	<b>SAMPLE PREPARATION MANUALS</b>	V. Haider, A. Decou, I. Ottenbacher 28. 2. 2011
University of Göttingen	<b>Heavy liquid separation</b>	Series editor: I. Dunkl

### **Equipments to be prepared:**

1 stand with 3 grippers  
 1 separating funnel  
 2 funnels  
 2 beakers (250 ml)  
 1 filter paper for heavy minerals: MN 1670 ¼ Ø 150mm [Macherey – Nagel]  
 1 filter paper for light fraction: coffee filter [ALDI] **or** MN 617 ¼ Ø 150mm  
 Drying chamber (T: 50 °C, marked by the blue arrow)  
 Measuring cylinder (20 ml or 25 ml)  
 Hot water cooker  
 'Big' beakers (2 and 5 l)  
 Atterberg cylinders

### **Chemicals:**

Heavy liquid solution (HL): Na-polytungstate ( $\rho$ : 2.82 – 2.85 g/ml)  
 Distilled water (VE-Wasser)

### **Obligatory labelling of the fractions:**

**S** ..... heavy mineral fraction      or S 2.68 on case of usage of unusual dense HL  
**L** ..... light mineral fraction

### **Procedure:**

#### **1) Before the heavy liquid (HL) is used for separation, the density must be determined**

Weight the empty, dry measuring cylinder and fill it with exactly 20 or 25 ml HL (room temperature) and weight the filled cylinder again. Calculate the density from the mass and the volume of the liquid. If the density is  $>2.88$  g/ml, then the HL should be diluted with distilled water until the HL has the required density.

#### **2) Preparation**

The filter papers for the heavy minerals have to be labelled by pencil with the sample name and the code, which is either **S** for heavy minerals, or **L** for the light fraction.

If you use coffee filter, then bend the lower part of it, otherwise the mass of the liquid and the grains opens the weakly closed sealing at the bottom.



Make the filter paper slightly wet by distilled water.

#### **3) Separation**

*Work continuously and fast, do not let it staying long. Keep in mind that the HL makes chemical reactions with the minerals, further it is drying by time.*

Close the stopcock of the funnel. Depending on the amount of sample, the funnel has to be filled with max. 200 ml of HL. If the sample amount is tinny the HL volume in the separating funnel can be strongly reduced.

First fill just a small part of the sample into the funnel to observe the sample behaviour. If the main mass of the sample floats and only a minor part sinks down, then the rest can be added to the heavy liquid in one step; otherwise it must be done gently in more small portions. In this case keep some break between the feeding steps. The maximum amount of sample per separating funnel should be not more than approximately 35 g. Otherwise the separation does not work well and it has to be repeated!

 	<b>SAMPLE PREPARATION MANUALS</b>	V. Haider, A. Decou, I. Ottenbacher 28. 2. 2011
University of Göttingen	<b>Heavy liquid separation</b>	Series editor: I. Dunkl

As soon as the sample is in the liquid, swing the funnel by holding the stopcock in the one hand and rotate gently the waist by the other hand. You can use also a glass rod for stirring. Depending on the amount of the sample this procedure is repeated several times until no more HM settles down. Shake or stir again only, when the L and S fractions are separated well.

Let the S fraction out only when above the heavy grains there is a clear layer of HL. The heavy fraction goes into the filter paper, which was prepared before. Open slowly the stopcock and close it as soon as the heavy mineral fraction is all in the filter.

The light fraction goes into an other prepared filter, for that the stopcock must be fully open. When the HL pass trough the filter very slowly, just knock the funnel into the funnel holder very gently. The vibration will compact the sample in the filter better so the HL can flow trough easier. Collect all HL and let the beakers dropping out completely empty. Save as much HL as possible!

If no more HL comes trough the funnels, then the collected HL should be refilled into the HL flask **as long as it is not contaminated by any oxides, colloids or fine particles!** Close the flask of the HL.

Boil distilled water. Wash the filters several times by hot distilled water. The diluted HL (no grains, no clay) goes into the big beaker. Rinse well the rest of HL from all funnels, beakers, etc. and fill this solution also to the big beaker.

#### 4) Recycling HL

**If the heavy liquid is contaminated by particles, the HL must be filtered.**

**If the diluted HL is contaminated by particles, then fill it into Atterberg cylinders. After a few days of sedimentation transfer the clear solution into the big beaker for evaporation.**

Label the big beakers as 'Poly-W' and place them on the hot plates in the fume-chamber. Set the temperature to be around 60-70 °C. The intensity of stirring should not be too strong, otherwise the liquid will spray out when it has low-level.

After pre-concentration to ca. 1/10 of volume make a day break, remove the magnetic stirrer and let sink the particles in the partly concentrated HL. Decant the clear part of the partly concentrated HL into a smaller beaker for final concentration. **Be careful at the final concentration; the solution should not dry in !**

Organize well the concentration of HL. One responsible person should carry on the procedure. Write his/her name on the fume-chamber.

#### 5) Post-work cleaning

Let the table surface, funnels, beakers, etc. clean after the separation.

Note:

- If some heavy liquid drop off, save it by using e.g. filter and wash it back into the Atterberg cylinder.
- Do not dilute or contaminate concentrated HL with aqua dist and other liquids, so it can be reused without additional cleaning processes.