the tubes, bins and cones via the PE wash bottle in a 2 l beaker with a filter attachment. The diluted heavy liquid is steamed at 85°C maximum until the wanted density is reached.

Diagram – heavy liquid separation



Etching

Required lab devices:

- 100 ml PE beakers
- Magnetic stir bars
- Hydrofluoric acid
- Magnetic stirrer (when several samples magnetic stirrer with 9 stirring spots)
- HCL 10%
- Canister for contaminated Hydrofluoric acid
- Wash bottle with de – ionized water
- acid – proof gloves, apron and protective goggles

ATTENTION! Wear acid – proof gloves, apron and protective goggles when working with Hydrofluoric acid. When skin comes into contact with Hydrofluoric acid, wash - no rubbing! - immediately with floating water and go immediately with an assistant to the hospital. Take at the same time the drug Calcium-Sandoz, which is in the first – aid kit left to the room 0.10 with you. Also in the first – aid kit is a calcium gel, which you out onto the concerned skin section.

- Transfer the sample from the beaker into the labeled PE beakers with Magnetic stir bars. Afterwards put the samples onto the magnetic stirrer placed in the flue and fill it with 40% Hydrofluoric acid until the samples are well covered with the reagent. Work very carefully to avoid every contact with the Hydrofluoric acid. Let the magnetic stirrer work 45 min. with interval stirring. Clean the bottle well with the Hydrofluoric acid under floating water.
- After 45 min. decant the Hydrofluoric acid in the therefore PE bottle and dilute the sample with 10% HCL and let it react for 30 min. with occasionally stirring. Rinse the sample afterwards very well (at least 8 times), whereas the water of the first two rinse procedures are also transferred into the PE bottle for used Hydrofluoric acid. Dry the samples at 59°C at maximum.

Sieving and packaging of the samples:

Required lab devices:

- Aluminum foil
- Labeled sample jars
- Film jars
- Light proof (black) sample bags
- DIN A4 paper
- Sieving cloth (90 µm) or 90 µm sieves
- Fixture for the sieving cloth

➢ Put the sieving cloth into the fixture (with high amounts of sample material use 90 µm sieves) and sieve the dried sample onto a sheet of paper. Everything which gets through the sieve can be rejected. Shake the sieving residues on a v – formed folded paper and transfer it in a labeled sample jar. Wrap the aluminum foil light proof around and pack it in a labeled film jar. Put several samples from one sample serial in light proof sample bags and label them.

4. Allocation for the coarse grain fraction measurement

Required lab devices:

- Aluminum plate
- Plate holder ‘A’ for allocation
- Plate holder ‘B’ for the finished allocated plates
- Hole mask
- Cone mask
- Sieve mask
- Top mask
- Silicon spray (which enables the mineral grains to adhere to the aluminum plates)
- Portion spoon for allocation of the aluminum plates
- Sample material, filled in cone tubes

Preparation of the aluminum plates:

- Assemble the plate holder ‘A’ with aluminum plates
- Put the hole mask on the plate holder ‘A’ and fix them together
- Spray the whole set with silicon spray, so that the aluminum plates are coated through the dot mask with silicon. CARE: do not use too much silicon
- Wait a few minutes until the silicon is slightly dried and remove the dot mask.
- Put on the plate holder ‘A’ first the cone mask, then the sieve mask and at last the top mask and fix everything together – dies forms the allocation unit.

Allocation of the aluminum plates:

- Fill the deepening of the portion spoon by dipping into the sample for filling the deepening well. Do not remove the spoon from the sample jar, but remove spare material by clinking the spoon slightly against the side of the sample jar.
- Move the filled spoon with a circular hand movement about 180° and put the sample material through the hole in the top mask onto the aluminum plate.
- When all plates are allocated, turn the allocation unit upside down and shake it slightly to remove spare material.
- Remove cone, sieve and top mask from the plate holder ‘A’ and shift the allocated plates from ‘A’ onto plate holder ‘B’ with the help of a pair of tweezers.
- Pack plate holder ‘B’ afterwards in a light proof bag (black photo bags) and is packed for transport to the measure room in a carton (special wooden box)